Residual Stress in Plasma Transferred Arc (PTA) of Ti-6Al-4V for Additive Manufacturing (AM)

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Thesis submitted to the University of Warwick for the degree of Doctor of Philosophy (PhD)

May 2020
Acknowledgement

“If we knew what it was we were doing, it would not be called research, would it?”

Albert Einstein

I would like to acknowledge WMG, University of Warwick for supporting this research work. Everyone at WMG, University of Warwick has been equally important for the positive outcome of this research work.

In particular my supervisors: Dr Greg Gibbons, Dr Darren Hughes and Professor Richard Dashwood, thank you for your continuous inspiration and support. Also, thanks to Professor Kevin Neailey for supporting me throughout the way.

Thanks to my mum and dad for unequivocal support and encouragement. I would be honoured to be even half as great parents as you have been.

Thanks to my best friend and gorgeous wife, Dr Hoda Amel. It was so reassuring to have a home supervisor! I could not have done it without you.

Hadi Moztarzadeh

May 2020
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Declaration

This thesis is submitted in partial fulfilment of the requirements for the degree of Doctor of Philosophy (PhD) and describes the work carried out from November 2014 to September 2019. Unless otherwise indicated, the research described is my own and not the product of collaboration. No part of this thesis has been submitted to any other university, or as any part of any other submission to the University of Warwick.

A large part of the research work described in this thesis is based on articles which have been, or are intended to be, published elsewhere. For each of them, the candidate (H. Mozantarzadeh) contributed principally to all aspects of the work described therein:

- Hadi Mozantarzadeh, Sampan Seth, Hoda Amel, Gregory J. Gibbons, “Plasma Transfer Arc (PTA) cladding for Additive Manufacturing (AM) of Metal Alloys”, The 18th International Conference on Machine Design and Production, Eskişehir, Turkey, July 2018
- Hadi Mozantarzadeh, Sampan Seth, Hoda Amel, Gregory J. Gibbons, “Investigating the Effects of Process Parameters on Residual Stress Evolution in Plasma Transfer Arc (PTA) cladding of Ti-6Al-4V”, The 18th International Conference on Machine Design and Production, Eskişehir, Turkey, July 2018
Abstract

Additive Manufacturing (AM) is known as an alternative method to conventional manufacturing processes, which enables design freedom, lightweighting solutions and resource efficiency for the high value manufacturing sector. As a valuable but costly material, titanium and its alloys have been the material of the choice for most of the metal-based AM processes. Plasma Transferred Arc (PTA) has been used as a high deposition rate AM technique to manufacture near net-shape parts. PTA AM of titanium alloys have presented a great business case to be adopted by different industrial sectors. However, similar to all metal-based AM techniques, PTA AM parts are affected by high thermal gradients and suffer from residual stress and associated distortion. The evolution of residual stress therefore remains a main challenge in commercialising PTA AM. Understanding the effect of the manufacturing process on the final state of the residual stress could result in manufacturing optimisation.

This thesis investigates the effects of the three main PTA AM process parameters, deposition strategy, dwell-time and energy density, on the evolution of residual strain/stress in PTA AM parts. For the first time, this work links manufacturing process and the final state of the residual stress, for PTA AM parts. Three residual stress measurement techniques, including two non-destructive diffraction-based techniques and one destructive contour method, are used to determine the level and the variation of the residual stress within the PTA AM parts, with different process parameters.

The results provide an understanding of the effects of the three process parameters (and their combinations) on the residual stress evolution in PTA AM parts. The applicability of the stress measurement techniques for PTA AM parts is demonstrated, where the contour method is shown to be a viable and straightforward technique to determine residual stress. Conversely, it is seen that diffraction techniques have experimental limitations for titanium PTA AM components, leading to increased measurement error. The residual stress/strain data show that the deposition strategy is the dominant process parameter. However, the dwell-time and the energy density are also contributor factors to the evolution of residual stress in the final component. The data presented identifies the critical combinations of the process parameters and their levels on the evolution of residual stress in PTA AM parts.
Abbreviations

3DP: 3-Dimensional Printing
AM: Additive Manufacturing
ASTM: American Society for Testing Materials
BCC: Body-Centred Cubic
BSE: Backscattered electrons
CAD: Computer-Aided Design
CAE: Computer-Aided Engineering
CAM: Computer-Aided Manufacturing
CCT: Continuous Cooling Transformation
CMT: Cold Metal Transfer
CNC: Computer Numerical Controlled
CPH: Close-Packed Hexagonal
CTE: Coefficient of Thermal Expansion
DAF: Direct Arc Fabrication
DED: Directed Energy Deposition
DLD: Direct Laser Deposition
DLF: Direct Laser Fabrication
DMD: Direct Metal Deposition
DMLS: Direct Metal Laser Sintering
DOE: Design of Experiment
EBM: Electron Beam Melting

EB-PBF: Electron Beam-Powder Bed Fusion

EBSD: Electron Back Scattered Diffraction

ESPI: Electronic Speckle Pattern Interferometry

GMAW: Gas Metal Arc Welding

HAZ: Heat-Affected Zone

ICP-MS: Inductively Coupled Plasma – Mass Spectrometry

IGF: Inert Gas Fusion

ISO: International Standard Organisation

LENS: Laser Engineering Net Shaping

L-PBF: Laser beam-Powder Bed Fusion

LWH: Length-Width-Height

MIG: Metal Inert Gas

PBF: Powder-Bed Fusion

PGFR: Plasma Gas Flow Rate

PTA: Plasma Transferred Arc

SEM: Scanning Electron Microscopy

SL: Scan Line

SLM: Selective Laser Melting

SLS: Selective Laser Sintering

SMD: Shaped Metal Deposition

TIG: Tungsten Inert Gas
Abbreviation

UTS: Ultimate Tensile Strength

WAAM: Wire Arc Additive Manufacturing

WFR: Wire Feed Rate
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Chapter 1 – Introduction
1.1. Context – scientific and industrial relevance

Additive Manufacturing (AM) technologies are of interest to many industrial sectors due to their lightweight design solutions, design freedom to manufacture complex geometries and resource efficiency. Metal-based AM techniques have been significantly evolving in line with the need for process optimisation as well as accommodating new materials developments.

As a category of metal-based AM technologies, Directed Energy Deposition (DED) techniques enable the creation of parts by melting material as it is being deposited on to a substrate. DED processes use a focused heat source (typically a plasma, laser or electron beam) to melt the feedstock material and build up three-dimensional objects in a layer-by-layer fashion. Commercial DED processes include a laser or electron beam to melt deposited powders or wires. In many ways, DED techniques can be used in an identical manner to laser cladding and plasma welding machines.

Unlike Powder Bed Fusion (PBF) techniques, DED processes are not used to melt a material that is pre-laid in a powder bed but are used to melt materials as they are being deposited. DED processes direct energy into a narrow, focused region, to heat deposited material and a substrate, melting material that is being deposited into the substrate’s melt pool. The process is repeated in a layer-by-layer fashion, depositing a number of layers on top of each other to manufacture a final part. As the layer deposition process burdens a very high thermal gradient, the manufacturing process consequently affects the final mechanical and material properties, as well as geometrical aspects of manufactured parts.

Plasma Transferred Arc (PTA) represents an efficient DED process to build parts based on the AM concept. In this process, a wire or powder is fed to the melt pool to create a deposited layer and follows the principle of the DED process. Hence, the deposition process induces high thermal gradients and affects the final mechanical and material properties. The wire-fed PTA AM is also categorised as a Wire Arc Additive Manufacturing (WAAM) technique.

Titanium and its alloys are increasingly used in different industrial applications, including: aerospace, marine, chemical industries and biomedical devices, owing to their exceptional properties such as high specific strength, low density, excellent corrosion and creep resistance and good biocompatibility. The PTA AM process offers the possibility of
Chapter 1 – Introduction

achieving large economies of scale, and as a cost effective manufacturing technology, thus
PTA AM of titanium alloys presents a great business case for a wide range of industrial
applications. Ti–6Al–4V is known as the most widely used titanium alloy and material of
choice for most metal AM techniques. As a dual phase titanium alloy, Ti–6Al–4V is
susceptible to processing conditions, specifically related to thermal gradients, and the
induced residual stresses during the manufacturing process.

Residual stresses are defined as the stresses that remain within a material or body after
manufacture and material processing in the absence of external forces or thermal
gradients. The engineering properties of materials and structural components, notably
fatigue life, distortion, dimensional shape and size accuracy and corrosion resistance can
be considerably influenced by residual stresses.

1.2. Research opportunity

While the WAAM technique in general has been examined under various process names,
e.g. Shaped Metal Deposition (SMD) and Direct Metal Deposition (DMD) relatively little
data is available in public domain in relation to residual stresses resulting from the
process, particularly in titanium alloys, during DED PTA AM.

As the PTA AM process involves a high thermal gradient, AM parts are affected by
distortion and residual stresses. For the purpose of functional parts in the high value
industrial sectors, understanding the effects of the manufacturing process on the final
characteristics of the manufactured part is essential. The final geometrical shape and size
accuracy of the PTA AM parts are dependent on the geometrical features as well as the
combination of process parameters.

There has been some development of empirical relationship between the WAAM process
parameters and built geometry. However, in most cases, it has been noted that it is not
feasible to make a direct link between process parameters and geometrical features and/or
defects. A fundamental understanding of residual stress formation during the
manufacturing process is a vital step to develop such a methodology.

1.3. Thesis structure

This thesis comprises of 10 chapters in total:
Chapter 1 – Introduction: this chapter introduces metal-based AM as well as PTA AM. Main challenges associated with the AM technologies were identified along with research opportunities, which was led to defining the aim of the research work.

Chapter 2 – Literature review: this chapter reviews the current research and development in metal-based AM, and specifically PTA AM. A review of the research in material science development related to welding and AM is covered, specifically for Ti-6Al-4V as the material of the choice for most of the metal-based AM technologies. The literature survey concludes with a review of the state-of-the-art residual stress measurement techniques, with a focus on applications and case studies related to AM.

Chapter 3 – Manufacturing of the samples and mechanical, material and process assessment: the aim of this chapter is to develop an understanding of the current set up for the PTA AM machine at the University of Warwick and to develop a methodology for manufacturing of the samples. This chapter also provides an analysis and assessment of the mechanical and material properties of the manufactured parts as well as thermal analysis to obtain an understanding of the effects of the PTA AM process parameters. Chapter 3 concludes with a sample matrix, identifying different combinations of the process parameters, for residual stress measurement, as the main objective of the research work.

Chapter 4 – Methodology for residual stress measurement: this chapter introduces the methodology to determine residual stress in PTA AM parts, as manufactured in Chapter 3. Three residual stress measurement techniques are outlined, based on the geometry and condition of the PTA AM samples. Two non-destructive, diffraction-based stress measurement techniques (neutron and synchrotron X-ray diffraction) are explained in details, which are used as complementary techniques. Furthermore, the experimental procedure for the destructive contour method is covered and a step-by-step process is laid out to provide a basis for the residual stress (and strain) results.

Chapter 5 – Results from neutron diffraction: this chapter summarises results from the neutron diffraction scanning/experiment on the PTA AM samples. The results are provided along the height and the length of the samples to enable further detailed discussions along with the results from other residual stress measurement techniques. The summary section of this chapter summarises the results to provide a basis for discussions along with the data from the other two stress measurement techniques.
Chapter 6 – Results from synchrotron X-ray diffraction: this chapter sums up the data from the synchrotron X-ray diffraction experiment. Residual strains and equivalent stress components are summarised along the length and height of the PTA AM samples. Sample choice for the synchrotron X-ray was based on the preliminary assessment and the data from neutron diffraction, as identified to be the ‘worst cases’ samples, in terms of residual stress. The summary section of Chapter 6 tabulates and represents the results graphically to provide a basis for more detailed discussions along with the data from the other two residual stress measurement techniques.

Chapter 7 – Results from contour method: this chapter summarises data from the contour method. All stresses along the ‘section of interest’ (according to the data from neutron and synchrotron X-ray) are summarised and contour data along the middle line of the cross section, equivalent to the scan lines for neutron and synchrotron X-ray, are plotted for different samples.

Chapter 8 – Validation of the plane stress condition: as the data from the three residual stress measurement techniques are available, further discussions are provided to show a detailed understanding of the issues and challenges associated with one of the main assumptions to calculate residual stress in this thesis; plane stress condition. Chapter 8 provides a detailed analysis of why it is important to consider an analysis of the plane stress condition and how it is implemented, based on the three stress measurement techniques.

Chapter 9 – Discussions of the results: the discussion chapter collates all data from the three stress measurement techniques (Chapters 5, 6 and 7) to provide a comprehensive overview of the effect of the PTA AM process and different combinations of the parameters on the evolution of the residual stress.

Chapter 10 – Conclusions and recommendations for future work: based on the discussions of the results in Chapter 9, Chapter 10 of the thesis summarises all the findings and highlights the contribution to knowledge. Chapter 10 concludes with three main proposed packages of work, as recommendations for follow up research works, based on this thesis.

The overall structure of the thesis is summarised in Table 1-1, where the chapters are identified along with their purpose and contribution to the overall thesis.
# Chapter 1 – Introduction

## Table 1.1 – Thesis structure

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Chapter 2 – Literature review and research question

2.1. Additive Manufacturing (AM)

2.1.1. Introduction to Metal Additive Manufacturing

In the manufacturing sector, global competitiveness demands a spectrum of issues regarding cost, performance, and environmental impact to be addressed for viable product realisation in the marketplace. Novel manufacturing and processing technologies enable the use of new materials and will open up opportunities for new product development, leading to the next-generation of sustainable manufacturing technologies to meet production requirements (Zhang, Xu and Wang, 2003).

Three-dimensional printing (3DP) originally (and still today) is a powerful tool for rapid prototyping; enabling visualisation of a component prior to manufacturing. Additive Manufacturing (AM) is the development of 3DP, using similar concepts, which aims to create functional end-use components. Over the past 30 years, AM technology has become an important fabrication process for manufacturing custom-made components (Ding et al., 2015).

According to ASTM, AM is defined as “a process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies. Synonyms are: additive fabrication, additive processes, additive techniques, additive layer manufacturing, and freeform fabrication” (ASTM F2792-12a). This definition is broadly applicable to all classes of materials, including metals, ceramics, polymers and composites. There are significant advantages for AM processes over conventional manufacturing routes, including optimised design of components (design freedom), efficient use of materials and reduced need for joining and assembly (Frazier, 2014). This process could also be complementary to the advances made in forging, casting, and hipping on a scalable level.

The history of metal-AM dates back to at least 1920 when Baker filed a patent for an arc welding process to make decorative articles (Baker, 1920). The current development focus of metal-AM is to produce complex shapes functional components that cannot be economically produced using conventional methods, to meet demanding requirements from aerospace, defence, automotive and biomedical industries (Gu et al., 2012). One of the obstacles in commercialisation of the AM techniques for real-world applications is their relatively low production volume capability.
The ASTM F42 standard introduces seven main categories for AM technologies: Vat Photopolymerisation, Material Jetting, Binder Jetting, Material Extrusion, Sheet Lamination, Powder Bed Fusion (PBF) and Directed Energy Deposition (DED) (Bott, 2014).

In metal-AM, two main components of a process are the type of material feedstock and the energy source used to build the part. Processes are broken down to powder and wire based on their input material and laser, electron beam and arc based on their energy source (Frazier, 2014; Ding et al., 2015).

Current state-of-the-art (commercialised) metal AM technologies (using metal-melting methods) are summarised in Figure 2-1, and some of the well-known commercialised metal-based AM technologies are presented in this section.

2.1.2. Powder Bed Fusion (PBF)

In powder-bed AM systems, an enclosed chamber encompasses the build area. By raking powder across the work area (onto a build substrate), a powder-bed is formed to create layers. The chamber is typically filled with an inert gas to prevent oxidation of metal powders or provide vacuum for operation.
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Two types of heat sources can be used for powder-bed systems: electron-beam (EB-PBF) or laser-beam (L-PBF). The powder layer thickness in L-PBF is typically between 20 μm and 50 μm, which gives relatively high feature resolution. The thickness of a single layer in EB-PBF is in the range of 20 to 100 μm, providing less resolution compared to L-PBF. However, compared to L-PBF, EB-PBF has shown a higher process speed. The other difference is the temperature of the build chamber. While in EB-PBF bed temperature is up to 700 °C, bed temperature in L-PBF only rises up to 200 °C in heated bed systems (Murr et al., 2010; Zah and Lutzmann, 2010; Dinwiddie et al., 2013).

Most of the powder-bed AM techniques utilise support structures when non-vertical geometries are being built. This is to provide a surface for overhanging structures to be built onto. They also hold the part down onto the build platform during the build process to prevent the residual stresses from allowing the part to bend (and thus colliding with the recoating system on subsequent layers). The support structure also facilitates heat transfer during the build process (Gibson, Rosen and Stucker, 2015a). An example of use of support structure is shown in Figure 2-2 (EOS, 2013).

![Figure 2-2 – Use of support structure for non-vertical geometries (EOS, 2013)](image)

In powder-bed AM systems, the deposition rates are relatively low. However, due to a focused heat source, a higher dimensional accuracy is achievable compared to other metal-based AM technologies (Zhang, Xu and Wang, 2003).

2.1.3. Directed Energy Deposition (DED)

The principle of Directed Energy Deposition (DED) processes is based on directing energy into a narrow focused region to melt the feed material to deposit a layer/bead, towards creating three-dimensional parts in a layer-by-layer fashion. This would provide a high deposition rate AM solution, with the material deposition/melting rate being up to 10 to 20 times higher than PBF techniques.
As this method has been predominantly used for manufacturing of metals, it is also referred to as metal deposition technology. However, in theory, this technique can work for a range of materials including polymers, ceramics and metal matrix composites (Gibson, Rosen and Stucker, 2015b).

Depending on the type of feed material and the energy source, these processes are also referred to as: Direct Laser Fabrication (DLF) (Wang, Mei and Wu, 2008), Direct Laser Deposition (DLD) (Baufeld, Biest and Gault, 2010), Direct Laser Forming (Arcella and Froes, 2000), Laser Engineering Net Shaping (LENS) (Rangaswamy et al., 2005), Direct Metal Laser Sintering (DMLS) (Simchi, Petzoldt and Pohl, 2003), Direct Arc Fabrication (DAF) (Song et al., 2005) and Shaped Metal Deposition (Merz, 1994a; Baufeld, Biest and Gault, 2010; Yilmaz and Ugla, 2016).

The DED process uses metal wire or powder as the material feedstock and the heat source can be laser beam, electron beam or plasma arc. The supplied material is fed directly into the molten pool created by the energy source. The feed rate is controlled and the heat source follows the pattern corresponding to the geometry of a single layer of the manufactured component (Pinkerton, 2010). Similar to PBF systems, to protect the molten pool and the deposited layer from reacting with Oxygen, a supply of shielding gas is used (Herderick, 2011; Frazier, 2014; Gibson, Rosen and Stucker, 2015a). A range of Oxygen shielding mechanisms has been developed and implemented based on standard free energy formation of oxides for different metals and the criticality of the final product. A schematic of a powder-feed laser DED process is shown in Figure 2-3.

Figure 2-3 – Schematic of a powder-based DED process (Merz, 1994b)
As the bed does not need to be filled with powder in the DED processes, the build volume can be larger than powder-bed systems. Another benefit of DED systems is their ability to be used to repair damaged components, as material can be applied to existing objects (Gu et al., 2012; Ding et al., 2015).

### 2.1.3.1. Wire-feed DED / Wire Arc Additive Manufacturing (WAAM)

Wire-feed AM, is a promising technology for producing larger features with moderate complexity due to its higher deposition rates and better supplied material quality compared to powder-based AM techniques (Ding et al., 2015). Metal-AM wires are lower in cost and are more readily available than metal-AM powders. The level of contamination in wire deposition is generally lower than powder, due to lower exposed surface area than powder. The use of material in the wire-fed AM process is more efficient than in powder-fed techniques, and wire-based AM technologies seem to offer higher repeatability levels, which makes wire-feed technologies more cost-competitive (Baufeld, Brandl and Van Der Biest, 2011). However, as the material deposition rate is larger for wire than for powder-based techniques, the melting and solidification phenomena cause considerable distortion leading to size and shape inaccuracy. Another issue with the wire-fed DED approach is reported to be poor surface finish of the produced parts due to the higher volume of the fed material in the weld pool, compared to powder-fed systems. On the other hand, wire-fed DED has higher material usage efficiency with up to 100% of the wire material deposited into the component. (Herderick, 2011; Gibson, Rosen and Stucker, 2015a). A schematic of a wire-fed AM system is shown in Figure 2-4 (Herderick, 2011; Frazier, 2014; Yehorov, da Silva and Scotti, 2019).

![Figure 2-4 – Generic illustration of an AM wire-feed system (Herderick, 2011; Frazier, 2014)](image-url)
Wire Arc Additive Manufacturing (WAAM), is established based on the use of either Gas Metal Arc Welding (GMAW) as the heat source and metal wire as the material feedstock. This technique consists of a combination of an external wire-feeding unit and a MIG/TIG electrode or arc-welding torch, mounted on either a robot arm or a CNC manipulator. The robot arm or CNC-bed is programmed to deposit beads of material as specified by the geometry of a part (Ding et al., 2015; Gibson, Rosen and Stucker, 2015b).

WAAM technologies have been utilised in various industries including aerospace, automotive, and rapid tooling. Some examples of components made by WAAM are shown in Figure 2-5. Due to less geometrical complexity and flexibility available in wire-base AM techniques, the majority of the components manufactured by WAAM have relatively simple geometries, such as cylinder, wall and stiffened panels. (Ding et al., 2015; Williams et al., 2015a).

![Metal components produced by Wire-feed AM](Williams et al., 2015b)

Plasma Transfer Arc cladding or (PTA), a particular variant of WAAM using plasma arc as the heat source, is known to offer higher deposition rates due to the higher power of the plasma arc; however, higher heat input creates molten pool instability and distortion.

Furthermore, the plasma gas flow might be affected by the orifice geometry, affecting the deposited bead geometry. As a result, PTA deposition can vary from one platform to another, and the deposited geometry may depend on the design of the torch and movement of either torch or table. There is also an effect from the plasma flow rate on bead geometry, which adds another level of complexity to process understanding and development (Martina et al., 2012).

However, PTA has shown elements of robustness, which makes it a favourable process for metal-AM. By using plasma arc as the heat source, the electrode is not exposed (as it is in TIG), and therefore electrode contamination is not so severe. However, in TIG welding, by observing the electrode tip, the creation of the arc could become more simplified and
potentially more effective. The torch-to-work piece distance is also much higher in PTA (7-8 mm) compared to TIG (3.5 mm), which should make the process less sensitive to electrode to part distance variation (Mok et al., 2008; Ding et al., 2015; Williams et al., 2015b).

There are still challenges in commercialising wire DED processes in general, and PTA AM in particular, including automation of CAD-to-part, shape and size accuracy and poor surface finish (Ding et al., 2015). These are mainly due to the fact that the number of process parameters, their interactions, and complicated effects of welding parameters on the properties of the deposits, makes the optimisation process a significant task (Keränen, 2010; Ding et al., 2011).

2.1.3.2. Hybrid Additive Manufacturing

In the past few years, a hybrid approach has emerged which implements the DED welding-based technique combined with a machining/milling process to increase accuracy and surface quality of the final part (the DED process can serve for feature addition to conventionally manufactured substrates/parts). The process entails a deposition stage followed by surface milling/conditioning, as schematically shown in Figure 2-6. This would allow producing parts with comparable mechanical and material properties to conventional subtractive manufacturing techniques, as discussed in Section 2.2. The successful fabrication of metallic parts using this technique, demonstrates the potential of the wire-based DED process for high volume AM (Song et al., 2005). A hybrid process with intermittent deposition and machining, also allows for correction of deposition inaccuracies such as over-deposition of material in between layers.

*Figure 2-6 – Process principle of hybrid manufacturing: 3D deposition and surface finishing*
2.1.4. Summary

In general, commercialised metal-based AM technologies are categorised into two types: Powder-Bed Fusion (PBF) and Directed Energy Deposition (DED). The material type is either in the form of powder or wire. Powder can be supplied on a bed which is the basis for the powder-bed fusion (PBF) process or can be deposited on to the build-plate which is called powder-feed Directed Energy Deposition (DED) technique. However, wire can only be supplied by feeding on to the build area to create layers which is defined as a wire-feed DED technology. In terms of heat source, three main sources have been used in AM techniques: Laser beam, Electron Beam and Arc. Figure 2-7 shows a comparison between the different metal-AM processes.

The speed of the manufacturing process is a common challenge in commercialising AM for production and the build chamber volumes limit the manufacturing of large components. DED technologies have significant advantages over PBF processes, as they offer relatively higher deposition rates and therefore faster manufacturing. Also, DED processes can be used in a hybrid manufacturing approach, enabling complex features or different materials to be deposited onto an existing component produced conventionally or to integrate manufacturing and post-process machining.

Wire-based DED offers even higher deposition rates compared to powder-fed DED. Also, the material usage efficiency is much higher in wire-fed processes, as there is more control on the deposition process than powder-based technologies. The PTA process offers a high deposition rate, which can speed up the metal-based AM process. As a DED process, the wire-feed PTA process provides lower contaminated deposition, better material quality and higher repeatability in comparison to powder-feed processes. However, the distortion and residual stresses remain a big challenge to the uptake of the PTA technology. This is due to the heating and cooling processes, which is the basis of all metal-based AM techniques and is affected by various processing parameters. Therefore, it is important to obtain an understanding of the effect of process parameters on the final state of residual stresses to achieve the optimised geometrical accuracy.
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2.2. Mechanical and material properties in DED AM

In this section, the material considerations for metal-based AM are discussed, with the focus on titanium alloys, specifically Ti-6Al-4V, as the material of choice for this research work. A literature survey highlights the current state-of-the-art applications of this promising metal alloy for Additive Manufacturing (AM) and its metallurgical requirements and material considerations.

2.2.1. Titanium alloys: material properties and microstructural considerations

Titanium has been recognized as an element (Symbol Ti; atomic number 22) for at least 200 years. Titanium is the fourth most abundant metal, comprising about 0.6% of the earth’s crust, next to aluminium, iron and magnesium (Lütjering and Williams, 2007).

Many industrial sectors, including aerospace, automotive and energy, use titanium alloys in different ways to benefit from their good combination of mechanical and physical properties: such as high specific modulus. Titanium alloys are also known to have good corrosion resistance, which makes them desirable for chemical and oil and gas industries as well as bio-applications. Another superior property of titanium alloys is their retention
of mechanical properties at elevated temperatures, enabling them to withstand and safely function at temperatures up to 600 °C (Poondla et al., 2009; Antonysamy, 2012). However, the high cost of titanium and its alloys has always been a limiting factor when considering it for industrial applications.

These limitations have called for advancements in manufacturing processes towards reducing the cost and developing economically affordable products made out of titanium and its alloys whilst maintaining the quality and performance of the output products (Lütjering and Williams, 2007; Poondla et al., 2009).

Development of innovative and affordable manufacturing technologies requires an understanding of the effect of the manufacturing process on the final mechanical and metallurgical properties of the product that determines its performance, including: hardness, tensile, fracture toughness, fatigue resistance, creep and fracture behaviour. The properties of titanium alloys can vary based on their production and thermo-mechanical treatments (Stanford and Bate, 2004; Lütjering and Williams, 2007; Poondla et al., 2009).

Titanium is an allotropic element, which adopts more than one crystal structure, depending on temperature. It has a Close-Packed Hexagonal (CHP) α crystal structure at low temperatures and a body-centred cubic (bcc) β structure appears at elevated temperatures above β-transus (980 °C ± 20 °C), until reaching the melting temperature of about 1650 °C. The schematic of unit cells for α-phase and β-phase are shown in Figure 2·8 (Lütjering and Williams, 2007; Antonysamy, 2012).
Alloying Ti alters the β-transus temperature and the volume fraction of α and β phases. Three main categories are defined for titanium alloys (i) α-phase, (ii) α+β, and (iii) β-phase alloys (Lütjering and Williams, 2007). Titanium alloying elements can be categorised into α stabilisers or β stabilisers based on the effect of the added alloying element on the β-transus temperature e.g. α stabilisers, stabilise the α phase by increasing the β-transus temperature. Important α-stabilising elements are Al, O, N, and C. Al is widely used as an α-stabilising element, since it has a large solubility in the α phase. β-stabilising elements include V, Mo, Nb and Ta (Lütjering and Williams, 2007; Antonysamy, 2012).

The α-phase titanium alloys are relatively weak in strength but offer good corrosion and creep resistance. A bimodal (α+β) titanium alloy contains one or more α-stabilizing elements together with one or more β-stabilizing elements. The α+β titanium alloys have been reported to offer a combination of ductility, strength and fatigue resistance. However, the amount of each phase is a decisive factor in such determination (Pederson, 2002; Poondla et al., 2009; Wang et al., 2013).

For a lamellar microstructure of a titanium alloy, the β-grain size within the dual-phase microstructure, and the size of the colonies of α-phase lamellae are reported to affect mechanical properties of the alloy. These parameters are shown in Figure 2.9 (a). An inter-lamellar interface (β-phase) can also be observed.
It is important to note that the microstructure and resulting mechanical properties of titanium alloys are complicated and highly dependent on their thermomechanical processing history and heat treatments (Lütjering, 1998). Increasing the cooling rate by 50% leads to refinement of microstructure, where both α-colony size and α-lamella thickness are reduced and more basket-weave or Widmanstätten-type microstructure appears, as shown in Figure 2·9 (b) (Sieniawski et al., 2013).

Titanium and its alloys are susceptible to interstitial elements that affect the mechanical properties and the microstructural phase balance. Table 2·1 shows a schematic relationship between the effect of alloying elements on phase distribution and selected properties of titanium alloys.
Chapter 2 – Literature review and research question

Table 2-1 – The effect of alloying elements on phase dominance and mechanical properties of titanium alloys (adapted from Dai et al., 2008)

<table>
<thead>
<tr>
<th>Alloys</th>
<th>Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>α alloys</td>
<td>Unalloyed titanium Ti-5Al-2.5Sn</td>
</tr>
<tr>
<td>Near-α alloys</td>
<td>Ti-8Al-1Mo-1V Ti-6Al-2Sn-4Zr-2Mo</td>
</tr>
<tr>
<td>α+β alloys</td>
<td>Ti-6Al-4V Ti-6Al-2Sn-6V</td>
</tr>
<tr>
<td>Near-β alloys</td>
<td>Ti-6Al-2Sn-4Zr-6Mo Tr-3Al-10V-2Fe</td>
</tr>
<tr>
<td>β alloys</td>
<td>Ti-13V-11Cr-3Al Ti-8Mo-8V-2Fe-3Al</td>
</tr>
</tbody>
</table>

- Higher density
- Increased heat treatment response
- Higher short time strength
- Increasing strain rate sensitivity
- Higher creep strength
- Ease of weldability

Ti-6Al-4V

Ti-6Al-4V is the most common titanium alloy that is well known in industrial applications for its high strength to weight ratio. The main application of Ti-6Al-4V is in the aerospace sector and the remainder is used in other industrial sectors such as automotive and medical applications.

Ti-6Al-4V alloy is an α+β titanium alloy with nominally 6 wt% aluminium as α-stabilizer and nominally 4 wt% vanadium as β-stabilizer. The microstructure consists mainly of the α-phase and some retained β-phase. Depending on processing conditions, mainly heating and cooling rates, different types of microstructure have been reported. The main type of microstructure in this alloy is globular or primary α. This type of microstructure is also called bi-modal when surrounded by Widmanstätten platelets. Basket-weave microstructure is described to have finer boundaries between α and β grains. Bi-lamellar is known as another type of microstructure, in which the retained β grains contain thinner secondary α platelets and lying between the α platelets is a Widmanstätten microstructure (Lütjering, 1998).

For slow cooling rates from the α+β region or above the β-transus temperature (980 °C ± 20 °C for Ti-6Al-4V), the β-phase mainly transforms into a globular α-phase. However, by increasing the cooling rate, the α-phase nucleation rate is increased towards the β-grain boundaries, which enhances the formation and growth of α-platelets into prior β-grain. The length and feature of these α platelets are determined by the cooling rate (Ahmed and Rack, 1998; Lütjering and Williams, 2007).
As a two-phase titanium alloy, the thermo-mechanical processes can affect the combination of α and β phases, in Ti-6Al-4V. In addition, microstructural characteristics of the alloy, such as grain size, β content and α-β phase distribution, influence its mechanical properties. Therefore, understanding the effects of thermal cycles on the microstructure is important in order to understand mechanical and material behaviour (Material Properties Handbook, 1994; Ahmed and Rack, 1998; Elmer et al., 2005).

In general, solidification behaviour controls the size and shape of the grains, and the extent of defects such as porosity and hot cracks, and ultimately the properties of a solidified metal. It also controls the primary microstructure formed in the melt pool during processes like welding and AM. The solidification mechanism involves heterogeneous nucleation and epitaxial growth and depends on the grain formation. The rate of the solidification boundaries formation resulted from localized melting and the thermal diffusivity of the material would affect the overall mechanism. During the laser welding process formation and solidification of the melted material, hydrodynamic movements in the melted material affect the solidification mechanism. Thus, in additive manufacturing process, for the first few layers, the grain size and formation could be affected by the layer formation and therefore directional solidification should be investigated. In deposition-based AM, the substrate condition could become an important factor too (Akman et al., 2009; Brandl et al., 2011).

Thus, it is important to understand the influence of various parameters such as temperature gradient, growth rate, and cooling process on development of the solidified microstructure as well as the solid phase growth and final phase distribution (Elmer et al., 2005; Frazier, 2014).

Experimental techniques, such as high energy synchrotron X-ray diffraction and electron backscattered diffraction (EBSD) have been used to understand recrystallisation and consolidation mechanisms (Malinov et al., 2002; Stanford and Bate, 2004; Elmer et al., 2005).

High temperature phase transformation microstructural evolution in Ti-6Al-4V was studied by using high temperature XRD (Pederson, 2002; Zheng et al., 2014). Continuous heating and cooling cycles was applied to the titanium sample, which was cut from a press-forged component of Ti-6Al-4V. Similar to Figure 2-9, the microstructure of the as-received material was reported as a typical Widmanstätten, consisting of primary α-phase surrounded by transformed β-phase. In order to understand the kinetics of the β → α
transformation, the titanium sample was heated up and cooled down to different temperatures and the microstructure was studied at each step (Pederson, 2002).

During the heating stage, the sample was continuously heated up to 950 °C (just below the β-transus temperature), and held for approximately 20 minutes to reach equilibrium of α and β phases. Then a step by step cooling process occurred to 900 °C, 795 °C, 715 °C and 610 °C, followed by equilibrium hold. The microstructure was observed at each step. Figure 2-10 shows the microstructural changes during β→α phase transformation (Pederson, 2002).

![Microstructural Changes](image1)

**Figure 2-10 – X-ray micrographs (280X) of the sample cooling down from 950 °C (and isothermally held) at (a) 900 °C, (b) 795 °C, (c) 710 °C and (d) 610 °C (Pederson, 2002)**

At the 900 °C, a coarse α-grain boundary was observed in a dense prior β-grain boundaries, while a Widmanstätten-type α-platelets precipitated within β-grain boundaries. These α-platelets could be interpreted as primary α-grains. At the 795 °C, again a dominant β-phase was observed with coarse Widmanstätten-type α-platelets. The prior β-grains were
almost more extended within the microstructure, which could be an indication of more stable formation of α+β microstructure. More dominant α-platelet appeared as the sample was cooled down to 700 °C and then 610 °C, where α-phase is surrounded by the stabled and extended β-grains. The differences in microstructure reported in different areas of the sample at each temperature could be related to uneven polishing during sample preparation. These results were slightly different from the Continuous Cooling Transformation (CCT) Diagrams, as published by Sieniawski et.al., in which the end of transformation from β to α phase was predicted to lie between 670 – 690 °C (Pederson, 2002; Sieniawski et al., 2013).

Therefore, it was shown that phase transformation and presence of α and β phases has a considerable effect on the microstructure of the material which determines the final mechanical and material properties. An understanding of the phase transformation phenomena could lead to tailoring the process towards desirable properties. (Jacobs and Kilduff, 2005; Lütjering and Williams, 2007).

2.2.2. AM process and the effect on titanium alloy’s microstructure and mechanical properties

The number of metal alloys, which have been commercially used for AM processes, is still limited as summarised in Table 2-2. Titanium and its alloys are of uttermost interest with regard to AM. Titanium alloys combine broad industrial application in high performance parts with high machining costs and long lead times in conventional processing. Hence, many business cases exist for AM of titanium that offer substantial cost advantages.

| Selected metal alloys commercially used in metal-AM (adapted and modified from Frazier, 2014) |
|-----------------|----------------|----------------|----------------|----------------|
| **Titanium**   | **Aluminium** | **Tool Steels** | **Super Alloys** | **Stainless Steel** |
| Ti-6Al-4V       | Al-Si-Mg      | H13            | Inconel 625     | 316 & 316L       |
| ELI Ti          | Al 6061       | Cermets        | Inconel 718     | 420             |
| CP Ti           | Al 2024 & 2319| Maraging 250 & 350 | Waspaloy       | 347             |

AM parts fabricated from the dual-phase (α-β) Ti-6Al-4V alloy have been investigated and today are widely used for commercial AM fabrication (Frazier, 2014; Herzog et al., 2016). A more comprehensive data on alloys used for AM processes can be found in (Gu et al., 2012; Frazier, 2014).
Due to the nature of AM process, repetitive heating and cooling of deposited layers causes melting and solidification within a moving melt pool. As a result, a complex, time-dependent temperature profile is generated within each layer. The material may experience repeated solid state and liquid-solid phase transformations.

Figure 2-11 shows a thermal profile for a single layer AM processed Ti-6Al-4V. In this graph, the temperature of the first layer is plotted while the individual layer is deposited (time index 1-2) and this is repeated for the second to seventh layers. This plot shows the temperature during deposition of the individual layer reached up to 2,216 °K and depending on dwell-time, cooled down to ~300 K.

During the deposition of the second layer, the first layer was re-melted causing another liquid-solid transformation. It shows this individual layer experienced two liquid-solid transformation and four α-β transformations. These profiles are dependent upon a number of variables including the AM equipment, the ‘dwell-time’ between layers, and the size of the manufactured part. (Kelly and Kampe, 2004; Wang, Mei and Wu, 2008).

The heat flow and cooling phenomenon in AM processes is directional, typically in the build direction, as shown schematically in Figure 2-12 (a), which causes directional microstructural formation such as columnar microstructures (Herzog et al., 2016). Repeated directional thermal cycles also have a possible complex effect, including microstructural differences between deposited layers (Mok et al., 2008; Frazier, 2014).
For AM processing of Ti-6Al-4V, large columnar grains have been observed growing in the \(z\)-direction (perpendicular to the build plane: XY) and in the direction of heat extraction/cooling as schematically shown in Figure 2-12 (a). This effect is shown in macrographic images in Figure 2-12 (b) from laser powder-fed and electron beam wire-fed AM Ti-6Al-4V (Vilaro, Colin and Bartout, 2011; Frazier, 2014).

![Macrographic images of AM Ti-6Al-4V](image)

\(a, b, c\)

Figure 2-12 – (a, b, c) Macrographic images of AM Ti-6Al-4V: (a) powder-feed single bead width (b) powder-feed three bead width, (c) electron beam wire-feed three bead width and (d) Schematic of build (and cooling) direction (Mok et al., 2008; Vilaro, Colin and Bartout, 2011; Frazier, 2014)

Optical micrographs of the same materials are presented in Figure 2-13. The microstructure of the laser powder-feed materials appears finer in structure, than those produced by the wire-feed system. This is probably due to the larger melt pool and slower cooling rates associated with wire-feed system (Frazier, 2014). It is reported that increasing the solidification rate causes an alteration in the microstructure from columnar to equiaxed grains and a finer microstructure (Saboori et al., 2017).
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Bagheri (Bagheri et al., 2015) reported a reduction in the length of the columnar grains by increasing the laser power in powder feed laser DED. Columnar grains were replaced by equiaxed α and β laths in samples produced by higher laser powers. They also reported negligible effect of powder feed rate on the microstructure, but observed a decrease in grain size by increasing the traverse speed. This was attributed to higher cooling rates due to faster travel of the head.

Prior β grains are considered to have a direct effect on mechanical properties of parts produced by AM. In DED of titanium alloys, ductility and toughness were reported to be higher in the build direction than the transverse directions, due to the growth of larger prior β grains in the build direction (Saboori et al., 2017).

Although a slight anisotropy is reported in yield strength and ultimate tensile strength associated with the build directions in AM, mechanical properties of the AM Ti-6Al-4V are reported to exceed the typical value of wrought and cast Ti-6Al-4V, as shown in Table 2.3 (Wang et al., 2013; Bagheri et al., 2015). Finer microstructures due to the higher cooling rates associated with DED processes are deemed the cause of such increases. The manufacturing imperfections such as voids and oxides inclusions, however, are considered to cause lower elongation to failure compared to case and wrought material (Bagheri et al., 2015).
Table 2-3 – Mechanical properties of DED AM Ti-6Al-4V (Wang et al., 2013)

<table>
<thead>
<tr>
<th>Property</th>
<th>Unit</th>
<th>AM (X-Y) plane</th>
<th>AM (Z) direction</th>
<th>Wrought</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield Strength</td>
<td>MPa</td>
<td>950</td>
<td>803</td>
<td>828</td>
</tr>
<tr>
<td>UTS</td>
<td>MPa</td>
<td>1033</td>
<td>918</td>
<td>879</td>
</tr>
</tbody>
</table>

Considering the hardness of AM deposited Ti-6Al-4V beads, the hardness of deposited beads and Heat-Affected Zone (HAZ) seems to be higher than the base material. In some cases, however, the α+β microstructure tends to exhibit various hardness values depending on the formation and amount of α and β phases. The hardness value for Ti-6Al-4V lies in the range of 340 to 450 HV, depending on the processing condition. The density and porosity level directly affects the hardness. Hence, if the AM deposition process parameters are set to produce a dense material, a higher hardness value could be achieved (Baufeld, Van Der Biest and Gault, 2009; Brandl et al., 2011). DED could potentially produce a denser bulk of material, due to layer on layer (and arguably into the previous layer) nature of the process.

Based on the literature, different processing and post-processing conditions are potentially resulted in various microstructures, causing a range of mechanical properties. Furthermore, due to the directional nature of the AM process as well as layer-upon-layer recrystallization and solidification, the evolution of microstructures becomes a major concern in metal-AM, which could lead to the presence of different phases, inducing residual stresses and ultimately affecting shape and size accuracy of the final part.

### 2.2.3. Summary

Ti-6Al-4V is two-phase alloy with both α and β phases present in the microstructure of the material. In an AM process, the cyclic heating and cooling phenomena could affect the density and distribution of the phases. Hence, such a processing condition could result in a range of mechanical and physical properties.

Transformation phase laws are defined for both heating and cooling stages and they are material/alloy dependent. Such phase transformation during the AM process could result in micro-stresses. The residual stress due to the phase distribution and transformation could be affected by such processing condition, depending on what type of residual stress is of interest to the design and functionality of the final AM component.
2.3. Residual stress in DED AM

2.3.1. Residual stress measurement techniques

Residual stresses are defined as the stresses that remain within a component after it has been manufactured and/or used in service in the absence of external loading condition or thermal gradients. The presence of residual stresses could lead to inhomogeneous (non-uniform) deformation resulting in unfavourable distortion in a component. Due to the repeated heating/cooling nature of the AM processes, residual stresses have significant effect on the geometrical status of a component (Withers and Bhadeshia, 2001a).

Three types of residual stresses can be defined:

- Type I: ‘macro’ residual stresses that develop in a component-level on a scale larger than the microstructural grain size of the material
- Type II: ‘micro’ residual stresses that vary on the scale of an individual grain size
- Type III: ‘micro’ residual stresses that exist within a grain, essentially as a result of the presence of dislocations and other crystalline defects

Different methods for determining residual stresses in different types of components have been developed in the past three decades. The measuring techniques can be generally classified into three categories: destructive, semi-destructive and non-destructive techniques, as summarised in Figure 2.14 (Rossini et al., 2012).

Destructive and semi-destructive methods are also called mechanical measurement or stress-relaxing techniques. These methods rely on the deformation of the original geometry of the component due to the release of residual stresses upon removal of material from the component. In semi-destructive methods, the destruction to the structure is not significant enough to affect the performance or the structure (Withers and Bhadeshia, 2001a).

Non-destructive methods typically consider microstructural interactions due to residual stresses within the part. Therefore a knowledge of the microstructural changes due to stress and strain is required for determining the final state of the residual stress within the component (Withers and Bhadeshia, 2001a).
In the current research project, diffraction techniques (neutron and X-ray) are considered as non-destructive methods to measure residual strains in AM parts and the Contour Method was used as a destructive method. Other non-destructive or destructive techniques cannot be used due to the material property or the geometry of the parts as further explained in Chapters 3 and 4.

Diffraction techniques measure residual stresses by investigating the changes in the microstructure of materials. This allows for a comprehensive detailed understanding of residual stresses within materials in macro and micro scales (Withers and Bhadeshia, 2001b; Withers, 2007).

There are also other less well-known, more specific, residual stress measurement techniques, such as Electronic Speckle Pattern Interferometry (ESPI), Raman spectroscopy and curvature layer removal, as described in the literature (Sglavo, Bonafini and Prezzi, 2005; Schajer, 2010; Rossini et al., 2012).
2.3.1.1. Diffraction techniques: neutron and synchrotron X-ray diffraction

Diffraction techniques are classified as non-destructive methods to measure residual stress in polycrystalline materials. A polycrystalline sample consists of small crystallites, of the order of few μm, randomly oriented with respect to each other.

Similar to X-ray diffraction, when a crystalline material is illuminated with a neutron beam of wavelength \( \lambda \), comparable with the inter-planer spacing \( d_{hkl} \), a diffraction pattern is observed in which, the position of each plane \((hkl)\) is obtained using the Bragg’s relation, as expanded in Equation 2·1.

\[
2d_{hkl} \sin \theta_{hkl} = n\lambda \quad \text{Equation 2·1 – Bragg’s law}
\]

The wavelength \((\lambda)\) can be calculated from the Planck-Einstein equation, as shown in Equation 2·2.

\[
E = h\nu = h\frac{c}{\lambda} \quad \text{Equation 2·2 – Planck-Einstein equation}
\]

Figure 2·15 shows angle \(2\theta_{hkl}\) at which the Bragg peak appears and is referenced to the incident beam direction. So, each \(d\)-spacing \((d_{hkl})\) is determined from the angle \(\theta_{hkl}\) at which the reflection is observed (Gnäupel-Herold et al., 2005).

![Figure 2·15 – Schematic illustration of Bragg scattering (Bragg’s law) (Gnäupel-Herold et al., 2005)](image)

The concept of diffraction techniques relies on the elastic deformations within a polycrystalline material and measuring internal strain. The deformations cause changes in the spacing of the lattice planes from their strain free value to a new value that
corresponds to the magnitude of the applied stress. This new spacing will be the same in any similarly oriented plane, with respect to the applied stress and the crystal lattice, therefore effectively acts as a small strain gauge (Withers, 2013).

The gauge volume over which the strain measurement is made is given by the intersection of the incident and diffracted beams (Figure 2-16), which is defined as the geometry over which the measurements are made.

While electron-diffraction has a very small penetration depth, neutron diffraction and high energy synchrotron x-rays have been used to provide penetration depth in the order of tens of millimetres (Reimers et al., 1999).

These two diffraction methods create different shapes of gauge volume. While lab-based x-ray is mainly used to measure stress/strain at the surface of the parts, high energy synchrotron x-rays and neutron diffraction are used to analyse residual stress in the bulk of components. Neutrons tend to give a diffraction angle of ~90° which leads to a near cubic gauge volume. Synchrotron x-rays tend to result in low scattering angles ~10° which provides an “elongated” gauge volume. The schematic of the two gauge volumes are shown in Figure 2-17 (Geneva Switzerland: ISO, 2001; Poulsen, 2004).
Ex-situ diffraction-based measurements of residual stresses within steel-based welds is fairly well-established and a standard has been associated to such measurements (Ahmed and Rack, 1998; Geneva Switzerland: ISO, 2001; Voisey et al., 2014). Ex-situ neutron diffraction was used to evaluate the level of residual stress, and the effect of phase transformation during cooling, on welded steel alloy, and results were compared with the same welded components of titanium alloys. For Ti-6Al-4V a high degree of texture in the
welded material made the diffraction results complicated, however the potential to indicate the presence or absence of α and transformed β phases in the weld area was shown (Voisey et al., 2014). Also ex-situ x-ray synchrotron and neutron diffraction scanning of titanium welds have shown the capability of these techniques to study phase transformation and to measure residual stresses in titanium alloys (Malinov et al., 2002; Hoye et al., 2014; Song, Paradowska and Dong, 2014).

In-situ study of microstructural evolution during welding has also been considered for observing phase transformation in Ti-6Al-4V. These experiments were conducted as material was being deposited, where relatively low heating rates were produced, to study the effect of welding parameters on the α→β transformation. The results however, were limited by long acquisition time per diffraction pattern, and the ability to study only one single, slow heating rate. To study the kinetics of the α→β transformation, in-situ x-ray diffraction was conducted on spot welds, where the heating and cooling rates were rapid, showing a set of kinetic parameters for the prediction of the α→β phase transformation at high heating rates (Elmer et al., 2005).

There is limited work on the use of diffraction techniques to measure residual stresses within metal AM components. Neutron diffraction has shown the capability of measuring residual strains in bulk components made by laser-based AM process of 316 stainless steel and Inconel 718 alloys (Rangaswamy et al., 2005; Hoye et al., 2014).

Residual stresses within wire-arc Ti-6Al-4V components were also determined by using neutron diffraction, showing the capability of the technique to evaluate the level of residual stresses within AM titanium components. However, the measurements were limited to one set of process parameters which could not be used to develop an understanding of the effect of AM process parameters on the stress development (Wang et al., 2013; Martina et al., 2016). The neutron diffraction technique was used to evaluate the role of phase transformations due to different heating and cooling rates on the resultant residual stresses in laser cladding of titanium and aluminium alloys (Cottam et al., 2014).

Therefore, diffraction-based techniques (neutron and synchrotron x-ray) could provide a reliable non-destructive solution for residual stress measurement in AM components, especially in crystalline materials and metal-based AM. Neutron and synchrotron x-ray diffraction are normally called complementary solutions for residual stress determination.
2.3.1.2. Contour method

The contour method is a relaxation method, which is based on the Solid Mechanics concept. The method enables a 2D residual stress map to be evaluated on a plane of interest. If a component containing residual stress is cut through a section, the stresses with force components acting on the cut surface will relieve and the stresses within the remaining material will redistribute to maintain the equilibrium of the whole part. The stress redistribution causes distortion across the cut surface which can be used to evaluate the level of the relieved (or initial) residual stresses (Prime, 2001).

This method is very similar to the sectioning technique as both use relaxation of stress due to removing material/cutting. However, the contour method provides higher spatial resolution and therefore a thorough understanding of the residual stress distribution (Withers, 2007; Rossini et al., 2012).

Since the contour method measures the relaxed strain across a distorted cut section, the quality of cutting plays a critical role in accuracy of the results. The cutting process usually consists of wire EDM (Electro-Discharge Machining) with precisely defined wire characteristics and cutting speed depending on the required spatial resolution, material type and geometry. Measuring the surface deformation is usually conducted by using a Coordinate Measurement Machine (CMM) or a laser profile-meter. The result will be a point cloud, which will be used against a virtual non-deformed surface to calculate the strain and resultant stress, typically using a Finite Element (FE) solver. The process of the contour method is shown schematically in Figure 2-18 (Schajer, 2010).

The use of the contour method has been broadened to particularly validate the FE results as it provides a fairly accurate 2D stress distribution across the cut cross section. Also, the contour method is being used to validate the results from other residual stress measurement techniques on welded components, mainly to support diffraction-based data (Prime et al., 2002, 2004). However, as the contour method is a destructive technique, typically this should be done as a complementary step, after non-destructive method/s are implemented.

The method has recently been used to validate residual stress data on titanium and steel-based AM components and has shown a good correlation with results from neutron and synchrotron x-ray diffraction. Such experiments have proven the capability of the contour method to measure residual stresses in AM parts (Hoye et al., 2014; Martina et al., 2016).
2.3.2. AM process and the effect on residual stress evolution

As discussed in the previous sections, AM processes cause large thermal gradients in components, which result in build-up of residual stresses, and ultimately occurrence of distortion, or in more catastrophic scenarios, cracks. This phenomenon is particularly more significant in larger components processed by DED technologies. However, each AM process includes a combination of process parameters, not all of which have the same effect on the evolution of residual stresses and distortions.

Denliger (Denlinger et al., 2015) studied the effect of changing the dwell time between layer depositions on distortion in powder-fed laser DED of Ti-6Al-4V and Inconel 625 thin walls. They used a laser displacement sensor to measure the distortion of a cantilever substrate during linear material deposition. Their results suggested that while distortion decreased in Inconel 625 with increased dwell times, elimination of dwell time resulted in a decrease in distortion in Ti-6Al-4V samples. Distortion is believed to be directly proportional to residual stress.

In another study, Moat et al (Moat et al., 2011) investigated the effect of laser pulse length and duty cycle (cycle length/pulse length) on the residual stress distribution in laser DED of Waspaloy powder using neutron diffraction and the contour method. They reported that increasing the laser pulse length resulted in increasing the stress gradient and the size of the region of maximum stress in the longitudinal direction, whilst duty cycle (dwell time) was shown to have a negligible effect on residual stress.
Residual stresses were shown to be uniaxial, along the height for thin wall and columnar samples made of 316 stainless steel and Inconel 718 using the LENS process. Samples showed a combination of compressive stresses in the core balanced by tensile stresses at the surface. It was also reported that the direction of the raster used to fill the columnar samples, did not have any notable effect of the magnitude of residual stresses. Stress profiles were discovered to be similar for both materials suggesting thermal gradients to have the main effect on evolution of residual stresses. Material's phase transformations were considered to have second order effects (Rangaswamy et al., 2005).

It was shown in the literature that residual stresses and distortions in DED processes could be reduced by weakening local thermal gradients. This is due to the fact that residual stress is a function of the mechanical properties of the material (Young’s modulus), thermal history (temperature difference) and physical properties of the material, relevant to the thermal properties, such as Coefficient of Thermal Expansion. The basic thermal strain (stress) equation is given as Equation 2-3.

\[
\Delta L = \alpha L_0 \Delta T \\
\epsilon = \frac{\Delta L}{L_0} = \frac{\sigma}{E} \\
\sigma = E \alpha \Delta T
\]

Where, \( \Delta L \) and \( L_0 \) are change in the dimension and the original dimensions, respectively and \( \epsilon \) denotes the strain due to the thermal difference of \( \Delta T \) and \( \sigma \) is the corresponding stress (thermal stress). \( E \) is the mechanical property of the material, Young’s modulus and \( \alpha \) is the linear thermal expansion coefficient of the material (physical property).

The thermal stress, due to the heating up the material would normally be followed by a cooling process. The Newton's law of cooling is given by Equation 2-4.

\[
T(t) = T_s + (T_0 - T_s) e^{-kt}
\]

Where \( T \) is the temperature and \( t \) is the cooling time of each layer. \( T_0 \) is the initial temperature (of the substrate or the previous layer before depositing a new layer) and \( T_s \) is the surrounding temperature (environment). The constant \( k \) is called the coefficient of the temperature change (cooling rate), which is determined based on materials/parts subject to cooling.
In principal, the fundamental thermal stress, Equation 2·3 can be combined with the Newton’s law of cooling, Equation 2·4, to describe the heating and cooling phenomena during the AM process.

However, in AM, a more complicated transient equation could be derived as the governing equation for the heat transfer and the cooling process. Such complex phenomena need a detailed mathematical approach to take account of the governing equation, as described in details in different numerical analysis of AM processes (Megahed et al., 2016; Motaman et al., 2020).

Understanding such phenomena could help in identifying the effect of different process parameters to quantify the range for the set of such parameters for different scenarios. Albeit, the processing parameters should be considered alongside processing condition, such as geometry and dimension of the manufactured part, to account for a detailed analysis of the effect of process parameters on the final state of residual stress.

To understand and control the effect of thermal stress, process parameters, e.g. input power and traverse speed, should be controlled and optimised. Furthermore, manipulating the process parameters to maintain a constant melt pool size and temperature throughout the part were considered to reduce residual stresses (Shamsaei et al., 2015).

Nickel et al (Nickel, Barnett and Prinz, 2001) reported the deposition pattern to have a significant effect on the evolution of thermal stresses and deflection of the manufactured part. They discovered that when depositing onto beam substrates, raster patterns perpendicular to the beam’s long axis result in lower deflection compared to patterns parallel to the beam’s long axis. In deposition onto plate cases, they showed spiral patterns scanned from the outside to the inside produced lower deflection than scanning from inside to outside or using raster patterns. This was probably due to the better management of the heat input and more uniform thermal distribution within the deposited layers.
2.3.3. Summary

Metal-AM processes involve relatively rapid heating and cooling processes (10^3 to 10^5 °C/min), giving a complex thermal history, directional thermal phenomena, repeated melting and rapid solidification during the manufacturing process. The conjoint effects of process parameters cause a complex microstructural formation and repeated solid-state phase transformations in different regions of the AM component. This results in a variation in mechanical and metallurgical properties and formation of residual stresses. These factors introduce complexities not typically found in conventional subtractive manufacturing processes. Understanding the residual stress formation phenomena during the AM process will lead to optimising and tailoring the manufacturing parameters sequencing towards an accurate production process.

There has been a great level of interest shown in the use of titanium and its alloys in different AM technologies, mainly because of the cost implication as well as geometrical and material properties requirements in a number of industrial sectors. Ti-6Al-4V has been the most widely used titanium alloy for industrial applications.

Titanium alloys are therefore described as being allotropic and can therefore exist in two or more crystal lattice forms within the same physical state. Ti-alloys of the α-β type, e.g. Ti-6Al-4V, have the advantage of existing as two-phases between room temperature and the fully transformed β-transus temperature circa 980 ± 20 °C.

A review of titanium alloys and Ti-6Al-4V has been conducted to choose the applicable methodologies in understanding the effect of PTA process on the metallurgical and mechanical properties of the material. As a two-phase metal alloy, the effect of repeated heating and cooling on Ti-6Al-4V will lead to a complex microstructural transformation and residual stress formation.

Furthermore, to investigate the evolution of microstructure and residual stress formation, a series of stress measurement techniques have been analysed and their applications for different joining, welding and AM processes are reviewed. Both ex-situ and in-situ analysis of residual stress formation support the methodology development to analyse the effect of PTA process parameters on the AM parts.
2.4. The research question and significance to the discipline

2.4.1. Research opportunity, novelty and principal hypothesis

The wire-based PTA process is a promising technology to be utilised as an AM technique due to a number of advantages over current powder-based DED AM techniques. However, thermal, mechanical and metallurgical phenomena and their couplings have an impact on the state of phase equilibrium and final residual stress of the PTA AM parts. An interdisciplinary approach is therefore required to investigate the significance of the manufacturing process in formation of residual stresses and the impact on the shape and size accuracy of the PTA AM components.

There has been limited published work done on the evaluation of the effects of DED AM process parameters on the geometrical aspects and mechanical performance of the AM components. The main focus of this research work is to develop a fundamental understanding of the PTA AM process towards automation and commercialisation. Hence, the research question is defined as the following:

- What is the relationship between the process parameters and residual stress state in wire-based PTA AM of Ti-6Al-4V?

2.4.2. Research objectives and roadmap

The aim and objectives of this research work can be identified according to the research question (above). A coherent study of residual stress evolution in a metal-based DED AM technique will be conducted with the focus on the PTA AM process. This will provide a baseline for a comprehensive evaluation of size and shape accuracy in a DED-process metal AM.

Considering affecting parameters from both PTA process and the AM process, a fundamental understanding of the conjoint effect will be investigated to develop a comprehensive methodology for predicting the performance of the PTA AM technology. This will result in a method to find a combination of process parameters for minimum residual stress evolution during the process.

The challenges and required considerations to address this research question are summarised in Figure 2-19.
Based on the research question, the following objectives are defined for this research work:

- To identify the PTA process parameters and develop an understanding of their significance in PTA AM
- To develop an understanding of the AM features (layer-by-layer manufacturing process) and associated parameters in PTA AM
- To develop a methodology to determine residual stress in PTA AM parts
- To investigate the conjoint effect of process parameters on the evolution of residual stresses in PTA AM of Ti-6Al-4V
Chapter 3 – PTA AM Process and Preliminary Assessment
3.1. Introduction

The main purpose of this chapter is to provide an understanding of the manufacturing process and process parameters and to define a methodology for the manufacturing the samples for the residual stress determination. The PTA AM process to build samples is explained in details and the range and the level of the process parameters are clarified. A second part of the methodology for the manufacturing of the samples is also defined, based on the process assessment as well as the investigation of the mechanical and material properties.

To manufacture the samples, the essential processing variables and their range are identified for the current PTA AM machine at WMG, University of Warwick. The work in the literature established a knowledge of the process parameters' windows for the metallic materials; such as nickel-based super alloys (Cooper, 2016) and titanium alloys (Martina, 2014). In addition, an investigation of the level and range of process parameters are conducted in this chapter, which led to the identification of the critical boundary conditions.

A series of mechanical and material characterisation techniques and dimensional measurements are employed to analyse and quantify the different mechanical and material properties, and to provide a foundation for the analysis in the subsequent chapters. The details of the experimental methodologies to determine the residual stress are explained in Chapter 4. Understanding the process parameters, the build methods and an initial assessment of the mechanical and material properties are covered in this Chapter.

3.2. PTA AM process

3.2.1. PTA AM at the University of Warwick

The principle of the PTA AM process is based on a wire-based PTA coupled with a 3-axes CNC bed to manufacture parts in a layer-by-layer fashion. Although the concept of the PTA AM process at WMG, University of Warwick, was similar to other WAAM process, the implementation was slightly different, as the CNC bed provided the geometrical movement of the deposition process as opposed to robot-arm-based WAAM processes (Martina et al., 2012; Martina, 2014). This approach could offer more accuracy and control for the manufacturing process. This method also provided a streamlined route towards
automation for the PTA AM techniques controlling the deposition pattern by using CNC-coding is believed to be less challenging than the computer programming required for the robot-arm motions.

However, the robot-arm could provide more complex motions and geometries and possibly more design freedom. An advanced multi-axis CNC machine could offer similar outcome to deposit complex geometries and patterns.

The PTA equipment used was a GAP 3002, constant voltage power source, using a wire feeder WF V1.0 equipped with a GAP E15N wire plasma torch to deposit beads/layers (all equipment supplied by Castolin Eutectic, UK). The CNC machine was a Bostomatic 32.2, enabling 3-axes motion of the bed (plus rotation which was not used in this work), as shown in Figure 3-1(a) and (b).

![Figure 3-1](attachment:image.png)

(a) PTA equipment

(b) CNC-bed and torch and wire-feeder

*Figure 3-1 · PTA AM set up at WMG, University of Warwick*

The wire spool and the roller system are attached to the top of the PTA equipment. As shown in Figure 3-2 the wire is fed through a roller system to straighten the wire before depositing.
Figure 3.2 – The wire-feeder: spool (left) and roller system (right)

The wire-feeder supplied a wire plasma torch through a wire feed nozzle attached to the front of the wire plasma torch and both remained fixed/stationary above the CNC-bed, while the bed moved during the deposition of layers. The bed movement provides the geometrical pattern of the deposited layers/beads as defined by the G-code, which also enabled different deposition strategies. A 150 mm long trailing argon shield was attached to the torch (and the wire-feeder) to minimise oxidation. The torch, the wire-feeder and the shielding device were shown in Figure 3.3.

The wire-feeder was calibrated at the beginning of each build by allowing 1 meter of the wire being fed (dry-fed) at the tip of the wire-feeder system while the time was measured (1m/min).

Figure 3.3 - The PTA Equipment: torch, wire-feeder and shielding device

The PTA AM setup was previously developed and investigated in an Innovate UK funded project (ACCLAIM) at WMG – University of Warwick. The effectiveness of the setup including the tailing shield to minimise oxidation while building 3D geometries was shown during that project.
3.2.2. Manufacturing process and parameters

As explained in Chapter 2, the PTA deposition process is similar to welding where a feed wire is melted to deposit a bead. Hence, the process parameters in PTA AM can be attributed to the welding process as outlined in a number of welding standards. The main process parameters are summarised in Table 3-1.

<table>
<thead>
<tr>
<th>Process Parameter</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Deposition/Weld Speed</td>
<td>m/min</td>
</tr>
<tr>
<td>Current</td>
<td>A</td>
</tr>
<tr>
<td>Voltage</td>
<td>V</td>
</tr>
<tr>
<td>Energy Density</td>
<td>J/m²</td>
</tr>
<tr>
<td>Plasma Gas Flow Rate (PGRF)</td>
<td>l/min</td>
</tr>
<tr>
<td>Wire Feed Rate</td>
<td>m/min</td>
</tr>
</tbody>
</table>

The concept of energy density in metal-AM was explained in Chapter 2. In PTA AM, energy density is defined as a resultant of current, voltage and deposition rate to provide a measure for the input energy during the process. Referring back to Section 2.1.3, this parameter can be defined as the required power to melt deposited wire and create a certain volume as a deposited bead per unit of time. Hence, the Equations 3-1 to 3-3 can be used to obtain the energy density in PTA AM.

\[
\text{Bead Width (m) \times Deposition rate (m/s) = Bead area per unit of time (m}^2\text{/s)}
\]

Equation 3-1

\[
P (\text{Power in Watt}) = V (\text{Voltage in Volt}) \times I (\text{Current in A})
\]

Equation 3-2

\[
\text{Energy Density} = \frac{\text{Power (Watt or J/sec)}}{\text{Bead Area per sec}} = \frac{J}{m^2}
\]

Equation 3-3

Plasma gas plays a role in the stability of the arc and the weld pool; hence, the Plasma Gas Flow Rate (PGFR) can affect the input energy density. Wire Feed Rate (WFR) determines the material usage for the deposition, which affects the volume of material deposited and the energy per unit volume within the deposited material.
The wire is typically fed at the front of the torch and deposition path to minimise the effect of preheating and the weld pool on the existing deposited beads. Usually, bead layers are deposited on a substrate of the same material. The substrate can be designed as part of the final component or can be detached at the end of the process. The schematic of the PTA AM process to create consecutive beads is shown in Figure 3-4.

![Schematic of the PTA AM process](Martina et al., 2012)

3.3. Manufacturing of the samples

In this section, the experimental matrix development for sample preparation for residual stress investigation is explained in detail. This is followed by using a similar and simplified methodology to manufacture samples for mechanical and material characterisation. PTA AM process parameters are explained in detail and other physical features are clarified, where necessary.

The processing window for ‘stable’ deposition of Inconel 625 (In625), using the PTA set up at WMG was previously investigated (Cooper, 2016), which has provided a baseline for setting the initial parameter levels for a Design of Experiment (DoE). A complementary study of the processing parameters for deposition of Ti-6Al-4V was conducted in this study as well as in an Innovate UK project at WMG (Accelerated Cladding and Integrated Machining - ACCLAIM).

In this research work, Grade 5 titanium alloy (Ti-6Al-4V) wire, 1 mm diameter (±0.1mm), is used as the feed material, to create beads on a Ti-6Al-4V substrate. Material properties are described in the next section (section 3.4).
3.3.1. Samples for residual stress measurement

As shown in Table 3·1, the variable parameters on the PTA set-up are the deposition (weld) speed, current, PGRF and wire feed rate. Deposition speed and current combined with the voltage can be interpreted as the energy density of the process. The voltage during the deposition process depends on the distance between the torch-tip and the work-piece. A constant distance of 7 mm was manually set-up (using a measured 7mm Allen key) for all depositions, which resulted in a nominal voltage of 25 V. The deposition speed was kept constant at 50 mm/min for all depositions. This is to produce a ~10 mm width beads during a simple linear deposition of Ti6Al4V. The PTA parameters and their values are given in Table 3·2. These considerations are based on the best practices (to enable building 3D geometries) and previous studies of the PTA deposition using the PTA AM machine at the University of Warwick.

<table>
<thead>
<tr>
<th>Process Parameter</th>
<th>Value/Range (Unit)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Voltage</td>
<td>25 (±2.5) (V)</td>
</tr>
<tr>
<td>Wire Feed Rate (WFR)</td>
<td>1 (±0.1) (m/min)</td>
</tr>
<tr>
<td>Plasma Gas flow Rate (PGFR)</td>
<td>0.4 (±0.02) (l/min)</td>
</tr>
<tr>
<td>Linear bead (weld) deposition speed (for both linear and zig-zag deposition strategies)</td>
<td>50 (±2) (mm/min)</td>
</tr>
</tbody>
</table>

It is important to note that the process parameters given in Table 3·2 are all nominal values based on the limitations of the WMG PTA equipment and averaging the measurements where applicable. The actual values might have varied slightly. For example, to control the voltage, the weld gap must have been adjusted in process to maintain a constant value (and thus a constant voltage). However, this was not possible and the voltage was measured at the beginning of each layer deposition and ultimately averaged to give the nominal value of voltage (tolerance of the voltage was ±2.5 V).

The range for energy density in this process is a resultant of current, as the other two parameters (voltage and deposition-speed) were kept constant (Equation 3·3). The current was set at the maximum (125 A) and minimum (50 A) values to give two levels for the energy density.
To take the AM aspect of the process into consideration, the ‘deposition strategy’ of the layers and the ‘dwell-time’ between layer depositions are considered as the other two process parameters.

There are different strategies to deposit beads: linear, zig-zag, circular, stepped linear, etc. as defined by a number of welding standards (AWS, 1994; ISO, 2009, 2013). The deposition strategy is mainly defined by moving either the work-piece or the torch to provide the geometrical pattern of the weld bead deposition. To investigate the effect of heat source and the input energy, two deposition strategies are employed to manufacture the samples: linear and zig-zag. The linear deposition is based on the deposition of the beads in a straightforward linear way. The zig-zag strategy was chosen based on the Nickel et al. study finding that patterns perpendicular to the long axis of the build plate, generate lower residual stresses (Nickel, Barnett and Prinz, 2001).

The deposition strategies are schematically shown in Figure 3-5. The parameters for the zig-zag deposition are also clarified in Figure 3-5. This is based on the welding standards labelling for the zig-zag (or oscillated) weld beads.

The zig-zag parameters were \( v_1 = 10 \text{ mm} \) (to provide a nominal bead width equivalent to the linear deposition), \( v_2 = 1 \text{ mm} \) and \( v_3 = 80/2 = 40 \text{ mm} \). These parameters combined with the linear torch/deposition speed of 1200 mm/min and 0.1 s dwell at the end of each side of the width, provided an equivalent of 50 mm/min linear bead deposition, comparable to the linear deposition strategy.

\[ \text{(a) Schematic of the deposition} \quad \text{(b) zig-zag parameters} \]

\[ \text{Figure 3-5 – Linear and zig-zag strategies} \]
The torch translation direction was unidirectional (one-way) for both linear and zig-zag deposition strategies, as shown in Figure 3·6 (a and b). All layers started from one end of the substrate and finished at the other end. This was to avoid causing damage to the torch and wire-feeder. The rotation of the wire-feeder could be an option to enable two-way deposition (bi-directional deposition), however, this would introduce a series of artefacts due to an un-balanced and non-symmetric deposition process, which has been avoided in this research work.

(a) One-way deposition: torch and wire Feeder
(b) Schematic of the unidirectional deposition
(c) Deposition direction and clamping of the substrate

Figure 3·6 – Deposition setup for both Linear and zig-zag strategies

The substrate was clamped at the middle point of each side, as shown in Figure 3·6 (c), by using two bolts and fixings on the CNC-bed. The middle-position of the clamps, rather than end clamping, was utilised based on previous trials, where cracks appeared, as a consequence of adding deposited layers, as shown in Figure 3·7. Cracking on deposited layers was mainly observed on large parts (longer length deposition) and has been considered as an indication of brittle oxide formation within the microstructure and residual stress. The middle-position clamps allowed the substrate to bend, across the length, and prevented cracking across the length of the beads.

There are a range of other process parameters could be considered for this set up process. For instance, the substrate temperature and the inter-pass temperature, as also mentioned in section 3.4, thermal analysis of the process. However, here the focus was on the actual parameters, which could be controlled and monitored via the PTA equipment or the CNC bed.
Figure 3.7 – Transverse cracks, observed during additive-layer deposition, mainly on large parts, the substrate was clamped at both ends

To take the inter-layer phenomena into consideration, effect of each layers’ thermal history was studied by choosing two dwell-times of 60 s and 180 s between layer depositions. The temperature of the layers was measured at a number of time points between layer depositions to understand the effect of dwell-time as a process parameter.

It is important to note that trial builds were carried out with dwell times between 30 s and 360 s, with 30 s intervals before choosing the final parameters. 60 s was the shortest dwell time, which allowed deposition of a full height wall (50 mm) while no significant dimensional/shape changes was observed between 180 s and 360 s. Hence, 60 s and 180 s were chosen as the two best candidates to study the effect of the dwell-time.

Table 3.3 summarises the process parameters and their range for this research study. Based on the current setup of the PTA equipment, and the CNC-bed, three process parameters were considered as variables to develop the matrix for manufacturing of the PTA AM samples to investigate the effects of them on residual stress development.

<table>
<thead>
<tr>
<th>Process Parameter</th>
<th>Unit</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Minimum</td>
</tr>
<tr>
<td><strong>Deposition Strategy</strong></td>
<td>-</td>
<td>Linear</td>
</tr>
<tr>
<td><strong>Energy Density</strong></td>
<td>(MJ/m²)</td>
<td>~135</td>
</tr>
<tr>
<td><strong>Dwell Time</strong></td>
<td>s</td>
<td>60</td>
</tr>
</tbody>
</table>
Based on the principle of the Design of Experiment (DoE), various combinations of the three process parameters were considered. The DoE matrix for residual stress determination was generated based on the three main process parameters and two levels (min and max) for each of them as summarised in Table 3-3. To create this matrix, the factorial design of experiment was used, enabling process parameters interaction effects to be understood. The required builds to capture the effects of process parameters are listed in Table 3-4, which includes the repeat samples to examine the consistency of the process. In total twelve samples were manufactured for the purpose of residual stress analysis. Eight original samples based on the combinations of the three process parameters and four repeats for samples with the low-level of the energy density.

The samples are labelled as ‘X-YYY-Z’ to denote the process parameters (deposition strategy-dwell time-energy density), as shown under the ‘Sample ID’ column in Table 3-4.

<table>
<thead>
<tr>
<th>Sample No. (run order)</th>
<th>Sample ID</th>
<th>Deposition strategy</th>
<th>Energy density (MJ/m²)</th>
<th>Dwell time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>L-060-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>60</td>
</tr>
<tr>
<td>(and repeat)</td>
<td>L-180-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>180</td>
</tr>
<tr>
<td>2</td>
<td>L-060-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>60</td>
</tr>
<tr>
<td>(and repeat)</td>
<td>L-180-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>180</td>
</tr>
<tr>
<td>3</td>
<td>Z-060-L</td>
<td>Zig-Zag (Z)</td>
<td>150 (Low)</td>
<td>60</td>
</tr>
<tr>
<td>(and repeat)</td>
<td>Z-180-L</td>
<td>Zig-Zag (Z)</td>
<td>150 (Low)</td>
<td>180</td>
</tr>
<tr>
<td>4</td>
<td>Z-060-H</td>
<td>Zig-Zag (Z)</td>
<td>300 (High)</td>
<td>60</td>
</tr>
<tr>
<td>(and repeat)</td>
<td>Z-180-H</td>
<td>Zig-Zag (Z)</td>
<td>300 (High)</td>
<td>180</td>
</tr>
</tbody>
</table>

The schematic geometry of a sample is shown in Figure 3-8(a), and an actual sample is shown in Figure 3-8(b). The substrate dimensions were 100 (length) × 50 (width) × 7mm (thickness). Deposition of the layers started at 10 mm into the plate, ending at 10 mm from the plates end (layer/bead length of 80 mm).
The width of the beads was affected by the welding speed and the wire feed rate. This was measured to be 10mm (±1mm) for the linear beads, based on the parameter values in Table 3-4. In theory, in zig-zag deposition, the width of the beads can be controlled by changing the $V_1$ parameter, which defines the movement of the bed along the X-axis, as shown in Figure 3-5. In terms of the height of the samples, the deposition of the layers was continued until a 50 mm (from the substrate) high wall was achieved.

However, it was observed that the height reduced along the length of the samples towards the end. This is reported to be due to the lack of the heat sink ahead of the deposited material (and the torch) when bead deposition is finished (Martina et al., 2016). This effect can be reduced by increasing the wire feed rate towards the end of each bead. This was, however, a manual and imprecise process in WMG’s PTA setup. It was hence decided to keep the wire feed rate constant in the manufacturing of all the samples.

In some samples, a ‘balling effect’ was also observed, mainly towards the end of the deposited beads, as shown in Figure 3-9.
The balling effect could be attributed to a number of issues, including: lack of diffusion, non-alignment and lack of heat sink. However, as this was observed for some of the samples and not for all, a further detailed analysis of the process parameters suggested that a combination of the input energy of deposition and the wire-feed rate play a significant role in causing the balling effect. However, in this work, as the wire-feed rate was maintained constant during the deposition, the balling effect could be linked to the energy density of the deposition process. As mentioned in Equation 3·4, in calculating the energy density, the voltage and the deposition speed were constant while the current was varied. It seems that the lower current (and therefore the lower energy density) provides a more stable bead deposition and less balling effect.

Due the balling effect and the height drop towards the end of the deposited walls, the ‘analysis section’ of the sample is chosen to be 20 mm inside the length of the deposited wall from the start-side of the deposition, section A·A, as shown in Figure 3·8. This section is chosen for the residual stress measurement along the height of the samples (for both non-destructive and destructive residual stress analysis) as well as microstructural analysis of the samples.

It should be noted that during the initial trial, a wider range of process parameters and dimensions were used, mainly for the purpose of the mechanical and microstructural analysis, to gain a wider understanding of the manufacturing process. It has been clarified where other process parameters were used to manufacture samples, mainly to build samples for mechanical characterisation, where larger deposited walls were required.
3.3.2. Samples for mechanical and material characterisation

For the purpose of material characterisation, samples similar to the samples for the residual stress measurement are manufactured. For mechanical properties, however, larger samples are required to extract standard test specimens, as specified by ASTM E8 standard. To determine mechanical properties, a number of PTA AM parts were built to extract standard tensile samples in two in-plane directions. A substrate of 220 mm×120 mm×15 mm (Length × Width × Height) was used to deposit 200 mm × 20 mm × 100 mm (Length × Width × Height) walls through deposition of ~70 layers (average layer thickness of ~1.5 mm). The mid-range values of the three variable process parameters are chosen to cover the most possible variation of the properties, rendering process parameters as described in Table 3-5. The same tolerances as Table 3-2 are applied to the PTA parameters.

<table>
<thead>
<tr>
<th>Process Parameter</th>
<th>Value/Range (Unit)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Voltage</td>
<td>25 (V)</td>
</tr>
<tr>
<td>Current</td>
<td>100 (A)</td>
</tr>
<tr>
<td>Wire Feed Rate (WFR)</td>
<td>1·2 (m/min)</td>
</tr>
<tr>
<td>Plasma Gas flow Rate (PGFR)</td>
<td>0·4·0.7 (l/min)</td>
</tr>
<tr>
<td>Linear deposition speed</td>
<td>50 (mm/min)</td>
</tr>
<tr>
<td>Deposition strategy</td>
<td>zig-zag (v₁=20 mm, v₂=1 mm, v₃=100·110 mm)</td>
</tr>
<tr>
<td>Argon shielding (shielding device)</td>
<td>25 (l/min)</td>
</tr>
<tr>
<td>Dwell-time</td>
<td>360 (s)</td>
</tr>
</tbody>
</table>

To build walls for tensile samples extraction, zig-zag deposition was used to take account of the complexity of deposition strategy. The zig-zag deposition has also shown more stability in producing defined bead widths which is an influential factor to provide stability of deposited layers as they stack on top of each other, enabling production of larger walls to the required height of 100 mm.
A longer dwell-time of $360 \text{ s}$ was chosen for these large samples, as the bead’s length was more than double that of the residual stress samples (Section 3.3.1). A longer dwell-time provided the opportunity to additively deposit layers on a steady basis until the wall was complete.

Due to the larger length of the deposited layers/walls, a higher rate of external argon shielding was used during deposition to avoid oxidation of the material. The mid-range of current (as the variable parameter for energy density) was used ($100 \text{ A}$) and the nominal voltage of the process was measured to be $25 \text{ V}$. The equivalent linear speed of deposition was set to $50 \text{ mm/min}$, same as the value used to make the residual stress samples.

To avoid the balling effect and shrinkage of material towards the end of deposited walls, the wire feed rate was gradually increased from $1 \text{ m/min}$ to $2 \text{ m/min}$ manually, to add more material towards the end of deposited beads and compromise for the lack of material ahead of the heat source towards the end of deposition.

Seven walls (1 to 7) were built to extract “tensile” samples for mechanical testing. The CAD drawings of the walls, including the schematic of the tensile samples, and the actual parts (walls 1 and 4) are shown in Figure 3·10. Walls 1, 2, 3, 5 and 6 were used to extract tensile samples in the vertical (height) direction and walls 4 and 7 were built to extract horizontal samples (longitudinal direction). In the vertical direction, three tensile samples were extracted from each wall and in the horizontal direction five tensile specimens were extracted, as schematically shown on the CAD drawings of the PTA AM walls.
a) Walls 1, 2, 3, 5 and 6 for vertical specimens

b) Walls 4 and 7 for horizontal specimens

Figure 3.10 – PTA AM walls for tensile specimens extraction
3.4. Thermal analysis – dwell-time

Understanding the thermal history of the deposited walls was of importance as it influences the microstructure, material properties and causes residual stress, and distortion. To obtain an understanding of the thermal gradients and their effects during the deposition process, the temperature of the substrate and deposited layers was measured using a BP17 infrared pyrometer (Trotec GmbH) before and after the layer deposition. The nominal error of the device was ±10 °C. The temperature of each layer was measured twice, once 10 s after depositing the layer and again 10 s before the end of the dwell-time and the deposition of the next layer.

The temperature data from the substrate was also recorded by attaching thermocouples on the substrate, 10 mm from each side of the substrate (start and stop side, with relation to the deposition), and 10 mm from the bead, as it is shown in Figure 3-11.

The thermocouples was attached to the substrate, by using a ceramic/cement based paste, also known as sandy cement. They were firstly glued then applied the thermally conductive high temperature cement paste. After spreading it on the wire, it needed to rest/dry up for a couple of hours. This approach allowed making sure that the wire was directly attached on the substrate. To calibrate the thermocouples and ensuring they are collecting the right temperature data, the temperature was measured by both thermocouples and by pyrometer at the beginning.

![Figure 3-11 – Thermocouple to collect temperature data from the substrate](image-url)
The temperature data from the substrate was collected for a zig-zag sample with the lower level of the energy density and the dwell-time of 180 s (Z-180-L). Figure 3-12 shows the temperature data collected by the “start” and the “end” sides’ thermocouples.

![Figure 3-12](image)

*Figure 3-12 – Temperature plot of the start and the end points of the substrate during the deposition of the five layers*

The equivalent linear motion of the torch (and wire-feeder) was 50 mm/min, hence deposition length of 80 mm (layer length) took 100 s. Adding the dwell-time of 180 s (and set-up time of up to 40 s) would give the ~320 s between the peaks in Figure 3-12.

From the thermocouple on the “start-side”, the temperature for the first layer reached ~450 °C, 10 s after starting the deposition, while the temperature at the “end-side” was still at around ambient temperature. After ~10 s (to 20 s), the temperature at the end-side raised rapidly and reached ~500 °C just before deposition stopped at the end side of the substrate. After the dwell time, the temperature on the start-side dropped to 170 °C and the temperature at the end-side was ~177 °C. As soon as the deposition of the second layer started, the temperature at the “start-side” rapidly reached ~500 °C. This shows how the pre-heated previous layer contributed to the thermal gradient of the whole component (including the substrate) and increased the overall temperature of the deposit. The same effect was observed at the “end-side” of the substrate where the top temperature towards the end of deposition reached ~600 °C towards the end of depositing the second layer (in comparison to 500 °C for the first layer).
The same phenomenon was observed for the third layer, when the top temperature was increased on both the start and the end sides by a magnitude of \(~50\text{-}75^{\circ}\)C. However, the thermal gradients seemed to become steadier towards the fourth and mainly fifth layers. This conforms the thermal history of individual layers as shown in the next section.

Figure 3·13 shows the temperature profile for the first 10 layers of four samples with the low level of energy density, with two different deposition strategies (linear and zig-zag) and two different dwell-times of 60 s and 180 s. The dashed and solid graphs correspond to the temperatures immediately after deposition and after the dwell-time, respectively.

---

**Figure 3·13 – Temperature profiles for the first ten layers for the samples with the low level of the energy density**

For all four samples, the temperature of the new layer increased as more layers were deposited and the cooling rate showed a consistent trend for both samples with shorter dwell-time and for both samples with longer dwell-time. A steady cooling rate was observed, while the rate became slower on the top layers. For the linear samples L·60 and L·180, the temperature of the layers after deposition were almost identical and followed the same trend, ranging from \(~270^{\circ}\)C up to \(~890^{\circ}\)C. This was expected as these samples were produced using the same deposition strategy. Linear motion of the heat source provided a similar effect of the melt pool on the deposited and previous beads, indicating a good repeatability between the two samples. However, the temperature data, 10 s after
deposition, for the zig-zag samples Z-60 and Z-180 were not consistent. Sample Z-60 showed a higher temperature of the layers after deposition. In fact, the case of zig-zag deposition, oscillating the melt heat source, and as a result the melt pool, seemed to cause a more uniform heat distribution and less thermal difference compared to linear deposition.

Unlike the temperature data after layer deposition, the data after passing the dwell-time was shown to be more dependent on the dwell time rather than the deposition strategy. For both samples L-60 and Z-60, with a dwell time of 60 s, the temperature of the layers before depositing the next one followed the same trend, and were in the same range, starting from ~150 °C for the first layer up to 552 °C and 492 °C for samples L-60 and Z-60, respectively. A similar trend was seen between samples L-180 and Z-180, with the longer dwell-time of 180 s. The lower temperature of the layers for the zig-zag sample could be attributed to the more uniform heat distribution and extraction in the case of oscillated melt pool. Hence, it seemed that in zig-zag deposition, a longer dwell-time would be more effective in cooling down the sample, than the linear deposition.

The cooling rate was considerably affected by the dwell time, as it is proportional to the specific heat capacity and the thermal conductivity of the material. During the cooling of each layer, it was observed that cooling was almost doubled in the first 60 s, compared to the third 60 s of dwell time.

### 3.5. Mechanical and material properties

#### 3.5.1. Chemical composition of wire

The material of choice for this work was dual phase (α-β) Ti-6Al-4V alloy. Chemical composition of the Ti64 wire was determined using Inert Gas Fusion (IGF), which is also referred to as the “Leco Test”. IGF provides a quantitative analysis of the concentrations of oxygen, nitrogen, and hydrogen (ONH) in ferrous and nonferrous materials.

Samples of wire were taken from the wire-feeder at three stages during manufacture of the samples, before starting the process, halfway through deposition of layers and at the end of the process. The samples were accurately weighed and placed in a pure graphite crucible in a fusion furnace with inert gas atmosphere. The averaged values for chemical composition of the wire samples from ICP-MS analysis (Inductively Coupled Plasma – Mass Spectrometry) are shown in Table 3-6.
Table 3.6 – Chemical composition of the Ti₆Al₄V wires

<table>
<thead>
<tr>
<th>Weight %</th>
<th>Ti</th>
<th>C</th>
<th>Fe</th>
<th>N</th>
<th>Al</th>
<th>O</th>
<th>V</th>
<th>H</th>
<th>other</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti₆Al₄V</td>
<td>Bal.</td>
<td>0.08</td>
<td>0.3</td>
<td>0.05</td>
<td>5.5-6.75</td>
<td>0.20</td>
<td>3.5-4.5</td>
<td>0.015</td>
<td>0.4</td>
</tr>
</tbody>
</table>

3.5.2. Mechanical properties of PTA AM samples

Tensile tests were conducted on the samples according to ASTM E8 / E8M-16a, in strain rate controlled method. Strain rate before and after the Yield point were 0.005/min and 0.050/min, respectively. The results for Young’s modulus, UTS and elongation for PTA AM walls No. 1 to 7, are summarised in Figure 3.14.

Results from walls 1, 2, 3, 5 and 6 are for tensile properties on the “vertical” (height) direction, and results from walls 4 and 7 are for the “horizontal” direction (along the length). From the tensile test results, consistent tensile properties can be assumed for both vertical and horizontal directions of the sample allowing for isotropic assumption for the stress-strain relationship. Intra-sample variation was observed for different mechanical properties and then considered towards averaging the results for a global set of tensile properties for PTA AM samples.

Comparing the tensile properties with Aerospace Materials Specifications (AMS 4911), the Young’s modulus \( (E) \) seems to be lower than the requirement for walls 3 and 5, while results for other five walls are in a good range and correlation. The average of \( E \) for samples from walls 4 and 7, (walls for horizontal/longitudinal samples) displayed slightly higher values than the other samples. The average \( E \) out of all seven PTA AM walls is 113.8 GPa. The average UTS for walls 6 and 7 were lower than the requirements by AMS 4911, mainly because one sample from each wall showed a poor tensile strength property and broken unexpectedly, dropping the average UTS for samples 6 and 7 to 759 MPa and 763 MPa, respectively. However, both walls showed a good elastic deformation which is represented by acceptable \( E \). Therefore, the average \( E \) from these two walls were counted towards the average \( E \) of the PTA AM samples, but their UTS were excluded. The average UTS from all samples from walls 1 to 5 was 980 MPa, which is in a good range of AMS 4911 specification. Some of the samples displayed poor elongation, which could be an indication of porosity and brittle failure.
**Chapter 3 – PTA AM Process and Preliminary Assessment**

**a) Young’s modulus (E)**

**b) Ultimate Tensile Strength (UTS)**

**c) Elongation**

*Figure 3-14 – Tensile test results from PTA AM samples – Mechanical properties a) Young’s modulus E, b) Ultimate Tensile Strength (UTS) and c) Elongation*
The fracture surfaces for samples T2.1 and T5.1 (vertical samples) and samples T4.1 and T4.2 (horizontal samples) were analysed, using SEM and optical microscopy. The SEM and optical micrographs are shown in Figure 3-15.

![SEM micrograph of T2.1 fracture surface](image1)

![Optical micrograph of T2.1 fracture surface](image2)

![SEM micrograph of T5.1 fracture surface](image3)

![Optical micrograph of T5.1 fracture surface](image4)

\[ a) \text{Tensile samples T2.1 (top) and T5.1 (bottom)} \]

Figure 3-15 - SEM (left-hand-side) and optical (right-hand-side) micrographs of fracture surfaces

The fracture surface of T2.1 indicates a fracture, which falls in the boundary of ductile and brittle, with very little deformation and some surface dimples. This correlates to the 2.3% elongation observed for sample T2.1 (average of 3.6% for wall 2), these low values link to potentially poor shielding allowing the ingression of interstitials into the bulk. The fracture surfaces of T5.1 shows ductile fracture, with clear indication of deformation and surface dimples. This correlates to the 10.8% elongation observed for this sample (average of 9% elongation for wall 5).

Mechanical properties of deposited walls (PTA AM samples) are summarised in Table 3-7. Also, nominal material properties of the wire and substrate (grade 5), as specified by the supplier, are given in Table 3-7.
Table 3-7 – Mechanical properties of Ti-6Al-4V

<table>
<thead>
<tr>
<th>Mechanical Property</th>
<th>Value (Unit)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Wire and substrate</td>
</tr>
<tr>
<td></td>
<td>(grade 5)</td>
</tr>
<tr>
<td>Ultimate Tensile Strength (UTS)</td>
<td>1050 (MPa)</td>
</tr>
<tr>
<td>Yield Strength</td>
<td>900 (MPa)</td>
</tr>
<tr>
<td>Modulus of Elasticity</td>
<td>113.8 (GPa)</td>
</tr>
<tr>
<td>PTA AM sample</td>
<td>950 (MPa)</td>
</tr>
<tr>
<td></td>
<td>880 (MPa)</td>
</tr>
<tr>
<td></td>
<td>113.8 (GPa)</td>
</tr>
</tbody>
</table>

In Table 3-7, the results from PTA AM walls are shown to be comparable for mechanical properties of the Ti-6Al-4V, grade 5. Unless otherwise stated, the mechanical properties for the PTA AM have been used as the benchmark values throughout this research work.

3.6. Microstructural analysis

During processing of the α+β titanium alloys, specifically in the presence of high thermal gradients and different cooling rates, a diverse microstructure could appear which results in a local variation of mechanical and material properties (Lütjering and Williams, 2007).

Following the establishment of the process window, different samples with various number of layers, heights and other processing parameters were manufactured for the purpose of microstructural analysis. Range of techniques were employed to conduct a series of analyses on different PTA AM samples.

3.6.1. Sample preparation and analysis

As a general guideline, sample preparation was required for all microstructural analysis techniques. This included polishing the surface of the sample to give the best reflection of interacting beams/lights and provide an accurate micrograph or other properties as being investigated. Sample preparation for metallographic investigation and microstructural characterisation was conducted according to material characterisation guidance by Buehler® SumMetTM (Buehller SunMet, 2013).

The samples were prepared for all microstructural analysis by mounting in conductive phenolic resin, grinding and polishing to a 1μm finish. Sample preparation can be summarised as follows:
• The cross-section of the samples was further sectioned to extract samples from top, middle and bottom of the samples. A minimum surface area of ~ 100 mm\(^2\) (the bead face in the direction of deposition) was chosen for investigation.

• The sectioned samples were mounted in a 40mm diameter conductive Bakelite. Normally, all three sections were kept on a single mounting for examination, although in some cases different sections were mounted separately.

• An Ecomet 250 grinder polisher (Buehler, UK) was used to prepare the samples. The following steps were followed:
  
  o A 320-grit SiC paper was used for initial grinding. A load of 27 N was applied on the sample in the specimen holder rotating at 60 rpm. The base with SiC paper was kept at a rotational speed of 300 rpm. The relative motion between the sample holder and base plate was kept complementary or same direction. This operation was water-cooled and was carried out until initial removal of surface deformations on the sectioned plane of the sample post cutting operation (1min to 1.5 min).
  
  o This was followed by a 9µm diamond suspension polish using a hard woven Ultra pad cloth. The base speed was reduced to 150rpm and same force was applied on the mounted sample. This operation was performed for 10min in counter clockwise relative direction of motion between base and sample holder.
  
  o The final polishing was performed using a 0.02-0.05µm water-based colloidal silica solution for 10 min. A soft synthetic porous ChemoMet cloth was used. The force on the samples was reduced to 22N. The other parameters were kept similar to previous step.

It should be noted that after each step, the samples were thoroughly cleaned.

Optical micrographs were obtained by using a Nikon Eclipse LV150N optical microscope (Nikon Metrology, UK).

A Zeiss Scanning Electron Microscope (SEM) Sigma Field Emission (FE-SEM) (Carl Zeiss Ltd, UK) with Angular selective Backscatter detector (AsB) was used to investigate the microstructure of the weld deposits. The AsB detector was selected to obtain a high atomic number contrast, using an accelerating voltage of 20kV. Backscattered Electron microscopy (BSE) was used to identify different chemical compositions and different phases within the dual-phase titanium weld deposits. Micrographs for three samples are
presented here: a two-layer sample (~3.5 mm in height), a seven-layer sample (~10 mm in height) and a complete sample for residual stress measurement (~50 mm in height, 26 layers).

### 3.6.2. PTA AM analysis – 2-layer sample

To gain a fundamental understanding of the microstructure, a two-layer PTA sample was produced using the linear deposition strategy, with the height of ~3.5 mm. The geometry of deposition and the process parameters were the same as sample 1 in Table 3-3 (sample ID: L-060-L). The sample was cut across its cross-section and prepared for microstructural analysis as explained in the previous section (3.6.1). Micrographs were obtained by using optical microscopy using polarised light from three regions of the sample to study the difference in microstructure between bottom, middle and top of the deposited layers, as schematically shown in Figure 3-16.

![Figure 3-16 – Schematic of a two-layer sample and scan areas for micrographs](image)

All micrographs from three regions were shown in Figure 3-17 (left-hand-side: a to c) with the higher magnification (right-hand-side: a’ to c’).
An increase in columnar $\beta$ grain size from the bottom to the top of the height can be observed in the sample, which is due to the link between epitaxial growth and heterogeneous nucleation. This can be attributed to the rate of solidification during the process as also reported by (Vilaro, Colin and Bartout, 2011).

Figure 3-17 (a') and (b') show higher magnification optical micrographs of the selected regions from the top and middle, which illustrate a finer lamella microstructure. This Widmanstatten or Basket weave type microstructure has been reported by previous researchers as a result of precipitation of $\alpha$ phase within the $\beta$ grains due to rapid cooling cycles during the process (Poondla et al., 2009).
A coarser microstructure was seen towards the bottom of the weld, which can be seen in Figure 3-17 (c'). Previous studies have shown that due to repeated thermal cycles in a multi-layer PTA deposition of Ti-6Al-4V (which causes re-heating of the previous layer), coarsening of α phase can occur as the temperature of the layers away from the heat source remains below the β-transus temperature (Addison et al., 2015).

3.6.3. PTA AM analysis – 7-layer sample

As the next step, a 7-layer sample was built with the same process parameters for the two-layer sample, as also described in Table 3-4 (sample ID: L-60-L), to study the effect of PTA AM process as the number of layers are added up. Deposition of seven layers resulted in a sample with ~10 mm height. The sample was cut across the cross section in order to gain an understanding of the microstructure from the top to the bottom of the wall, in different heights.

Backscattered Electron (BSE) images were obtained from four different regions of across the height of the sample, as schematically shown in Figure 3-18. Scanning of the sample was conducted on the top layer, on the middle of the height, on the intersection of the deposited layers with the substrate. Images were also obtained from the substrate. The micrographs are shown in Figure 3-18.
Similar to the 2-layer sample, a fine microstructure of mainly α-phase can be observed in the bottom layers (section D) while the α-lamellae are separated by very fine columnar β-grains in boundaries. However, the β-microstructure is not noticeable on the bottom layer. Moving upwards to the top layers, an increase in precipitated β-grain boundaries within the α-microstructure can be observed. This has resulted in a basket-weave (Widmanstätten-type) formation of α-CPH crystals as observed in the two-layer sample.

Figure 3-18 – Backscattered electron micrograph of cross-section of Ti 6Al 4V seven layer weld deposit as shown on the left. A’ is the higher magnification of the region A
3.6.4. PTA AM analysis – 27-layer samples

To understand the microstructure of the PTA AM parts, which were built for residual stress measurement, two 50 mm tall PTA AM samples, were sectioned across the same scan-line for stress measurement (Chapter 4). The scan-line along the height of the sample is shown schematically in Figure 3-8. The two samples were labelled L180L (linear, low energy density) and Z-180-H (zig-zag deposition strategy, high energy density), as specified in Table 3-4.

The sample was sectioned to cut a section of 10 mm from the “start-side” of the deposition, as shown in Figure 3-19.
Samples were extracted from the top layers, the middle section and the bottom of the cross section of the deposited wall. The samples were polished and prepared for microstructural analysis as described in samples preparation (3.6.1), and micrographs were obtained, as shown in Figure 3·20 and Figure 3·21. A slight porosity was observed on the micrographs from the top and the middle of the cross section of the linear sample, L·180·L. Hence, Omnimet image analysis software (Buehler) was used to compute the porosity levels using the object selection and area fraction (%) method.

![Figure 3·20 – Backscattered electron micrographs from three regions across the cross section along the height (Z-direction) of a 50mm PTA AM Ti-6Al-4V sample (L·180·L)](image)

In general, a fully lamellar α + β microstructure could be observed on the cross section of the linear sample (L·180·H). The sample from the bottom region of the cross-section seems to have a ‘fine’ α + β lamellar microstructure.
Moving upwards to the middle section of the sample, the precipitated β-grains become more lengthy and visible and surrounding the primary α-phase. The α-platelet remained as the dominant phase, though.

On the top section however, the retained long β-grain boundaries become even more considerable although it is still precipitated within α-phase. The porosity level seemed to increase from bottom to the top region. On the bottom region, an equivalent of 0.84% porosity was observed while it becomes 0.94% for the middle region and just over 1.02% within top layers.

The shape of the build geometry can be described in relation to the wire feed rate, volume of the material deposited and the energy input per unit length of deposited layer, as outlined in Equation 3-4 (Martina, 2014).

\[
\text{Volume rate} \left( \frac{\text{mm}^3}{\text{s}} \right) = \frac{(\pi \times r^2 \times \text{WFR})}{\text{Deposition speed} \left( \frac{\text{mm}}{\text{s}} \right)} \quad \text{Equation 3-4}
\]

Where the WFR represents the Wire Feed Rate (in mm/s) and r is the radius (curvature) of the top of the bead as being deposited.

Further details on the effect of the deposition process and relationship between the geometrical aspects of the deposited layers and the process parameters can be found in (Martina, 2014).
A similar trend on the microstructure of the zig-zag sample was observed. Starting from the bottom of the cross section, a fully lamellar “fine” α+β microstructure was observed. The α-phase seemed to dominate the microstructure in a basket-weave-type microstructure with very thin grain boundaries as β-phase. This could be an indication of the recrystallization of precipitated β-grains into α-microstructure and is also observed in conventional Ti-6Al-4V microstructure in the literature (Poondla et al., 2009; Antonysamy, 2012). Moving to the middle section, precipitated primary α-phase was visible which could be related to more effects from the β-phase in comparison to the bottom section. This type of microstructure was also reported as an “acceptable” arrangement for Ti-6Al-4V alloy, where the heating and cooling rate could affect the final microstructure
(Antonysamy, 2012; Wang et al., 2013). The top section also showed a lamellar α+β microstructure with more evident and lengthy β-grains; surrounding the equiaxed α-phase, which was cooled down from the β-phase field.

Considering the porosity, the overall porosity level in the zig-zag sample was less than 0.5%, which was lower than the linear sample. This could be due to the higher energy input for the Z-180-H than the L-180-L sample.

Long columnar β-grains was also reported as an effect of AM process for PBF and DED processes. Williams et al (Williams et al., 2015b) described AM of titanium alloys as a micro-casting process, resulting in long columnar grains that are highly textured and could potentially lead to anisotropy in mechanical and material properties. The repeated thermal cycles induced by the deposition of each layer cause local variations of the microstructure, with inconsistent lamellae size. These features are undesirable from a design point of view and may limit the implementation of AM. This effect is more considerable for DED AM processes (specifically for PTA AM) as a higher deposition rate causes more heating and cooling phenomena. Rolling, heat treatment or other post processing of deposited layers has been proposed as a solution to produce a refined microstructure in deposited Ti-6Al-4V (Martina et al., 2012; Wang et al., 2013; Martina, 2014). However, it is not always possible to integrate such post-processing for an AM process. During trials in this work, it is evident that the combination of processing parameters could have a major effect on this, and by choosing the right set of parameters, semi-uniform, isotropic material properties can be achieved without the need for further “post-processing”.

3.7. Summary

This chapter provided a foundation to study the effect of PTA AM process on the microstructural evolution and residual stress formation. Understanding the concept of this DED AM technique was the main aim where the current set up for the PTA AM process at WMG, University of Warwick was explained in detail and the set of process parameters was explored. A preliminary assessment of the mechanical properties and microstructural characteristics for the PTA AM samples made out of Ti-6Al-4V was conducted.
To investigate the evolution of residual stresses, three main parameters and their levels were chosen to establish the process parameters matrix employed to manufacture samples. In total, eight different combinations of process parameters (and their levels) were chosen to investigate their conjunctive effects on residual stress evolution. Twelve samples were manufactured (eight main samples and four repeats from the lower energy density level) to determine distribution and level of residual stresses within them.

A similar matrix was chosen for mechanical and material characterisations, although some additional combinations of process parameters and their levels were chosen to manufacture extra samples and justify the processing parameter window. A series of samples were manufactured for the purpose of mechanical testing and microstructural characterisation. Samples with different heights (number of layers) were manufactured to examine the microstructure of the PTA AM parts in different regions along the height of deposited layers. A two-layer (~3.5 mm height), a seven-layer (~10 mm height) and a 26-layer (50 mm height) sample was made and prepared for microstructural analysis. Different microstructural analysis techniques were used: polarised microscopy and Scanning Electron Microscope (SEM) (backscattered electron detector) images of the microstructure.

The micrographs indicated that there were more uniform microstructure, basketweave (Widmanstätten) α-phase on the lower layers while the length and presence of columnar prior-β grains increased towards the upper regions. A coarser Widmanstätten α-phase was observed on the upper layers surrounded by thicker and longer β-grains extended on the microstructure. Although different combinations of process parameters showed a various extend and density of each phase on the final microstructure, it was shown to be a similar finer α-lamella on the bottom layers, transformed-β microstructure and more β-phase precipitated towards the upper layers in all samples.
Chapter 4 – Methodologies and Assumptions to Determine Residual Stress in PTA AM Parts
4.1. Introduction

An understanding of the PTA AM process was obtained in Chapter 3, where mechanical and microstructural properties of the PTA AM parts was also analysed. Chapter 3 showed the capability of the PTA AM technique to build parts with mechanical properties very similar to conventional techniques for Ti-6Al-4V and therefore the mechanical properties was identified.

There is however, a further need to analyse the evolution of residual stress to ensure the geometrical, shape and size accuracy of the PTA AM manufactured parts. Understanding the level and variation of residual stress and potential link to the processing parameters would allow one to manage the effect of the manufacturing process on the final shape and size accuracy of PTA AM parts. In an automatic AM process, the ideal outcome would be a replication of the precise geometry/dimensions as defined by the CAD model. However, the heating and cooling phenomena combined with additive layer/material processing, typically leads to significant distortion in metal-AM parts, driven by residual stress.

Residual stresses are self-equilibrating stresses occurring in a component after removing the original cause/s of the stress, such as external forces, heat gradient and mismatch of thermal coefficients during cooling or heating of multiphase materials. A fundamental understanding of residual stress formation during the manufacturing process is a vital step to develop such a methodology.

This Chapter outlines the three methodologies to determine the level and the variation of the residual stress in PTA AM parts manufactured in Chapter 3. This would allow investigation and discussion of the potential effect of the manufacturing process on the final state of residual stress.
4.2. Neutron diffraction to determine residual stress

4.2.1. The principle of neutron diffraction

Diffraction-based techniques utilise Bragg’s law to quantify the relationship between the atomic d-spacing ($d_{hkl}$) of certain lattice planes to the diffraction angle ($2\theta_{hkl}$) at which the radiation is scattered coherently and elastically for a given wavelength of the radiation. Comparing the d-spacing of strained lattice with un-strained lattice could result in determination of residual stress. As explained in chapter 2, based on Bragg’s law, the angle at which any given diffraction peak occurs is related to d-spacing and incident wavelength (Equation 4-1).

$$n\lambda = 2d_{hkl} \cdot \sin\theta_{hkl}$$  \hspace{1cm} Equation 4-1 – Bragg’s law

Where ‘n’ is an integer and ‘$\lambda$’ is the neutron wavelength in Å (i.e. $10^{-10}$ m). The spacing between the planes in the atomic lattice is ‘d’ (same unit as ‘$\lambda$’) and the angle between incident and diffracted beams is $2\theta$.

In a diffraction-based technique, the lattice spacing of crystal planes acts as a strain gauge. Stress causes changes in lattice spacing resulting in a different diffraction angle. The magnitude of strain is determined by comparing the diffraction angle against a known (stress-free) sample. A gauge volume is established via definition of the incident and diffracted beam, which captures the lattice structure. The gauge volume in neutron diffraction is almost cubic in shape, as schematically shown in Figure 4-1. The technique was covered in full details in the literature review (Chapter 2).

Figure 4-1 – Schematic of the incident and diffracted beam and formation of a near-cubic gauge volume in neutron diffraction (Watkins et al., 2013)
Strain can be calculated along the direction of the \( \mathbf{Q} \)-vector, as shown in Figure 4·1. This direction is typically set to be one of the principal axes of the stress/strain state. The strain-free and strained lattice (d-spacing) are schematically shown in Figure 4·2, which shows how a strained lattice affects the angle between the incident and scattered beam (\( 2\theta \)). By measuring the angle (\( \theta \)) and considering the reference value (based on a strain-free sample), strain can be determined for each (principal) direction. Measurement of the angle \( (2\theta \) or \( \theta \)) can be repeated for other principal directions (at the same point) to give a full picture of the strain state at one point in a component or specimen.

\[ \varepsilon = \frac{d}{d_0} - 1 = \frac{\sin\theta_0}{\sin\theta} - 1 \]

Equation 4·1 – Strain – d-spacing
The obtained principal strain values \((\varepsilon_x, \varepsilon_y, \varepsilon_z)\) at each point, can be converted to principal stress values \((\sigma_x, \sigma_y, \sigma_z)\), by using Hooke’s law, as given in Equation 4·3.

\[
\sigma_x = \frac{E}{(1 + v)(1 - 2v)} [(1 - v)\varepsilon_x + v(\varepsilon_y + \varepsilon_z)]
\]

\[
\sigma_y = \frac{E}{(1 + v)(1 - 2v)} [(1 - v)\varepsilon_y + v(\varepsilon_x + \varepsilon_z)]
\]

\[
\sigma_z = \frac{E}{(1 + v)(1 - 2v)} [(1 - v)\varepsilon_z + v(\varepsilon_x + \varepsilon_y)]
\]

\[\text{Equation 4·3 – Hooke’s law}\]

Where \(\sigma_{(x,y,z)}\) is the (principal) stress (along the direction as defined by \(\text{Q·vector}\)), \(v\) is the Poisson ratio, \(E\) is the Young’s modulus and \(\varepsilon_{(x,y,z)}\) is the strain (along the direction of the \(\text{Q·vector}\)). The uncertainties for the strains \((\Delta \varepsilon)\) and stress \((\Delta \sigma)\) can be given by Equation 4·4 (Szost et al., 2016).

\[
\Delta \varepsilon = \sqrt{\frac{d_0^2 \Delta d^2 + d^2 \Delta d_0^2}{d_0^4}}
\]

\[\text{Equation 4·4 – Uncertainties for strain and stress}\]

\[
\Delta \sigma_{ij} = \frac{E}{(1 + v)(1 - 2v)} \left[ (v - 1)^2 \Delta \varepsilon_{ij}^2 + v^2 (\Delta \varepsilon_{jj}^2 + \Delta \varepsilon_{kk}^2) \right]
\]

It should be noted that all the stress-strain relationships of continuum mechanics were obtained by considering the average or ‘macroscopic’ deformations and stresses within the elastic limit of the material (Young’s modulus of \(E\) and Poisson ration of \(v\)). However, in determination of strains (and stress) by diffraction techniques, the strain state is evaluated with respect to a particular family of planes, which implies that the microstructural formation (microscopic strain and stress) affects the overall stress state. Hence, the real stress value at each point depends on microstructural properties. In such cases the use of microscopic Young’s modules \((E_{hkl})\) and microscopic Poisson’s ratio \((\nu_{hkl})\) of a plane with Miller indices “hkl” has been suggested, (Lorentzen et al., 2005).

Depending on the geometry of samples and the stress component/s of interest, typically, points of interest are measured in three principal directions to provide a full stress state at each point. This is conducted by rotating the sample such that the \(\text{Q·vector}\) is aligned with each principal direction. Hence, the strain component for the associated principal direction is determined.
4.2.2. Plane stress condition and reference d-spacing ($d_0$) in neutron diffraction

To calculate strain as a function of 2θ, a reference value (2θ₀) should be used, which is logically derived from an unstrained crystal structure. In reality this is a difficult process as all materials have a level of strain due to interatomic forces and a balance of entropy.

The choice of strain-free inter-planar distance ($d_0$) (or corresponding 2θ₀) has been reported as the main issue with diffraction techniques, as covered in the literature review (Chapter 2). Different methods have been used to ensure the accuracy of data, typically dependent on the geometry of samples and measurement/scanning strategy. However, in most cases it remains a challenge to justify the choice of reference value based on only one set of experiments, mainly due to possible microstructural changes and/or the formation of the gauge volume to capture microstructural features. It is therefore recommended to conduct neutron and synchrotron X-ray diffractions as complementary techniques, due to their corresponding gauge volume shapes (nearly cubic versus extended) and potentially providing a more comprehensive overview of the strained lattice (Rossini et al., 2012). In most cases, other experimental data (e.g. from destructive methods) and/or simulation-based solutions have supported results from neutron and synchrotron X-ray diffractions to ensure the accuracy of final results (Lorentzen et al., 2005)(Lorentzen et al., 2005; Rossini et al., 2012).

Typically, in thin samples, when the thickness is smaller than the planar dimensions, the stress is assumed not to change along the thickness (between the two opposite parallel surfaces. This assumption results in a “plane stress” or “biaxial stress” conditions, which means the stress along the direction perpendicular to the planar surface is assumed to be negligible (out-of-plane stress being zero). In such cases, applying the plane stress condition could provide a solution to determine the reference d-spacing.

In plane stress condition, the out-of-plane stress is assumed to be zero to determine the reference lattice parameter and strain can be calculated based on the average lattice parameters across all scan-points, for a specific material (here Ti-6Al-4V). The global coordinate system for the PTA AM samples is shown in Figure 4-3.
By assuming the plane stress condition on Y-Z plane and X-axis being normal to the surface, the out-of-plane component of stress and strain can be rewritten as Equation 4-5.

\[
\sigma_{xx} = \frac{E}{(1 + \nu)(1 - 2\nu)}[(1 - \nu)\varepsilon_x + \nu(\varepsilon_y + \varepsilon_z)] = 0
\]

Equation 4-5 – out-of-plane stress and strain

\[
\varepsilon_{xx} = \frac{-\nu(\varepsilon_y + \varepsilon_z)}{(1 - \nu)}
\]

By applying plane stress condition, the in-plane stress components were calculated using the reduced Hook’s law, as Equation 4-6.

\[
\sigma_x = 0
\]

\[
\sigma_{yy} = \frac{E}{1 - \nu^2}(\varepsilon_{yy} + \nu \varepsilon_{zz})
\]

Equation 4-6 – Hooke’s law (plane stress)

\[
\sigma_{zz} = \frac{E}{1 - \nu^2}(\varepsilon_{zz} + \nu \varepsilon_{yy})
\]
4.3. Residual stress determination by synchrotron X-ray diffraction

4.3.1. The principle of synchrotron X-ray diffraction

High energy synchrotron X-ray can provide an alternative or complementary solution to determine residual stresses in polycrystalline materials. The concept is similar to neutron diffraction and is based on directing a beam on a component and measuring the angular distribution of the radiation diffracted from the material.

The high energy photons in synchrotron X-rays can range between 20 – 300 keV. The relatively high energy leads to very low scattering angles, typically ranging from about 10° at moderate energies (~25 keV) to about 4° at higher energies (~80 keV). As a consequence of low scattering angles obtained using high energy X-rays, an elongated gauge volume, normally a diamond-shape is formed as opposed to the near-cubic gauge volume in neutron diffraction. The schematic of a low scattering diffraction and the elongated gauge volume is shown in Figure 4-4. Typically, the nominal gauge width is approximately 5–20 times that of the height. The synchrotron X-ray diffraction is considered as a complementary technique to other non-destructive (and even destructive) methods to evaluate residual stresses.

![Schematic of the incident and diffracted beam and formation of an elongated gauge volume in synchrotron X-ray diffraction in two directions of a sample](image)

*Figure 4-4 – Schematic of the incident and diffracted beam and formation of an elongated gauge volume in synchrotron X-ray diffraction in two directions of a sample (Withers and Schajer, 2013)*
By using Bragg’s law (Equation 4-1), the “d-spacing” is determined against the unstrained lattice structure, as schematically shown in Figure 4-2. The associated strain and stress can be calculated by using Equation 4-2 and Equation 4-3, in three principal directions.

High energy X-rays are becoming more increasingly available for materials science investigations, for example beamlines ID11, ID15 and ID22 at the ESRF, Grenoble, France. The penetration power of X-rays in the range 50–100 KeV allows a novel solution for the elucidation of high resolution stresses. Through the use of motorised incident and detector slit systems, the typical diffraction geometry can be readily modified so that high resolution residual strain can be measured.

4.3.2. Plane stress condition and reference d-spacing (d₀) in synchrotron X-ray diffraction

Similar to neutron diffraction, to determine residual strain (and stress) by synchrotron X-ray diffraction, a reference value for the strain-free lattice parameter (d₀) is required to enable calculating strain.

Typically, in synchrotron X-ray diffraction, only two components of the strain are determined. This is mainly due to the low scattering angle, causing elongated gauge volume, in which does not allow formation of the gauge volume along the length, penetrating inside the sample. The strain components are calculated by using Bragg’s law and strain – d-spacing relationship, Equation 4-2. By applying the plane stress condition and referring to Equation 4-5, the in-plane stress components are calculated using the reduced Hook’s law as in Equation 4-6. As only two strain components can be determined, the strain-free d₀ cannot be calculated, so no variation of the d₀ can be applied.

Scanning for two principal directions is conducted by rotating the samples in two directions to give strain (Q-vector) in two in-plane directions (Y and Z), assuming a plane stress condition, the out-of-plane stress is zero (σₓ=0).

As there was no data for out-of-plane strain (σₓ=0), the reference lattice parameter is calculated by referring to the assumed the unstrained inter-planar distance (d₀) from neutron diffraction data. As the first step, an average value of the d-spacing were calculated from all scan points on each sample and then compared to the reference d-spacing from neutron diffraction to adjust it accordingly.
4.4. Contour method: a mechanical stress measurement technique

4.4.1. The principle of the contour method

Mechanical methods (also called stress-relaxing methods) are used to analyse the stress-relaxation produced in metal parts when a layer of material or clamps or any other type of constrains are removed (Prime, 2001).

The advantage of the contour method over neutron diffraction is the capability of the technique to evaluate stress within the substrate as well as the wall, which is not possible to achieve using neutron diffraction due to the maximum depth the neutron beam can travel into the material and the location of the gauge volume. However, this is a destructive method. The generic process of the contour method to determine residual stress is summarised in Figure 4-5.

![Figure 4-5 – The process of contour method](image)

The cutting process is known as the most critical step of the process, as it provides the relaxed contours on the cross section, for stress analysis. The typical approach to the cutting process is using wire EDM (Electric Discharge Machining) to allow deformed contours being generated in the cut surface with maximum accuracy and detail. The cutting direction is an important factor. The aim would be a smooth cutting path to minimise the potential effect of wire-bouncing (wire effects) during the cutting process. Another critical consideration is the change in the geometry of the cross section being cut. Such geometrical changes could produce artefacts on the relaxed contours, which needs to be considered carefully during the analysis stage.

Direction of cutting on a PTA AM sample is schematically shown in Figure 4-6. A constant power was used throughout the cutting process to avoid any unpremeditated changes of the relaxed contours (deformed surface). Change of the cross section could have an influence on the final contours. To minimise this effect, use of sacrificial materials is considered.
Chapter 4 – Methodologies and Assumptions to Determine Residual Stress in PTA AM Parts

Figure 4.6 – Wire EDM cutting process for contour method

The cut section was scanned on a Coordinate Measurement Machine (CMM) to read the relaxed contours. The scanning process was conducted for both sections (both surfaces) of the samples and then the results were averaged to provide a comprehensive set of data and to minimise any errors. A Renishaw TP8 touch probe scanner (Renishaw UK, New Mills, UK) was used to read the data across the perimeter of both parts of the cut section. The surface of both cross sections was scanned using a laser scanner (Micro-Epsilon UK Ltd, Birkenhead, KK) to provide a grid of data across the cross section. One part of the cut cross section and the touch probe and laser scanner are shown in Figure 4.7.

Figure 4.7 – Scanning process; touch probe (left) and laser scanner (right) to measure the surface deformation

The next step was to correct the data for the perimeter data to eliminate the probe radius. Then the contours (deflections) from both surfaces are averaged which resulted in a single point cloud of the relaxed contour. The point cloud was then imported into a Finite Element code (CAE/ABAQUS 6.13) to read the deformation and stress contours.
4.4.2. Section-cut and plane stress condition in contour method

As explained in Chapter 3, the contour method provides stress info perpendicular to the cut-section. Hence, it is critical to obtain an understanding of the stress state and ensure that the correct stress component is chosen when the section is cut. Since contour method is a destructive technique, the same specimen/section cannot be used for stress determination in other directions, but the sample should be cut again according to the stress component/s of interest.

This limitation makes the contour method a complementary technique to determine residual stress. Data from the contour method is typically compared against data from non-destructive methods to provide a comprehensive understanding of the stress variation in components.

Considering plane stress condition in both neutron and synchrotron X-ray diffraction, the contour method is utilised to determine stress along the length of the samples (Y-direction), as the stress component of interest.

Yet again, a second cut was also defined to provide more details about the out-of-plane stress component (σₓ=0), again as a complementary step for further discussion around the choice of reference d-spacing, as explained in Chapter 8.

4.5. Residual stress/strain data (and comparison)

The residual stress determination techniques provide a series of data along different directions (horizontal and vertical) within the PTA AM parts, as explained in more details in the subsequent results chapters. In presenting the data in this thesis, the strain and calculated stress are presented along different scan-lines across individual samples (Chapters 5, 6 and 7). The data for samples are then superimposed to enable comparison between samples, with regard to the overall variation of the stress (and strain) and to discuss the potential effect of any individual and/or combination of process parameters.

Furthermore, the maximum, the minimum and the mean values of the stress (and strain) data, along different scan-lines in different directions are presented (in the results Chapters 5, 6 and 7) and compared where applicable (in the discussion Chapter 9).
4.6. Summary

The three methods, which will be used to measure residual stress in this research work, were explained in details in this Chapter.

The following three Chapters; 5, 6 and 7, will present results from these techniques. In presenting results, the maximum, minimum and mean values across data points are calculated and presented to summarise the data (stress and strain) based on each measurement technique. This data will be used to analyse the evolution of residual strain and stress and discuss differences between samples as well as the effect of process parameters.

All three residual stress measurement techniques assumed a plane stress condition for all stress calculations. This assumption led to stress concentration in two in-plane principal detections (Y and Z) while assuming negligible stress on the out-of-plane direction ($\sigma_x=0$). Further discussions and justifications for the plane stress condition and the reference d-spacing ($d_0$) consideration and assumption is provided in details in Chapter 8.
Chapter 5 – Neutron Diffraction to Determine Residual Strain and Stress in PTA AM Parts
5.1. Introduction

As a non-destructive diffraction-based technique, neutron diffraction provides information about lattice d-spacing. Comparing the strained d-spacing with the unstrained crystal structure provides an understanding of the residual strain/stress.

Following the methodology of neutron diffraction in Chapter 4, this chapter provides a detailed overview of the experimental procedure undertaken during the neutron diffraction experiment at the Institut Laue Langvein (ILL), Grenoble, France.

Considering the plane stress condition in PTA AM walls, the strain was calculated by using Bragg’s law as explained in Chapter 4. The corresponding residual stress results were calculated by using Hooke’s law for the plane stress condition and considering mechanical properties as explained in Chapter 3.

The sample matrix for residual stress measurement was discussed in Chapter 3 (Table 3.4) and it is repeated here in Table 5.1. As mentioned previously, the samples were labelled with the processing parameters to make discussions clearer. “Repeat samples” were also manufactured to investigate the repeatability of the process, and labelled with “R”. Also, for each residual stress measurement technique, the measurement (or scanning) on at least one sample was repeated, to verify the results from each technique.

This chapter covers the experimental procedure and the results from the neutron diffraction studies of twelve samples from Table 5.1.

The results from this chapter will be discussed in Chapter 9 along with the results from the other two residual stress measurement techniques (synchrotron X-ray and contour method) to investigate the link between the PTA-based additive-layer process parameters and the evolution of residual stress in PTA AM parts.
### Table 5-1 – The samples matrix for residual stress analysis

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Sample ID</th>
<th>Deposition strategy</th>
<th>Energy density (MJ/m²)</th>
<th>Dwell time (s)</th>
<th>Neutron diff.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>L-060-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>60</td>
<td>✓</td>
</tr>
<tr>
<td>2</td>
<td>L-180-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>180</td>
<td>✓</td>
</tr>
<tr>
<td>3</td>
<td>L-060-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>60</td>
<td>✓</td>
</tr>
<tr>
<td>4</td>
<td>L-180-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
</tr>
<tr>
<td>5</td>
<td>Z-060-L</td>
<td>Zig-zag (Z)</td>
<td>150 (Low)</td>
<td>60</td>
<td>✓</td>
</tr>
<tr>
<td>6</td>
<td>Z-180-L</td>
<td>Zig-zag (Z)</td>
<td>150 (Low)</td>
<td>180</td>
<td>✓</td>
</tr>
<tr>
<td>7</td>
<td>Z-060-H</td>
<td>Zig-zag (Z)</td>
<td>300 (High)</td>
<td>60</td>
<td>✓</td>
</tr>
<tr>
<td>8</td>
<td>Z-180-H</td>
<td>Zig-zag (Z)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
</tr>
<tr>
<td>2 (repeat)</td>
<td>R-L-180-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>180</td>
<td>✓</td>
</tr>
<tr>
<td>4 (repeat)</td>
<td>R-L-180-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
</tr>
<tr>
<td>6 (repeat)</td>
<td>R-Z-180-L</td>
<td>Zig-zag (Z)</td>
<td>150 (Low)</td>
<td>180</td>
<td>✓</td>
</tr>
<tr>
<td>8 (repeat)</td>
<td>R-Z-180-H</td>
<td>Zig-zag (Z)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
</tr>
</tbody>
</table>

#### 5.2. Neutron diffraction scanning procedure

Neutron strain scanning was carried out using the SALSA instrument (Strain Analyser for Large Scale Applications) at the ILL (ILL experiment: 1-04-86). A neutron wavelength of 1.69 Å was used and the titanium α-phase (101) peak at ≈41.22° 2θ was selected for the scans (Voisey et al., 2014). The gauge volume was defined by vertical and horizontal collimators, giving an effective gauge volume of 2 mm³.

The suggested plane by the ISO standard is α (103) for titanium. However, the (101) plane was shown to give the highest peak strength and visible reflection which was present through the whole sample (along different scan lines/points) in the angular diffraction. However, it was acknowledged that it does not justify the choice of this plane for diffraction to calculate the ‘actual’ stress value. As it was explained in the methodology Chapter 4, the comparison of strain magnitudes was considered as the first step, to give
Chapter 5 – Neutron Diffraction to Determine Residual Strain and Stress in PTA AM Parts

authenticity to the discussions. The stress variation was considered as the main point of comparison and discussion to evaluate the effect of combination of processing parameters on the evolution of the residual stress.

In total, twelve samples were scanned via neutron diffraction. Eight samples, with different combination of process parameters, plus four 'repeat' samples. For all samples, two main in-plane scan lines (along the length and height) were chosen with a number of scan points, as shown in Figure 5-1, to give an overview of the level of the residual stress along different directions. An additional vertical scan line (along the height) was also chosen with fewer scan points, as a validation step to investigate the variation of residual stress along the height of the samples. The coordinate system for scanning each point was aligned with the coordinate system of the manufacturing process, as explained in chapters 3 and 4 (Y: longitudinal direction of deposition, Z: transverse direction of deposition or height of the sample and X: normal to the deposited wall).

![Figure 5-1 – Schematic of the part and neutron diffraction measurement points and definition of the coordinate system](image]

Scan line 1

A total of nine points were scanned for this scan line, from along the height of the samples. The scan line was located 20 mm inside the wall, from the lateral edge on the start side (side at which the deposition started) of the deposited walls. The scan points started from 10 mm below the reference point (Z-coordinate of -10 mm from the reference) and were 5 mm apart with the last point at the intersection of the wall and the substrate (Z-coordinate of -50 mm). Scanning the first scan point at 10 mm below the reference point
(top surface) was to eliminate the artefact from neutron diffraction scanning, as the area of interest was the main wall. It is assumed that the few top layers could be machined/removed after manufacturing, as is also explained in Chapters 2 and 3.

**Scan line 2**

This scan line was along the length of the deposited samples/walls, on the longitudinal direction of the beads. Nine points were scanned along this scan line, from the lateral edge of the deposited wall to the middle of the samples (40 mm into the wall). The scan line was 25 mm below the reference point (Z-coordinate of 25 mm), started from the edge of the wall and were 5 mm apart from each other, with the last point at the middle of the wall. Again, the choice of this scan line was to focus on the area of interest, starting from the start side of the sample (where the layer deposition occurred stably). The other side of the wall, showed a decline in the flat surface of the layer, as discussed in Chapter 3.

**Scan line 3**

This is the extra scan line along the height of the sample, parallel to scan line 1, 10 mm shifted longitudinally down the length of the deposited wall (from the start side of the sample). Five points were scanned along this scan line, starting 10 mm below the top surface (same height as the first scan point for scan line 1). The scan points were 10 mm apart until the intersection of the deposited wall and the substrate.

Also for two samples, L-180-H (4) and Z-180-L (6), a “finer” scan line was chosen along the height of the sample, coincident with scan line 1, with scan points being 1 mm apart. Hence, a total of fifty (50) scan points were scanned along the height of the two samples.

To scan through the points, the samples were mounted on a hexapod reference table and scanned through the neutron beam, as shown for one of the samples in Figure 5-2. By rotating the sample, each point was scanned in three principal directions of strain; on the principal directions: longitudinal (Y), transverse (Z) and normal (X), as explained in Figure 5-1. Therefore, the sample was rotated to align the Q-vector with the associated direction of strain. The set-up to scan each point through three principal directions is schematically shown in Figure 5-2. Measurement time was set to be 10 minutes per point (on each scan point in each direction).

The raw data from SALSA was processed using a dedicated piece of software developed at the ILL, called LAMP; which is a data handling programme (LAMP, 2006) (Hughes et al., 2006).
(a) Mounting a sample on the sample stage

(b) Sample rotation to scan along the desired Q-vector; view from top (left hand-side) and view from side (right hand-side)

Figure 5.2 – Experimental set-up (a) mounting a sample on the stage (hexapod) and (b) sample rotation for neutron diffraction measurements along two principal directions: longitudinal strain, and transverse strain

In presenting the results from the neutron diffraction scanning, the scanned 2θ are plotted for two samples, while the measured strain and corresponding stress are presented for all samples. Furthermore, the maximum tensile and compression values and mean of the strain data and associated stress magnitudes are presented for all samples/scan-lines, which will provide a basis for direct comparison between samples.
5.3. Results

The residual strain and stress results for all samples scanned by neutron diffraction are presented in this section, to show the level and distribution of residual strain/stress. Residual strain was calculated based on the assumed $2\theta_0=41.22^\circ$. The results are given for two in-plane components of strain (longitudinal: Y and transverse: Z), which is required to calculate the two in-plane stress components, according to the two-dimensional Hooke’s law and the plane stress condition (out-of-plane stress: $\sigma_{zz}=0$).

It must be noted that all data ($2\theta$, strain and residual stress) are plotted on similar scales (for all samples/scan lines) to provide a basis for further discussions along with the data from other residual stress measurement techniques.

5.3.1. Sample L-060-L (1)

For sample L-060-L (1), the measured $2\theta$ is shown in Figure 5-3, along two in-plane principal directions (Y and Z). The strain and stress results from the top to the bottom of the height, along the vertical scan lines 1 (nine scan points) and 3 (five scan points) are shown in Figure 5-4, for the two in-plane principal directions: longitudinal (Y) and transverse (Z).

![Figure 5-3 – $2\theta$ along the height (scan lines 1 and 3) of sample L-060-L (1) in two in-plane directions: longitudinal (Y) and transverse (Z) (average error: ±0.005) (as-welded)](image-url)
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Figure 5-4 – Results along the height of sample L·060·L (1): scan line 1 (9 scan points) and scan line 3 (5 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
A good correlation between the results from scanlines 1 and 3 can be observed (Figure 5·4). The slight difference between the results can be justified by the 10 mm distance between the two vertical scan lines. Scan line 1 was closer to the start edge of the deposited wall compared to scan line 3, hence higher stress levels.

The same set of data along the length of the sample was given for scan line 2 (nine scan points) halfway through the height of the sample. The measured $2\theta$ along the length of the sample L·060·L (1), horizontal scan line 2, are shown in Figure 5·5. The strain and stress results are plotted in Figure 5·6.

*Figure 5·5 – $2\theta$ along the length (scan line 2) of sample L·060·L (1) in two in-plane directions: longitudinal (Y) and transverse (Z) (average error: ±0.005)*
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Figure 5.6 – Results along the length of sample L-060-L (1) (average error: ±250×10⁻⁶)

a) Residual strain along the length of sample L-060-L (1) (average error: ±250×10⁻⁶)

b) Residual stress along the length of sample L-060-L (1) (average error: ±50 MPa)

Longitudinal strain (εy) scan-line 2
Transverse strain (εz) scan-line 2

Longitudinal stress (σy) scan-line 2
Transverse stress (σz) scan-line 2

Distance from the start-side lateral edge (mm)
The stress and strain variations along the height appeared to be greater than along the length of the sample, according to Figure 5-5 and Figure 5-6. This was the case for both strain/stress components (longitudinal and transverse). Although, the transverse strain and stress components ($\varepsilon_z$ and $\sigma_z$) were lower than the longitudinal components ($\varepsilon_y$ and $\sigma_y$).

The strain (and stress) was comparable along scan lines 1 and 3. Along the height of the sample, strain (and stress) was compressive at the top layer while becoming a tensile stress and became compressive moving to the middle of the height. The longitudinal residual stress profile became tensile at the intersection of the substrate and the deposited wall. The maximum tensile stress occurred at 15 mm below the top layer with the magnitude of ~314 MPa. The maximum compressive stress was shown to be at the bottom-middle of the sample at the height of 30 mm below the top surface of the wall, with the magnitude of ~310 MPa.

Along the length of the sample, the horizontal scan line 2, the longitudinal component showed a tensile stress near the lateral edge, while the transverse component appeared to be compressive stress near the lateral edge, although it did not follow a visible trend. The corresponding scan points on the vertical and horizontal scan lines (25 mm below the reference point on the vertical scan line 1 (and 3) versus 20 (and 30 mm) from the lateral surface towards the inside of the sample on the horizontal scan line 2) showed a similar stress level. The maximum tensile stress was 156 MPa and the maximum compressive stress is ~158 MPa.

### 5.3.2. Sample L-180-L (2) and repeat sample R-L-180-L (R2)

Sample L-180-L (2) was manufactured using the same processing parameters as sample L-060-L (1), except for dwell-time, where a triple dwell-time of 180 s was implemented. Residual strain/stress profiles along the height (scan lines 1 and 3) and length (scan line 2) of the sample are shown in Figure 5-7 and Figure 5-8, respectively.

Also, residual stress results for the repeat sample R-L-180-L (R2) are plotted in Figure 5-9, along both the height and the length of the sample.
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a) Residual strain along the height of sample L-180-L (2) (average error: \( \pm 250 \times 10^{-6} \))

b) Residual stress along the height of sample L-180-L (2) (average error: \( \pm 50 \) MPa)

Figure 5.7 – Results along the height of sample L-180-L (2): scan line 1 (9 scan points) and scan line 3 (5 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
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Figure 5-8 – Results along the length of sample L·180·L (2): scan line 2 (9 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) 

- **a)** Residual strain along the length of sample L·180·L (2) (average error: ±250×10⁻⁶)

- **b)** Residual stress along the length of sample L·180·L (2) (average error: ±50 MPa)

![Graph showing residual strain and stress along the length of sample L·180·L (2)]
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Figure 5.9 – Residual stress in two in-plane directions: longitudinal (Y) and transverse (Z) for repeat sample R·L·180·L (sample R2) a) along scan line 1 (height) and b) along scan line 2 (length)
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From the top to the bottom of the height of the sample (scan lines 1 and 3), the longitudinal residual strain ($\varepsilon_y$) started from a tensile status at the top scan point (10 mm below the top surface) and became compressive towards the middle of the height. The strain changes to tensile status towards the bottom of the height (the intersection of the deposited wall and the substrate). A similar trend was observed for the stress level, where the longitudinal stress component was +51 MPa at 10 mm below the top layer and the stress became compressive towards the middle of the sample (~266 MPa at the height of 30 mm below the top surface). Again, a tensile stress appeared towards the bottom of the sample with the stress of ~55 MPa at the intersection of the deposited wall and the substrate. Results along both scan lines 1 and 3, along the height of the sample, showed a comparable trend (Figure 5-7).

For the longitudinal stress component ($\sigma_y$), the maximum (tensile) stress was +120 MPa at 15 mm below the top surface and the minimum compressive stress was ~266 MPa through the middle of the height, at the height of 30 mm below the top surface. The maximum and minimum stresses for the transverse stress component ($\sigma_z$) were 23 MPa tensile stress at a height of 15 mm below the top layer and ~269 at a height of 30 mm below the top layer, same as the longitudinal stress component.

The strain variation along the length of the sample was lower than for the strain variation along the height of the sample. Strain was compressive for both longitudinal and transverse strain components at the lateral edge of the sample, being ~1230 ($\varepsilon \times 10^{-6}$) and ~2159 ($\varepsilon \times 10^{-6}$), respectively. Strain became tensile towards the first half of the sample while the strain variation was much less than the strain variation along the height. Considering the plane stress condition, the stress status followed the same trend for both longitudinal and transverse stress components. The maximum longitudinal stress magnitude along the length of the sample was 331 MPa at the lateral edge. However, disregarding the lateral edge effect, the maximum tensile stress (longitudinal component) occurred 15 mm into the sample with a magnitude of 67 MPa and the minimum compressive stress was observed to be ~97 MPa, at 10 mm into the horizontal scan line 2 (Figure 5-8).

A good correlation could be observed between the stress results for samples L-180-L (2) and R-L-180-L (R2), along both the height and the length of the samples. Stress along the height started with a tensile stress of +65 MPa at the top scan points (10 mm below the top surface) and became compressive towards the middle, showing a compressive stress of ~102 MPa at 25 mm below the top layer and +84 MPa at 30 mm below the top surface.
Considering the maximum and minimum stress magnitudes along the height and length, similar to sample L-180-L (2), the variation of stress appeared to be higher along the height than the length of the sample. The maximum (tensile) stress along the height was +188 MPa while the maximum (tensile) stress along the length was +106 MPa. The same scenario appeared to be the case for the maximum compressive stress along the height and the length of the sample, being -169 MPa and -83 MPa, respectively.

5.3.3. Sample L-060-H (3)

Linear deposition strategy was used to build sample L-060-H (3) and the dwell-time was kept at 60 s. The sample was, however, built by using the higher level of the heat source’s energy density, compared to samples L-060-L (1) and L-180-L (2).

The results along both in-plane scan lines (vertical: scan lines 1 and 3 and horizontal: scan line 2) for sample L-060-H (3), are shown in Figure 5-10 and Figure 5-11, respectively. The results include residual strain data for both in-plane components (longitudinal and transverse) and corresponding residual stresses, along both height (vertical scan line) and length (horizontal scan line).

![Graph showing residual strain along the height of sample L-060-H (3)](image)

*a) Residual strain along the height of sample L-060-H (3) (average error: ±250×10⁻⁶)*
\[ \text{Residual stress along the height of sample L-060-H (3) (average error: ±50 MPa)} \]

Figure 5-10 – Results along the height of sample L-060-H (3): scan line 1 (9 scan points) and scan line 3 (5 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
a) Residual strain along the length of sample L-060-H (3) (average error: ±250×10⁻⁶)

![Graph showing residual strain along the length of sample L-060-H](image)

b) Residual stress along the length of sample L-060-H (3) (average error: ±50 MPa)

Figure 5-11 – Results along the length of sample L-060-H (3): scan line 2 (9 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress

Along the height of the sample L-060-H (3), the variation of both longitudinal stress (σ_y) and transverse stress (σ_z) were in agreement between both scan lines 1 and 3.

The strain at the first scan points, 10 mm below the top surface was compressive for the longitudinal component (ε_y=-921 ε×10⁻⁶), and tensile for the transverse stress component (ε_z=206 ε×10⁻⁶). The corresponding stress state was compressive for both stress components, with -110 MPa for the longitudinal stress (σ_y) and -15 MPa for the transverse stress component (σ_z). The stress state became tensile towards the bottom of the wall for the longitudinal stress component, where the transverse stress component showed a slight tensile status at that point. Again, the variation of the longitudinal stress seemed to be more considerable than the transverse stress, along the height of the sample.

The strain/stress behaviour along the length of the sample, horizontal scan line 2, seemed more stable than the vertical scan lines 1 and 3. The maximum magnitude of the strain was near the edge, with a value of 1028 (ε×10⁻⁶) at the lateral edge for the transverse
strain ($\varepsilon_z$) and 5 mm from the lateral edge for the longitudinal strain ($\varepsilon_y$), with a value of $1138 \times 10^{-6}$.

5.3.4. Sample L·180·H (4) and repeat sample R·L·180·H (R4)

Sample L·180·H (4) was manufactured by using the same process parameters as sample L·060·H (3), with one exception: a longer dwell-time of 180 s. A repeat of this sample was also built, and labelled as R·L·180·H (R4). The results are shown in Figure 5·12, for the vertical scan lines 1 and 3 (along the height), and the strain results for a finer scan line, coincident with scan line 1 are included. The results for the horizontal scan line 2 (along the length) are plotted in Figure 5·13. The same plane stress condition was applied to calculate stresses and the errors were given for stress values. Also, the residual stress results for the repeat sample R·L·180·H (4), along both vertical and horizontal scan lines, are plotted in Figure 5·14.

*a) Residual strain along the height of sample L·180·H (4) (average error: $\pm 250 \times 10^{-6}$)*
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b) Residual stress along the height of sample L·180·H (4) (average error: ±50 MPa)

Figure 5.12 – Results along the height of sample L·180·H (4): scan line 1 (9 scan points) and scan line 3 (5 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
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a) Residual strain along the length of sample L·180·H (4) (average error: ±250×10⁻⁶)

b) Residual stress along the length of sample L·180·H (4) (average error: ±50 MPa)

Figure 5·13 – Results along the length of sample L·180·H (4): scan line 2 (9 scan points)
in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
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Scan lines 1 (9 scan points) (average error: ±50 MPa)

Scan lines 2 (9 scan points) (average error: ±50 MPa)

Figure 5.14 – The level/variation of residual stress on the longitudinal (Y) and transverse (Z) directions for repeat sample R-L-180-H (sample R4) a) along scan line 1 b) along scan line 2

The results for sample L-180-H (4) had a similar trend to sample L-060-H (3), with a compressive stress at the top and changing towards a tensile stress at the bottom of the deposited wall. However, towards the middle of the wall, the residual stress data was more similar to the data for sample L-180-L (2), where the only difference between the two samples was the energy density. The results for the repeat sample R-L-180-H (R4), however, seemed to be more similar to sample L-180-L (2). Again, the only difference between the two samples is the level of energy density. Overall, a similar trend for residual stress profiles in samples L-180-H (4) and R-L-180-H (R4) could be observed.

Along the height of sample L-180-H (4), residual strain (and stress) appeared to have a similar trend, along both scan lines 1 and 3. The longitudinal component of residual strain ($\varepsilon_y$) starts from compressive strain of -2061 ($\times 10^6$) and changed to tensile strain of +672 ($\times 10^6$) at 30 mm below the top surface and again fall down to the compressive stress of -1104 ($\times 10^6$) and again went back to tensile strain towards the substrate. The
corresponding residual stress ($\sigma_y$) followed the same trend, starting from a compressive stress of $-300$ MPa at 10 mm below the top layer, followed by a tensile stress of $+113$ MPa at 5 mm above the substrate, however ending with a compressive stress of $-87$ MPa at the intersection of the wall and the substrate. This seemed to be due the effect of the transverse component of the residual strain. The transverses strain were fluctuating along the scan line 1, showing a mainly compressive strain along the height. This trend caused the overall longitudinal stress ($\sigma_z$) become compressive at the bottom of the wall. For the transverse component, the minimum compressive strain ($\varepsilon_z$) was $-1313 \times 10^{-6}$ at the bottom of the wall from scan line 1 and $-1639 \times 10^{-6}$ from scan line 3. The corresponding transverse ($\sigma_z$) stresses were $-178$ MPa for scan line 1 and $-216$ MPa for scan line 3. Longitudinal strain results for the finer scan through scan line 1 were in agreement with the results from the coarser scan.

Along the length of sample L-180-H (4), scan line 2, a more steady strain (and stress) state could be observed from the lateral edge towards the middle of the sample with only one outlier strain of $1494 \times 10^{-6}$ at 15 mm inside the wall. However, considering the higher error at this point for the corresponding stress, these values cannot be trusted.

Considering the results for the repeat sample (R-L-180-H) (R4), a similar trend to sample L-180-H (4) could be observed along the height, while the variation of stress seemed to be less along the length. This confirms that the outlier value of strain (and stress) along the horizontal scan line 2 for sample L-180-H (4), cannot be trusted.

In both samples L-180-H (4) and R-L-180-H (R4), the longitudinal stress ($\sigma_y$) seemed to be the dominant stress component, while the transverse stress ($\sigma_z$) had a similar trend but with less variation between the maximum and minimum values in the repeat sample compared to the original sample.

### 5.3.5. Sample Z-060-L (5)

The zig-zag deposition strategy was utilised to manufacture sample Z-060-L (5). The dwell-time was 60 s and the lower level of energy density was used similar to the linear version of this sample: L-060-L (1).

Results along the height of the sample (scan lines 1 and 3) are shown in Figure 5-15 and results along the length of the sample (scan line 2) are plotted in Figure 5-16 for both in-
plane directions (longitudinal and transverse directions), employing the same plane stress condition for stress calculations.

![Graph showing residual strain and stress](image)

**a) Residual strain along the height of sample Z-060-L (5) (average error: ±250×10⁻⁶)**

![Graph showing residual stress along the height of sample Z-060-L (5) (average error: ±50 MPa)](image)

**b) Residual stress along the height of sample Z-060-L (5) (average error: ±50 MPa)**
Figure 5.15 – Results along the height of sample Z-060-L (3): scan line 1 (9 scan points) and scan line 3 (5 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z).

a) Residual strain along the length of sample Z-060-L (5) (average error: ±250×10⁻⁶)

b) Residual stress along the length of sample Z-060-L (5) (average error: ±50 MPa)
The results along the height of the sample seemed to follow the same trend along scan lines 1 and 3. Again, the longitudinal strain ($\varepsilon_y$) and stress ($\sigma_y$) seemed to be dominant compared to the transverse components. Also, variation of the strain/stress seemed to be higher along the height (scan lines 1 and 3) than the length (scan line 2) of the sample.

Along the height, strain (and stress) started with a compressive state at the top scan points, 10 mm below the top surface, for both longitudinal ($Y$) and transverse ($Z$) components; although the magnitude of the transverse components seemed to be lower than the longitudinal ones. Unlike other samples, the stress and strain state remained compressive through the height until towards the bottom the wall, where it grew rapidly to a tensile state at 40 mm below the top surface and the maximum tensile strain of 1591 ($\times10^{-6}$) and tensile stress of +167 MPa, at the intersection of the wall and the substrate. There was probably more tensile stress within the substrate to equate the effect of compressive stress and give equilibrium throughout the whole sample. This will be further investigated in the subsequent chapters, by the contour method.

Residual strains/stresses along the horizontal scan line 2 seemed to have a more stable behaviour. It started with a tensile stress at the edge of the sample, showed a slight compressive, and then slight tensile behaviour as shown up to 40 mm from the edge of the deposited wall.

5.3.6. Sample Z-180-L (6) and repeat sample R-Z-180-L (R6)

Results for sample Z-180-L (6) along scan lines 1 and 3 (height) and scan line 2 (length) of the sample are given in Figure 5·17 and Figure 5·18, respectively. Also, the stress results for repeat sample R-Z-180-L (R6) are plotted in Figure 5·19.

A fine scan line coinciding scan line 1 was also scanned on this sample, only for the longitudinal component $2\theta_y$ (and strain), with 1 mm step-size between scan points along the height, from top surface (height of zero from the reference point) to the intersection of the wall with the substrate. The strain results are included in Figure 5·17.
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a) Residual strain along the height of sample Z-180-L (6) (average error: ±250×10^6)

b) Residual stress along the height of sample Z-180-L (6) (average error: ±50 MPa)

Figure 5.17 – Results along the height of sample Z-180-L (6): scan line 1 (9 scan points) and scan line 3 (5 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
Figure 5.18 – Results along the length of sample Z-180-L (6): scan line 2 (9 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
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Figure 5-19 – Residual stress on the longitudinal (Y) and transverse (Z) directions for repeat sample R-Z-180-L (R6) a) along scan line 1 b) along scan line 2
In sample Z-180-L (6), results along scan lines 1 and 3 seemed to follow the same trend, although the level of strain (and stress) seemed to be slightly higher on the scan line 3 scan points compared to the corresponding scan points on scan line 1. Also, the strain and stress variation seemed to be more dominant for the longitudinal (Y) components.

Along the height of the sample, both longitudinal and transverse strain (and stress) started with a slight compressive state at the scan points 10 mm below the reference point and then became tensile at 15 and 20 mm below the top layer. The strain and stress trend was followed by a compressive state at the middle of the sample (25 and 30 mm below the reference point). Moving towards the intersection of the substrate and the deposited wall (40 to 50 mm below the reference point), the strain (and stress) state seemed to move towards a tensile behaviour. Although it remained slightly compressive on scan line 1, it showed a tensile stress on scan line 3.

Along the length of the sample, a more stable stress trend could be observed along the horizontal scan line 2. This seemed to be the case for both in-plane stress components (longitudinal: $\sigma_y$ and transverse: $\sigma_z$). The longitudinal stress ($\sigma_y$) started with a negative compressive stress and showed a very slight variation along the half-length (scan line 2). A similar slight variation was observed for the other component of the stress ($\sigma_z$), however it started with a positive tensile stress at the lateral edge and moved to a compressive state for the rest of the scan line 2.

Stress results from the repeat sample; R-Z-180-L (R6) were similar to the trend from results for sample Z-180-L (6), along both horizontal and vertical scan lines (1 and 3), although slight differences could be seen between the two set of results. The stress results from the repeat sample showed slightly less variation compared to sample Z-180-L (6). This was the case for both horizontal and vertical scan lines and at some of the scan points, the transverse component showed a more dominant effect.

Along the height of the repeat sample, both longitudinal ($\sigma_y$) and transverse ($\sigma_z$) stress components started with a compressive state going into a tensile behaviour and shifting to a compressive state at the middle of the sample. The stress statutes then reached to a slight tensile stress at the intersection of the wall and the substrate, which was opposed to the stress state for the same scan points from sample Z-180-L (6).

The maximum and minimum stress values for both stress components were slightly less in the repeat sample compared to the original sample Z-180-L (6).
Along the horizontal scan line 2 in the repeat sample, the stress variation became even less significant in the repeat sample R-Z-180-L (R6) compared to sample Z-180-L (6). Similar to the original sample Z-180-L (6), the transverse component of the strain (and stress) seemed to play a more dominant role in stress variation along the length of the sample.

5.3.7. Sample Z-060-H (7)

To manufacture sample Z-060-H (7), the zig-zag strategy was utilised to deposit beads while the dwell-time was kept to the minimum of 60 s and the higher energy density was set on the heat source.

Results along the height of the sample, vertical scan lines 1 and 3, are given Figure 5-20 and results along the length of the sample, horizontal scan line 2 are plotted in Figure 5-21. Average errors are given for both strain and stress data.

a) Residual strain along the height of sample Z-060-H (7) (average error: ±250×10^-6)
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b) Residual stress along the height of sample Z-060-H (7) (average error: ±50 MPa)

Figure 5-20 – Results along the height of sample Z-060-H (7): scan line 1 (9 scan points) and scan line 3 (5 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress

a) Residual strain along the length of sample Z-060-H (7) (average error: ±250×10⁻⁶)
b) Residual stress along the length of sample Z-060-H (7) (average error: ±50 MPa)

Figure 5-21 – Results along the length of sample Z-060-H (7): scan line 2 (9 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress

The trend for the strain results (and associated stresses) along the height of sample Z-060-H (7) seemed to be similar for both scan lines 1 and 3, although the variation of the data was slightly less along the scan line 3. Once again, the variation of the longitudinal strain (and stress) seemed to be more than the transverse components of strain and stress.

Along the height of the sample, on the horizontal scan line 1, the longitudinal strain (and stress) starts with a minor compressive state at the top scan points, 10 mm below the reference point. Moving towards the bottom of the wall, a tensile state is observed at 15 and 20 mm below the reference point, where it started to decline to a compressive strain (and stress) again. Eventually the longitudinal data showed a tensile profile towards the bottom of the deposited wall, with the maximum strain of 1815 (×10⁻⁶) at 40 mm below the top surface and associate stress of 221 MPa at that point.

Along the horizontal scan line 2, the transverse component of the strain (and stress) seemed to have a higher variation than the longitudinal components, although the overall variation was far less than the vertical scan lines, along the height of the sample. This
confirmed the slight domination of the transverse components over the longitudinal components, along the horizontal scan line.

5.3.8. Sample Z-180-H (8) and repeat sample R-Z-180-L (R8)

Residual strain and stress results for sample Z-180-H are plotted in Figure 5-22 and Figure 5-23 for the vertical and horizontal scan lines, respectively. The strain and stress data along all scan lines are given for the longitudinal and the transverse components, and stress data were calculated by using the same plane stress condition.

Furthermore, the residual stress results for the repeat sample R-Z-180-H (R8) are shown in Figure 5-24. The strain were scanned along two scan lines (1 and 2) and the stress results are shown for the two in-plane components.

\[ \varepsilon \times 10^{-6} \]

a) Residual strain along the height of sample Z-180-H (8) (average error: \( \pm 250 \times 10^{-6} \))
Chapter 5 – Neutron Diffraction to Determine Residual Strain and Stress in PTA AM Parts

b) Residual stress along the height of sample Z-180-H (8) (average error: ±50 MPa)

Figure 5.22 – Results along the height of sample Z-180-H (8): scan line 1 (9 scan points) and scan line 3 (5 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress

a) Residual strain along the length of sample Z-180-H (8) (average error: ±250×10⁻⁶)
b) Residual stress along the length of sample Z-180-H (8) (average error: ±50 MPa)

Figure 5.23 – Results along the length of sample Z-180-H (8): scan line 2 (9 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress

a) Scan lines 1 (9 scan points) (average error: ±50 MPa)
b) Scan lines 2 (9 scan points) (average error: ±50 MPa)

Figure 5-24 – The level/variation of residual stress on the longitudinal (Y) and transverse (Z) directions for repeat sample R-Z-180-H (R8) a) along scan line 1 b) along scan line 2

Similar to other samples, the data along the height of sample Z-180-H (8) were in agreement between the two parallel vertical scan lines 1 and 3. Along the height of the sample, the longitudinal strain (εy) had a clear dominant effect up to near the intersection of the wall and the substrate. The longitudinal strain (and stress) started from a mild tensile status and the tensile strain becomes more considerable until the middle of the sample. Then a compressive strain (and stress) appeared in the middle of the sample towards the bottom part of the wall, where the minimum compressive strain occurred at 45 mm below the reference point, with a magnitude of -871 (×10⁶). The transverse strain (and stress) followed a similar trend towards the middle of the wall, where the transverse strain became a more considerable tensile strain.

Along the length of sample Z-180-H (8), both longitudinal and transverse components of strain showed a mild variation from the edge of the sample towards inside the wall. Longitudinal strain (εy) started with a tensile state and declined quickly as the scan points are moved inside the sample. In this sample, the variation of the stress and strain seemed to be slightly more along the length than the height of the sample.
Considering the data from the repeat sample R-Z-180-H (R8), the results for the vertical and horizontal scan lines conformed to the data from the original sample, although there are some differences between some of the individual scan points.

Along the height of the repeat sample, the strain and stress started with a slight compressive state, which was different from the original sample Z-180-H (8). Moving on towards the middle of the wall, however, the strain and stress followed a similar trend to the original sample Z-180-H (8). The stress state was tensile until the middle of the sample, where it declined to a compressive stress state until 40 mm above the substrate. The stress showed a very slight tensile behaviour at this point and continued to a compressive stress until the end of the wall. The longitudinal component showed a more considerable variation along the height of this repeat sample.

Along the height of the repeat sample, stress started with a tensile status for both longitudinal and transverse components at the edge of the wall. This was similar to the original sample Z-180-H (8). Data along the horizontal scan line showed a similar slight variation for both in-plane stress components until the middle of the wall.

5.4. Summary

Chapter 5 provided the results from neutron diffraction scanning of the samples. In total, twelve samples were scanned along vertical (height) and horizontal (length) scan lines. Eight samples with different combination of processing parameters and four ‘repeat’ samples to investigate repeatability of the manufacturing process.

Neutron diffraction provided an understanding of the level and variation of residual stress within PTA AM parts. However, it was shown that results from neutron diffraction, for some of the samples, could be difficult to interpret (or repeated) with confidence. The main reason was the incoherent diffraction for titanium, as the gauge volume from neutron diffraction could not capture multi-phase microstructure, effectively. This was predominantly the issue, where the large grain sizes and boundaries prohibited the effectiveness of the near-cubic gauge volume, produced by incident and diffracted beams in neutron diffraction.

It should be noted that position the sample to scan the correct direction of $2\theta$ and hence calculate the correct corresponding strain and stress is an important step in conducting neutron diffraction scanning. The alignment between the coordinate system for the
sample’s manipulator and the sample itself should be carefully considered and implemented throughout the experiment to render the human errors.

The results from neutron diffraction showed an acceptable level (lower than the material yield strength) of stress magnitudes and variation along both the height and the length of the deposited walls. Also the stress variation along the height (and length) of the repeat samples showed a good correlation with the original samples. Although the maximum and minimum (and average) of the stress variation seemed to be different between the original and repeat samples. It was therefore evident that the results from neutron diffraction need more in-depth analysis and a complementary set of data, from other methods.

Severe set of process parameters, where the effect of heat input or the distribution of the input energy becomes less controllable could cause such issues. It was shown that the data validity is limited by incoherent scattering from titanium, as also reported in the literature (Chapter 2).

The strain results for all twelve samples scanned via neutron diffraction are summarised in Table 5.2, for the main scan line along the height (scan line 1, 9 scan points), for both strain components: longitudinal strain ($\varepsilon_x$) and transverse strain ($\varepsilon_z$). Also, the same set of data are presented for stress results in Table 5.3.

The results along the length (scan line 2 with 9 scan points) are also summarised in Table 5.4 and Table 5.5, for strain and stress data, respectively.

Also, the strain results along the height (vertical scan line 1) and the length (horizontal scan line 2) are summarised in a bar chart, in Figure 5.25 and Figure 5.26, respectively. The summary of the strain results are based on maximum tensile/compression values and mean of the data points along the main vertical and horizontal scan lines, as described in Chapter 4 (section 4.5). This is to represent the variations of the residual strain/stress within the PTA AM samples and enable comparison between different samples.

- Generally, all samples show a tensile stress towards the top (10 mm below the reference point), a compressive stress towards the middle and then again a tensile stress towards the bottom of the wall (intersection with the substrate). The top layers however were not scanned by neutron, but they were scanned by synchrotron X-ray and the results are presented in the next chapter (Chapter 6). Also, the stress distribution (for the longitudinal stress component) is presented
for the entire cross section and was captured by the contour method and is presented in Chapter 7.

- However, some samples did not show tensile stress towards the top (10 mm below the top surface). Mainly for zig-zag samples, a considerable compressive stress is observed at 10 mm below the top layer.

- The variation of the longitudinal components of strain ($\varepsilon_y$) and stress ($\sigma_y$) was shown to be higher than the variation of the transverse components of strain ($\varepsilon_z$) and stress ($\sigma_z$), along both vertical (height) and horizontal (length) scan lines.

- It was shown that the transverse stress components ($\sigma_2$) become more considerable throughout the zig-zag samples. This is probably caused by the zig-zag deposition strategy as it made the out-of-plane direction (along the width of the deposited beads) to play a role in stress development within each bead and consequently within the whole sample.

- The variation of stress and strain along the height was shown to be more considerable than along the length of the samples. It was expected considering the deposition direction in manufacturing the samples.

- The results from neutron diffraction along the height are from 10 mm below the top surface to the intersection of the wall and the substrate, so they do not represent the data for the first 10 mm of the wall from the top surface. This should be noted when comparing the strain (and stress) variation between data from different residual strain/stress measurement techniques. Data for the first 10 mm of the wall from the top were captured/analysed by the other two residual stress measurement techniques, as provided in the two subsequent chapters, for the synchrotron X-ray (Chapter 6) and contour method (Chapter 7).
Table 5.2 – Summary of strain results along the height (scan line 1) from neutron diffraction (all strain data $\times 10^6 \varepsilon$) (average error: $\pm 250 \times 10^6$)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Strain component</th>
<th>Min</th>
<th>Max</th>
<th>Difference</th>
<th>Mean</th>
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<td>L-060-L (1)</td>
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Table 5.3 – Summary of stress results along the height (scan line 1) from neutron diffraction (all stress data in MPa) (average error: ±50 MPa)

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<th>Difference</th>
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**Table 5.4 – Summary of strain results along the length (scan line 2) from neutron diffraction (all strain data in ×10^6) (average error: ±250×10^6)**

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<th>Sample ID</th>
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Table 5.5 – Summary of stress results along the length (scan line 2) from neutron diffraction (all stress data in MPa) (average error: ±50 MPa)

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<thead>
<tr>
<th>Sample ID</th>
<th>Stress component</th>
<th>Min</th>
<th>Max</th>
<th>Difference</th>
<th>Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>L-060-L (1)</td>
<td>$\sigma_y$</td>
<td>-311</td>
<td>314</td>
<td>625</td>
<td>-22</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-335</td>
<td>221</td>
<td>555</td>
<td>-129</td>
</tr>
<tr>
<td>L-180-L (2)</td>
<td>$\sigma_y$</td>
<td>-266</td>
<td>121</td>
<td>387</td>
<td>-11</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-269</td>
<td>23</td>
<td>293</td>
<td>-126</td>
</tr>
<tr>
<td>L-060-H (3)</td>
<td>$\sigma_y$</td>
<td>-110</td>
<td>260</td>
<td>370</td>
<td>101</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-133</td>
<td>217</td>
<td>350</td>
<td>32</td>
</tr>
<tr>
<td>L-180-H (4)</td>
<td>$\sigma_y$</td>
<td>-301</td>
<td>113</td>
<td>414</td>
<td>-67</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-272</td>
<td>69</td>
<td>341</td>
<td>-108</td>
</tr>
<tr>
<td>Z-060-L (5)</td>
<td>$\sigma_y$</td>
<td>-368</td>
<td>167</td>
<td>535</td>
<td>-124</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-285</td>
<td>52</td>
<td>337</td>
<td>-146</td>
</tr>
<tr>
<td>Z-180-L (6)</td>
<td>$\sigma_y$</td>
<td>-322</td>
<td>73</td>
<td>395</td>
<td>-129</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-298</td>
<td>19</td>
<td>318</td>
<td>-149</td>
</tr>
<tr>
<td>Z-060-H (7)</td>
<td>$\sigma_y$</td>
<td>-154</td>
<td>229</td>
<td>384</td>
<td>31</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-98</td>
<td>240</td>
<td>338</td>
<td>4</td>
</tr>
<tr>
<td>Z-180-H (8)</td>
<td>$\sigma_y$</td>
<td>-105</td>
<td>224</td>
<td>329</td>
<td>23</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-141</td>
<td>77</td>
<td>218</td>
<td>-26</td>
</tr>
<tr>
<td>R-L-180-L (R2)</td>
<td>$\sigma_y$</td>
<td>-104</td>
<td>224</td>
<td>327</td>
<td>43</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-151</td>
<td>85</td>
<td>237</td>
<td>-64</td>
</tr>
<tr>
<td>R-L-180-H (R4)</td>
<td>$\sigma_y$</td>
<td>-196</td>
<td>253</td>
<td>449</td>
<td>-3</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-173</td>
<td>136</td>
<td>310</td>
<td>-30</td>
</tr>
<tr>
<td>R-Z-180-L (R6)</td>
<td>$\sigma_y$</td>
<td>-222</td>
<td>125</td>
<td>347</td>
<td>-58</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-226</td>
<td>60</td>
<td>286</td>
<td>-77</td>
</tr>
<tr>
<td>R-Z-180-H (R8)</td>
<td>$\sigma_y$</td>
<td>-157</td>
<td>152</td>
<td>310</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-172</td>
<td>115</td>
<td>288</td>
<td>1.5</td>
</tr>
</tbody>
</table>
Chapter 5 – Neutron Diffraction to Determine Residual Strain and Stress in PTA AM Parts

(a) Minimum, maximum and average, longitudinal strain ($\varepsilon_y$)

(b) Minimum, maximum and average, transverse strain ($\varepsilon_z$)

Figure 5.25 – Strain data, from neutron diffraction, along the height (scan line 1) (a) longitudinal strain ($\varepsilon_y$) and (b) transverse strain ($\varepsilon_z$)
Chapter 5 – Neutron Diffraction to Determine Residual Strain and Stress in PTA AM Parts

(a) Minimum, maximum and average, longitudinal strain ($\varepsilon_y$)

(b) Minimum, maximum and average, transverse strain ($\varepsilon_z$)

Figure 5.26 – Strain data, from neutron diffraction, along the length (scan line 2) (a) longitudinal strain ($\varepsilon_y$) and (b) transverse strain ($\varepsilon_z$)
Chapter 6 – Synchrotron X-Ray Diffraction to Determine Residual Strain and Stress in PTA AM Parts
6.1. Introduction

Neutron diffraction provided an understanding of the level and variation of residual stress within PTA AM parts. However, the long scan-time per point required for neutron diffraction resulted in a set of coarse scan-lines across the height and length of the samples. Also, as explained in chapter 2 (literature review), incoherent diffraction for titanium has been reported as a common issue in interpreting data from neutron diffraction.

As another non-destructive technique, synchrotron X-ray diffraction was used to determine residual stress in PTA AM parts. High energy synchrotron X-ray can provide a solution to determine residual stress in crystalline materials. The concept is similar to neutron diffraction and is based on directing an X-ray beam onto a component and measuring the angular distribution of the diffracted radiation from the material.

Considering the higher energy of the X-ray beam, it could produce a longer/wider gauge volume which is thought to capture more detail within the sample. Also, the higher energy of the incident beam means deeper penetration of the beam and hence a more accurate set of data. This can resolve the incoherent scattering issues as arose in neutron diffraction for titanium (Malinov et al., 2002).

Typically, the nominal gauge width is approximately 5–20 times that of the height. Therefore, synchrotron X-ray diffraction is considered as a complementary technique to other non-destructive (and even destructive) methods to evaluate residual stresses.

Central facilitates provide high energy X-rays for materials science investigations. Beamlines ID11, ID15 and ID22 at the ESRF, Grenoble, France, are well-known examples in Europe. The penetration power of X-rays in the range 50–100 keV allows a novel solution for the elucidation of high resolution stresses. Through the use of motorised incident and detector slit systems, the typical diffraction geometry can be modified so that high resolution residual strain can be measured.

Within the context of understanding of the evolution of residual stress, the aim is to investigate any potential link between the manufacturing process parameters and the state of the residual stress within the primitive structures. The focus is on the effects of heating and cooling phenomena combined with additive layer deposition processing.
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The samples’ matrix for residual stress measurement was given in chapters 4 and 5, and is repeated here, in Table 6·1, with the addition of the relevant column for the experimental undertakings in this Chapter. As previously explained, the samples are labelled and referred to with respect to their processing parameters and “repeat samples” are also labelled with “R”. The three main process parameters were defined to be deposition strategy, energy density and dwell-time between layer depositions, as covered in details in Chapter 3 (section 3.3).

In presenting data from synchrotron X-ray diffraction, the neutron data for the main vertical and horizontal scan-lines, coincident with the two fine scan-lines for the X-ray, are also included for all samples.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Sample ID</th>
<th>Deposition strategy</th>
<th>Energy density (MJ/m²)</th>
<th>Dwell time (s)</th>
<th>Neutron diff.</th>
<th>Sync X-ray</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>L-060-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>60</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>2</td>
<td>L-180-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>180</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>L-060-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>60</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>4</td>
<td>L-180-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>180</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Z-060-L</td>
<td>Zig-zag (Z)</td>
<td>150 (Low)</td>
<td>60</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>6</td>
<td>Z-180-L</td>
<td>Zig-zag (Z)</td>
<td>150 (Low)</td>
<td>180</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Z-060-H</td>
<td>Zig-zag (Z)</td>
<td>300 (High)</td>
<td>60</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>8</td>
<td>Z-180-H</td>
<td>Zig-zag (Z)</td>
<td>300 (High)</td>
<td>180</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2 (repeat)</td>
<td>R-L-180-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>180</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4 (repeat)</td>
<td>R-L-180-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>6 (repeat)</td>
<td>R-Z-180-L</td>
<td>Zig-zag (Z)</td>
<td>150 (Low)</td>
<td>180</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8 (repeat)</td>
<td>R-Z-180-H</td>
<td>Zig-zag (Z)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
<td>✓</td>
</tr>
</tbody>
</table>
6.2. Synchrotron X-ray diffraction scanning procedure

Typically, in synchrotron X-ray diffraction, only two components of the strain are determined. This is mainly due to the low scattering angle, which results in the formation of an elongated gauge volume. The low scattering angle does not allow formation of the gauge volume along the length of the samples due to penetration depth. The strain components are calculated by using Bragg’s law (Equation 4·1) and strain – d-spacing relationship (Equation 4·2), as explained in Chapters 3 and 4.

By applying the plane stress condition, the in-plane stress components are calculated using the reduced Hooke’s law, as in Equation 6·1. The errors have been calculated by using the same formulae as explained in chapter 4.

\[
\sigma_{xx} = 0
\]
\[
\sigma_{yy} = \frac{E}{1 - v^2} (\varepsilon_{yy} + v\varepsilon_{zz})
\]
\[
\sigma_{zz} = \frac{E}{1 - v^2} (\varepsilon_{zz} + v\varepsilon_{yy})
\]

Equation 6·1 – Hooke’s law (plane stress)

Where \(\sigma_{xx}\) is the out-of-plane stress and \(\sigma_{yy}\) and \(\sigma_{zz}\) are the longitudinal and transverse stress components, respectively.

Measurements were made at beamline ID22 of the ESRF (experiment No. MA 3411). The X-ray beam energy provided at ID22 was 60.001 keV with wavelength of \(\lambda=0.20678\) Å.

The objective of this experiment was to gain a complementary knowledge of the distribution of the residual stresses within the Ti-6Al-4V PTA AM parts. This will be achieved by comparing and contrasting obtained X-ray diffraction data with neutron diffraction and contour method results. Therefore, the choice of samples (process parameters) and scan-lines correspond to those used in the neutron diffraction scanning experiments. Due to the fairly quick scan time by synchrotron X-ray diffraction (24 seconds per point), a larger number of scan points were chosen along all scan-lines and additional scan-lines (next to the original ones) were instigated to provide more details about the level of residual stress.

However, due to the elongated gauge volume as described earlier, the strains were determined in two principal directions along all scanlines, assuming a “plane stress” condition.
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Considering the dual-phase crystal structure of Ti-6Al-4V, with hexagonal (α) and cubic (β) crystal structures (as explained in chapter 2, section 2.2.1), and the wavelength of the high energy synchrotron X-ray (\(\lambda = 0.20678 \, \text{Å}\)), the positions of the α peak and their corresponding 2θ can be calculated, which allows the scanning of the 2θ to be determined, accordingly. It was done by using Dragon software, version 5.01 at ESRF ID22, on 24 July 2017, for 2θ between 0° and 10°, which determined the 2θ for the α (101) peak, to be 5.2846°.

The α (101) peak showed the strongest diffraction peak. This can be checked against the position of the α (101) peak in the X-ray diffraction pattern from Cu-Kα (2θ\text{Cu-Kα}= 41.21987°), the position of the same peak, α (101), in synchrotron X-ray was determined to be 2θ\text{sync. X-ray}=5.3128°. Therefore, scanning for each point was performed from 2θ=5.24° to 5.4° with a scan velocity of 0.5°/min.

Considering the geometry of the PTA AM samples and the plane stress assumption, the scanning lines were defined along two in-plane directions: along the height and length of the samples, as shown in Figure 6·1. Three scan lines along the height of the samples (vertical) and three horizontal scan lines along the length of the deposited walls were considered, making a total of six scan lines in two in-plane directions. The dimensions and the distance between the scan lines are shown in Figure 6·1 and the number of scan points on each scan line is summarised in Table 6·2.

![Figure 6·1 – Schematic of a sample and scan lines for the synchrotron X-ray diffraction](image)
One “fine scanline” was defined along both the height and the length of the samples (scanlines 1 and 2, respectively). The step distance between each scan point along these fine scanlines was 0.5 mm. The other two scanlines for each scan direction were considered as “coarse scanlines” (scanlines 3 and 4 along the height and scanlines 5 and 6 along the length of the samples). The step distance between the scan points was 5 mm along each of the coarse scanlines.

For each sample, a total of the 222 points were scanned in two directions (444 scans). The scan-time at each point was 24 s, which shows the considerable difference between the neutron (600 seconds per point) and synchrotron X-ray diffraction techniques. The scan-lines are summarised in Table 6-2.

<table>
<thead>
<tr>
<th>Along the height (vertical scan-lines)</th>
<th>Along the length (horizontal scan-lines)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Red (fine)</td>
<td>Scanline 1</td>
</tr>
<tr>
<td>Blue</td>
<td>Scanline 3</td>
</tr>
<tr>
<td>Yellow</td>
<td>Scanline 4</td>
</tr>
</tbody>
</table>

The incident beam was set to give a width of ~1.4 mm and a height of ~1 mm and a matchstick through the thickness of the deposited walls, although the actual gauge volume extended the width (~12 mm across the elongated length of the gauge volume), as is shown schematically in Figure 6-2. In this set-up, the Q-vector is aligned to the direction of the height of the sample (Z-direction), which means the Z-component of the strain is measured.

The data from neutron diffraction provided a set of results across 9 scan points along the length, while the synchrotron data provided results for 101 scan points. Also, the scan-line for the neutron diffraction lined up to the middle of the sample, while the horizontal scan-line for the synchrotron X-ray diffraction continued up to 50 mm inside the sample.
By rotating the samples in two directions, each point was scanned to give strain along two in-plane directions (Q-vector in Y and Z directions), assuming a plane stress condition, the out-of-plane stress is zero ($\sigma_{xx}=0$).

By using these scan configurations and considering the three main process parameters for PTA AM, a total of six samples were scanned, as specified in Table 5-1. The main focus for this experiment was investigating the effects of deposition strategy, and the dwell time and the energy density deposition parameter levels (constants in this experiment) were chosen for the most severe stress distribution scenario, according to the results from neutron diffraction (shorter dwell-time of 60 s and higher energy density level of ~300 J/m$^2$).
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(a) Scanning for longitudinal $2\theta_y$

(b) Scanning for transverse $2\theta_z$

Figure 6.3 – Sample set-up on the Synchrotron X-ray beamline at ID22, ESRF: scanning along two in-plane directions, measuring $2\theta$ along (a) the length ($Y$) and (b) the height ($Z$) of samples
All six samples: 1, 3, R4, 5, 7 and R8 were scanned by synchrotron X-ray diffraction. The results for samples R4 and R8 provided an understanding of the repeatability of the manufacturing process. Also, the scans, along the main vertical and horizontal scan-lines, were repeated for sample 7, to ratify the results, by using a finer scan points (step distance of 0.5 mm) along all six scanlines. (2θ₀ of 5.3128°)

6.3. Results

The residual strain and stress results for six samples, scanned via synchrotron X-ray diffraction, are presented in this section. A total of six samples were scanned on ID22: four samples with lower dwell time of 60 s (with high and low energy density levels and linear and zig-zag deposition strategies) and two repeat samples with the dwell-time of 180 s, built by the higher energy density and linear or zig-zag deposition strategy.

For each sample, the two principal strains (ε_Y and ε_Z) are given for one ‘fine’ scan-line (vertical or horizontal) plus the same results for the other two ‘coarse’ scan-lines along vertical or horizontal directions. The resultant stresses are calculated by Hooke’s law for plane stress condition (Equation 4.2) for both in-plane principal stresses (σ_Y and σ_Z).

To give more in-depth perception of data, for both samples L-060-L (1) and Z-060-L (5), the scanned 2θ values are also given along the main vertical scan-lines (fine scan-lines 1 and 2), in both in-plane directions (2θ_Y and 2θ_Z).

The results from all six samples provided a complementary set of data to the results from neutron diffraction and will be later validated and compared with the results from the contour method. The analysis will lead to a comprehensive understanding of the effects of the process parameters on the evolution of residual stress in PTA AM.

For all samples, the strain results from neutron data are also included, for the main scan-lines along the height of the sample (scan-line 1) and along the length of the sample (scan-line 2). It must be noted that there is no intention to make a direct comparison of the data from different residual stress measurement techniques. However, such comparison is given as an indication of the conformity of the diffraction-based techniques to determine residual strain/stress.
6.3.1. Sample L-060-L (1)

The measured 2θ values for scan lines 1 are given in Figure 6-4. Residual strains and corresponding residual stresses along the height (vertical scan lines 1, 3 and 4) are given in Figure 6-5, where the data from neutron diffraction along the same scan line are also included. As it can be seen, only 9 points were scanned via neutron diffraction, while 101 points were scanned via synchrotron X-ray diffraction.

It must be pointed out that the synchrotron X-ray scanning through the fine scan line 1, along the height of sample L-060-L (1), was repeated to ensure repeatability of data capturing.

![Graph showing 2θ along the height for sample L-060-L (1) in two in-plane directions: longitudinal (Y) and transverse (Z) (average error: ±0.0005°)](image)

*Figure 6-4 – 2θ along the height (scan line 1) for sample L-060-L (1) in two in-plane directions: longitudinal (Y) and transverse (Z) (average error: ±0.0005°)*
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Figure 6.5 – Results along the height of sample L·060·L (1): scan line 1 (101 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain (and data from neutron diffraction scan line 1) and b) residual stress
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a) Residual strain along the height of sample L-060-L (1) (average error: ±500×10^{-6} \varepsilon)

b) Residual stress along the height of sample L-060-L (1) (average error: ±50 MPa)

Figure 6.6 – Results along the height of sample L-060-L (1): scan lines 3 and 4 (11 scan points each) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
Along the height of the sample, a partial correlation can be seen between the data from synchrotron X-ray and neutron diffraction, along scan-line 1. Similar to results from neutron diffraction, the longitudinal components of stress ($\sigma_y$) and strain ($\varepsilon_y$) have slightly more variation and dominant effect than the transverse components of stress ($\sigma_z$) and strain ($\varepsilon_z$).

Also, a reasonable correlation could be observed from data along all vertical scan-lines, the fine scan-line 1 and the coarse scan-lines 3 and 4. However, there was a mismatch between the data from the fine scan-line 1 and the coarse scan-lines 3 and 4, at ~20 mm below the top surface. The jump in the strain components only occurred along the fine scan-line, as already reported by data from neutron diffraction, but not along the parallel coarse scan-lines. This could be considered a local effect on the scan points along scan-line 1, as also observed from neutron diffraction data, and could be related to the local observable defect on that area in sample L-060-L (1), as covered in the manufacturing of samples section, in chapter 3.

Along scan-line 1, both longitudinal and transverse strains started with a compressive status at the top surface and rising up very slightly towards the middle of the sample. There was a jump in the longitudinal strain ($\varepsilon_y$) at ~20 mm below the reference point, however the strain declined again towards the middle of the sample. At 40 mm below the reference point, towards the bottom of the deposited wall there was another jump in the longitudinal strain.

The strain and stress trend along the other two vertical scan-lines (3 and 4) are plotted for 11 scan points. The longitudinal strain components ($\varepsilon_y$) jumped to a tensile state just below the top surface (5 mm below the top surface) and then declined to a compressive state up to 40 mm above the substrate, where it jumped to another tensile state. The transverse component of the strain ($\varepsilon_z$) showed a generally compressive state up to the middle of the sample towards the bottom the wall, where it jumped to a tensile state and then drops to a compressive state at the intersection of the wall and the substrate.

The same set of results were given along the length of the sample for horizontal scan-lines 2, 5 and 6. The measured 2$\theta$ along the fine scan-line 2 are plotted in Figure 6·7 and residual strains and corresponding stresses are given in Figure 6·8, for the fine scan-line 2, and in Figure 6·9 for the coarse scan-lines 5 and 6.
Strain data from neutron diffraction for the main scan line along the length (scan line 2, at the middle of the sample) is given in Figure 6.8.

*Figure 6.7 – $2\theta$ along the length (scan line 2) of sample L-060-L (1) in two in-plane directions: longitudinal (Y) and transverse (Z)*
a) Residual strain along the length of sample L-060-L (1) (neutron data for scan-line 2 are included) (average error: ±500×10^-6 ε)

b) Residual stress along the height of sample L-060-L (1) (average error: ±50 MPa)

Figure 6.8 – Results along the length of sample L-060-L (1): scan-line 1 (101 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain (and data from neutron diffraction scan-line 2) and b) residual stress
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**Figure 6.9** – Results along the length of sample L-060-L (1): scan lines 5 and 6 (11 scan points each), in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) level/variation of residual stress.

a) Residual strain along the length of sample L-060-L (1) (average error: ±500×10⁶ ε)

b) Residual stress along the length of sample L-060-L (1) (average error: ±50 MPa)
Along the length of sample L-060-L (1), the strain and stress variation seemed to be smaller than the scan-lines along the height of the sample.

Results along the fine scan-line 2, with 101 scan points, showed a good agreement with the results from neutron diffraction up to half way through the length of the sample. The longitudinal strain ($\varepsilon_l$) showed a tensile state towards just below the top layer while the transverse strain showed a compressive state below the top surface. A jump in longitudinal strain could be seen towards the position of the vertical scan-line 1, 20 mm into the sample. The jump to tensile strain was more intense for the longitudinal component ($\varepsilon_l$) than the transverse component ($\varepsilon_t$). However, this jump in the strain was observed along the other two parallel scan-lines along the length (scan-lines 5 and 6) which confirmed the severe effect of the local defect in the sample at that point at the intersection of the vertical scan-lines 1 and the horizontal scan-line 2.

Overall, the general trend for the longitudinal strain (and stress) seemed to have a higher variation than the transverse components of strain and stress, which was in agreement with the results from neutron data. However, as previously mentioned in the analysis of the data from synchrotron X-ray diffraction, the direct comparison between the results from neutron diffraction and synchrotron X-ray diffraction cannot be made, as they are not really like-to-like data.

Overall, the stress and strain along the coarse scan-lines 5 and 6 showed a more moderate trend compared to the fine scan-line 2.

**6.3.2. Sample L-060-H (3)**

The two in-plane components (longitudinal and transverse) of residual strain and associated residual stresses along the height of the sample L-060-H (3) are given along the height of the sample, for the fine scan-line 1 and the coarse scan-lines 3 and 4, in Figure 6-10 and Figure 6-11, respectively.
a) Residual strain along the height of sample L-060-H (3) (neutron data for scan-line 1 are included) (average error: $\pm 500 \times 10^{-6}$)

b) Residual stress along the height of sample L-060-H (3) (average error: $\pm 50$ MPa)

Figure 6:10 – Results along the height of sample L-060-H (3): scan-line 1 (101 scan points) in two in-plane directions: longitudinal ($Y$) and transverse ($Z$) a) residual strain (and data from neutron diffraction scan-line 1) and b) residual stress
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Figure 6-11 – Results along the height of sample L-060-H (3) (average error: \( \pm 500 \times 10^{-6} \) ε)

- **a) Residual strain along the height of sample L-060-H (3) (average error: \( \pm 500 \times 10^{-6} \) ε)**

- **b) Residual stress along the height of sample L-060-H (3) (average error: \( \pm 50 \) MPa)**
Along the height of the sample, a good correlation could be observed between the data from synchrotron X-ray and neutron diffraction, from 10 mm below the reference point down to half-way through the height of the sample. Based on the data from synchrotron X-ray diffraction, the longitudinal component of the strain ($\varepsilon_y$) showed slightly higher variation than the transverse component of the strain ($\varepsilon_z$), as also observed from strain data obtained by neutron diffraction.

Along the fine scan-line 1, both components of the in-plane stresses started with a slight tensile state at the top of the sample, although it dropped for the first few layers below the top surface. At 10 mm below the reference point, the longitudinal stress ($\sigma_y$) dropped to a negative value (compressive state). It jumped up again at 15 mm below the top layer, and showed a fluctuation tensile stress up to 10 mm above the substrate (40 mm below the top surface), where it dropped again to a compressive stress and remained at the same state for almost 5 mm (45 mm below the reference point). The longitudinal stress ($\sigma_y$) made a jump at this point to a tensile state shooting up to almost 400 MPa at the intersection of the wall and the deposited wall. However, the transverse strain ($\varepsilon_z$) and stress ($\sigma_z$) remained compressive towards the end of the wall. Although a similar trend was observed between the data from neutron diffraction and synchrotron X-ray diffraction, the two set of data showed discrepancy towards the end of the wall, from 35 mm below the reference point down to the intersection of the wall and the substrate. The longitudinal strain ($\varepsilon_y$) from neutron diffraction showed a tensile state, however synchrotron X-ray data went to a very slight negative value (compressive) and jumped up to a much higher level of the tensile strain towards the end of the wall.

Considering the results along the other two vertical scan-lines (5 and 6), a smoother variation of the longitudinal and transverse components of strains and stresses was observed, although a similar trend could be deliberated for both longitudinal and transverse components (of strain and stress).
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Figure 6.12 – Results along the length of sample L·060·H (3): scan·line 1 (101 scan points) in two in·plane directions: longitudinal (Y) and transverse (Z) a) residual strain (and data from neutron diffraction scan·line 2) and b) residual stress

a) Residual strain along the length of sample L·060·H (3) (neutron data for scan·line 2 are included) (average error: ±500×10⁻⁶ ε)

b) Residual stress along the length of sample L·060·H (3) (average error: ±50 MPa)
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a) Residual strain along the length of sample L·060·H (3) (average error: ±500×10⁻⁶ ε)

b) Residual stress along the length of sample L·060·H (3) (average error: ±50 MPa)

Figure 6.13 – Results along the length of sample L·060·H (3): scan·lines 5 and 6 (11 scan points each), in two in·plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
The strain (and stress) variation along the length of the sample seemed to be almost half of the height of the sample. Considering the strain data from neutron diffraction and synchrotron X-ray diffraction, a similar trend could be observed, although the magnitudes (and state) of strains were slightly different, mainly inwards to the wall. The neutron data was available for half of the length (40 mm from the lateral edge of the wall), while 50 mm of the horizontal scan-line 2 was scanned via synchrotron X-ray diffraction.

Along the length of the sample, fine scan-line 2, both components of strain ($\varepsilon_y$ and $\varepsilon_z$) showed a tensile status at the lateral edge of the wall. As moving inwards to the wall, both components dropped to a compressive strain and rose to a tensile status at 10 mm inside the wall. Both strain components showed a fairly stable variation along the horizontal scan-line. The variation of the strain along the length seemed to be lower than what was predicted by neutron diffraction.

The results along the coarse scan-lines 5 and 6 showed a similar trend, starting from a tensile status at the lateral edge and falling to a very slight negative (compressive) state, while showing a steady trend along the two coarse scan-lines. The transverse component of strain along scan-line 6 seemed to have a jump at 20 mm inside the wall, which was an obvious outlier.

**6.3.3. Sample R-L-180-H (R4)**

Results along the height of the repeat sample R-L-180-H (R4) are summarised in Figure 6-14 for the fine scan-line 1 and in Figure 6-15, for the coarse scan-lines 5 and 6. The strain data from neutron diffraction are included along the height, to give a comparison of the data from neutron and synchrotron X-ray diffraction.

Strain and stress data along the length of the repeat sample are also given in Figure 6-16 and Figure 6-17, for the fine scan-line (2) and the coarse scan-lines (5 and 6), respectively.
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a) Residual strain along the height of sample R·L·180·H (R4) (neutron data for scan-line 1 are included) (average error: \(\pm 500 \times 10^{-6}\) c)

b) Residual stress along the height of sample R·L·180·H (R4) (average error: \(\pm 50\) MPa)

Figure 6-14 – Results along the height of sample R·L·180·H (sample R4): scan-line 1 (101 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain (and data from neutron diffraction scan-line 1) and b) residual stress
a) Residual strain along the height of sample R·L·180·H (R4) (ave. error: ±500×10⁻⁶ ε)

b) Residual stress along the height of sample R·L·180·H (R4) (ave. error: ±50 MPa)

Figure 6.15 – Results along the height of sample R·L·180·H (sample R4): scan lines 3 and 4 (11 scan points each) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
Results along the height of sample R-L-180-H (R4), a reasonable similarity could be observed between strain data from neutron diffraction and synchrotron X-ray.

Along the fine vertical scan-line 1, both in-plane components of strain started with a very slight tensile state at the top surface, then immediately dropped to a negative (compressive) state. Then the status of strain (and stress) rose to a positive (tensile) state at ~15 mm below the top surface. The strain state seemed to be fluctuating towards the middle of the height, where both components showed mostly a compressive state up to 10 mm above the substrate (40 mm below the reference point). At the intersection of the wall and the substrate, the neutron data suggested a positive tensile strain for the longitudinal component of strain ($\varepsilon_y$) while the synchrotron data suggested a slight compressive status for the same component of strain. Both longitudinal (\(\sigma_y\)) and transverse (\(\sigma_z\)) stress components seemed to drop from a very high tensile state to a slight compressive state, which suggested the tensile behaviour was more credible.

Results along the other two vertical scan-lines (3 and 4) also showed a good correlation with the data from the fine scan-line 1. However, more fluctuations could be seen in the middle region of the height, where the longitudinal stress ($\sigma_y$) reached a maximum of 517 MPa at 30 mm below the top layer. Also, results along both scan-lines suggested a tendency towards tensile stress at the intersection of the wall and the substrate. The destructive contour method (chapter 7) provided more detailed stress profile across the whole height of the sample, including within the substrate, which could be illuminating in interpretation strain and stress behaviour within the sample.

Furthermore, based on the data from all there vertical scan-lines, the variation of the longitudinal components of strain (and stress) seemed to have a more dominant effect than the transverse components.
a) Residual strain along the length of sample R·L·180·H (R4) (neutron data for scan-line 2 are included) (average error: ±500×10^{-6} \varepsilon)

b) Residual stress along the length of sample R·L·180·H (R4) (average error: ±50 MPa)

Figure 6·16 – Results along the length of sample R·L·180·H (sample R4): scan-line 1 (101) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain (and data from neutron diffraction scan-line 2) and b) residual stress
a) Residual strain along the length of sample R·L·180·H (R4) (ave. error: ±500×10^-6 με)

b) Residual stress along the length of sample R·L·180·H (R4) (ave. error: ±50 MPa)

Figure 6.17 – Results along the length of sample R·L·180·H (sample R4): scan-lines 5 and 6 (11 scan points each), in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
Similar to other samples, results along the length of sample R-L-180-H (R4) indicated that the strain and stress variations along the length was smaller than along the height of the sample.

Along the fine scan-line 2, the strain results from synchrotron X-ray seemed to be comparable to the strain results from neutron diffraction, for both longitudinal ($\varepsilon_y$) and transverse ($\varepsilon_z$) components. Strain started with a tensile state at the lateral edge and dropped to a compressive state as moving inwards along the fine scan-line. Strain and stress fluctuated along scan-line 2, while showing a tensile behaviour half-way through the length of sample. The longitudinal strain and stress ($\varepsilon_y$ and $\sigma_y$) seemed to have less dominant effect than the transverse components ($\varepsilon_z$ and $\sigma_z$) along scan-line 2, as the transverse strain showed more fluctuating behaviour.

Results from the coarse scan-lines along the length (scan-lines 5 and 6) were relatively in agreement with the results from the fine scan-line 2, although more fluctuation could be seen along the coarse scan-lines. Both components of strain (and stress) started from higher magnitudes at the lateral edge and remained mainly tensile up to 10 mm inside the wall. A slight compressive behaviour could be seen at that point and both components rose again throughout the sample, showing mainly a positive (tensile) state. The results from the coarse scan-lines seemed to be in a better correlation with the results from neutron diffraction, along the length, where a mostly tensile strain (and stress) was predicted.

### 6.3.4. Sample Z-060-L (5)

For sample Z-060-L (5), the scanned 20 values along the height of the sample were plotted for two in-plane components (Y and Z) in Figure 6-18. The scanned 20 values along the scan-line 1, showed more variation towards the bottom of the wall (near the substrate). This variation seemed to be higher for the longitudinal component ($2\theta_y$). The scanned 20 values were used to calculate strain, based on the Bragg’s law.

Residual strains and associated residual stresses plotted along the height of the sample are given for the vertical fine scan-line 1 in Figure 6-19, where the strain data from neutron diffraction are also included on the same scan-line (for nine scan points by neutron diffraction). Also, along the height of sample Z-060-L (5), two coarse scan-lines were scanned (3 and 5) and the strain and stress results are given in Figure 6-20.
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It must be pointed out that the scan through the fine scan line 1, along the height, in sample Z-060-L (5), was repeated to ensure repeatability of data capturing.

Figure 6.18 – 2θ along the height (fine scan line 1) of sample Z-060-L (5) in two in-plane directions: longitudinal (Y) and transverse (Z)

Figure 6.19 – Residual strain (ε) from neutron data in the longitudinal and transverse directions for sample Z-060-L.
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a) Residual strain along the height of sample L Z-060-L (5) (neutron data for scan-line 1 are included) (average error: ±500×10^{-6} ε)

![Graph showing longitudinal and transverse stress](image)

b) Residual stress along the height of sample Z-060-L (5) (average error: ±50 MPa)

Figure 6-19 – Results along the height of sample Z-060-L (5): scan-line 1 (101 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain (and data from neutron diffraction scan-line 1) and b) residual stress

![Graph showing longitudinal and transverse strain](image)
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a) Residual strain along the height of sample Z-060-L (5) (average error: ±500×10⁻⁶ ε)

b) Residual stress along the height of sample Z-060-L (5) (average error: ±50 MPa)

Figure 6-20 – Results along the height of sample Z-060-L (5): scan lines 3 and 4 (11 scan points each) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress

Along the height of sample Z-060-L (5), a good correlation could be observed between the strain data from neutron diffraction and synchrotron X-ray, from data obtained on the scan line 1. Similar to the data from neutron diffraction, the strain results along the fine scan line 1 suggested a higher variation of the longitudinal component (ε_y) than the transverse component (ε_z).

Along the fine scan line 1, the strain data showed a relatively large compressive magnitude at the top of the wall until almost halfway through the height, for both strain components. At the height of 20 mm below the reference point, a slight tensile state could be seen for the longitudinal strain (ε_y), although the corresponding stress magnitude remained compressive, due to the effect of transverse strain (ε_z) at that point. The transverse strain had a jump at ~30 mm below the top layer, reaching a maximum of ~2000 (ε×10⁻⁶) and then falling down again. The longitudinal strain (ε_y) started rising up in the second half of the wall towards the intersection of the wall and the substrate, at the height of ~35 mm below the top layer. However, the transverse strain (ε_z) declined to
a compressive state falling down to the minimum of $2200 \times 10^{-6}$ at $\sim 40$ mm below the reference point and $1600 \times 10^{-6}$ at the intersection of the wall and the substrate. The corresponding stresses followed the same trend, mostly showing a compressive-tensile behaviour along scan-line 1, from top of the sample towards the bottom of the wall.

A similar trend could be observed along scan-lines 3 and 4, shifted by 10 mm on either side of the fine scan-line 1. Again, a mainly compressive-tensile strain behaviour was observed from top to the bottom of the wall, mainly for the longitudinal components of strain ($\varepsilon_y$) and stress ($\sigma_y$). Although the transverse components were following a similar trend to scan-line 1, their magnitudes descending with a slightly lower rate than the magnitudes for scan-line 1 and landed at a slightly lower residual strain (and stress) values at the bottom of the wall.

A similar set of results are provided along the length of sample Z-060-L (5). The scanned 2θ for both in-plane components are given in Figure 6-21, for the fine scan-line 2.

Residual strains and resultant residual stress results along the length of the sample are given in Figure 6-22 for the fine scan-line 2 and in Figure 6-23, for the coarse scan-lines 5 and 6, shifted by 10 mm above and below of the middle scan-line 2.

From the 2θ graphs in Figure 6-21, a slightly higher variation/fluctuation of the 2θ could be observed nearer to the edge of the deposited wall, up to $\sim 7$ or 8 mm inside the wall. The variation of the transverse component of 2θ ($Z$) seemed to be slightly higher than the longitudinal component ($Y$).
Figure 6.21 – $2\theta$ along the length (scan line 2; 101 scan points) of sample Z-060-L (5) in two in-plane directions: longitudinal (Y) and transverse (Z)
a) Residual strain along the length of sample Z-060-L (5) (neutron data for scan-line 2 are included) (average error: ±500×10⁻⁶ ε)

b) Residual stress along the length of sample Z-060-L (5) (average error: ±50 MPa)

Figure 6.22 – Results along the length of sample Z-060-L (5): scan-line 2 (101 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain (and data from neutron diffraction scan-line 2) and b) residual stress
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a) Residual strain along the length of sample Z·060·L (5) (average error: ±500×10⁻⁶ ε)

![Residual strain graph]

b) Residual stress along the length of sample Z·060·L (5) (average error: ±50 MPa)

![Residual stress graph]

Figure 6.23 – Results along the length of sample Z·060·L (5): scan lines 5 and 6 (9 scan points each), in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
In sample Z-060-L (5), similar to the linear samples, the variation of strain and stress along the length seemed to be much less than the height of the sample. Strain variation and resultant stresses showed a relatively stable trend, especially towards the inside of the wall. Also, unlike the linear samples, the longitudinal strain ($\varepsilon_y$) seemed to be the dominant component along the length of the sample compared to the transverse strain ($\varepsilon_z$).

Results along fine scan-line 2 started from a relatively large tensile strain (and stress) at the start-side of the lateral edge and dropped to a compressive state 10 mm inwards the scan-line. The strain (and stress) components rose again to a tensile state for almost 5 mm of the scan-line and dropped down to a slight compressive state for almost the rest of the horizontal scan-line.

The strain and stress trends along the coarse scan-lines 5 and 6 had the same trend along the length of the sample, showing a tensile state at the lateral edge and mainly slight compressive state for the rest of the two scan-lines. Only one exception was observed on scan-line 5, where a jump can be seen at 20 mm inside the wall. This jump in the strain (and associated stress) could be interpreted as an outlier, as not observed along the other horizontal scan-lines, neither from neutron diffraction nor from Synchrotron X-ray.

6.3.5. Sample Z-060-H (7)

Two in-plane components of strains and resultant residual stresses are plotted along the height of the sample, for the fine scan-line 1 in Figure 6-24 and for the coarse scan-lines 3 and 4, in Figure 6-25.

The strain data from neutron diffraction for the same sample is also included along scan-line 1, with less number of scan points (nine points) from neutron diffraction scanning of the same sample.
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Figure 6.24 – Results along the height of sample Z-060-H (7): scan line 1 (101 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain (and data from neutron diffraction scan line 1) and b) of residual stress
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**a) Residual strain along the height of sample Z-060-H (7) (average error: ±500×10^{-6} \; \varepsilon)***

![Graph showing residual strain](image)

**b) Residual stress along the height of sample Z-060-H (7) (average error: ±50 MPa)**

![Graph showing residual stress](image)

*Figure 6-25 – Results along the height of sample Z-060-H (7): scan lines 3 and 4 (11 scan points each) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress*
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Along the height of the sample, scan-line 1, the strain data from synchrotron X-ray and neutron diffraction showed an agreement, for both strain components. Also, considering the variation of strain (and stress) for both in-plane components, the longitudinal strain seemed to have a higher variation along the height of the sample and a more dominant effect.

Along the fine scan-line 1, both longitudinal and transverse strains started with a negative magnitude at the top surface and remained compressive for the first 10 mm below the reference point. Then a slight tensile strain could be seen for both components, although the fluctuation was slightly higher for the transverse strain ($\varepsilon_z$). The transverse strain and stress showed a steady trend for the rest of scan-line, almost towards the bottom of the height, where it declined to a negative (compressive) state at the intersection of the wall and the substrate. The longitudinal strain ($\varepsilon_y$), however, rose towards the middle of the height and reached a tensile value of $\sim$1900 ($\varepsilon\times10^6$) at 10 mm above the substrate and eventually reached to the maximum of $\sim$3000 ($\varepsilon\times10^6$) at the intersection of the wall and the substrate. The longitudinal components of strain and stress ($\varepsilon_y$ and $\sigma_y$) showed a mainly compressive-tensile behaviour from top to the bottom of the height, along the scan-line 1.

The strain and stress results along the other two vertical scan-lines (coarse scan-lines 3 and 4) showed an overall agreement with the data from the fine scan-lines, although more fluctuations could be seen mainly for the transverse strain and stress components. Also, a clearer/higher tensile behaviour could be observed along both scan-lines 3 and 4, from top to the bottom of the height of the sample.

The same set of results are given along the length of sample Z-060-L (7) in Figure 6-26 for the fine scan-line 2 and in Figure for the coarse scan-lines 5 and 6. Strain data from neutron diffraction are also included in Figure 6-26, for nine scan points from neutron diffraction scanning.
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Figure 6.26 – Results along the length of sample Z-060-H (7): scan-line 1 (101 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain (and data from neutron diffraction scan-line 2) and b) residual stress.
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Figure 6.27 – Results along the length of sample Z-060-H (7): scan lines 5 and 6 (9 scan points each), in two in-plane principal directions: longitudinal (Y) and transverse (Z) a) residual strain and b) level/variation of residual stress

a) Residual strain along the length of sample Z-060-H (7) (average error: ±500×10⁻⁶ ε)

b) Residual stress along the length of sample Z-060-H (7) (average error: ±50 MPa)
Similar to other samples, the variation of both in-plane components of strain (and stress) (longitudinal: Y and transverse: Z) seemed to be much less along the length than the height of sample Z-060-L (7). Strain data along the horizontal scan-line 2 showed a good correlation with the data from neutron diffraction for the same scan-line.

Along the fine scan-line 2, a steady trend for both strain components could be observed. Both longitudinal and transverse strains (ε_Y and ε_Z) showed a tensile behaviour at the lateral edge and decline as moving inwards the sample. The fluctuation of both components of strain and stress was less than what observed along the height.

The results along the coarse scan-lines 5 and 6 showed a similar trend to the fine scan-line 2, where a tensile status was observed at the lateral edge and a steady variation for both in-plane components can be seen along the scan-lines.

Similar to other zig-zag samples, along the length of this zig-zag sample, it seemed that the variation of the longitudinal component of strain (and stress) was less than the transverse components of strain (stress).

6.3.6. Sample R-Z-180-H (R8)

Results for the repeat sample R-Z-180-H (sample R8) are summarised along the height of the sample in Figure 6-28 for the fine (vertical) scan-line 1 and in Figure 6-29 for the coarse (vertical) scan-lines 3 and 4.

The strain data from neutron diffraction along scan-line 1 are also included in Figure 6-28, for the less number of scan points (nine scan points) from neutron diffraction scanning of the same sample.
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a) Residual strain along the height of sample R·Z-180·H (R8) (neutron data for scan-line 1 are included) (average error: ±500×10^{-6} ε)

b) Residual stress along the height of sample R·Z-180·H (R8) (average error: ±50 MPa)

Figure 6.28 – Results along the height of sample R·Z-180·H (R8): scan-line 1 (101 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain (and data from neutron diffraction scan-line 1) and b) residual stress
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**a) Residual strain along the height of sample R-Z-180-H (R8) (ave. error: ±500×10⁻⁶ ε)**

**b) Residual stress along the height of sample R-Z-180-H (R8) (ave error: ±50 MPa)**

Figure 6.29 – Results along the height of sample R-Z-180-H (R8): scan lines 3 and 4 (11 scan points each) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
In the repeat sample R-Z-180-H (R8), the correlation between strain data from neutron diffraction and synchrotron X-ray seemed to be less clear, especially towards the middle of the height, where the strain data from neutron predicted a mainly compressive strain for the longitudinal component, while the synchrotron X-ray scanning predicted a steady tensile strain for the same component. However, the overall trend for both in-plane components of strain seemed to correlate between the data from two techniques.

Along the fine scan-line 1, neglecting the data from the top layer (surface effects), both strain components seemed to be in a slight compressive state towards the top of the wall and showed a steady state of a tensile behaviour towards the bottom of the wall, where a decline occurred just above the substrate, at ~40 mm below the reference point. Although the transverse strain ($\epsilon_z$) showed a fluctuating behaviour at the middle region of the height, the overall variation of the longitudinal strain ($\epsilon_y$) seemed to be higher than the transverse strain ($\epsilon_z$) along the fine scan-line 1. The state of both components of stress along the vertical scan-line was a reflection of such higher variation for the longitudinal component. However, the overall difference between the longitudinal and transverse components of strain (and stress) seemed to be less than other samples.

The results along the coarse scan-lines 3 and 4, along the height of the sample, showed a similar trend. A steady state of longitudinal strain (and stress) could be observed at the middle of the sample, although the stress state for both scan-line showed a tensile behaviour at the bottom of the deposited wall.

The same set of results are given along the length of the repeat sample in Figure 6-30 for the fine scan-line 2 and in Figure 6-31 for the coarse scan-lines 5 and 6. Strain data from neutron diffraction are also included in Figure 6-30 for nine scan points from neutron diffraction scanning.
a) Residual strain along the length of sample R-Z-180-H (R8) (neutron data for scan-line 2 are included) (average error: \(\pm 500 \times 10^{-6}\) ε)

b) Residual stress along length of sample R-Z-180-H (R8) (average error: \(\pm 50\) MPa)

Figure 6.30 – Results along the length of sample R-Z-180-H (R8): scan-line 1 (101 scan points) in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain (and data from neutron diffraction scan-line 2) and b) residual stress
a) Residual strain along the length of sample R-Z-180-H (R8) (ave. error: \( \pm 500 \times 10^{-6} \) \( \varepsilon \))

b) Residual stress along length of sample R-Z-180-H (R8) (ave. error: \( \pm 50 \) MPa)

Figure 6-31 – Results along the length of sample R-Z-180-H (R8): scan lines 5 and 6 (9 scan points each), in two in-plane directions: longitudinal (Y) and transverse (Z) a) residual strain and b) residual stress
The trends for strain data along the horizontal scan line 2 seemed to be comparable to the strain data from neutron diffraction, for both in-plane components (Y and Z), although similar to the data along the height of the sample, the actual values were in different status (tensile for the synchrotron X-ray data versus compressive for the neutron data). Also, the difference between the variations of strain along the height and along the length seemed to be less visible in this sample. The variation of the strain components along the length was slightly less than the variation of the strain along the height. However, the difference between the data along the height and along the length from neutron data was clearer.

Along the horizontal scan line 2, both strain components (ε_Y and ε_Z) started with a tensile strain at the lateral edge and declined to a compressive strain ~10 mm inside the wall. Then both components showed a semi-stable trend inwards up to the middle of the length, where a tensile behaviour arises.

Little difference can be observed between the variations of the strain components along this scan line, which makes it difficult to judge the dominant component. However, considering the semi-steady trend for both strain components, it is not critical to make such a judgement, as both stress components have a slight variation along the length. Similar variation for stress components along the fine scan line 2 was observed.

The trend for strain (and stress) components from the coarse scan lines 5 and 6, along the length of the sample seemed to be in a good agreement with the strain (and stress) data from the fine scan line. However, there was a slightly higher variation seen along the two coarse scan lines, especially towards the middle of the height of the sample.
6.4. Summary

This chapter provided the full set of results from synchrotron X-ray diffraction experiment, at ESRF, Grenoble, France. Six samples were scanned via synchrotron X-ray diffraction. All samples were scanned through three scan lines along the height (one fine and two coarse scan lines) and three scan lines along the length (one fine and two coarse scan lines). X-ray scanning was repeated through the vertical fine scan line (height) for samples L-060-L (1) and Z-060-L (5).

The strain results for all 6 samples scanned via synchrotron X-ray diffraction are summarised in Table 5-2, for the main (fine) scan line along the height (scan line 1, 101 scan points), for both strain components: longitudinal strain ($\varepsilon_y$) and transverse strain ($\varepsilon_x$). Also, the same set of data are presented for stress results in Table 5-3.

The results along the length (scan line 2 with 101 scan points) are also summarised in Table 6-5 and Table 6-6, for strain and stress data, respectively.

Also, the strain results along the height (vertical scan line 1) and the length (horizontal scan line 2) are summarised in a bar chart, in Figure 5-25 and Figure 5-26, respectively.

- A good correlation was observed between the strain data from neutron diffraction and synchrotron X-ray diffraction. However, some discrepancy could be observed likely to be related to the misplacement of the sample on the stage and the mismatch of the coordinate system.
- The position of the sample on the sample manipulator is a critical step in setting up the experiment for the scanning of the samples. The alignment of the coordinate systems between the sample’s reference coordinate axes and the machine set up should be carefully conducted to deliver a reliable set of data. Rendering human error should be of utmost importance when setting up the sample could. However, it should be noted that by introducing the stress variations as point of comparison between different samples, the effect of such error on the measured/scanned data has been minimised, as individual data points have not been compared. This has been the case for the data collected by both neutron diffraction and synchrotron X-ray diffraction (as already covered in Chapter 5, section 5.4).
- A more effective gauge volume in synchrotron X-ray could be interpreted from the results, as the gauge volume was matchstick (as opposed to near cubic).
• Similar to the data from neutron diffraction, synchrotron X-ray results proved that the variation of stress along the vertical scan-lines (along the height of the samples) was higher than along the length of the samples (horizontal scan-lines). This made the vertical scan-lines to be of more interest to investigate the evolution of residual stress in PTA AM samples.

• Also, similar to neutron data, results from synchrotron X-ray diffraction confirms that the main component of stress seems to be on the Y-direction.

• The magnitude and variation of stress seemed to be higher in the Y-direction (along the length of deposited beads).

• Four main samples scanned by synchrotron X-ray diffraction were the samples with the lower dwell-time (60 seconds), as it was identified to have more severe effect on the residual stress evolution and distribution within parts. Also, two repeat samples with the higher level of the energy density were scanned to examine a more hectic combinations of process parameters (with regard to the level of the energy density).

• For the samples manufactured by linear deposition, both longitudinal (Y) and transverse (Z) components of strain and stress seemed to be high and playing a role in the overall stress distribution, while the variation and difference between stress values seemed to be higher on the longitudinal component of stress ($\sigma_y$), along both the height and length of the samples.

• For the samples manufactured by zig-zag deposition however, the longitudinal components of strain and stress showed a higher variation than the transverse components. Although, the variation of transverse strain and stress along the length was slightly higher than the longitudinal components.
Table 6-3 – Summary of strain results along the height (scan line 1) from synchrotron X-ray diffraction (all strain data ×10^6 ε) (average error: ±250×10^6)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Strain component</th>
<th>Min</th>
<th>Max</th>
<th>Difference</th>
<th>Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>L-060-L</td>
<td>εy</td>
<td>-1741</td>
<td>3286</td>
<td>5027</td>
<td>64</td>
</tr>
<tr>
<td></td>
<td>εx</td>
<td>-1718</td>
<td>3411</td>
<td>5129</td>
<td>185</td>
</tr>
<tr>
<td>L-060-H</td>
<td>εy</td>
<td>-1326</td>
<td>3790</td>
<td>5116</td>
<td>408</td>
</tr>
<tr>
<td></td>
<td>εx</td>
<td>-1897</td>
<td>2591</td>
<td>4488</td>
<td>527</td>
</tr>
<tr>
<td>Z-060-L</td>
<td>εy</td>
<td>-1744</td>
<td>2897</td>
<td>4641</td>
<td>-36</td>
</tr>
<tr>
<td></td>
<td>εx</td>
<td>-2197</td>
<td>2459</td>
<td>4656</td>
<td>-793</td>
</tr>
<tr>
<td>Z-060-H</td>
<td>εy</td>
<td>-1943</td>
<td>3185</td>
<td>5129</td>
<td>251</td>
</tr>
<tr>
<td></td>
<td>εx</td>
<td>-1717</td>
<td>2314</td>
<td>4030</td>
<td>-70</td>
</tr>
<tr>
<td>R-L-180-H</td>
<td>εy</td>
<td>-1280</td>
<td>3871</td>
<td>5152</td>
<td>465</td>
</tr>
<tr>
<td></td>
<td>εx</td>
<td>-1404</td>
<td>1908</td>
<td>3312</td>
<td>15</td>
</tr>
<tr>
<td>R-Z-180-H</td>
<td>εy</td>
<td>-1893</td>
<td>1825</td>
<td>3718</td>
<td>376</td>
</tr>
<tr>
<td></td>
<td>εx</td>
<td>-748</td>
<td>2538</td>
<td>3286</td>
<td>686</td>
</tr>
</tbody>
</table>
Table 6.4 – Summary of stress results along the height (scan-line 1) from synchrotron X-ray diffraction (all stress data in MPa) (average error: ±50 MPa)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Stress component</th>
<th>Min</th>
<th>Max</th>
<th>Difference</th>
<th>Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>L-060-L (1)</td>
<td>$\sigma_y$</td>
<td>-261</td>
<td>464</td>
<td>725</td>
<td>17</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-276</td>
<td>392</td>
<td>668</td>
<td>27</td>
</tr>
<tr>
<td>L-060-H (3)</td>
<td>$\sigma_y$</td>
<td>-197</td>
<td>416</td>
<td>613</td>
<td>76</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-185</td>
<td>308</td>
<td>493</td>
<td>86</td>
</tr>
<tr>
<td>Z-060-L (5)</td>
<td>$\sigma_y$</td>
<td>-270</td>
<td>309</td>
<td>579</td>
<td>-39</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-289</td>
<td>323</td>
<td>412</td>
<td>-104</td>
</tr>
<tr>
<td>Z-060-H (7)</td>
<td>$\sigma_y$</td>
<td>-251</td>
<td>353</td>
<td>605</td>
<td>29</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-153</td>
<td>298</td>
<td>445</td>
<td>20</td>
</tr>
<tr>
<td>R-L-180-H (R4)</td>
<td>$\sigma_y$</td>
<td>-189</td>
<td>481</td>
<td>671</td>
<td>61</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-162</td>
<td>308</td>
<td>470</td>
<td>22</td>
</tr>
<tr>
<td>R-Z-180-H (R8)</td>
<td>$\sigma_y$</td>
<td>-274</td>
<td>275</td>
<td>500</td>
<td>78</td>
</tr>
<tr>
<td></td>
<td>$\sigma_z$</td>
<td>-173</td>
<td>339</td>
<td>512</td>
<td>105</td>
</tr>
</tbody>
</table>
Table 6.5 – Summary of strain results along the length (scan line 2) from synchrotron X-ray diffraction (all strain data in $\times 10^{-6}$) (average error: $\pm 250 \times 10^{-6}$)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Strain component</th>
<th>Min</th>
<th>Max</th>
<th>Difference</th>
<th>Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>L-060-L (1)</td>
<td>$\varepsilon_y$</td>
<td>-1741</td>
<td>3286</td>
<td>5027</td>
<td>65</td>
</tr>
<tr>
<td></td>
<td>$\varepsilon_z$</td>
<td>-1718</td>
<td>3411</td>
<td>5129</td>
<td>185</td>
</tr>
<tr>
<td>L-060-H (3)</td>
<td>$\varepsilon_y$</td>
<td>-1325</td>
<td>3790</td>
<td>5116</td>
<td>408</td>
</tr>
<tr>
<td></td>
<td>$\varepsilon_z$</td>
<td>-1897</td>
<td>2591</td>
<td>4488</td>
<td>527</td>
</tr>
<tr>
<td>Z-060-L (5)</td>
<td>$\varepsilon_y$</td>
<td>-1744</td>
<td>2897</td>
<td>4641</td>
<td>-37</td>
</tr>
<tr>
<td></td>
<td>$\varepsilon_z$</td>
<td>-2197</td>
<td>2459</td>
<td>4656</td>
<td>-793</td>
</tr>
<tr>
<td>Z-060-H (7)</td>
<td>$\varepsilon_y$</td>
<td>-1943</td>
<td>3185</td>
<td>5129</td>
<td>251</td>
</tr>
<tr>
<td></td>
<td>$\varepsilon_z$</td>
<td>-1717</td>
<td>2314</td>
<td>4030</td>
<td>-69</td>
</tr>
<tr>
<td>R-L-180-H (R4)</td>
<td>$\varepsilon_y$</td>
<td>-1280</td>
<td>3871</td>
<td>5152</td>
<td>465</td>
</tr>
<tr>
<td></td>
<td>$\varepsilon_z$</td>
<td>-1404</td>
<td>1908</td>
<td>3312</td>
<td>15</td>
</tr>
<tr>
<td>R-Z-180-H (R8)</td>
<td>$\varepsilon_y$</td>
<td>-1893</td>
<td>1825</td>
<td>3718</td>
<td>375</td>
</tr>
<tr>
<td></td>
<td>$\varepsilon_z$</td>
<td>-748</td>
<td>2538</td>
<td>3286</td>
<td>686</td>
</tr>
</tbody>
</table>
Table 6.6 – Summary of stress results along the length (scan line 2) from synchrotron X-ray diffraction (all stress data in MPa) (average error: ±50 MPa)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Stress component</th>
<th>Min</th>
<th>Max</th>
<th>Difference</th>
<th>Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>L-060-L (1)</td>
<td>σ_y</td>
<td>-261</td>
<td>464</td>
<td>725</td>
<td>17</td>
</tr>
<tr>
<td></td>
<td>σ_z</td>
<td>-276</td>
<td>392</td>
<td>668</td>
<td>27</td>
</tr>
<tr>
<td>L-060-H (3)</td>
<td>σ_y</td>
<td>-197</td>
<td>416</td>
<td>613</td>
<td>76</td>
</tr>
<tr>
<td></td>
<td>σ_z</td>
<td>-185</td>
<td>308</td>
<td>493</td>
<td>86</td>
</tr>
<tr>
<td>Z-060-L (5)</td>
<td>σ_y</td>
<td>-270</td>
<td>309</td>
<td>579</td>
<td>-40</td>
</tr>
<tr>
<td></td>
<td>σ_z</td>
<td>-289</td>
<td>323</td>
<td>412</td>
<td>-104</td>
</tr>
<tr>
<td>Z-060-H (7)</td>
<td>σ_y</td>
<td>-251</td>
<td>354</td>
<td>605</td>
<td>29</td>
</tr>
<tr>
<td></td>
<td>σ_z</td>
<td>-153</td>
<td>298</td>
<td>450</td>
<td>20</td>
</tr>
<tr>
<td>R-L-180-H (R4)</td>
<td>σ_y</td>
<td>-189</td>
<td>481</td>
<td>671</td>
<td>61</td>
</tr>
<tr>
<td></td>
<td>σ_z</td>
<td>-163</td>
<td>308</td>
<td>470</td>
<td>22</td>
</tr>
<tr>
<td>R-Z-180-H (R8)</td>
<td>σ_y</td>
<td>-274</td>
<td>276</td>
<td>550</td>
<td>79</td>
</tr>
<tr>
<td></td>
<td>σ_z</td>
<td>-173</td>
<td>339</td>
<td>512</td>
<td>105</td>
</tr>
</tbody>
</table>
Chapter 6 – Synchrotron X-ray Diffraction to Determine Residual Strain and Stress in PTA AM Parts

Figure 6.32 – Strain data, from synchrotron X-ray diffraction, along the height (vertical scan line 1) (a) longitudinal strain ($\epsilon_y$) and (b) transverse strain ($\epsilon_z$).
Chapter 6 – Synchrotron X-Ray Diffraction to Determine Residual Strain and Stress in PTA AM Parts

Figure 6.33 – Strain data along the length (horizontal scan line 2) (a) longitudinal strain ($\varepsilon_y$) component (b) transverse strain ($\varepsilon_z$) component
Chapter 7 – Residual Stress Measurement in PTA AM Parts by Contour Method
7.1. Introduction

Non-destructive diffraction-based techniques provided an understanding of the level and the variation of residual strain/stress along the height and the length of the samples. Although both neutron and synchrotron X-ray diffraction provided detailed information about local strain (and stress) values (at scan-points), it is challenging to make a rational judgement of the stress state in the full-scale component as changes occur across the whole part. Another issue with diffraction-based techniques is the beam penetration depth and the formation (and location) of the gauge volume which makes such techniques incapable of determining strain (and stress) within the substrate of the parts; hence, no data can be obtained or extrapolated via neutron or synchrotron X-ray diffraction in the substrate. Also, the simplification and assumption for the plane stress condition proves to be a challenging process, mainly due to the choice of the reference value for the d-spacing \( d_0 \), as also mentioned in the literature. As residual stresses are self-equilibrating stresses occurring in a component, it is useful to have a full picture of the stress distribution to make a full-scale judgment of residual stress evolution. As explained in literature review (chapter 2), typically, residual stresses are categorised in three main forms: macro-stress, interphase stress, and intra-phase stress. Normally, the sum of these three contributes to the total residual stress within a component.

The contour method provides a more straightforward methodology to determine residual stress in a component. The advantage of the contour method over diffraction-based methods is its capability to evaluate stress within the whole cut cross section. In the case of PTA AM, it means the method enables measurement within the substrate as well as the deposited wall. However, this is a destructive method.

The samples’ matrix for residual stress measurement was given in chapters 4, 5 and 6 and is repeated here in Table 7.1: including samples information for the contour method. The samples are labelled with the processing parameters, as described before. The “repeat samples” were also manufactured and labelled with “R” to investigate the repeatability of the process. In total, the contour method was used to investigate residual stress in five samples, all manufactured by using the higher level of energy density. Two linear samples with the 60 s and 180 s dwell time, two zig-zag samples with the shorter and longer dwell-time and one repeat zig-zag samples with 180 s dwell-time.
As previously explained in the methodology chapter (Chapter 4), the aim is not to provide a comparison between different residual stress measurement techniques, but to investigate the level and variation of residual stress in PTA AM primitive geometries, via different methods to provide a baseline to understand any potential links between the PTA AM process parameters and the state of residual stress in PTA AM parts. This would allow customisation of the process to manufacture optimised parts with the desired mechanical/material properties and geometrical accuracy.

In presenting the data from the contour method, the stress results from neutron and synchrotron X-ray data, for the main vertical scan-line (scan-line 1 for both techniques) are also included for all samples, where such data were available.

**Table 7-1 – The residual stress measurement matrix**

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Sample ID</th>
<th>Deposition strategy</th>
<th>Energy density (MJ/m²)</th>
<th>Dwell time (s)</th>
<th>Neutron diff.</th>
<th>Sync X-ray</th>
<th>Contour method</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>L-060-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>60</td>
<td>✓</td>
<td>✓</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>L-180-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>180</td>
<td></td>
<td></td>
<td>✓</td>
</tr>
<tr>
<td>3</td>
<td>L-060-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>60</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>4</td>
<td>L-180-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
<td></td>
<td>✓</td>
</tr>
<tr>
<td>5</td>
<td>Z-060-L</td>
<td>Zig-zag (Z)</td>
<td>150 (Low)</td>
<td>60</td>
<td>✓</td>
<td>✓</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Z-180-L</td>
<td>Zig-zag (Z)</td>
<td>150 (Low)</td>
<td>180</td>
<td></td>
<td></td>
<td>✓</td>
</tr>
<tr>
<td>7</td>
<td>Z-060-H</td>
<td>Zig-zag (Z)</td>
<td>300 (High)</td>
<td>60</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>8</td>
<td>Z-180-H</td>
<td>Zig-zag (Z)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
<td></td>
<td>✓</td>
</tr>
<tr>
<td>2 (repeat)</td>
<td>R-L-180-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>180</td>
<td></td>
<td></td>
<td>✓</td>
</tr>
<tr>
<td>4 (repeat)</td>
<td>R-L-180-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
<td></td>
<td>✓</td>
</tr>
<tr>
<td>6 (repeat)</td>
<td>R-Z-180-L</td>
<td>Zig-zag (Z)</td>
<td>150 (Low)</td>
<td>180</td>
<td></td>
<td></td>
<td>✓</td>
</tr>
<tr>
<td>8 (repeat)</td>
<td>R-Z-180-H</td>
<td>Zig-zag (Z)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
<td></td>
<td>✓</td>
</tr>
</tbody>
</table>
7.2. Contour method experimental procedure

The process of the contour method to determine residual stress was summarised in the methodology chapter 4. As explained, the contour method is a geometry-dependent technique, which can be applied to simple geometries and is not suitable for parts with complex shapes or features. This is mainly due to the cutting process as a critical step in the method. The section geometry must be simplified as much as possible to reduce the effects of the cutting process.

From the diffraction-based techniques, it was concluded that the variation of stress (and strain) is higher along the height than the length of the samples. Therefore, the contour method was used to investigate stress variation along the height.

7.2.1. Cutting the samples

The cutting process is known as a critical step of the contour method as it provides the contours on the sample’s surface of interest for analysis. To obtain the section with maximum accuracy and details of the contours, the Wire EDM (Wire Electric Discharge Machining) process is recommended (Prime, 2001).

Change of the cross section could influence the final contours. To minimise this effect, use of sacrificial materials is considered, which is glued to the sample. By adding sacrificial material, a uniform cross section is created for the cutting process, to minimise the effect of wire travel across the cut-surface. The cutting direction is also an important factor to minimise the potential effect of wire-bouncing during the cutting process. A constant power was used throughout the cutting process to avoid any unpremeditated changes of the relaxed contours.

The addition of the sacrificial material and the direction of cutting on a sample is schematically shown in Figure 4-6. A wire EDM machine model Agie Charmilles FI440 (GF Machining Solutions Ltd, Coventry, UK).

The cut section is coincident with the main vertical scan-lines from both neutron (scan-line 1: 9 scan-points) and synchrotron X-ray diffraction (scan-line 1: 101 scan-points) to produce results along the height of the sample, at the same section scanned via diffraction techniques, as also shown in Figure 4-6.
(a) Cut section coincident with neutron and synchrotron X-ray scan line 1

(b) Addition of sacrificial material

(c) Direction of cutting

(d) Cutting process through the section (and sacrificial material)

*Figure 7.1 – Wire EDM cutting process for contour method*

The two counterparts of a cut sample is shown in Figure 7.2.
7.2.2. Scanning to map deformation (strain)

To map deformation, the scanning is conducted for both counterparts of the cut section (both surfaces) and then the results were averaged to minimise any errors and artefacts. A touch probe scanner Renishaw TP8 (Renishaw plc, Wootton-under-Edge, UK) was used to scan along the perimeter of both parts of the cut section. The surface of both cross sections was scanned using an optoNCDT2200 (Micro-Epsilon UK Ltd, Birkenhead, UK) laser scanner to provide a grid of data across the cross section. The scanning process for the two counterparts of a sample and the touch probe and the laser scanner are shown in Figure 4-7.
The scanned point cloud from each surface is corrected, to take the effect of the probe radius into account and eliminate any artefacts which is believed to be due to the wire effect (such as possible wire bouncing). Then the contours (deflections) from both surfaces are averaged which results in one set of point cloud as the contour. The point cloud is then imported to a Finite Element package (CAE/ABAQUS 6.13) to read deformation and stress contours.

Second cut

To investigate the level of stress on the out-of-plane direction, the second-cut methodology was applied to two samples. Referring back to the basics of the contour method, typically second-cut (or multiple-cut) is applied to investigate strain and stress in a second (and third) direction of the sample’s geometry, as schematically shown in Figure 7-4.

![Figure 7-4 – The schematic of the multiple-cut contour method](image)
The first-cut provides information on the longitudinal stress component and the second-cut provides data along the normal direction of the deposited wall, as schematically shown in Figure 7-5. Capturing and interpreting data along the edges must be taken carefully as typically there are overlap effects along multiple-cut edges. Adding the relaxed normal residual stress from the first cut will give the initial normal residual stress.

\[\text{(a) First contour cut}\\
\text{(b) Second contour cut}\\
\]

*Figure 7-5 – The schematic of the contour cut to measure (a) longitudinal and (b) normal deformation and stress*

The choice of samples for the second cut was based on the perceived “worst” variation of strain (and stress) from neutron diffraction and synchrotron X-ray diffraction, along the height of the samples (vertical scan-line 1). Hence, samples 3 and 7 were chosen for the second cut to investigate the level (and possibly the variation) of the out of plane stress (normal) and explore the validity of the plane stress assumption. The results from the second cut are presented and discussed in Chapter 8.
Chapter 7 – Residual Stress Measurement in PTA AM Parts by Contour Method

7.3. Contour results

The results from the contour method are presented in this section to show the level and distribution of residual strain/stress.

From neutron and synchrotron data, the variation of the strain and stress along the height of the PTA AM samples was observed to be more considerable than along the length of the samples. In this Chapter, results along the height from the section coincident with the scan line 1 for both neutron and synchrotron X-ray are presented. To make the data comparable, the strain and stress data along the middle line across the cut cross section was plotted for all samples, as schematically shown in Figure 7-6. It should be noted that the stress data from neutron and synchrotron X-ray were calculated based on two in-plane stresses (longitudinal and transverse strains), however the stress profile from the contour method is solely based on contour profile perpendicular to the cross cut section. This makes the variation of strain and stress profiles along a certain path, across the cross section, being similar, as both reflect one component of strain and stress (longitudinal).

To present the data, the same scale for the strain and stress axes was chosen to make the results comparable to the data from the diffraction techniques. As seen in the contour results, much less variation has been observed in both strain and stress values from the contour data.

Figure 7-6 – An example of the FE results across the cross section and the location of the middle line, where the data are plotted along
It is shown that the variation of the strain and stress along the width of the sample is negligible which is in agreement with the plane stress condition. Strain and stress data across the width of a sample plus data along the normal direction, from the second cut, are presented in Chapter 8, as part of a more detailed investigation and discussion of the plane stress condition.

7.3.1. Sample L-060-H (3)

The results from the Finite Element (FE) model of the cross section, from the first contour cut, for sample L-060-H (3) are shown in Figure 7.7, to show the variation and the level of strain and stress across the whole section, including the substrate.

![Figure 7.7 – FE results for strain and stress contours across the cross section of the first cut from sample L-060-H (3)](image)

To study the level and variation of the longitudinal strain ($\varepsilon_y$) and stress ($\sigma_y$) along the height of the sample, data along the middle-line of the cross section are plotted (Figure 5.4). The strain data from both neutron and synchrotron X-ray diffraction are also included. The contour method provided data from the substrate, which gives a full overview of the strain and stress across the whole cross section. However, it should be noted that only one component of the strain (and stress) can be observed from the contour method data (only normal to the cross section, which is equivalent to the longitudinal strain and stress along the length of the deposited wall/sample).
Figure 7.8 – Results for sample L-060-H (3) (a) residual strain and (b) residual stress in longitudinal direction (perpendicular to the cross section) from the top to the bottom of the middle line of the cross section.
A good correlation can be observed between the results from contour data and diffraction methods, although it should be noted that the number of data points is different between the three methods. The contour data were provided across the whole cut section and rendered to reflect the data along the middle scan-lines for diffraction methods.

The strain data from all three methods appeared to be in the same region. Similar to the strain (and stress) data from diffraction techniques, the strain (and stress) start from a compressive state at the top surface and become tensile at ~10 to 15 mm below the top layer. Moving towards the middle of the height, a compressive state can be seen for both strain and stress. Then, a tensile behaviour appears towards the bottom of the wall, at the intersection of the deposited wall and the substrate.

The longitudinal strain (εy) along the height of the cross section had a minimum of -1560 (ε×10⁶) at the top surface (just 1 mm below the reference point) and reached a maximum of 2544 (ε×10⁶) just below the wall (as the deposited material entered the substrate), at the height of 53 mm below the top surface of the wall.

The maximum compressive stress (σy) of -184 MPa occurred at the top of the wall, and the maximum tensile stress (σy) of 512 MPa occurred towards the bottom of the wall, within the substrate. The stress at the intersection of the wall and the substrate showed a similar value of 416 MPa to the data synchrotron X-ray diffraction (but slightly higher than the stress result from neutron diffraction)

As an advantage of the contour method over diffraction techniques, the strain (and stress) profile within the substrate was also provided which suggested a considerable tensile behaviour within the substrate. This tensile strain (and stress) could justify the relatively high level of compressive strain and stress on the top surface of deposited walls to provide an overall equilibrium of strain (and stress) status.
7.3.2. Sample L-180-H (4)

The contour method was conducted on another linear sample, L-180-H (4), with the higher level of energy density and a longer dwell-time of 180 s. This sample was also scanned via neutron diffraction, but not via synchrotron X-ray.

The contours for stress and strain distribution for sample L-180-H (4) are shown in Figure 7-9. Compared to sample L-060-H (3), a finer middle line point was obtained across the cross section, due to a slightly finer mesh size for this sample. Hence, data along the middle line across the cross section was captured along 172 nodes.

As expected, and briefly explained in the diffraction chapters (chapters 5 and 6), most of the corresponding compressive strain (and) stress appeared within the substrate, which was not scanned by either of the diffraction techniques, due to the limitation for the penetration depth of the neutron/X-ray beam.

![Figure 7-9](image)

(a) Strain contour repeat sample L-180-H (4)  
(b) Stress contour repeat sample L-180-H (4)

Figure 7-9 – FE results for strain and stress contours across the cross section of the first cut from sample L-180-H (4)

The stress and strain components perpendicular to the cut section ($\sigma_y$ and $\varepsilon_y$) along the middle line across the cross section of the sample are plotted in Figure 7-10.
Figure 7-10 – Residual strain/stress results along the middle line of the cross section for sample L-180-H (4) (including data from neutron diffraction) along the longitudinal direction (perpendicular to the cut-section) (a) residual strain and (b) residual stress
Similar to the linear sample L-060-H (3), the variation of strain and stress predicted by the contour method shows a reasonable correlation with the data from neutron diffraction. Both techniques predict similar trends mainly towards the bottom of the wall, although, there seems to be a large variation in strain and stress at a 10mm depth from the top layer. It should be noted that the contour data was extracted (rendered) from the contours across the whole cut section whereas the neutron data was acquired from local scan points along the middle line of the widths of the wall (cross section). The stress from neutron diffraction was calculated by using reduced form of the Hooke’s law, using two in-plane components of strain (longitudinal and transverse).

According to the contour data for strain and stress, the strain and stress seemed to be fully compressive at the top of the sample (stress of 535 MPa). Moving down the wall, along the middle line of the cross section, the stress transitioned to a tensile state 10 mm below the top layer, which is contrary to the neutron data. The stress remained tensile until 35 mm below the reference point, where it starts to decline again to a compressive state. The same trend could be observed from the neutron data. The stress showed a slight compressive behaviour at 40 mm down the reference point. The trend and values for both stress and strain seemed to be comparable between the neutron and contour methods, at the middle of the height, while the level and variation of stress seemed to be smaller from the contour data.

Towards the bottom of the wall, the stress increased to a tensile behaviour, rising up to more than 500 MPa within the substrate. This is equivalent to the results from neutron diffraction for the scanned point at 45 mm below the top surface. At the intersection of the wall and the substrate, the contour method predicted a positive (tensile) behaviour whereas the neutron data predicted a compressive strain (and stress). However, the tensile behaviour (from the contour data) seemed to be more reasonable than the compressive stress predicted by neutron diffraction.
7.3.3. Sample Z-060-H (7)

The Finite Element (FE) results for strain and stress distribution across the cross section from the 1st cut, for sample Z-060-H (7) are shown in Figure 7-11, to show the variation and the level of strain and stress across the cut section. The same scale is used to show both strain and stress distribution. The contour method provided strain and stress data within the substrate, as well.

![Strain and Stress Contours](image)

*Figure 7-11 – Finite Element (FE) results for strain and stress contours across the cross section of the first cut from sample Z-060-H (7)*

The longitudinal component of the stress ($\sigma_y$) and the strain ($\varepsilon_y$) along the middle line of the cross section were plotted in Figure 7-12, for 182 data points along the height of the cross section. Also, data from the coincident scan-line for neutron and synchrotron X-ray diffraction were also included. Contour data are also plotted against a secondary axis to magnify the trend for both stress and strain results.
Figure 7-12 – Residual strain/stress results along the middle line of the cross section for sample Z-060-H (7) (including data from neutron diffraction) along the longitudinal direction (perpendicular to the cut-section) (a) residual strain and (b) residual stress
Similar to the linear samples, for the zig-zag sample Z-060-H (7), a reasonable correlation can be observed between the trends from the contour data and diffraction-based results along the height of the sample. Although, the level and variation of the data from the contour method seemed to be slightly less compared to the results from either of the diffraction-based techniques.

As shown in Figure 5.10, both stress and strain start from a compressive state at the top of the sample. Their behaviours shift to a tensile state at 10 mm below the top layer, as also predicted by both neutron and synchrotron X-ray. The stress measured by the contour method was ~250 MPa at the top surface.

Towards the middle of the height, both stress and strain showed less variation, remaining in a tensile state at ~100 MPa for the stress component and ~550 ($\epsilon \times 10^6$) for the strain component. At ~35 mm below the reference point, stress (and strain) start to decline towards a compressive value up to a position 10 mm above the substrate (40 mm below the reference point). The same trend was observed from the neutron data, but shifted by 5 mm along the height, starting at 30 mm below the top surface.

Moving down the height, towards the intersection of the deposited wall and the substrate, the stress rises up rapidly to a tensile state, which seems to be similar to the predicted trend from synchrotron X-ray diffraction, although the neutron diffraction data predicted a slight decline in the stress (and strain) at the intersection of the wall and the substrate.

The stress within the substrate increased and reached a maximum tensile stress of ~450 MPa at the bottom of the substrate, which was also seen from the Finite Element (FE) contour. The equivalent strain at the bottom of the substrate was ~2800 ($\epsilon \times 10^6$).
7.3.4. Sample Z-180-H (8) and repeat sample R-Z-180-H (R8)

The contour method was used for both samples Z-180-H (8) and R-Z-180-H (R8). Repeat samples were manufactured to investigate repeatability of the manufacturing process. Neutron data were also obtained for both samples. However, synchrotron X-ray was conducted on the repeat sample R-Z-180-H (R8) only. The contour maps for stress and strain across the cut section are shown in Figure 7-13 for both original and repeat samples.

![Contour maps for stress and strain](image)

(a) Strain contour  
(b) Stress contour

*Figure 7-13 – Finite Element (FE) results for strain and stress contours across the cross section from the first cut for sample Z-180-H (8)*

Residual strain and stress results along the middle line of the cross section for both samples are shown in Figure 7-14 and Figure 7-15, where neutron and X-ray data are also included, where available. All results represented the longitudinal components, for both strain and stress ($\sigma_y$ and $\varepsilon_y$).
Figure 7-14 – Residual strain/stress results along the middle line of the cross section for sample Z-180-H (8) (including data from neutron diffraction) along the longitudinal direction (perpendicular to the cut-section) (a) residual strain and (b) residual stress.
Figure 7-15 – Residual strain/stress results along the middle line of the cross section for sample R-Z-180°-H (R8) (including data from neutron diffraction) along the longitudinal direction (perpendicular to the cut-section) (a) residual strain and (b) residual stress
A good correlation can be observed between the results obtained via the contour method for both original sample Z-180-H (8) and repeat sample R-Z-180-H (R8). Also, same as for the other samples, the contour data seemed to be in agreement with both neutron and synchrotron X-ray diffraction results.

As shown in Figure 7-14 and Figure 7-15, in both samples, the stress (and strain) started from a compressive state at the top of the deposited wall and shifted to a tensile behaviour at ~10 mm below the top surface. With a slight variation, the stress remained tensile until the middle of the height, where the stress value reached a maximum of ~120 MPa at ~20 mm below the top layer. This is in a good agreement with results from both neutron and synchrotron X-ray diffractions for both original and repeat samples Z-180-H (8) and R-Z-180-H (R8).

Both neutron and synchrotron x-ray predicted a compressive stress state at the intersection of the wall and the substrate, however the contour data suggest a tensile stress around that region with more considerable tensile stress as you move into the substrate.

Comparing the contour data between the original and repeat samples: it seemed the variation was slightly less in the repeat sample, which was contrary with the prediction from neutron diffraction. Neutron data suggested that the variation was slightly higher in the repeat sample R-Z-180-H (R8) than the original sample Z-180-H (8).

### 7.4. Summary

The destructive contour method provided stress (and strain) results across the cut sections. As suggested by both neutron and synchrotron X-ray diffraction, the variation and level of residual stress was higher along the height than the length of the PTA AM samples. Hence, the contour method was implemented to obtain an understanding of the level and variation of residual stress along the height of the samples.

The contour method utilised the concept of solid mechanics to calculate the stress (and strain). The method provided residual stress (and strain) results based on the relaxed contours (after cutting the sections), across the whole cross section. The method thus enabled investigation of the level and the variation of the residual stress across the section of interest. To study the variation of the stress along the height, the middle line along the
height was chosen, presumably coincident with the location of the scan points (the centre of the gauge volume) in diffraction-based techniques.

The cutting process proved to be a critical step in implementing the contour method. In fact, through this step, the contours are generated, which signifies the internal residual stress/strain within the sample. The cutting process, however, involves a wire moving along the surface to cut through the section. Such a physical movement of the wire should be carefully controlled and monitored so that does not introduce artefacts, or if it does, the effects should be rendered during the analysis.

The contour method also provided data within the substrate where it could not be obtained via diffraction techniques. Hence, a clearer overview of the stress distribution was attained by the contour method. This is clearly an advantage over the diffraction-based techniques, as the contour method offers a more detailed analysis of the residual stress within the samples.

The results for the strain and stress for all samples measured by the contour method are summarised in Table 7-2 and Table 7-3. The samples were chosen based on the high level of the energy density to obtain deformation contours plus one repeat sample to investigate repeatability.

Generally, the residual stress/strain variations from the contour method seemed to be slightly of a lower level than the results obtained by diffraction-based techniques. However, considering the trend of the stress (and strain) variations along the height of the cross section, a good correlation observed between the results from the contour data and diffraction-based data. The stress (and strain) behaviour seemed to follow the logical compressive status at the top of the samples and become tensile towards the bottom of the height of the samples.
### Table 7.2 – Summary of the strain results along the middle line across the cut section from contour method (all strain data $\varepsilon \times 10^6$)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Strain component</th>
<th>Min</th>
<th>Max</th>
<th>Difference</th>
<th>Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L\cdot060\cdot H$ (3)</td>
<td>$\varepsilon_y$</td>
<td>9.21</td>
<td>2087</td>
<td>3009</td>
<td>791</td>
</tr>
<tr>
<td>$L\cdot180\cdot H$ (4)</td>
<td>$\varepsilon_y$</td>
<td>2061</td>
<td>1008</td>
<td>3069</td>
<td>265</td>
</tr>
<tr>
<td>$Z\cdot060\cdot H$ (7)</td>
<td>$\varepsilon_y$</td>
<td>1074</td>
<td>1815</td>
<td>2890</td>
<td>265</td>
</tr>
<tr>
<td>$Z\cdot180\cdot H$ (8)</td>
<td>$\varepsilon_y$</td>
<td>871</td>
<td>1753</td>
<td>2625</td>
<td>280</td>
</tr>
<tr>
<td>$R\cdot Z\cdot180\cdot H$ (R8)</td>
<td>$\varepsilon_y$</td>
<td>865</td>
<td>1102</td>
<td>1968</td>
<td>-8</td>
</tr>
</tbody>
</table>

### Table 7.3 – Summary of the stress results along the middle line across the cut section from contour method (all data in MPa)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Stress component</th>
<th>Min</th>
<th>Max</th>
<th>Difference</th>
<th>Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L\cdot060\cdot H$ (3)</td>
<td>$\sigma_y$</td>
<td>-82</td>
<td>227</td>
<td>310</td>
<td>46</td>
</tr>
<tr>
<td>$L\cdot180\cdot H$ (4)</td>
<td>$\sigma_y$</td>
<td>244</td>
<td>247</td>
<td>492</td>
<td>45</td>
</tr>
<tr>
<td>$Z\cdot060\cdot H$ (7)</td>
<td>$\sigma_y$</td>
<td>119</td>
<td>235</td>
<td>354</td>
<td>23</td>
</tr>
<tr>
<td>$Z\cdot180\cdot H$ (8)</td>
<td>$\sigma_y$</td>
<td>244</td>
<td>249</td>
<td>493</td>
<td>14</td>
</tr>
<tr>
<td>$R\cdot Z\cdot180\cdot H$ (R8)</td>
<td>$\sigma_y$</td>
<td>68</td>
<td>204</td>
<td>272</td>
<td>18</td>
</tr>
</tbody>
</table>
Chapter 8 – On the Question of Plane Stress Condition
Chapter 8 – On the Question of Plane Stress Condition

8.1. Introduction

All stresses in this research work were calculated based on the plane stress condition. In both diffraction-based methods, neutron and synchrotron X-ray, the two in-plane strain components (\(\varepsilon_N\) – longitudinal and \(\varepsilon_T\) – transverse) were determined by scanning in two directions, so the \(Q\)-vector remained in-plane. Hooke’s law was then used to calculate both in-plane stress components (\(\sigma_N\) and \(\sigma_T\)). The contour method was also used to determine longitudinal stress (\(\sigma_N\)) and the equivalent strain (\(\varepsilon_N\)), as the longitudinal direction (Y) was believed to be the more considerable in-plane direction.

In analysing diffraction-based data, to calculate strain from \(d\)-spacing, the reference value for \(d\)-spacing (\(d_0\)) was required to enable using Bragg’s law (Chapter 4). The reference \(d\)-spacing (\(d_0\) or \(2\theta_0\)) either is already known for a strain-free lattice or should be determined. However, \(d_0\) depends on a number of parameters. For example, for titanium, the chemical composition of \(\alpha\)-phase has significant effect on reference \(d\)-spacing (\(d_0\)). Thermal history of the material also affects the \(d\)-spacing. Localised microstructural changes could also become the source of unstable and variable \(d_0\) within the material. Furthermore, residual stress could affect the localised \(d\)-spacing. Overall, three main issues related to determining the reference lattice \(d\)-spacing (\(d_0\)) are: crystal size (microstructural features), chemistry of phases and residual stresses. Hence, the optimum method for determining the reference \(d\)-spacing (\(d_0\)) depends on the particular material, process or application. Methods include (Gnäupel-Herold et al., 2005):

- Measurement in the material at a position known to exhibit negligible strain
- Measurement on a representative powder of the material
- Measurement on small coupons cut from large blocks of the material
- Calculation of \(d_0\) by imposing force and stress equilibrium

The additive-layer process causes microstructural changes, such as extension of grain boundaries and change in the lattice parameter. Such microstructural variations typically occurs along the height as more layers are deposited and stacked on top of each other (rather than within an individual layer). Local variations in the microstructure leads to a variation in \(2\theta_0\) values, which means a unique value, could not be used for reference \(d\)-spacing (\(d_0\) or \(2\theta_0\)). The variation of \(2\theta_0\) could be related to different thermal phenomena for PTA AM.
Typically, using a powder particle is suggested to provide a zero stress field. However, preparing such a small sample and ensuring the correct scanning proved to be a complicated process to implement. Moreover, in PTA AM, the powder could not be representative of all material changes across the whole samples as microstructure is changing.

In the current research work, in order to calculate $2\theta_0$ and ultimately residual stress values at each point, a plane stress condition was assumed, based on the small thickness of deposited walls compared to the other two dimensions (length and height). Therefore, the out-of-plane stress was assumed to be zero ($\sigma_z=0$) and $2\theta_0$ values for each point were determined and adjusted to obtain zero stress in the out-of-plane direction (X). Then, the reference d-spacing ($d_0$ or $2\theta_0$) was determined by considering the plane stress assumption. Furthermore, a strain-free sample was cut from one of the samples to scan through the height of the sample and investigate any potential variation of d-spacing. All methods in this research work suggest a single reference value for d-spacing ($d_0$ or $2\theta_0$) across all scan-lines.

To validate the plane stress condition, this chapter provided a comprehensive overview of such an assumption and how it was implemented in different techniques, to provide a basis for the discussion of all results in Chapter 9.

8.2. Plane stress condition based on diffraction-based techniques: reference d-spacing ($2\theta_0$)

In both neutron and synchrotron X-ray diffraction, the position of the gauge volume and scattered and diffracted beams determined the measured d-spacing and the direction of the associated strain component. As explained in Chapter 4, in both techniques, the two in-plane directions for d-spacing were scanned and Bragg’s law was used to determine the associated strain components. Then Hooke’s law, based on plane stress condition, was applied to calculate the two in-plane stress components.

To determine the reference d-spacing, $d_0$ (and associated $2\theta_0$), different methods were suggested in the literature (Rangaswamy et al., 2005; Wang et al., 2017; Hönnige et al., 2018). In this chapter, three methods are used to determine the reference d-spacing ($2\theta_0$) and examined the authenticity of the plane stress assumption, accordingly.
8.2.1. Method 1 – Averaging the scanned \( 2\theta \) across the whole sample to obtain the reference \( 2\theta_0 \)

An obvious and straightforward approach to decide the reference value \( 2\theta_0 \) would be an average of all scanned \( 2\theta \) values along different scan lines in the sample. Starting with neutron data, all scanned \( 2\theta \) values along all the scan lines: vertical scanlines 1 and 3 and horizontal scan line 2, were averaged to obtain a reference value per sample. Then, all averaged \( 2\theta \) values from all samples were averaged to obtain one reference \( 2\theta_0 \) value for all strain calculations via Bragg’s law. Ultimately, both in-plane strain components were used to calculate in-plane stress components by applying the reduced form of Hooke’s law as explained in Chapter 4.

The reference value for synchrotron X-ray diffraction was set to the equivalent value to the reference value for neutron diffraction.

8.2.2. Method 2 – From neutron diffraction: scanning the out-of-plane \( 2\theta \) and making the out-of-plane stress to zero to determine the reference d-spacing (\( 2\theta_0 \))

This method was used to support the implementation of the plane stress condition as explained in method 1 (and in Chapter 4).

For all eight main samples (Table 4.1), \( 2\theta \) in the out-of-plane direction was also scanned. All measured \( 2\theta \) values in the out-of-plane direction were then used to calculate strain in the third direction. All three components of strain enabled the calculation of all three stress components. The stress results were then used to make out-of-plane stress (\( \sigma_x \)) zero to meet the plane stress assumption. To conduct that, all individual measured \( 2\theta_x \) (along the out-of-plane direction) were adjusted to obtain zero value for the out-of-plane stress (\( \sigma_x = 0 \)). The process was applied for each individual scan point along the height (along the main scan line 1) for all eight samples, resulting in correction of \( 2\theta_x \) for all samples for all scan points along the main scan line 1 (height of the samples).

Figure 8·1 and Figure 8·2 show the variation of \( 2\theta_0 \) values for eight main samples, along the height (scan lines 1) and length (presumably within an individual layer: scan line 2), respectively. Very slight variation in \( 2\theta_0 \) values confirms the validity of the assumption for microstructural variation. By assigning these adjusted \( 2\theta_0 \) values, the assumption of plane stress condition was met.
Chapter 8 – On the Question of Plane Stress Condition

**Figure 8.1** – The change of $2\theta_0$ along scan line 1 (height of the sample) in samples 1 to 8

**Figure 8.2** – The change of $2\theta_0$ along scan line 2 (length of the sample) in samples 1 to 8
As shown in Figure 8-1, for the eight main samples the variation of the adjusted $2\theta_0$ was negligible. The variation could therefore be referred to the microstructural variations along the height of the deposited walls. Hence, the assumption for plane stress condition based on one single value of $2\theta_0$ was justified by referring to the microstructural variations due to different heating and cooling phenomena or in most general term the effect of manufacturing process.

The same process for the results along the length of the samples showed a similar paradigm. The out-of-plane $2\theta$ was scanned for all individual scan points and $2\theta_0$ was decided based on the adjusted value to force the out-of-plane stress to zero ($\sigma_z=0$). The negligible difference between the values for the adjusted $2\theta_0$ proved that there is no global variation of the $2\theta_0$ value across the sample and therefore the choice of “one” magnitude for the reference d-spacing was valid.

It should be noted that scanning of the out-of-plane d-spacing $2\theta$ was not possible in synchrotron X-ray diffraction, due to the low scattering angle and the elongated shape of the gauge volume. Plane stress condition was applied by using the reduced version of Hooke’s law for the two in-plane stress components.

8.2.3. Method 3 – From synchrotron X-ray diffraction: scanning a strain-free sample to understand the variation and determine the reference d-spacing value ($2\theta_0$)

A standard approach to determine the reference value for d-spacing/$2\theta_0$ is scanning a strain-free sample. Preparing a strain-free sample, however, is a complicated process. Typically, the strain-free sample is cut from the actual sample with the minimum possible thickness along the direction of interest to measure strain/stress.

A strain-free sample, with the thickness of 3 mm, was cut from sample L-060-H (3), as one of the worst cases for strain (and stress) variation. A scan-line was defined along the height of the sample at the middle of the cross section and the sample was scanned via synchrotron X-ray beam with a nominal wavelength of $\lambda=0.3545$ Å.
Figure 8·3 – Strain free sample cut from sample L·060·H (3) with a thickness of 3 mm and the scan line along the height

The strain free sample was scanned along the scan line at the middle section (Figure 8·3) through consecutive scan points, 1 mm apart from each other, from the top to the bottom of the sample. The same α (101) plane was chosen for the scans due to a visible and strong diffraction. The first and last scan data were omitted from the results as believed to be affected by non-complete gauge volume, located outside the sample.

Also, considering the shape of the gauge volume in synchrotron X-ray, scanning outside the sample along the middle line was likely along the vertical scan line. Thus, a slight variation of the scanned d-spacing (and therefore 2θ₀) was expected. Although, the sample’s thickness was cut to be 3 mm and beam size covers up to 2 mm along at each scan point, dis-positioning or grains out of the gauge volume could be the source of such uncertainty.

The magnitudes and variation of the scanned 2θ along the scan line are plotted in Figure 8·4, where the equivalent strain values were also calculated and plotted for the scan points along the vertical scan line.
Figure 8.4 – (a) $2\theta_y$ and (b) strain (longitudinal component: $\gamma$) results along the height of the strain-free sample (Figure 8.3) from synchrotron X-ray diffraction
As shown in Figure 8·4, the magnitudes of the scanned $2\theta$ from the top to the bottom of the strain-free sample clearly showed a low variation of the $2\theta$. To convert the scanned $2\theta$ to strain, Bragg’s law was used. Averaging all scanned $2\theta$ (method 1), a reference value for $2\theta$ was determined to enable calculation of the strain by using Bragg’s law.

Strain data based on the scanned $2\theta$ also confirmed the negligible strain variation along the height of the strain-free sample. Also, the magnitudes of strain were shown to be of a lower scale compared to the longitudinal strain from the main PTA AM part.

Combining the analysis based on the neutron data for the eight main samples and synchrotron X-ray data for the strain-free sample confirmed that small change in the reference d-spacing ($d_0$ or $2\theta_0$) could be a possibility when moving from the centre of a bead to the sides. However, large scale variation of the reference value is unlikely and could be ignored.

Therefore, the negligible strain variation (and magnitudes) provides a justification for no considerable local variation of the reference d-spacing ($2\theta_0$) which allowed for presenting results based on the two in-plane components.

**8.3. Plane stress condition based on contour method**

With the contour method, all strain and stress data were obtained for the component along the direction perpendicular to the cut surface, i.e. longitudinal stress ($\sigma_y$) and strain ($\varepsilon_y$). However, unlike diffraction-based techniques, the contour method provided stress (and strain) results across the whole cut section, as explained in the methodology Chapter 4 and presented in the contour method’s results (Chapter 7).

**8.3.1. Cut section across the width of the sample (first-cut)**

Contour method data across the whole cross section could provide another justification for the plane stress condition. To examine it, three paths were chosen at different heights along the height of the cross section, as shown in Figure 8·5. The stress data along the three paths were plotted in Figure 8·6, to investigate any variation of the stress data along the width of the deposited wall.
As shown in Figure 8.6, stress variation along the width, along each path seemed to be negligible, which indicates no significant effect of the width of the sample on the evolution of residual stress.
This was the case for all three paths along the height of the sample. The stress magnitudes at each height also conform to the original perception of the stress distribution along the height of deposited samples. A compressive stress was observed towards the top of the wall, low tensile stress towards the middle (at the height of 30 mm below the top surface) and a higher tensile stress magnitude at the bottom, nearer to the intersection of the deposited wall and the substrate.

The negligible stress variation along the width of the sample (from first cut), evince the insignificance of the width compared to the length and the height of the sample and shows that the geometry of the deposited wall plays a role in the evolution of residual stress in PTA AM samples.

### 8.3.2. Cut section across the length of the sample (second-cut)

The second cut methodology was also conducted to investigate the level/variation of the out-of-plane stress component ($\sigma_z$), as described in Chapter 4 and the results from contour method in Chapter 7.

Sample L-060-H (3) was cut along the length of the sample, second cut, as shown in Figure 8-7. The second cut was conducted on the “start-side” of the deposited wall to provide a full height of the deposited wall and minimise the effects of the end-side, as much as possible.

(a) Schematic of the second cut
(b) First and second cut on the sample and associated stress components

Figure 8·7 – Second contour cut (a) schematic of the second cut and (b) second cut on the sample

As already explained for the contour method methodology (Chapter 4), the scanned point cloud was imported into a Finite Element manipulator to read deformation and stress along the direction perpendicular to the cut surface, which in this case is the out-of-plane stress $\sigma_n$ (normal stress).

The stress contour for the cut surface is shown in Figure 8·8. The overall range of stress was shown to be lower than the stress distribution across the first cut section through the width of the cross section of the deposited wall. A path was defined along the height of the cut surface, to investigate the level and variation of residual stress across the second contour cut.

Stress variation along the path is plotted in Figure 8·9, from the top to the bottom of the sample, including within the substrate. As the stress nearer to the first cut section seemed to be of higher magnitudes, the path was located near the edge of the first cut to consider the worst case scenario for the stress distribution.
Chapter 8 – On the Question of Plane Stress Condition

Figure 8.8 – Stress distribution across the second cut section

Figure 8.9 – Out-of-plane stress ($\sigma_x$) along the height of the sample, near the edge of the cut section (as shown in Figure 8.8)

The out-of-plane (normal) stress ($\sigma_x$) profile along the height of the second cut showed a negligible magnitude and variation compared to the longitudinal stress ($\sigma_y$) from the first
cut. Similar to the longitudinal stress ($\sigma_y$), the out-of-plane stress ($\sigma_x$) seemed to be more considerable within the substrate than the deposited wall. This shows the validity of the plane stress condition for the deposited wall, but not for the substrate.

**8.4. Summary**

Plane stress assumption was the main part of the methodology to calculate in-plane stress components from the three residual stress measurement techniques. The justification for the plane stress assumption was discussed in Chapter 4, where the geometrical aspects of the deposited wall was introduced as the main reasoning behind such assumption.

Following presenting the results in Chapters 5, 6 and 7, the validity of the assumption was discussed in more detail in Chapter 8 to justify the consistency of the approach to implement the plane stress condition for both diffraction-based techniques, neutron and synchrotron X-ray diffraction. The justification was also extended to the contour method to show the validity of the approach in considering the longitudinal stress component ($\sigma_y$) from the first contour cut, for the purpose of discussing the results in Chapter 9.

With regard to the diffraction-based techniques, it was important to realise the significance of having one value for the reference d-spacing (d$_0$ or 2$\theta_0$) in calculating all strains and stresses. This led to the definition of the three methods to determine the reference d-spacing (d$_0$ or 2$\theta_0$).

Based on the neutron diffraction data, no global variation of 2$\theta_0$ was observed along the height of the samples (vertical scan-lines) and along the length of the samples (the horizontal scan-lines).

Also, based on synchrotron X-ray scanning of the strain-free sample, a negligible local variation of the scanned 2$\theta_0$ was observed along the height of the sample, which allowed taking one value for the reference 2$\theta_0$ for all strain calculations.

Based on the first cut in the contour method, no global variation of the longitudinal stress along the width of the deposited wall was observed, which indicated no effect of width compared to the length and the height of the deposited walls.

Also, from the second contour cut, the out-of-plane stress ($\sigma_x$) magnitudes and variation along the height of the sample was shown to be negligible compared to the contour data.
for the longitudinal stress ($\sigma_y$). This has suggested negligible local effects of the out-of-plane stress and hence confirmed the validity of the plane stress assumption.

Analysis of the plane stress condition in this chapter validated the discussion of the results in Chapter 9 (next chapter), which is based on the two in-plane stress components, longitudinal stress ($\sigma_y$) and transverse stress ($\sigma_z$) components.
Chapter 9 – The Effect of the Process Parameters on the Evolution of Residual Stress in PTA AM
9.1. Introduction to the discussions

The diffraction-based techniques provided an understanding of the level/variation of the residual stress along both in-plane directions (height and length) through a number of scan points along different scan lines. Also, the contour method provided stress (and strain) data across the cross section coincident with the main vertical scan line (from the top to the bottom of the height) for neutron and synchrotron X-ray diffraction.

Following presenting of the results from the three residual stress measurement techniques, this chapter provides a full discussion of the results, to investigate the effect of the three process parameters: deposition strategy, dwell-time and energy density, on the residual stress state.

The samples matrix for residual stress analysis is repeated here, in Table 9-1, which summarises all experimental undertakings, for stress determination in PTA AM parts.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Sample ID</th>
<th>Deposition strategy</th>
<th>Energy density (MJ/m²)</th>
<th>Dwell time (s)</th>
<th>Neutron diff.</th>
<th>Sync X-ray</th>
<th>Contour method</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>L-060-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>60</td>
<td>✓</td>
<td>✓</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>L-180-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>180</td>
<td>✓</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>L-060-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>60</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>4</td>
<td>L-180-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
<td></td>
<td>✓</td>
</tr>
<tr>
<td>5</td>
<td>Z-060-L</td>
<td>Zig-zag (Z)</td>
<td>150 (Low)</td>
<td>60</td>
<td>✓</td>
<td>✓</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Z-180-L</td>
<td>Zig-zag (Z)</td>
<td>150 (Low)</td>
<td>180</td>
<td>✓</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Z-060-H</td>
<td>Zig-zag (Z)</td>
<td>300 (High)</td>
<td>60</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>8</td>
<td>Z-180-H</td>
<td>Zig-zag (Z)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
<td></td>
<td>✓</td>
</tr>
<tr>
<td>2 (repeat)</td>
<td>R-L-180-L</td>
<td>Linear (L)</td>
<td>150 (Low)</td>
<td>180</td>
<td>✓</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4 (repeat)</td>
<td>R-L-180-H</td>
<td>Linear (L)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
<td>✓</td>
<td></td>
</tr>
<tr>
<td>6 (repeat)</td>
<td>R-Z-180-L</td>
<td>Zig-zag (Z)</td>
<td>150 (Low)</td>
<td>180</td>
<td>✓</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8 (repeat)</td>
<td>R-Z-180-H</td>
<td>Zig-zag (Z)</td>
<td>300 (High)</td>
<td>180</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
</tbody>
</table>
Although differences were observed between the residual strain/stress results from neutron diffraction and synchrotron X-ray diffraction, both techniques showed similar trends and variations of residual stress among all samples. Also, the stress (and strain) data from the contour method conformed to the trends predicted by diffraction-based techniques.

It should be noted that comparing between different residual stress measurements techniques was not the purpose of this research work; but rather to obtain an understanding of the level and the variation of the residual stress and comparing between samples with different process parameters. The aim was to obtain a fundamental understanding of the potential effect of the manufacturing process on the evolution of the residual stress. The level of the residual stress was similar between the data from different measurement techniques. Also, the interpretation of the results from both neutron and synchrotron X-ray diffraction as well as the contour method, indicated similar trends from different combinations of process parameters.

To discuss the effect of each of the three altering process parameters (deposition strategy, dwell-time and energy density), the results from all three residual stress determination techniques (neutron diffraction, synchrotron X-ray diffraction and contour method) have been discussed for available samples/set of data. The results from the diffraction-based techniques were compared by superimposing the actual strain (and stress) data for the pair of the samples with one different level of process parameters, while the other two parameters were the same. This would allow understanding of the variation of the residual strain/stress evolution in samples and make a possible link to the process parameters and their combinations.

A schematic of a PTA AM sample with associated neutron scan-lines along the height (vertical scan-line 1) and the length (horizontal scan-line 2) of the samples is shown in Figure 9-1. A schematic of a sample with the associated vertical scan-line 1 and horizontal scan-line 2 for synchrotron X-ray diffraction, along the height and the length of the sample is shown in Figure 9-2. Also, a schematic of a PTA AM sample with the associated cut section along the height of the sample, for the contour method, is shown in Figure 9-3.
As it was shown in Chapters 5 and 6, the variation of the longitudinal strain ($\varepsilon_y$) and stress ($\sigma_y$) were higher than the variation of the transverse components of strain ($\varepsilon_z$) and stress ($\sigma_z$), along both the vertical (height) and the horizontal (length) scan lines. This was believed to be due to the deposition direction as was reported in previous works and
generally for welding beads. The presence of additional cooler material ahead of the weld pool in the longitudinal direction restricting expansion and contraction of the weld as it solidifies, caused higher levels of the stress in the longitudinal direction (Coules et al., 2012).

Also, it was shown that the variation (and the magnitude) of the strain and stress data were higher along the height (from the top to the bottom) than along the length of the samples. This was attributed to the larger thermal gradients during the cyclic heating and cooling of the samples and has been reported in studies for different materials and DED processes (Rangaswamy et al., 2005; Moat et al., 2011; Wang et al., 2017).

Along the height of the deposited walls, from the top to the bottom (along the height), all samples exhibited longitudinal tensile stress ($\sigma_y$) towards the top of the deposited wall and changed to compressive stress towards the middle of the height and to the tensile stress towards the bottom of the wall (intersection with the substrate). This was also the case from all the contour method data. The tensile stress towards the bottom of the deposited walls continued within the substrate and the magnitudes increased towards the bottom of the substrate, significantly.

The longitudinal tensile residual stresses ($\sigma_y$) near the top of the samples could be explained by the occurrence of the thermal shrinkage due to the localised thermal contraction, as each layer cooled down on top of the previous layer. This contraction would cause local distortion of each individual layer and ultimately the deposited wall/sample, which was constrained by the relatively thick substrate. Such phenomena resulted in introducing tensile stresses in the transverse direction towards edges and balancing compressive stresses in the inner sections of the wall.

Similar phenomena was reported by Rangaswamy et al for 316 stainless steel and Inconel 718 samples produced by the LENS process (Rangaswamy et al., 2005) and Moat et al. for Waspaloy samples produced by laser direct metal deposition (Moat et al., 2011).

The maximum, the minimum and the average of the longitudinal strain ($\varepsilon_y$) data for all samples, based on the two diffraction-based stress measurement techniques are summarised in Figure 9-4 (a and b).
Chapter 9 – The effects of process parameters on the evolution of residual stress in PTA AM

(a) Neutron diffraction results – Maximum, minimum and average strain values, longitudinal strain ($\varepsilon_y$)

(b) Synchrotron x-ray diffraction – Maximum, minimum and average strain values, Longitudinal strain ($\varepsilon_y$)

Figure 9.4 – Longitudinal strain ($\varepsilon_y$) data along the height (scan line 1) (a) Neutron and (b) Synchrotron x-ray diffraction
The data from the contour method are also summarised as in Figure 9·5. The maximum and minimum values for the longitudinal strain, perpendicular to the cut surface, were in the same range as the data from the diffraction-based techniques.

However, it should be noted that the data from the contour method were obtained by applying a different assumption, as there was no data available for the transverse components of strain ($\varepsilon_z$) and stress ($\sigma_z$).

The following sections discuss in more detail the effect of each processing parameter on the evolution of residual stresses based on the strain data presented in Figure 9·4 and Figure 9·5 and stress data, which are presented in their associated sections.

Figure 9·5 – Longitudinal strain ($\varepsilon_y$) data from the contour method along the height (coincident with the vertical scan line 1 for diffraction-based data)
9.2. The effect of the deposition strategy

To investigate the effect of the deposition strategy along the height of the samples, the residual strain and stress results are compared along the height for the three sets of results, from neutron diffraction, synchrotron X-ray diffraction and contour method. The residual stress data for the longitudinal stress \( \sigma_y \) are compared between samples with different deposition strategy, but with the same dwell time and the same level of the energy density. Table 9-2 summarised the pairs to be compared, based on the data from the three residual stress measurement techniques.

To enable a direct comparison between two samples, from neutron diffraction data, the residual stress results for the pairs of the samples with different deposition strategy (but the same dwell time and the same level of energy density) are presented in Figures 9-6, 9-7, 9-8 and 9-9.

The same approach is repeated for the samples scanned via synchrotron X-ray, where the residual stress results are superimposed for the pairs of the samples with different deposition strategy (but the same dwell time and the same level of the energy density). The results for the pairs of the samples are plotted in Figures 9-10, 9-11 and 9-12.

Furthermore, the data from the contour method enabled direct comparison between two pairs of the samples, as summarised in Table 9-2. The comparison between the pairs of the samples, based on contour data, are shown in Figures 9-13 and 9-14.

<table>
<thead>
<tr>
<th>Samples pairs</th>
<th>Neutron diff</th>
<th>Sync X-ray</th>
<th>Contour method</th>
</tr>
</thead>
<tbody>
<tr>
<td>L or Z -060-L</td>
<td>Samples 1 &amp; 5 (Figure 9-6)</td>
<td>Samples 1 &amp; 5 (Figure 9-10)</td>
<td>-</td>
</tr>
<tr>
<td>L or Z -180-L</td>
<td>Samples 2 &amp; 6 (Figure 9-7)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>L or Z -060-H</td>
<td>Samples 3 &amp; 7 (Figure 9-8)</td>
<td>Samples 3 &amp; 7 (Figure 9-11)</td>
<td>Samples 3 &amp; 7 (Figure 9-13)</td>
</tr>
<tr>
<td>L or Z -180-H</td>
<td>Samples 4 &amp; 8 (Figure 9-9)</td>
<td>Samples R4 &amp; R8 (Figure 9-12)</td>
<td>Samples 4 &amp; 8 (Figure 9-14)</td>
</tr>
</tbody>
</table>
Chapter 9 – The effects of process parameters on the evolution of residual stress in PTA AM

Figure 9.6 – Residual stress variations from neutron diffraction along the height (scan-line 1) for the samples with the dwell-time of 60 s and the low level of the energy density: linear sample L-060-L (1) versus zig-zag sample Z-060-L (5)

Figure 9.7 – Residual stress variations from neutron diffraction along the height (scan-line 1) for the samples with the dwell-time of 180 s and the low level of the energy density: linear sample L-180-L (2) versus zig-zag sample Z-180-L (6)
Figure 9.8 – Residual stress variations from neutron diffraction along the height (scan-line 1) for the samples with the dwell-time of 60 s and the high level of the energy density: linear sample L-060-H (3) versus zig-zag sample Z-060-H (7)

Figure 9.9 – Residual stress variations from neutron diffraction along the height (scan-line 1) for the samples with the dwell-time of 180 s and the high level of the energy density: linear sample L-180-H (4) versus zig-zag sample Z-180-H (8)
The results from neutron diffraction confirm a relatively low stress level towards the middle of the height, while the tensile stress appears towards the bottom of the wall, at the intersection of the deposited wall and the substrate.

Comparing the neutron diffraction data for the sample with different deposition strategies suggests that generally the linear deposition strategy causes a slightly more stress variation of the residual stress, as shown between all four pairs of the samples, Figure 9-6, Figure 9-7, Figure 9-8 and Figure 9-9. The difference between samples L-060-L (1) and Z-060-L (5) (Figure 9-6), and between samples L-060-H (3) and Z-060-H (7) (Figure 9-8), seems to be more visible with the typical trend for the compressive residual stress at the top, slight tensile stress, just below the top and a compressive stress at the middle and then a higher tensile stress at the bottom of the wall.

For the other two pairs, the difference is less visible, but could be argued that a slightly more variation can be seen for the linear samples than the zig-zag samples (Figure 9-7 and Figure 9-9).

As the number of neutron scan points is low (as explained in Chapter 5) and the range does not cover the first 10 mm of the top of the height, looking into the other sets of results from synchrotron X-ray diffraction and the contour method could help to investigate the potential effect of the deposition strategy better.

Figures 9-10, 9-11 and 9-12 provide a similar comparison between the three pairs where the synchrotron X-ray data were available.
Chapter 9 – The effects of process parameters on the evolution of residual stress in PTA AM

Figure 9-10 – Residual stress variations from synchrotron X-ray diffraction along the height (scan line 1) for the samples with the dwell time of 60 s and the low level of the energy density: linear sample L-060-L (1) versus zig-zag sample Z-060-L (5)

Figure 9-11 – Residual stress variations from synchrotron X-ray diffraction along the height (scan line 1) for the samples with the dwell time of 60 s and the high level of the energy density: linear sample L-060-H (3) versus zig-zag sample Z-060-H (7)
Chapter 9 – The effects of process parameters on the evolution of residual stress in PTA AM

Figure 9.12 – Residual stress variations from synchrotron X-ray diffraction along the height (scan-line 1) for the samples with the dwell-time of 180 s and the high level of the energy density: linear sample R·L·180·H (R4) versus zig-zag sample R·Z·180·H (R8)

From the three pairs of the samples, based on the synchrotron X-ray diffraction data, a similar residual stress trend to the data from neutron diffraction can be observed.

Considering each pair, the linear samples show a clearer variation of the residual stress than the zig-zag samples. This is more visible for the first pair of the samples, L·060·L (1) and Z·060·L (5), as in Figure 9·10 and for the second pair of the samples, in Figure 9·11. For both pairs, the dwell time was 60 s. So, it could be argued that the linear deposition resulted in a slightly more visible variation than the zig-zag deposition.

However, the third pair of the samples, R·L·180·H (R4) and R·Z·180·H (R8) shows less difference between the variations of the residual stress. For this pair of the samples, the dwell time was 180 s, which could be the reason for the less varying residual stress distribution. The longer dwell time could allow the more effective cooling of the deposits and therefore compromised the effect of the deposition strategy.
For the data from the contour method, the data along the middle line from the top to the bottom of the height, across the cut surface were attributed to the main vertical scan-line 1 in diffraction-based techniques (as explained in Chapter 7). The residual stress data, from the contour method, for the two pairs of the samples with different deposition strategies (linear vs. zig-zag), but the high level of the energy density and the short dwell time of 60 s, are presented in Figure 9.13, to provides a comparison between samples L-060-H (3) and Z-060-H (7).

Furthermore, the contour data enabled a comparison between the linear sample L-180-H (4) and the zig-zag sample Z-180-H (8), with the high level of the energy density and the longer dwell time of 180 s, is given in Figure 9.14.
Figure 9.13 – Residual stress results from the contour method for sample with different deposition strategy, samples L-060-H (3) (linear) and Z-060-H (7) (zig-zag)

Figure 9.14 – Residual stress results from contour method for samples with different deposition strategy, samples L-180-H (4) (linear) and Z-180-H (8) (zig-zag)
Considering the comparison of the data between different samples, from all three residual stress measurement techniques, enables the discussion of the effect of the deposition strategy.

From the stress data from both neutron and synchrotron X-ray diffractions, both diffraction-based techniques provided a similar trend for the residual stress variation from the top to the bottom of the samples, for all samples. Typically, the stress state is compressive at the top and shows a slight tensile behaviour just below the top few layers. The stress becomes compressive towards the middle of the deposited wall and then move up to the tensile state as moving down towards the intersection of the wall and the substrate.

From the diffraction-based techniques, a slightly higher stress variation could be seen for the samples with the linear deposition strategy, compared to the samples with the zig-zag deposition strategy. However, the actual stress data, the maximum and the minimum and the average of the stress data seem to be similar for most of the pairs of the samples, based on the deposition strategy.

The variations and the magnitudes of stresses (and strains) for the samples manufactured with the same dwell-time and energy density but the linear deposition strategy seemed to be higher than the samples with the zig-zag deposition strategy. This generally could be due to the difference in the rate of solidification as well as the pattern for the cooling which also affects the solidification pattern in linear beads versus the zig-zag deposited beads, as explained in the literature (Chapter 2) (Das et al., 1998).

According to the stress variations (Figure 9-6 to Figure 9-12) from diffraction-based data for the low energy density samples with the short dwell-time of 60 s, the deposition strategy could slightly influence the evolution and variation of the residual stress. In such a case, the zig-zag deposition resulted in a lower residual stress variation than the linear deposition strategy. This was also the case for the two samples with low energy density but the longer dwell-time of 180 s, samples L-180-L (2) and Z-180-L (6), based on the neutron data, in Figure 9-8.

Considering the stress data from diffraction based techniques (Figure 9-6 to Figure 9-12) as well as the contour data (Figure 9-13 and Figure 9-14), in samples with the higher level of energy density and the shorter dwell-time of 60 s, the difference between the residual stress variation between the linear and the zig-zag samples was shown to be negligible.
This was shown in the data from the diffraction-based techniques as well as the data from the contour method. This could be related to the effect of the energy density, as the higher energy input in the linear deposition caused a similar effect in distributing the heat as in the zig-zag deposition strategy, and therefore alleviated the effect of the deposition strategy.

In samples with the higher level of the energy density and the longer dwell-time of 180 s, the deposition strategy seemed to have a more considerable effect on the evolution of residual stress. In this case, the zig-zag sample, Z-180-H (8) showed less variation of the residual stress (and strain) than the linear sample, L-180-H (4), as it was shown by the data from both diffraction-based techniques (Figure 9-9 from neutron and Figure 9-12 from synchrotron X-ray) as well as the contour method (Figure 9-14). The longer dwell-time probably compromised the effect of the higher level of the energy density and hence made the deposition strategy a significant/dominant process parameter.

The results between the two repeat samples R·L-180-H (R4) and R·Z-180-H (R8), with the same dwell-time of 180 s and the same higher level of the energy density suggested a slightly different trend, though. The residual stress variations suggested very little difference between the two samples, according to the data from synchrotron X-ray diffraction, as shown in Figure 9-12.

However, comparing the variations of the residual stress along the height of the two samples (from top to the bottom of the walls), it showed that the stress variation was more fluctuating in the linear sample than the zig-zag sample. Hence, it seems that the linear deposition strategy could lead to a more considerable effect in this case. A similar argument could be made to justify the role of the deposition strategy in this case. It is likely that the longer dwell-time of 180 s allowed a more stable cooling of the deposits in both the linear and the zig-zag depositions and resulted in a more dominant effect for the deposition strategy. The zig-zag deposition then helped to create a more uniform thermal distribution and the evolution of the residual stress than the linear deposition. This effect was shown to be more considerable towards the middle of the height of the deposited walls than the top or the bottom of the walls, for both samples.

For the pairs of the samples compared based on the contour data, similar to the justification for the results from the neutron and the synchrotron X-ray diffraction, it seemed that the combination of the shorter dwell-time of 60 s and the higher level of the
energy density made the effect of the deposition strategy less significant, meaning that both the linear and the zig-zag deposition strategies resulted in a similar stress state.

Considering the higher quality and more reliable data from the contour method, in general, less effects of the deposition strategy was observed on the level and the variation of the residual stress, as shown in Figure 9-13 and Figure 9-14. It could be argued that the variation of the residual stress is insensitive to the deposition strategy.
Chapter 9 – The effects of process parameters on the evolution of residual stress in PTA AM

9.3. The effect of the dwell-time

A similar approach is taken to investigate the effect of the dwell-time on the evolution of the residual stress. The results from the three residual stress measurement techniques are analysed in more detail to understand any potential effect of dwell-time on the final state of residual stress within the PTA AM samples.

All discussions are based on the longitudinal components of stress ($\sigma_y$), as the dominant component compared to the transverse stress ($\sigma_z$). The stress results provided in results chapters (5, 6 and 7) are paired based on the dwell-time for the PTA AM samples and discussed accordingly.

The main scan-line along the height was scan-line 1 with 9 and 101 scan points for neutron and synchrotron diffraction, respectively. Also contour data are discussed based on the cut section coincident with scan-line 1 for diffraction-based techniques, and the results along the middle-line of the cross section were analysed to render a similar data set to the data from the diffraction-based techniques.

Residual stress results from neutron diffraction, synchrotron X-ray diffraction and the contour method, along the height of the samples (vertical scan-line 1) are paired for the samples with 60 s and 180 s dwell-time, while the other two parameters were the same and are summarised in Table 9.3 and the superimposed results are presented subsequently.

<table>
<thead>
<tr>
<th>Samples pairs</th>
<th>Neutron diff</th>
<th>Sync X-ray</th>
<th>Contour method</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L_{060}$ or $L_{180}$</td>
<td>Samples 1 &amp; 2 (Figure 9-15)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$L_{060}$ or $H_{180}$</td>
<td>Samples 3 &amp; 4 (Figure 9-16)</td>
<td>Samples 3 &amp; R4 (Figure 9-19)</td>
<td>Samples 3 &amp; 4 (Figure 9-21)</td>
</tr>
<tr>
<td>$Z_{060}$ or $L_{180}$</td>
<td>Samples 5 &amp; 6 (Figure 9-17)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>$Z_{060}$ or $H_{180}$</td>
<td>Samples 7 &amp; 8 (Figure 9-18)</td>
<td>Samples 7 &amp; R8 (Figure 9-20)</td>
<td>Samples 7 &amp; 8 (Figure 9-22)</td>
</tr>
</tbody>
</table>
Figure 9.15 – Residual stress variations from neutron diffraction data along the height (scan line 1) for samples with linear deposition strategy and the low level of the energy density: sample L-060-L (1) versus sample L-180-L (2)

Figure 9.16 – Residual stress variations from neutron diffraction data along the height (scan line 1) for samples with linear deposition strategy and the high level of the energy density: sample L-060-H (3) versus sample L-180-H (4)
Chapter 9 – The effects of process parameters on the evolution of residual stress in PTA AM

Figure 9.17 – Residual stress variations from neutron diffraction data along the height (scan-line 1) for samples with zig-zag deposition strategy and the low level of the energy density: sample Z-060-L (5) versus sample Z-180-L (6)

Figure 9.18 – Residual stress variations from neutron diffraction data along the height (scan-line 1) for samples with zig-zag deposition strategy and the high level of the energy density: sample Z-060-H (7) versus sample Z-180-H (8)
Figure 9-15, 9-16, 9-17 and 9-18 provided the superimposed data for the pairs of the samples where the only difference in each pair was the dwell time (60 s versus 180 s).

It should be noted that the neutron data are limited to the nine scan points from the top to the bottom of the height (scan line 1) and started from 10 mm below the top surface to the intersection of the deposited wall and the substrate.

Overall, the neutron data show the typical stress variation from the top to the bottom of the height in samples. However, comparing the pairs of the samples suggest less difference between samples with the 60 s and the samples with the 180 s dwell times. The two linear samples with the lower level of the energy density and the alternating dwell-time (60 s vs 180 s) was shown to have the highest stress variations among the four combinations of process parameters, as shown in Figure 9-15.

It seems that the longer dwell time of 180 s could result in slightly less variation in the residual stress compared to the 60 s, for the linear samples (Figure 9-15 and Figure 9-16). Also, the difference is slightly visible for the pair of the samples in Figure 9-17, where the zig-zag deposition strategy was implemented alongside the lower level of the energy density. However, the difference between the sample pairs manufactured via zig-zag deposition and the higher level of the energy density is not observable (Figure 9-18). This is probably due to the more dominant effect of the energy density (heat input) than the dwell time in the last pair.

The same analogy is implemented for the pairs of the samples with different dwell times, based on the synchrotron X-ray diffraction data. The results for the two available pairs are plotted in Figures 9-19 and 9-20.
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**Figure 9-19** – Residual stress variations from synchrotron X-ray diffraction along the height (scan-line 1) for the samples with the linear deposition strategy and the high level of the energy density: sample L-060-H (3) versus sample R-L-180-H (R4)

**Figure 9-20** – Residual stress variations from synchrotron X-ray diffraction along the height (scan-line 1) for the samples with the zig-zag deposition strategy and the high level of the energy density: sample Z-060-H (7) versus sample R-Z-180-H (R8)
Similar to the neutron data, the synchrotron X-ray data suggest little visible difference between the samples with the 60 s and 180 s dwell times.

For the two linear samples with the higher level of the energy density, as the data superimposed and plotted in Figure 9-19, the overall trend is the same for both samples. Although, the residual stress is slightly more fluctuating for sample R-L-180-H (R4) compared to sample L-060-H (3), there is no visible difference in the overall variation between the two samples. Similar to the argument made based on the neutron diffraction data, the combination of the linear deposition and the higher level of the energy density dominated the aggressive heat input and the effect of the dwell time has been negligible.

The two zig-zag samples, with the high level of the energy density however shows a slightly observable difference in terms of residual stress variation, as results are plotted together in Figure 9-20. In this case, sample Z-060-H (7) with the 60 s dwell time shows a slightly more variation than sample R-Z-180-H (R8). It could be argued that the zig-zag deposition provided a more uniform heat input and therefore the effect of the longer dwell time in alleviating the residual stress became slightly more visible. The trend of the stress distribution is still similar to other samples, as already covered.

The residual stress data from the contour method are also plotted together for each pair of the samples, with the same deposition strategy and energy density, but alternating dwell time of 60 s versus 180 s, as shown in Figures 9-21 and 9-22.
Figure 9.21 – Residual stress results from contour method for samples with different dwell times (60 s versus 180 s), sample L-060-H (3) and sample L-180-H (4)

Figure 9.22 – Residual stress results from contour method for samples with different dwell times (60 s versus 180 s), sample Z-060-H (7) and sample Z-180-H (8)
According to the stress variations, paired based on the dwell-times of 60 s and 180 s, the effect of the dwell-time is more observable for the samples with the low level of the energy density than the samples with the high level of the energy density. In such cases, the effect of the dwell-time was more considerable on the linear samples with the lower energy density than the zig-zag samples with the lower energy density, as it was shown for samples L-060-L (1) and L-180-L (2), or the repeat sample R-L-180-H (R4), from the three residual stress measurement techniques.

On the other hand, the effect of the dwell-time on the samples with the high level of the energy density was not as significant as the samples with the low level of the energy density. It was shown by the maximum strain (and stress) values, as shown in Figure 9-4, from neutron and synchrotron X-ray diffractions results as well as from the contour data, summarised in Figure 9-5.

This was the case for both of the linear samples, L-060-H (3), L-180-H (4) and both of the zig-zag samples, Z-060-H (7), Z-180-H (8), shown for all four samples, based on the contour data as summarised in Figure 9-21 and Figure 9-22. It seemed that the higher level of the energy density caused a more dominant effect than the dwell-time, in terms of the heat input and the evolution of the residual stress. Consequently, the dwell-time did not get a chance to control or affect the heating and cooling phenomena and the residual stress evolution.

In the samples with the linear deposition with the higher energy density, as shown in Figure 9-16, Figure 9-19 and Figure 9-21, for samples L-060-H (3) and L-180-H (4), the longer dwell-time of 180 s could result in a slightly more unstable stress formation than the shorter dwell-time of 60 s. It seemed that the combination of the dwell-time of 60 s and the higher level of the energy density could generate a more effective inclusion of the deposited layers. Therefore, the shorter dwell-time of 60 s slightly lessened the residual stress evolution or at least the variation/distribution of the stress, compared to the sample with the longer dwell time of 180 s.

In the zig-zag deposition, the strain variation was shown to be higher in the samples with the shorter dwell time of 60 s than the samples with the longer dwell-time of 180 s. This was aligned with the expected effect of the dwell-time, as the shorter dwell-time produced a more unstable and a more varied stress field than the longer dwell-time. It appeared that the zig-zag deposition could control the effect of the dwell-time and revert it to the typical outcome, as it was not the case for the pair of linear samples.
As discussed for the effect of the deposition strategy, the zig-zag deposition compromised the effect of the dwell-time when the higher level of the energy density (heat input) was utilised. It was shown by referring to the data from all three residual stress measurement techniques, for the two zig-zag samples Z-060-H (7) and Z-180-H (8). Comparing the stress variations for the two zig-zag samples, based on the neutron and the synchrotron X-ray diffraction (Figure 9-18 and Figure 9-20) confirmed that the variations was in the same range for both of the samples (the shorter of 60 s and the longer dwell-time of 180 s). It was also shown by the residual stress variations from diffraction-based techniques (Figure 9-18 from neutron diffraction and Figure 9-20 from synchrotron X-ray diffraction) as well as the stress data from the contour method for the two samples (Figure 9-22).

This was believed to be due to the more dominant effect of the zig-zag deposition strategy than the linear deposition strategy in providing a stable and uniform thermal gradient during the layer deposition process. As the higher level of the energy density was compromised by the zig-zag deposition, the deposition strategy seemed to have compromised the effect of the shorter dwell-time and therefore led to less residual stress formation.

Overall, the effect of the dwell time seemed to be less visible on the residual stress variation than the effect of the deposition strategy. Also, it is appeared that the effect of the dwell time could be compromised by the other two process parameters.
9.4. The effect of the energy density

The data from both diffraction-based techniques, neutron and synchrotron X-ray diffraction, are used to analyse the effect of the energy density. All samples measured by the contour method were manufactured by utilising the high level of the energy density, as explained in Chapter 7. So, the contour data could not be used to investigate the effect of the level of the energy density. The discussions in this section are therefore based on the neutron and synchrotron X-ray diffractions results.

From the neutron diffraction data, the stress variations are compared for four pairs of the samples, where the only difference in each pair is the level of the energy density. The four pairs of the samples, based on the neutron diffraction data are summarised in Table 9-4.

The same approach is repeated based on the data from the synchrotron X-ray diffraction. Considering the number of the samples scanned via synchrotron X-ray (Table 9-1) two pairs of the samples were available to compare and discuss the potential effect of the energy density, as summarised in Table 9-4.

<table>
<thead>
<tr>
<th>Samples pairs</th>
<th>Neutron diff</th>
<th>Sync X-ray</th>
</tr>
</thead>
<tbody>
<tr>
<td>L-060-L or H</td>
<td>Samples 1 &amp; 3 (Figure 9-23)</td>
<td>Samples 1 &amp; 3 (Figure 9-27)</td>
</tr>
<tr>
<td>L-180-L or H</td>
<td>Samples 2 &amp; 4 (Figure 9-24)</td>
<td>-</td>
</tr>
<tr>
<td>Z-060-L or H</td>
<td>Samples 5 &amp; 7 (Figure 9-25)</td>
<td>Samples 5 &amp; 7 (Figure 9-28)</td>
</tr>
<tr>
<td>Z-180-L or H</td>
<td>Samples 6 &amp; 8 (Figure 9-26)</td>
<td>-</td>
</tr>
</tbody>
</table>

The subsequent Figures show the superimposed results for the pairs of the samples, as summarised in Table 9-4, based on the neutron diffraction data (Figures 9-23, 9-24, 9-25 and 9-26) and synchrotron X-ray data (Figures 9-27 and 9-28).
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Figure 9-23 – Residual stress variations from neutron diffraction along the height (scan-line 1) for the samples with the linear deposition strategy and the dwell time of 60 s, sample L·060·L (1) versus sample L·060·H (3)

Figure 9-24 – Residual stress variations from neutron diffraction along the height (scan-line 1) for the samples with the linear deposition strategy and the dwell time of 180 s, sample L·180·L (2) versus sample L·180·H (4)
Figure 9-25 – Residual stress variations from neutron diffraction along the height (scan-line 1) for the samples with the zig-zag deposition strategy and the dwell time of 60 s, sample Z-060-L (5) versus sample Z-060-H (7)

Figure 9-26 – Residual stress variations from neutron diffraction along the height (scan-line 1) for the samples with the zig-zag deposition strategy and the dwell time of 180 s, sample Z-180-L (6) versus sample Z-180-H (8)
Similar to the other two process parameters, the trend for the residual stress variation seems to be comparable between each pair and among all samples, based on the data from neutron diffraction.

For the linear samples with the 60 s dwell time (Figure 9-23) as well as the two zig-zag samples with the 60 s dwell time (Figure 9-25) there seem to be a higher variation for the samples with the higher level of the energy density compared to the samples with the lower level of the energy density. As mentioned for the effect of the dwell time, the shorter dwell time of 60 s causes a slightly less heat uniformity and therefore the effect of the lower level of the energy density compared to the higher level of the energy density becomes more considerable.

The difference in the stress variation is not so much visible between samples in the other two pairs, manufactured by the linear deposition strategy and 180 s dwell time (Figure 9-24) and the zig-zag deposition strategy and the 180 s dwell time (Figure 9-26). Similar to the discussions for the effect of the dwell time, the effect of the of the longer dwell time of 180 s counteracted the effect of the energy density and allowed the sample to absorb and maintain the same level of the heat input and consequently a similar residual stress variations for the two samples.

Referring to the Table 9-4, the synchrotron data enabled the comparison between samples in two pairs, as superimposed and plotted in Figure 9-27 for the two linear samples and in Figure 9-28 for the two zig-zag samples, with the two different levels of the energy density (low versus high).
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Figure 9-27 – Residual stress variations from synchrotron X-ray diffraction along the height (scan-line 1) for the samples with the linear deposition strategy and 60 s dwell time: sample L·060·L (1) versus sample L·060·H (3)

Figure 9-28 – Residual stress variations from synchrotron X-ray diffraction along the height (scan-line 1) for the samples with the zig-zag deposition strategy and 60 s dwell time: sample Z·060·L (5) versus sample Z·060·H (7)
Synchrotron X-ray data also confirm the justification made based on the neutron diffraction data. The variation of the residual stress between samples in each pair seems to be negligible in each pair, while both pairs show a similar trend of the residual stress distribution from the top to the bottom of the height.

Based on the variation of the residual stress for the two linear samples with the dwell time of 60 s (Figure 9-27), a slightly visible difference between the two samples could be observed. It seemed that the lower level of the energy density could not be compromised by the effect of the shorter dwell-time of 60 s, which caused the stress evolution becomes more unstable, as it was shown in the samples with the lower level of the energy density. The effect of the deposition strategy seemed to be of less significance in such a case. The combination of the short dwell-time of 60 s and the low level of the energy density already caused an unstable layer deposition process and the higher variation of the residual strain/stress, consequently.

Furthermore, the difference between the two zig-zag samples with the 60 s dwell-time, but alternating level of the energy density; samples Z-060-L (5) and Z-060-H (7) was shown to be in the same range, from the synchrotron data, as shown in Figure 9-28. This is similar to the comparison made by the neutron diffraction data, Figure 9-25. From both sets of data, the effect of the energy density seemed to be slightly less in the zig-zag deposition case. The sample with the low level of the energy density showed a slightly higher stress variation. However, in general the difference between the two samples is negligible. It could be interpreted that the effect of the zig-zag deposition strategy, which is oscillating the deposits, counteracted the effect of the energy density (heat input), by imposing a more uniform heat distribution.

As it was shown by the neutron data, for the longer dwell-time of 180 s, for both the linear (Figure 9-24) and the zig-zag deposition strategies (Figure 9-26), the difference between the samples with the low and the high level of the energy density seemed to be less significant and the residual stress variations seemed to follow a similar trend. Although, there was a slightly more variation of strain for the sample with the higher energy density, sample Z-180-H (5), than the sample with the lower level of the energy density; sample Z-180-L (6), as it was shown in Figure 9-26. It could be interpreted that the lower level of the energy density resulted in a less stress variation compared to the higher level of the energy density, in this case. The zig-zag deposition strategy and the 180 s dwell-time provided a more stable deposition condition and therefore the effect of the energy
density would be realised as expected; the higher the energy input, the higher the variation of the final residual stress/strain state would be.

In general, the effect of the energy density seems to be of less significance than the effect of the deposition strategy and to some extend dependent on the effect of the combination of the process parameters and how they would affect the overall heat input as well as the heat distribution during the deposition and after/before depositing each individual layer. Considering the similar argument to the effect of the dwell time, it could be argued that overall the evolution of the residual stress would be insensitive to the level of the energy density, based on the two levels as defined in the current research work.
9.5. Summary of discussions

As outlined by the aim and objectives of the thesis, a series of experimental methodologies were defined and undertaken to investigate the effect of process parameters on the evolution of the residual strain/stress within Ti-6Al-4V PTA AM samples. The methodology to manufacture was samples was defined based on the PTA process parameters (heat input and deposition strategy) as well as the AM features for the process (deposition strategy and dwell-time). In total, three main process parameters were considered and altered to investigate the effect of them on the residual stress evolution: deposition strategy, dwell-time and energy density.

Discussions were focused on the effect of each of the process parameters where the data were grouped/paired to understand the effect of each individual parameter. Furthermore, considering the combined effect of process parameters, the data were discussed based on two and/or three process parameters together, where applicable.

The stress results from the three residual stress measurement techniques showed a good correlation and a satisfactory level of validation. However, it was acknowledged that some experimental limitation affected the data gathering and discussions for each of the measurement techniques. Specifically, the implementation of the diffraction-based techniques was shown to be more challenging. It was noted that the validity of neutron data could be limited by incoherent scatter from titanium, as reported in the literature (Chapter 2). Also, the elongated gauge volume from the synchrotron X-ray diffraction, could be the source of experimental errors (Chapters 2 and 4).

The higher quality data from the contour method provided a more stable and reliable set of results. The contour data seemed to show a similar trend for the four samples with different combinations of process parameters. In fact, based on the contour data, the residual stress evolution within the PTA AM samples is insensitive to the process parameters, within the defined range of the manufacturing parameters in this work. However, looking into more detailed analysis from diffraction-based techniques, a link could be drawn between the process parameters and the final state of the residual stress.

Overall, there seemed to be a good agreement between the set of data with regard to the level and variation of the residual stress across the whole sample (global data). However, the data from individual scan points do not necessarily follow the same trend / magnitudes and hence could be argued that the local confidence of the data is not evinced.
As the PTA AM process involves a high thermal gradient and also considering the higher rate of material input/deposition compared to other AM processes (powder bed), the management of the heat input and the heating and cooling cycles for the material could impact the level and the variation of the final state of the residual stress.

In general, a more stable deposition process could alleviate the level of residual stress within the PTA AM samples. This included zig-zag deposition strategy versus linear deposition, as the oscillation of deposition process could distribute the heat input more effectively. Also, the longer dwell-time versus the shorter dwell-time could provide a more stable cooling condition for deposited material, although the shorter dwell-time could result in more materials inclusion, in some cases. Also, the lower the energy input, the more stable material deposition would be (e.g. less materials shrinkage).

However, as it was shown for some of the samples, combination of the processing parameters could provide a different effect of the parameters and therefore it is important to consider all of the processing parameters together when discussing the effect of them on the evolution and final state of the residual stress. Moreover, the variation (distribution) of residual stress could be affected by process parameters, as it was showed by data from different samples.

Overall, PTA AM proved to be a very complicated AM technique, in terms of heating and cooling cycles involved in the process and the resulting residual stress state. However, it provides some advantages and benefits compared to typical powder bed AM processes, the high rate of material deposition combined with the high energy input normally leads to a series of complicated phenomena which affects the final state of the manufactured parts.

Whilst this research work provided a foundation for residual stress analysis in PTA AM, the limitations and constraint should be carefully studied and considered for any similar empirical and/or simulation methodology implementation.

A summary of the effect of processing parameters are provided in the conclusion Chapter 10, along with some future work recommendations to investigate the influence of processing condition in more details.
Chapter 10 – Conclusions and Future Works
10.1. Conclusions

As a metal-based AM technique, the use of Plasma Transferred Arc (PTA) for the relatively low cost deposition of metals is attracting interest from the high value manufacturing sector. The wire-based PTA AM provides a higher deposition rate compared to Powder-Bed Fusion (PBF) AM processes. The techniques could resolve the rate of AM processing to build parts. In addition, the wire-based technique lends more control over the deposition than the powder-fed process. The PTA AM technique is also a viable choice for multi-material AM processes (wire and/or powder fed). There is also increasing interest shown in titanium alloys as a lightweight metal for their excellent specific physical and mechanical properties. PTA AM of titanium alloys provides an excellent business case for a wide range of industrial applications.

The main key challenge in implementing any metal-based AM technology is the microstructural and mechanical properties of the final part. It is therefore vital to understand the effect of AM processing on the performance of the final AM component. Furthermore, another challenge related to AM components is the shape and size accuracy of the final part compared to the original Computer-Aided Design (CAD) model. These two challenges associated with metal-based AM processes are the main barriers in commercialising AM technologies. Hence, for establishing AM as a viable production technology, knowledge of physical/mechanical effects during the manufacturing process is essential to determine the integrity and geometrical accuracy of the final product.

The higher deposition rate in PTA AM introduces high gradient heating and cooling processes. The stress evolution during the process causes shape and size inaccuracy and residual stress state within the final AM part. Consequently, residual stress formation and distortion is intensified in the PTA AM products and typically controlling the evolution of residual stress becomes more challenging. Understanding the level and variation of the final state of residual stress could lead to optimising the process or design to manufacture reliable final components. Understanding and possibly controlling of residual stresses is of utmost importance for functional components in all industrial sectors.

The PTA AM process combines aspects of welding process (used for joining materials), in depositing material, as well as AM process in additive layer deposition and stacking them
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on top of each other. This would make the interdependencies of the processing parameters a complex landscape.

The evolution of residual stress in the PTA AM process depends on both PTA aspects of the process as well as the AM aspects of the process. There are a number of processing parameters associated with the PTA AM technique, such as: metal deposition strategy, dwell-time between layers deposition and the thermal gradient (depending on the level of energy input) during deposition and cooling of the layers.

In this thesis, three main processing parameters were studied in detail to understand their effects on the final state of residual stresses in PTA AM parts. The choice of the three processing parameters was based on the current PTA AM machine at WMG, University of Warwick and the controllability of the processing parameters. The three parameters were deposition strategy, dwell-time between layers’ deposition and the level of energy density of the deposition process.

For each processing parameter, two different levels were applied to build parts with different combinations of the processing parameters. Two deposition strategies were linear versus zig-zag depositions. Two dwell-times of 60 s and 180 s were used between the layers depositions and two levels of energy density was used to deposit PTA AM walls. Hence, 8 main samples were built to reflect on all possible combinations of the processing parameters. Repeat samples were also manufactured to examine the repeatability of the process.

To investigate the level and variation of residual stress within the manufactured parts, three main residual stress measurement techniques were utilised. Two diffraction-based techniques: neutron and synchrotron X-ray diffraction as non-destructive methods and contour method as a destructive method.

All calculations for the stress results were based on the plane stress assumption, assuming the out-of-plane stress to be zero. The associated discussions about the validity of the plane stress assumptions (Chapter 8) showed that the level and variation of the out-of-plane stress was negligible compared to the two in-plane stress components.

For the first time, a coherent study of the effect of the processing parameters on the evolution of residual stress in PTA AM of Ti-6Al-4V was delivered in this thesis, addressing the research question in Chapter 2. The results and methodology from this
work are believed to be state-of-the-art as no previous similar data were available in public domain literature.

The main findings from the residual stress analysis are summarised as below:

- Investigating the microstructure of the deposited walls showed a relationship between the geometrical aspects of the process (position of the deposited layer and deposition strategy) and the microstructural evolution. The further away from the substrate the effect of high thermal gradients became more visible in the form of extended grain size towards the top of the build.
- The variation of the strain (and stress) along the height was shown to be more significant than along the length of the deposited walls. This was expected to be the case based on the way the layers were deposited (Chapter 3), along the length of the substrate.
- The two strain components were determined via diffraction-based techniques, the longitudinal component ($\varepsilon_x$), along the length of the deposited walls and the transverse component ($\varepsilon_z$) from top to the bottom of the height of the deposited walls. However, the contour method only provided the longitudinal components of the strain ($\varepsilon_x$) and stress ($\sigma_y$).
- It was shown that the plane stress condition is a valid assumption, in measuring/determining the residual stress state within the PTA AM parts. Two equivalent stress components were calculated based on the plane stress condition and reduced Hooke’s law, longitudinal stress component ($\sigma_y$) and transverse stress component ($\sigma_z$), assuming the out-of-plane stress component was zero ($\sigma_z=0$).
- It was shown that the longitudinal component of strain ($\varepsilon_x$) and stress ($\sigma_y$) have more considerable magnitude (and variation) than the transverse components ($\sigma_z$ and $\varepsilon_z$). This was expected considering the geometrical aspects of the layer deposition process.
- The diffraction-based techniques provided strain and stress data at individual scan points, which were chosen along vertical and horizontal scan lines for the PTA AM walls.
- Destructive contour method applies a straightforward solid mechanics methodology to measure relaxed contours across the cut section.
- The data from the contours provided stress (and strain) results across the whole cut section, rather than individual scan points. However, contour method could only provide data for the longitudinal components of the stress ($\sigma_y$) and strain ($\varepsilon_x$),
as the longitudinal component is perpendicular to the cut section, and therefore is relaxed after the cutting process.

- The main aim in implementing different residual stress measurement techniques was to understand the level of residual stress along the height of deposited walls. The magnitudes were not as important, in this research work, as understanding the variation of residual stress. Understanding the variation of the residual stress could lead to methodologies to control both variation and magnitudes.

- Although, a comparison between results from different techniques was provided, comparing between different techniques, or examination and validation of residual stress measurement techniques was not the subject of this thesis. The main aim was to understand the variation of residual stresses along the height of the samples and relate it to the processing parameters.

- The contour data, showed a similar trend (and level/magnitude) for the residual stress distribution within the samples with the high level of the energy density. This would suggest that the evolution of residual stress within the PTA AM samples is insensitive to the process parameters, within the range for the manufacturing condition. It should be noted that the contour method was only applied to the samples with the high level of the energy density.

- However, considering the data from diffraction-based techniques and delving into the contour method data more carefully, it could be argued that each of the three process parameters could affect the level/variation of residual stress.

- Generally, the deposition strategy have a considerable/dominant effect on the deposition process and therefore in the level and the variation of residual stress. The zig-zag deposition was shown to help reduce residual stress compared to the linear deposition strategy. This was due to more uniform heat distribution, as the heat source (and wire-feeder) was oscillating to deposit layers. Linear deposition was shown to cause more localised heat input, which would destabilise the additive layer process.

- However, it was observed that combining the effect of the deposition strategy and the other two process parameters, this effect could be reversed, as other process parameters were also responsible for heat distribution and solidification of deposited layers.

- It was shown that the longer dwell-time of 180 s could be more effective in alleviating residual stress than the shorter dwell-time of 60 s. This could be related
to the thermal history of the deposited layers. The longer dwell-time could stabilise the thermal gradients and provided a more uniform solidification process.

- However, it is important to understand that longer dwell-time is not always a cost-effective case for industrial applications. Therefore, it is important to identify a “range” for the dwell-time in which the process could produce parts with “acceptable” levels/variation of residual stress (depending on the required safety factor for the parts).

- Similar to the deposition strategy, it was shown that the effect of the dwell-time could also be compromised when it is combined with other process parameters. It is therefore necessary to investigate its effect alongside other processing parameters, which was done for the first time in this thesis.

- Energy density was shown to affect the level/variation of residual stress within the parts. Generally, the higher the level of energy input, a higher variation of residual stress was produced in the final parts. For this reason, contour method was only conducted on the parts with the higher level of energy density.

- It was shown that the combination of linear deposition strategy and the higher level of energy input could produce less variation of residual stress compared to the combination of zig-zag deposition strategy and the higher level of energy density. The localised effect of linear deposition could provide a more effective material inclusion in the case of higher level of energy density than the oscillated zig-zag deposition process.

- The zig-zag deposition strategy however showed to be more effective in reducing residual stress than linear deposition, when it is combined with the lower level of energy density. The heat distribution produced by the zig-zag deposition strategy became more stable and therefore led to less residual stress evolution.

- The effect of dwell-time appeared to be more considerable when it was combined with deposition strategy than the level of energy density. In both linear and zig-zag deposition, the shorter dwell-time of 60 s resulted in more variation of residual stress than the longer dwell-time of 180 s. Less waiting time before depositing the next layer caused less stabilised heat distribution and as a consequence more residual stress produced in the final part, regardless of the deposition strategy.

- However, the effect of dwell-time could be compromised when it is considered combined with the level of energy density. Once again, the level of energy density seemed to cause more sever heating phenomena where the dwell-time could not easily cope with the thermal gradients, to stabilise it.
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- As the higher level of energy density was shown to prevail over the deposition strategy and the dwell-time in producing a higher level and variation of residual stress, all samples with the higher level of energy density were chosen for the contour method. The results from contour method proved that the difference between residual stress variations in samples with the higher level of energy density seemed to be less obvious.

- The contour method provided a more straightforward approach to measure stress/strain in PTA AM samples than the two diffraction-based techniques. Also, the contour method is less expensive and laborious to be implemented. As the data from contour method was shown to be reliable, it is suggested to be the first option to investigate residual stress in such parts. However, it should be noted that contour method is a destructive technique, which should be taken into account in choosing the option.

The link between the processing parameters and final residual stress state in PTA AM parts was shown to be predictable. The effect of processing parameters was mainly related to the heating and cooling phenomena associated with the process. Understanding the link between the processing parameters and the thermal gradients could help to manage the level and variation of the residual stress. Such understanding will lead to optimising the process and eventually incentivise implementation of the PTA AM as a viable manufacturing technique to produce functional parts for real world applications.

Whilst this research work provided a foundation for residual stress analysis in PTA metal-AM, the limitations and constrains should be carefully studied and considered for any similar empirical and/or simulation methodology implementation.
10.2. Recommendations for future work

As the PTA AM process is a relatively new AM technique, there is vast opportunity to investigate the process further to ensure it would deliver functional parts for industrial applications. This research work provided a coherent study of the effect of processing parameters on the evolution of residual stress within the PTA AM parts, with a focus on three main processing parameters. However, three main areas have been identified as the subject of further complementary research study to the current research work:

**Process Parameters:** as explained in the manufacturing parts and methodology chapters (Chapters 3 and 4), the processing parameters are a combination of the PTA process and AM processing conditions. This was shown to lead to a complex landscape in identifying and studying the effect of processing condition. In this research work, the effects of three parameters (deposition strategy, dwell-time and energy density) were investigated, where two different levels were considered for each processing parameter. Extending the variability of the three parameters would lead to better understanding of potential effects and solutions for the whole process. This could mainly be the case for the deposition strategy and the energy density, as a wider range of dwell-time was analysed to define a processing window for the manufacturing of the samples.

Furthermore, other processing parameters should also be investigated to understand their effects on the final residual stress state within PTA AM parts. For example, the wire-feed rate and the transverse speed of the deposition process play a crucial role in the amount of the material on the bed and affects the overall manufacturing process. Understanding the effect of the wire-feed rate and the transverse speed of the deposition process combined with a closed-loop system, integrated with the Artificial Intelligence (AI) and deep machine learning could therefore be part of the further research and development for the PTA AM.

A further detailed analysis of the process parameters could include gas purity, chemical composition of welding wire, electrode diameter, shape and chemistry, polarity, electrical waveform as well as zig-zag deposition parameters, such as frequency, amplitude and dwell times. Also, in this research work, to adjust the energy density, the current (I) on the PTA system was adjusted based on the current set up of the PTA AM machine. Considering the fact that the voltage of the PTA process could also affect the overall
energy density, and hence the heat input, makes it a potential processing parameter for further investigation.

In terms of thermal analysis of the PTA AM process, a more rigorous approach could be adopted to obtain a more detailed set of temperature data. A suggested approach would be looking into the temperature data from each individual bead as being deposited, by a thermal camera. The in-situ analysis of the thermal data could the development of a simulation model for the process. Such analysis could also support phase transformation analysis, which is directly related to the thermal history, and hence develop a detailed understanding of the residual stress (type II).

Also, regarding the thermal history, the type and the rate of the cooling (passive cooling versus assisted cooling process, for instance) could be considered as another factor to influence the solidification pattern and the final state of residual stress. This could be referred back to the heat input and the geometry and/or features for an individual bead to be deposited.

The geometrical aspects play a significant role in influencing the final geometrical and mechanical properties of manufactured parts. For example, a discrepancy between the widths of deposited walls by different deposition strategies was observed which could affect the final width of deposited walls. Understanding the geometrical effects could therefore help to build parts that are more reliable and could be the subject of further research.

In that respect, the relevance of the geometrical aspects to the assumption of the plane stress condition could be examined in more detail to understand the safe width range for such assumption. The further research should address how the profile of the strain/stress is changing along the width of deposited walls and how the width size is important in such assumption.

Furthermore, investigating the geometry of the deposited layer, as they are stacked up on top of each other, could lead to better understanding of the effect of the deposition process on the shape and size accuracy and link it to the residual stress. As briefly covered in Chapter 3, the shape of an individual bead could be described in terms of process parameters such as the deposition rate and Wire Feed Rate (WFR). Such analysis could lead to more reliable deposition process and hence alleviating residual stress, resulting in a more dimensional accuracy.
Microstructure and phase transformation: microstructural analysis of the PTA AM parts is a complementary research study to the current research work. The relevance of the microstructural features to the residual stress evolution process has been studied for dual phase materials, such as Ti-6Al-4V. It was shown that such study could support understanding of the residual stress evolution mechanisms and provide further details on how to control such mechanisms by referring back to the process parameters.

As a dual-phase alloy, phase transformation plays a significant role in the evolution of residual stress in Ti-6Al-4V. Therefore, phase transformation analysis could support understanding of the residual stress formation in any processing of the titanium alloy. As PTA AM is a relatively complicated manufacturing process, it is recommended that in-situ phase transformation analysis could lead to a better understanding of the effect of thermal history in residual stress evolution. This is the basis for Electro Thermal Mechanical Testing (ETMT). A state-of-the-art test rig for such experimental set up is available at ESRF and can be used to investigate the phase transformation in the titanium alloy during the PTA AM process.

Simulation: the residual stress data from this research work can be used to validate a Finite Element model for the PTA AM process. A thermo-mechanical Finite Element (FE) analysis of the PTA AM process to predict the properties of the PTA AM parts. Phase transformation and associated residual stress formation should be incorporated into both static and dynamic FE models.

The combination of experimental and simulation results could provide a foundation for a comprehensive understanding of the microstructural evolution in the PTA process and provides a foundation for wider applications of this metal-based AM technique.

Furthermore, the implementation of the Integrated Computational Materials Engineering (ICME) could potentially establish a series of relationships between material microstructure, manufacturing process and mechanical properties. By integrating the advantageous series of the experimental data from this research work, the ICME approach could deliver a validated software solution for component design and manufacture through PTA AM. Combining the ICME technique with Artificial Intelligence (AI) and deep machine learning could lead to a novel manufacturing solution for industry.


