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**PRESSURE ASSISTED SEGREGATION IN SQUEEZE  
CAST ALUMINIUM ALLOYS**

**Author: DAVID JOHN BRITNELL**

**Submitted for the Degree of Doctor of Philosophy in  
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**UNIVERSITY OF WARWICK**

**DEPARTMENT OF ENGINEERING**

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**To Sam**

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## **DECLARATION**

The work presented in this thesis is the authors own work and the author declares that no part of this thesis has been previously used or published by the author. Work of other authors is fully referenced within the text.

## SUMMARY

A study has been conducted into Pressure Assisted Macrosegregation during the squeeze casting of an aluminium alloy, LM25, and an aluminium based silicon carbide particulate metal matrix composite. The squeeze casting process is particularly prone to this type of segregation which results in large areas of solute rich material in the final casting.

The fundamental causes of the segregation have, until now, not been fully understood. This has restricted the use of squeeze casting as it was believed that the cause was a fundamental manufacturing weakness.

An existing squeeze casting facility at Warwick was used to produce cast samples for study. The facility produces simple tensile test bars. Inserts for the die were designed to specifically produce the segregation within the castings. Thermocouples were threaded through the die wall and placed within the casting to enable the recording of the thermal history during solidification. Samples were taken from the castings, mounted, ground and polished for study via an optical microscope.

A mechanism for the formation of the segregation is proposed. The primary causes of the segregation are shown to be surface tension, of the molten alloy, and the design of the component. The widespread belief that this form of segregation is a fundamental weakness of the squeeze casting process is therefore shown to be false.

A theoretical model to a first approximation of the segregation, using the Young and Laplace equation is given. Using the pressure difference across a thin film the minimum web width, or radius of a corner, for no significant segregation can be found.

The model is expanded and presented in a graphical form which is easy to understand and provides the designer with some tools to enable a sound casting to be produced. By sensible design the segregation phenomenon can be significantly reduced and in some cases eliminated completely.

# CHAPTER 1

## INTRODUCTION

### 1.1 WHY SQUEEZE CAST?

Traditionally it has been the aerospace industry, and the military, that has led the way in research into specialised materials and processes, [1, 2]. Strong, low weight materials and components that perform at their physical limits have all, in general, been first produced for use in the aerospace industry. In recent years, however, the need for such components and materials has grown within other industries.

The motor industry, in particular, is engaged in the search for lighter, stronger, more wear resistant components, and for lightweight high temperature materials, for use in the vehicles they produce, [3]. The driving force behind this is the need to significantly cut pollution by producing more fuel efficient vehicles. Rover Group is one of the companies engaged in research in this area. One process in particular that can produce stronger lighter components, in comparison to other processes using the same material, is squeeze casting, [4]. Squeeze casting is a process whereby near net shape components are produced directly from the molten state. Unlike other casting processes, in squeeze

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casting the molten alloy is subjected to a high externally applied pressure which is sustained until the component is completely solid.

The pressure helps to produce a very rapid heat transfer, between the alloy and the die by forcing the alloy into intimate contact with the die wall. The rapid solidification achieved produces a solid with a very fine microstructure leading to greatly enhanced mechanical properties, over conventionally cast pieces. The applied pressure is also high enough to prevent the formation of virtually all gas and shrinkage porosity.

There are two other significant benefits when using the squeeze casting process. The squeezing of the alloy, is carried out in closed polished dies. The dies impart a very good surface finish to the component, on a par with pressure die casting  $0.4$  to  $3.2\text{ }\mu\text{m}$ , [5]. Dimensional accuracy is also very good,  $0.2\text{mm}/100\text{mm}$ , [5].

The squeeze casting process is currently used commercially in the USA, Europe and Japan for the production of disc brake callipers [6], and engine pistons, [7, 8, 9]. The imposition of tighter emission controls on vehicles will inevitably lead to many more parts of vehicles being replaced with lighter components.

Other components made from metal matrix composites which have enhanced mechanical properties, e.g. high temperature resistance, wear resistance and tensile strength will also play a big part in emission control. The processing of MMC's is very difficult because of their inherent advantages. Squeeze casting is capable of producing components from these difficult to handle materials.

In an effort to meet the tighter emission controls being introduced a great deal of research into the production of components by the squeeze casting process, amongst others, is required. Research into the squeeze casting process is almost certainly well advanced in many commercial organisations. It is the squeeze casting process which is to be studied in this work, or at least one particular aspect of the process.

To enable the production of good quality squeeze castings a tight control over several process parameters is required. The principal parameters are melt temperature, die temperature, applied pressure, duration of applied pressure and the metering of the molten material. Poor control of these parameters can lead to defects within the casting, [10].

The most common defects found in squeeze castings, are; oxide inclusions, underfilling/overfilling, cold laps, hot tearing, sticking, centreline segregation, case debonding, extrusion and extrusion segregation. The fundamental processes at work behind some of these defects are not, at present, fully understood. This is especially true of extrusion segregation. The squeeze casting process is particularly prone to this type of defect which has to date received limited attention, [11]. Macrosegregation caused by this type of defect can have a significant effect on the mechanical properties of the final casting.

If the segregation appears on or close to the surface, of the component, a machining operation is required to remove it. Not only does this necessitate a larger casting, to allow for the machining, but two of the benefits of squeeze casting, good surface finish and good dimensional tolerances are effectively lost.

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Having established that there is a significant lack of understanding, of the processes behind the particular form of extrusion segregation found in squeeze casting, the main thrust of this work is to provide that understanding.

## **1.2 OBJECTIVES OF THE STUDY.**

The main objectives of the study can be set out as follows:

1. To produce extrusion segregation, in an aluminium alloy, in a controlled manner to enable the phenomenon to be studied in a systematic and scientific way.  
The effects, on the segregation, of pouring temperature, applied pressure and die geometry will be studied.
2. To propose a theoretical explanation of the processes involved.
3. To produce a mathematical model of the macrosegregation phenomenon.
4. To verify the model with experimental work as closely as possible using the equipment available.

5. To produce design criteria for the reduction, or in certain circumstances elimination, of segregation within squeeze castings.

### **1.3 METHODOLOGY**

The first part of the project involved a literature survey to provide a good grounding for the rest of the research work on pressure assisted segregation during squeeze casting. This work is presented in chapters 2 through to 4.

The experimental stage, as laid out in chapter 5, followed. The need was to produce and study the segregation in depth. A squeeze casting facility was designed and installed at Warwick University as part of two previous research projects, [12, 13]. The facility is capable of producing a simple test bar 150 mm long, 24 mm wide and 15 mm thick.

Modifications, to parts of the squeeze casting facility, formed the first part of the experimental stage. Die inserts were designed and installed to provide the means for the segregation to be produced in a controlled way. Thermocouples were introduced into the die cavity so that the temperature profile of the solidifying component could be recorded. The thermocouples can be placed at any point within the casting.

This then allowed further study of the phenomenon and in particular the effect of die design.

The effects of temperature, pressure and geometry on the segregation were all studied by using the thermal traces from the embedded thermocouples and by metallographic examination.

An aluminium alloy LM25, (Al-7%Si-Mg) was used in the main part of the work, with further work then carried out on an aluminium based particulate metal matrix composite. This further work studied the effect of the segregation on the particulates. The results and analysis of the experimental work is presented in chapters 6 through to 9.

In chapter 10 a theoretical mechanism is proposed to explain the formation of pressure assisted segregation within the squeeze casting process. A first approximation model of the segregation mechanism is produced and tested using pure zinc and pure aluminium. Design principles are then discussed which allow the control of the segregation and the production of sound castings.

Project conclusions and recommendations for further work are presented in chapter 11.



# **CHAPTER**

## **2**

### **SQUEEZE CASTING.**

#### **2.1 INTRODUCTION**

This chapter gives a comprehensive account of squeeze casting covering areas such as; history and development, the benefits and disadvantages of the process, the process parameters and the defects that can occur in the final component. The two main variants of the process are discussed, namely the direct method and the indirect method and some comparisons are drawn between them.

#### **2.2 THE BASICS OF THE PROCESS**

Squeeze casting is so called because liquid metal is solidified under sustained high pressure in a closed die i.e. the melt is squeezed.

In the basic process a metered amount of molten alloy, with minimal superheat, is drawn from a holding furnace. The alloy is transferred to a lubricated and preheated die. A load is then applied, via the die, to the molten alloy. The load is maintained during the solidification of the alloy. The die is then opened and the solid component ejected.

The process is also called by various other names which seem to depend upon the route the research has taken, e.g. extrusion casting [14], liquid extrusion [15], pressure crystallisation [16], squeeze forming [2] and liquid metal forging [17].

The squeeze casting process is really a hybrid of die casting and closed die forging. It combines the ease of casting with the mechanical integrity of forging to produce a very high quality product.

In conventional die casting processes the interface between the die and the molten alloy is not ideal. The dies are, usually, highly polished and coated with a die release agent such as graphite or mica which produces a thermal barrier between the die and the molten material. The viscosity of the molten material also causes a non ideal contact with the die surface, especially if the component and thus the die is of an intricate design. As the molten alloy solidifies and cools the solidifying metal tends to shrink away from the die wall. This produces an air gap, between the die wall and the solidifying component which significantly reduces the cooling rate. A lower cooling rate leads to an increase in the grain size of the microstructure which can adversely affect the mechanical properties of the final component.

In the squeeze casting process, however, the interface is almost ideal because of the application of the load which forces the molten alloy and the mould wall into intimate contact. This intimate contact greatly increases the heat transfer rate across the melt/mould interface and as the load is applied during the whole solidification cycle there is no shrinkage away from the mould walls. The fast

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heat transfer rate is, therefore, also maintained during the whole cycle. The result is a casting which has a fine grained microstructure in comparison to other forms of casting.

The fine grained microstructure gives the casting increased mechanical properties approaching those found in a forging. Unlike a forging, however, the properties are isotropic.

Most casting processes have porosity, to a greater or lesser degree, in the final casting. Squeeze casting, by the application of the load, eliminates virtually all porosity by not allowing it to form, [10], and also closes down any porosity induced by the pouring of the molten alloy.

The final product is thus fully formed with close tolerances, a very good surface finish, enhanced mechanical properties which are mostly isotropic and with a virtual lack of porosity.

### **2.3 HISTORY AND DEVELOPMENT**

There is a misconception that the squeeze casting process is a modern invention. While it is true that the vast majority of the research into the field has only been carried out during the last thirty years or so the concept of applying pressure to a solidifying metal goes back much further.

The earliest known reference to the concept of applying pressure to a solidifying metal was by James Hollingrake in 1819 [18]. Hollingrake filed a patent in which he states that by the application of pressure, "I produce a peculiar

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soundness of external surface and uniform closeness of texture commensurate to the force applied.....and which pressure may be continued until the fluid metal is sufficiently fixed and fully set". It is not known, however, what, if any experimental work was performed at that time. Some years later, in 1878 in Russia, Chernov [19] suggested that molten metal in a mould could be subjected to steam pressure.

The first, known, recorded work, done on the solidification of metals under pressure, was carried out in Germany in 1931 [16]. The work studied the effect of pressure on the solidification of aluminium silicon alloys. Around this time work was also underway within the Soviet Union into various forms of the squeeze casting process [14]

The 1960's saw a great deal of research initiated into the process throughout the U.S.A., Europe and Japan [2, 6, 9]. The initial research was confined to the aerospace industry in its search for ever stronger and lighter materials. Recently, however, the automotive industries have become more involved in squeeze casting research [6, 7]. The automotive industries also want lighter and stronger materials to help counter the effects of pollution by increasing fuel efficiency [20].

The past twenty years have also seen squeeze casting research into the effects of a ceramic reinforcement being incorporated into the component [1, 2, 6, 7, 21, 22]. The reinforcement can take the form of, continuous or chopped, individual ceramic fibres [23, 24, 25] a preformed porous ceramic insert [26, 27] or a ceramic particulate [28, 29]. These combined materials, consisting of an alloy

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matrix and a ceramic reinforcement, are called metal matrix composites. The manufacture and processing of metal matrix composites via squeeze casting is today a very large area of research [24, 25, 27, 30, 31, 32, 33].

Even though there is some commercial exploitation of the squeeze casting process [2, 6, 7, 9] a great deal of research is still required in many areas of the process. Research, into the squeeze casting process, is very much at the forefront of technology.

### **2.4 TWO VARIATIONS**

Two distinct variations of the squeeze casting process have emerged from the research: the indirect and the direct processes. The main difference between the two is in the way in which the pressure is applied to the melt. As the names of the processes imply the indirect method applies the pressure to the solidifying alloy indirectly via more material in a feed sprue whereas the direct method applies the pressure directly to the solidifying alloy via a split die mounted on the platens of a press. The indirect process has gained favour in Japan [6], whereas the direct process is favoured in Europe and the U.S.A.

### **2.4.1 The Indirect Process**

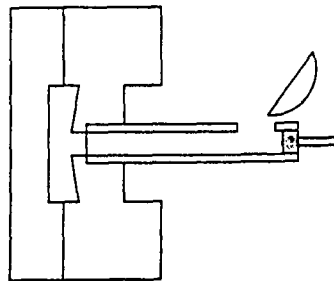
The indirect process is similar in operation to pressure die casting. The main difference is the injection of the molten alloy into the die. In the indirect squeeze casting process it is injected in a slow controlled way followed by the application of a high pressure whereas in die casting the injection rate is relatively high [5].

Molten alloy is drawn from a holding furnace and poured into a feed sprue, figure 2.1a. The alloy is then slowly injected in a controlled manner, into a closed, lubricated and preheated die, by the action of a ram, 2.1b. On complete filling of the die the ram applies a high pressure to the melt which is sustained throughout the solidification of the alloy, 2.1c. The die is then opened and the component ejected, 2.1d.

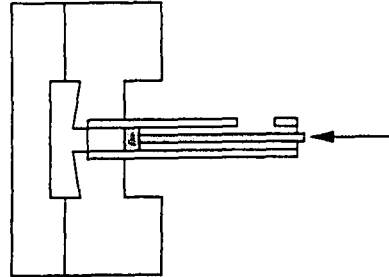
### **2.4.2 The Direct Process**

In the direct process, molten alloy is drawn from a holding furnace and poured into the bottom half of an open, lubricated, preheated die, figure 2.2a. The die is fixed to the top and bottom platens of a press. The top half of the die, the punch, is then brought down into contact with the molten alloy by the action of the press, 2.2b. A high pressure is then applied to the alloy and is sustained throughout the solidification of the alloy, 2.2c. The die is then opened and the component ejected, 2.2d.

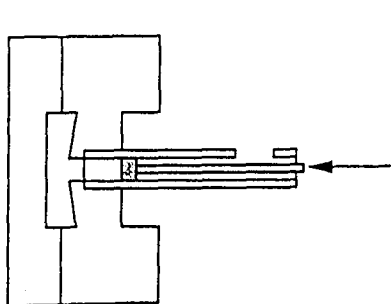
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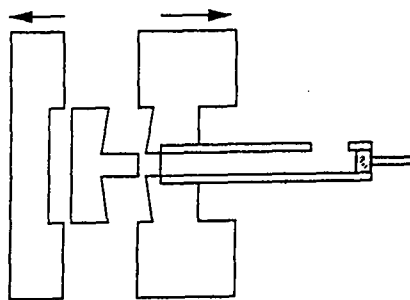
a. Fill feed sprue with sufficient molten alloy.



b. Inject alloy slowly into the die.



c. Apply a high load via the ram. Sustain until component is solid.

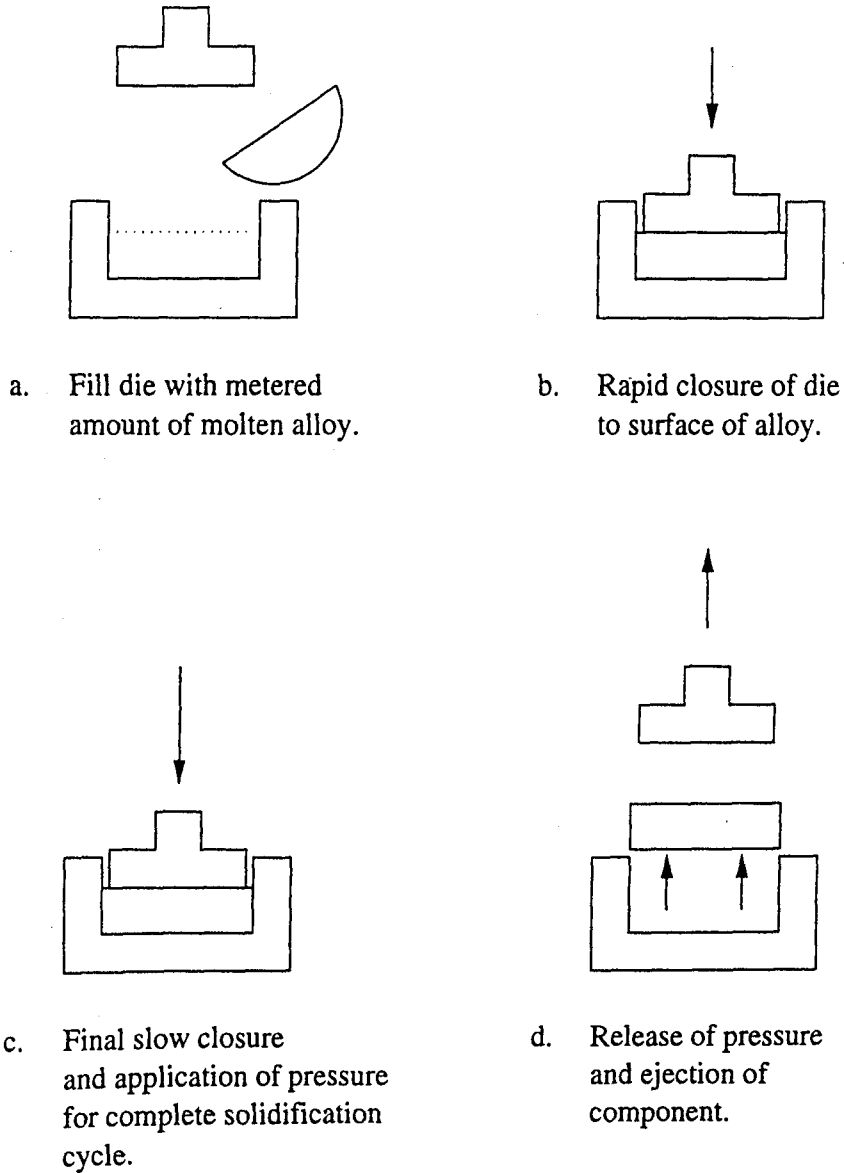


d. Release load, open die and eject component.

**Figure 2.1**

*Schematic Diagram of the Indirect Squeeze Casting Process*

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**Figure 2.2**

*Schematic Diagram of the Direct Squeeze Casting Process*

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## **2.5. ADVANTAGES OF THE SQUEEZE CASTING PROCESS**

The squeeze casting process has significant advantages over other casting processes. The main ones are given below.

### **2.5.1 Porosity**

The application of a high pressure during solidification greatly reduces or eliminates virtually all porosity within the casting.

Shrinkage porosity is eliminated by the forced movement of low melting point liquid into any voids that may have formed during the solidification process [35] and gas porosity is reduced as the applied pressure is high enough to suppress the growth of gas bubbles [10].

### **2.5.2 Mechanical Properties**

Data is available in the literature to show that squeeze cast components have enhanced mechanical properties such as increases in elongation and tensile strength, over conventionally cast components made from the same alloy.[2, 36, 37, 38] Consequently recycled and lower grade casting materials can also be used to produce components with the same mechanical properties as high grade alloys that have been sand cast [36].

### **2.5.3 Near Net Shape**

As the casting is solidified within a die under pressure it will assume, very closely, the shape and dimensions of the die. If the die, therefore, is of a high dimensional accuracy with a good surface finish the casting will also inherit these characteristics, [4, 6, 17].

### **2.5.4 Fluidity of the Alloy**

As the applied pressure will force the alloy to flow into all areas of the die, it has been thought up until now that the alloy is not required, necessarily, to have good fluidity. This, therefore, gives the process the ability to produce components from the usually non castable wrought alloys and the difficult to process MMC's, [21, 27, 31, 32, 33, 39].

### **2.5.5 Material Wastage**

As there is no feeder or running system required the material usage is almost 100%. The indirect process however does have a small amount of material within the feed sprue that is removed from the component and is therefore lost, [2, 5, 27].

amount of material within the feed sprue that is removed from the component and is therefore lost, [2, 5, 27].

#### **2.5.6 Manufacture and Processing of MMC's**

One area where squeeze casting will be at the forefront is in the manufacture and processing of metal matrix composites. Whether the ceramic reinforcement is in the form of a preformed insert, continuous fibres, chopped fibres or particulates squeeze casting has the potential to produce a finished component in one step. This will eliminate the need for expensive and difficult machining operations on the MMC, [1, 20, 40].

### **2.6 DISADVANTAGES OF THE SQUEEZE CASTING PROCESS**

Against the advantages, of the process, consideration must be given to the disadvantages. Some of these are given below.

#### **2.6.1 Capital Cost**

The capital cost of the equipment is very high for this process. A large capacity press is required; the size of the press will limit the size of the casting that can be produced. A complete handling facility for

molten material is also required. The dies are expensive and they have a relatively short life. This is because of the high loads involved, and the thermal shocks to which they are subjected. A typical die life of 15000 units has been quoted [9].

### **2.6.2 Design Restrictions**

The die has to be able to withstand the high pressures generated and therefore the complexity of the casting is restricted in comparison to some other casting processes, e.g. investment casting, [5]

### **2.6.3 Accurate Metering**

An accurate metering system is required to enable dimensionally accurate castings to be produced, [9, 41, 42]. This is especially true for the direct method, which in comparison to the indirect method, has virtually no margin for error. The mechanical properties can also vary dependent on the amount of material, [39].

### **2.6.4 Machinability**

Savas and Altintas [43] have found, in their study, that the machinability of certain aluminium alloys decreases if they are

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squeeze cast. The alloys studied were Al-5%Mg, Al-10%Mg and Al-4.5%Cu-0.5%Bi. Machinability decreased because of porosity elimination coupled with the coagulation of the  $Mg_2 Al_3$  phase in the Al-Mg alloys and the destruction of the bismuth rich particles in the Al 4.5 % Cu 0.5% Bi alloy.

### **2.6.5 Casting Practices**

Perhaps the biggest drawback of squeeze casting, at the present time, is that the components made by this process are not generally designed with this process in mind. The components, including those with ceramic reinforcements, tend to be adaptations of components previously made by other casting methods. This is not to say they necessarily need radical redesigning. However if the component was designed at the outset, with squeeze casting as the manufacturing route, it is possible that a greater complexity and standard of components could be made.

### **2.7 PROCESS PARAMETERS**

To achieve consistent castings there are a number of process parameters that need close control. The main ones are; volume of molten alloy, pouring temperature, die temperature, applied pressure and pressure duration.

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### **2.7.1 Volume of Molten Alloy**

Section 2.6.3 has already stated the need for an accurate metering system. The volume of the melt also controls the closing limits for the dies, in the direct squeeze casting process. Thus the final size of the component, in the direct process, is dependent on the volume of molten alloy initially placed in the open die, [9].

### **2.7.2 Pouring Temperature**

A too high pouring temperature will have an adverse effect on the die life, as well as extra fuel costs. As the die life is already relatively short the pouring temperature should be as low as possible, [5, 9].

A too low pouring temperature, however, can cause a significant increase in the amount of premature solidification. This in turn can lead to problems with cold laps [10].

Typically the pouring temperature is 100° to 800° C, [5], above the liquidus temperature, the actual value being dependent on the alloy involved.

### **2.7.3 Die Temperature**

For aluminium alloys die temperatures within the range 200<sup>o</sup> to 300<sup>o</sup> C are quoted, [5]. The top punch is usually 20<sup>o</sup> C below that of the bottom die, to allow for clearance in use, [12].

### **2.7.4 Applied Pressure**

Various factors will influence the amount of applied pressure required to produce a sound casting. These include the casting shape, the casting size, the type of microstructure required, the mechanical properties required of the final component and the alloy used in manufacture.

For each casting there is a minimum pressure below which the desired effects of refined microstructure and enhanced mechanical properties are not produced and a maximum pressure above which there is no further improvement, [39, 44]. A range of pressures, between 30 and 150 MPa, for the squeeze casting of most components, is given in the literature, [5, 6, 7, 39, 44].

### **2.7.5 Pressure Application Time**

As the pressure must be maintained during the solidification cycle, the dimensions and geometry of the casting will determine the pressure application time. Once the casting has fully solidified maintaining the pressure has no more material effect. The only effect of maintaining the pressure is to increase the cycle time, [5, 9, 17].

The delay between charging the die with molten alloy and the application of the pressure can have a profound effect on the casting. A long delay can result in a greatly increased amount of premature solidification. As a result this can have a large adverse effect on the quality of the casting [10].

## **2.8 CASTING DEFECTS**

As with all casting processes, defects within the final casting are possible especially if the process parameters are not tightly controlled.

Rajagopal and Altergott [10] say they have identified eleven types of defect within squeeze castings. These are oxide inclusions, porosity, underfilling, cold laps, hot tearing, sticking, centreline segregation, case debonding, blistering, extrusion debonding and extrusion segregation. Most of these defects can be found in other forms of casting so only extrusion debonding and extrusion segregation are dealt with here as they are more common in squeeze casting,

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compared to other forms of casting. The root cause, of these two defects, is related to mechanisms inherent in the squeeze casting process.

### **2.8.1 Extrusion Debonding**

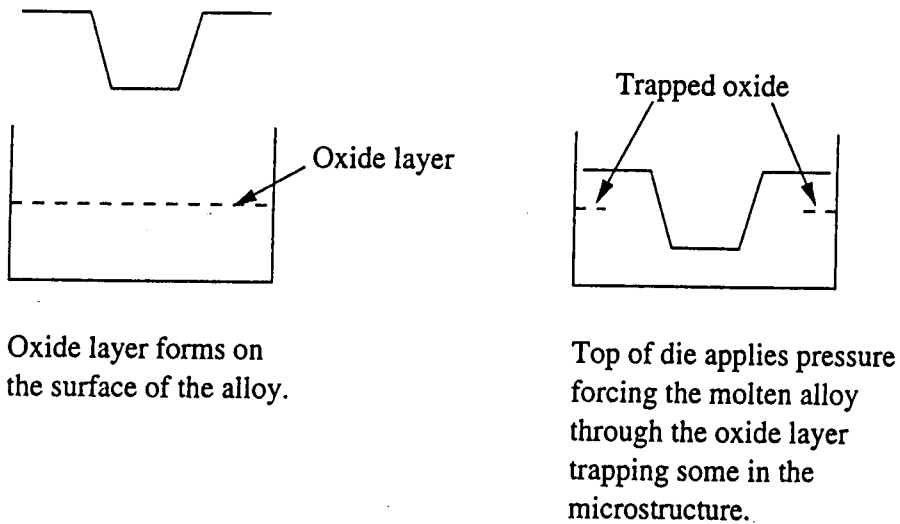
It is fair to say that this defect is confined, in the main, to the direct squeeze casting process. It is caused by oxidation of the surface of the molten alloy within the die. An oxide layer forms in the time delay between the molten alloy being poured into the die and the die closing. Application, of the pressure, then causes molten alloy to be extruded past, or through, the oxide layer trapping the oxide layer within the casting, figure 2.3. Extrusion debonding can happen where metal flow is required upwards into a void within the top die. This can be into a web, flange or even a corner of the casting, [10].

In the indirect process there is little surface area of the molten alloy exposed to the atmosphere for an oxide layer to form. However, any that does form will tend to be broken up and carried into the body of the casting on injection into the die. This can lead to one of the other defects, i.e. oxide inclusions. Factors affecting this have been looked at by Komatsu et. al. [6].

A number of steps can be taken to reduce the possibility of extrusion debonding within the direct squeeze casting process [10]:

- 1) Minimise the time between melt pouring and die closure.
- 2) Squeeze cast with any webs or flanges, that form a part of the casting, in the bottom part of the die.

- 3) Perform the whole casting process in an inert atmosphere.



**Figure 2.3**

*Extrusion of Molten Alloy Through Oxide Layer Causing Extrusion*

*Debonding, [10]*

### **2.8.2 Extrusion Segregation**

Extrusion segregation is material segregation in the casting on a macroscopic scale. In squeeze casting this form of segregation can be a common fault [10]. Microsegregation, that is material segregation between the dendrites, is, however, virtually non-existent because of the relatively rapid cooling associated with the process [4].

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Macrosegregation is caused when the applied load forces a lower melting point constituent of the alloy to flow through a network of dendritic growth, brought about by rapid heat loss from the melt into the die, into any voids within the casting. Macrosegregation is covered in depth in chapter 4 as it constitutes a large part of the current research work. The causes of the phenomenon are explored and the flow of the low melting point constituent is shown by the use of cooling curve data obtained from experimental work.

## **2.9 SQUEEZE CASTING AND METAL MATRIX COMPOSITES**

Metal matrix composites are a relatively new class of material that comprise of a ceramic material in a metallic matrix. They have been under development for between 25 to 30 years [45, 46]. Originally developed for aerospace and military applications they are now, as the price of the ceramic reinforcement and of the processing come down, beginning to expand into other industries. The automotive industry is just one that is committed to research into metal matrix composites today.

It is the ceramic reinforcement that gives metal matrix composites their advantages, over the base metals. These advantages include increased strength, increased elastic modulus, higher toughness and impact properties. It is these same ceramic reinforcements that make metal matrix composites notoriously difficult to manufacture and to process. Squeeze casting, with its unique property of producing near net shape components in one step from a liquid, is a

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process that is capable of producing fairly complex near net shape components using metal matrix composites. As a consequence the squeeze casting process is bound to have an influence on some aspects of metal matrix composite research in the future.

## **2.10 COMPOSITION OF METAL MATRIX COMPOSITES**

### **Matrix alloy.**

A large number of different alloys can be used as the matrix [47]. However, an alloy of aluminium or magnesium is usually chosen because the benefits of the increase in the strength to stiffness ratio attainable outweigh the increase in production costs. Aluminium alloys are by far the most common.[45, 48].

### **Reinforcement.**

Originally the reinforcement was only in the form of continuous fibres. Now, however, other forms of reinforcement are used. These include preformed inserts [26], chopped fibres [24], whiskers [49] and particulates [50]. A vast range of reinforcement materials exist. The predominant materials used are SiC, Al<sub>2</sub>O<sub>3</sub> and TiB<sub>2</sub> [45, 48].

## **2.11 SQUEEZE CASTING MMC's**

Many components made using a reinforcement in a metallic matrix can only be made via the squeeze casting process. The process of squeeze casting composites is basically the same as for the base alloys. Metal matrix composites can be split into two types for the purpose of this exercise; those where the reinforcement is of a discontinuous form giving a homogeneous material and those that require infiltration of a preform or fibres. The main problems encountered in their processing are highlighted in the following sections.

### **2.11.1 Discontinuous Reinforcement.**

With the reinforcement being homogeneously spread throughout the alloy the final casting also has a homogeneous structure. A number of problems have to be overcome to enable a sound casting to be made [27, 51].

In the initial melting a degassing agent is not used, as is usual for aluminium alloys, as this tends to remove the ceramic reinforcement along with the gas and other dross [40]. The melt requires stirring to prevent settling of the reinforcement over time [52, 53]. The stirring also helps to keep the reinforcement homogeneously spread, within the molten alloy. Stirring can, however, introduce air bubbles into the melt by the action of the paddle [54]. The reinforcement can also degrade over time if it is kept wetted by the alloy [55].

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Because the reinforcement does not melt but stays as a solid particle or fibre the composite fluidity is relatively poor. This is a major problem for most casting techniques. However, as stated in section 2.5.1 squeeze casting does not require fluidity in the melt so the process is well suited to the manufacture and processing of MMC's.

### **2.11.2 Infiltration of Preforms and Fibres**

At the present time the infiltration of preforms and continuous fibres is the more common way to produce a component incorporating a reinforcement. The preforms or continuous fibres allow the reinforcement of specific areas in a component. Uniformity of structure is also easier to obtain than with particulates or discontinuous fibres. Components made this way include pistons [7, 9], military vehicle wheels [2] and disc brake callipers [6].

For these composites the molten alloy is required to fully infiltrate the composite structure. Composites of this type are usually cast using the direct process as the preforms or fibres have to be placed into the die prior to casting. The action of the top die forces the molten alloy to flow into the porous structure of the reinforcement. Continued application of pressure forces the alloy to fully wet the surface of the reinforcement thereby producing a strong bond between the reinforcement and the matrix alloy. The pressure is again held until full solidification has occurred which, as previously stated, produces a fine grained structure in the alloy.

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# **CHAPTER**

## **3**

### **SOLIDIFICATION OF ALUMINIUM SILICON ALLOYS**

#### **3.1 INTRODUCTION**

The microstructure and mechanical properties, of the final casting, are, to a very great extent, dependent on the solidification cycle. A search through the literature shows that a great deal of work on the solidification of metals and alloys has been carried out and there are a number of books on the subject [56, 57, 58, 59, 60].

As this chapter provides the background for the work on macrosegregation, which is in the following chapters, the relevant parts of the solidification cycle are presented. Nucleation, dendritic growth and solidification under pressure are covered in this chapter. Macrosegregation during solidification will be covered in chapter 4.

### 3.2 NUCLEATION

Nucleation sites are required to enable the growth of a solid to form from a liquid [56]. These nucleation sites can be imperfections on the die wall, the coating used on the die or particles, either natural or added, within the melt itself. There are two distinct types of nucleating behaviour.

If the material spontaneously nucleates without any help from outside agents it is said to nucleate homogeneously [56]. Aluminium naturally contains a number of nucleation particles per unit volume [61]. This form of nucleation, however, requires a large driving force to become active[56]. The driving force is the undercooling of the melt, and in the case of homogeneously nucleating aluminium is 3-4 °C [61]. Large grains result from this form of nucleation due to the large driving force required and the relatively few nucleation sites available.

The term potency is used to describe the amount of undercooling required to activate the nucleating agent. The smaller the amount of undercooling required the higher the potency of the nucleating agent.

In commercial aluminium alloys nucleation particles are usually added in the form of a master alloy. This form of nucleation is termed heterogeneous. The particles, such as titanium-boride or titanium-carbide, have a high nucleating potency requiring an undercooling of as little as 0.1 °C [61]. A high nucleating potency allows many more Al grains to nucleate, with less undercooling, in comparison to homogeneously nucleating aluminium.

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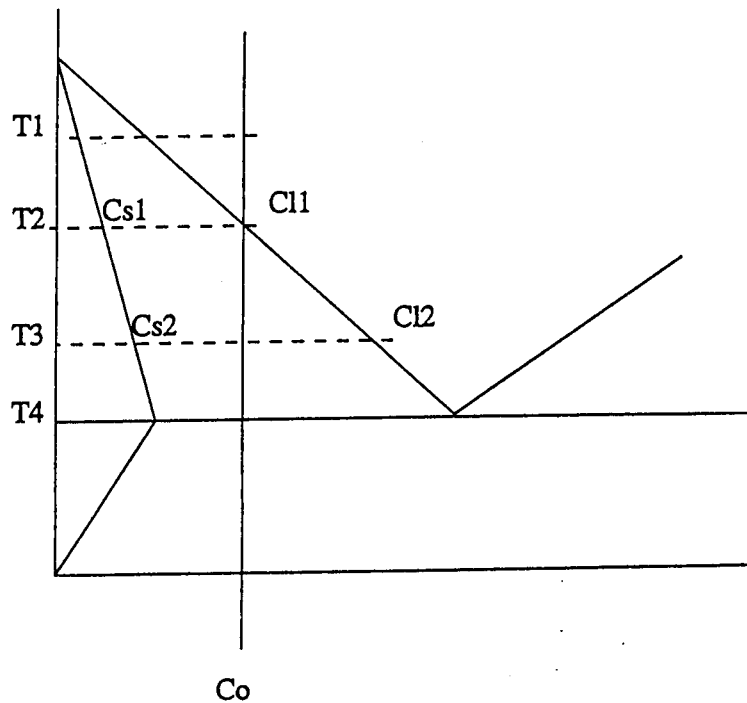
Thus by the addition of the nucleating agents it is possible to refine the structure of the casting thereby greatly improving the mechanical properties of the final casting. In general the larger the number of nucleating sites, within the melt, the finer the microstructure, as long as the degree of undercooling is maintained.

### **3.3 DENDRITIC GROWTH**

The undercooling described in section 3.2 is known as thermal undercooling, as it relies on a difference in temperature, i.e. a thermal gradient, to drive the solidification. During dendritic growth however another type of undercooling is also present which has a significant role in the solidification process; constitutional undercooling. In constitutional undercooling it is a difference in the composition of the liquid in comparison to the composition of the solid that provides the basis of the driving force [62].

In the vast majority of practical castings freezing occurs from the outside inwards. This is because the heat from the molten alloy is lost through the die, or mould, wall to the, relatively cooler, surrounding area. There is, therefore, a temperature gradient through the casting with the lowest temperature at the die wall and the highest temperature towards the centre [63]. The molten alloy, therefore, starts solidifying from the die wall and the solidification front progresses inwards as the heat is lost.

As an alloy solidifies the composition of the liquid, at the solid/liquid interface, changes. Figure 3.1 shows, schematically, part of a eutectic phase diagram. An alloy of composition  $C_o$  will be fully molten at temperature  $T_1$ .



**Figure 3.1**

*Schematic Diagram of a Eutectic Phase Diagram Showing the Liquidus and Solidus Lines*

When the temperature falls to  $T_2$  the alloy will start to solidify. The solid will have a composition  $C_{s1}$  and the liquid, at the solid/liquid interface, will have a composition  $C_{l1}$ . As the temperature falls further to  $T_3$  the solid will have

composition  $C_{s2}$  and the liquid composition  $C_{l2}$ . When the temperature reaches  $T_4$  the remaining liquid freezes to form a eutectic solid.

In an aluminium alloy the first phase to solidify is  $\alpha$  aluminium. From a nucleation point, on or close to the die wall, the  $\alpha$  aluminium begins to solidify out of the liquid. The grains, or cells, of  $\alpha$  aluminium, grow in preferred directions [56], and as the cells are enriched in the primary aluminium the remaining liquid becomes enriched in the solute. If the solidification front is stable cells will form with the cell boundaries enriched with solute.

Figure 3.2 shows schematically the enrichment of the liquid between growing cells.  $C_e$  is the eutectic composition,  $C_t$  is the composition at the cell tips,  $C_l$  is the average liquid composition and  $C_o$  is the initial composition of the alloy.  $X$  is the distance along the growth axis.

The characteristics, of the solidified casting, can be significantly changed by the formation of relatively large areas of solute enrichment. This type of solute enrichment is called macrosegregation. It is almost always an adverse change but can in some instances be beneficial depending on the properties required of the final casting.

If the equilibrium liquidus temperature remains below the actual temperature, as in figure 3.3, then no constitutional undercooling exists and the solidification is driven by the temperature gradient alone. There will, however, still be a difference in composition of material after solidification between the middle of the growing cell and its edges, because of solute rejection.

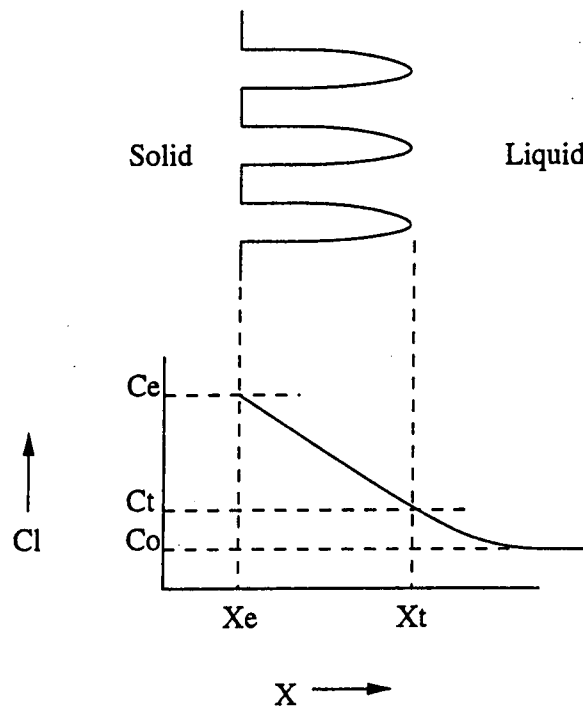
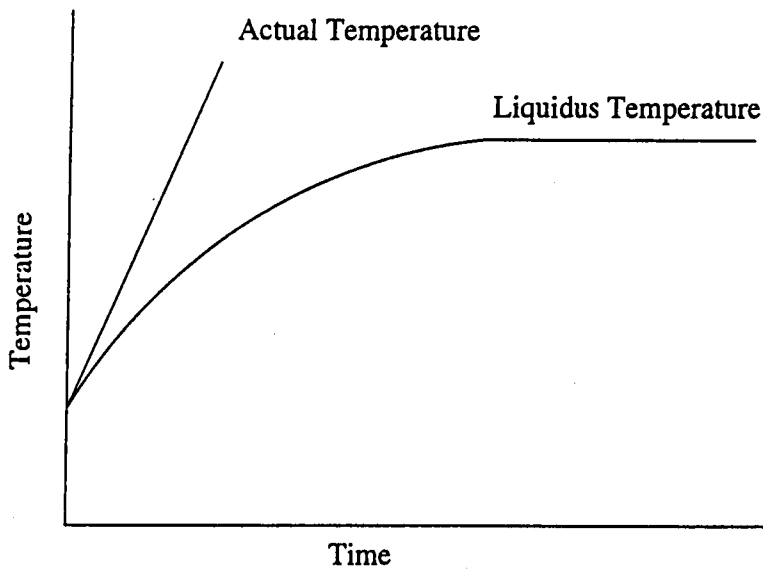


Figure 3.2

*Cells Growing in a Stable Front and Solute Distribution at the Solid/Liquid Interface. [56]*

This type of segregation, which is on a scale smaller than the primary dendrite arm spacing, is called microsegregation [56].

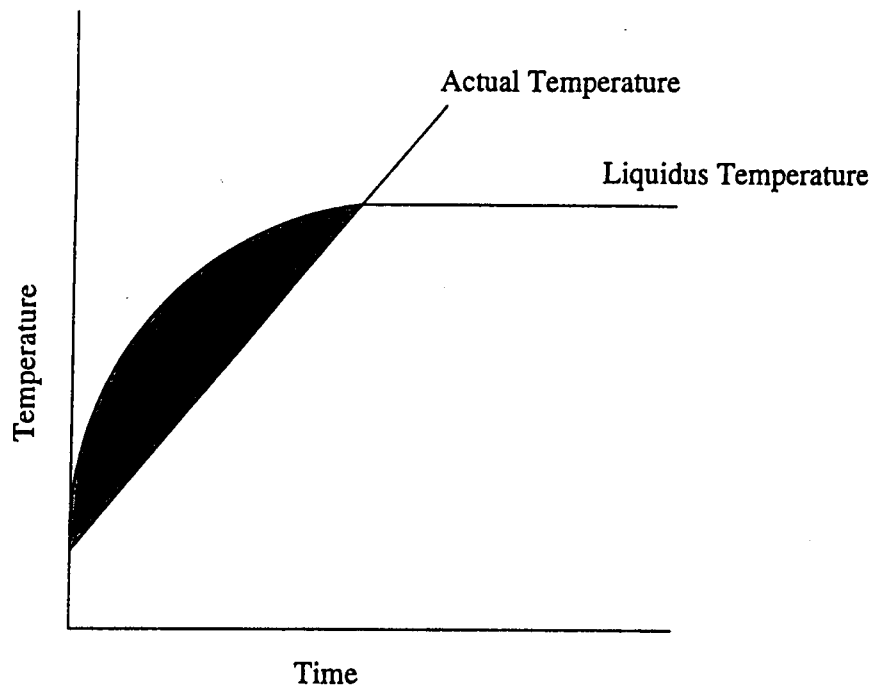


**Figure 3.3**

*Liquidus Temperature is Below the Actual Temperature at All Points Therefore  
there is no Constitutional Undercooling*

If, however, the actual temperature falls below the equilibrium liquidus temperature, as in figure 3.4, then there exists an undercooling of the melt. The shaded area on the graph indicates the extent of the undercooling. This undercooling of the melt allows a growing cell to grow faster than its neighbours if the conditions of temperature and composition are right.

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**Figure 3.4**

*The Shaded Region Shows the Amount of Constitutional Undercooling Existing as the Actual Temperature of the Melt is Lower than its Freezing Point*

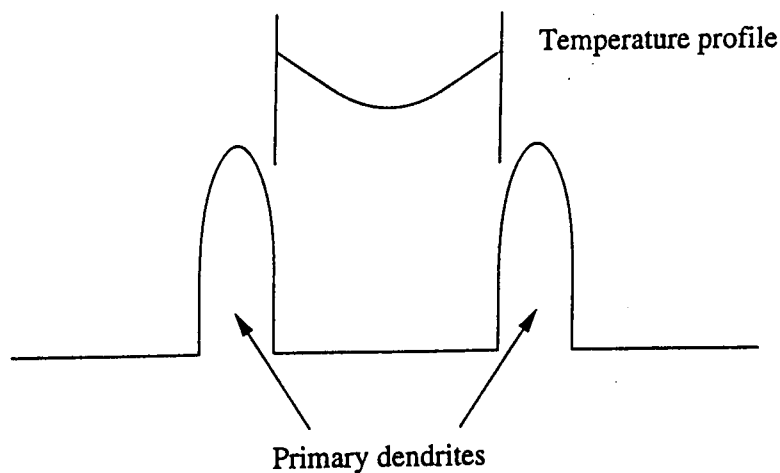
As more solute is rejected into the liquid, ahead of the solid/liquid interface the equilibrium liquidus temperature becomes higher than the actual temperature, figure 3.4, thereby providing a progressively larger amount of undercooling. This in turn drives the solidification of the  $\alpha$  aluminium at a faster rate than the surrounding cells. The cell then grows into the layer of liquid that is at a lower

---

temperature than its freezing temperature. The structure resulting from this type of freezing is called columnar dendritic, with each cell becoming a primary dendrite arm. The word dendrite comes from the Greek word dendra meaning tree like or branching.

The dendrites grow in the opposite direction to the temperature gradient and will only continue to grow into the remaining liquid if the equilibrium liquidus temperature of the liquid is above the actual temperature. If the actual temperature is above the equilibrium liquidus temperature the growing dendrite will melt back to the solid/liquid interface.

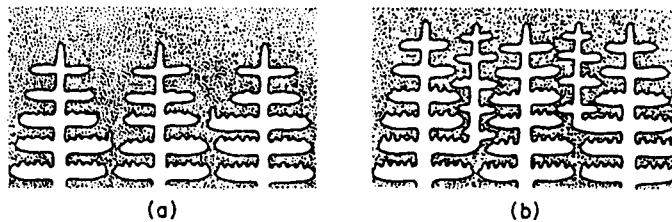
When two, or more, dendrites grow from adjacent nucleation points the liquid between them has a temperature gradient, [63]. The liquid adjacent to the



**Figure 3.5**

*Temperature Profile Between Primary Dendrites*

dendrites increases in temperature in comparison to the liquid in the centre, figure 3.5. The increase in temperature, adjacent to the dendrite, is due to the release of latent heat of solidification from the solidifying dendrite. The liquid is also enriched in the solute being rejected from the growing dendrite. The combination of the temperature gradient between the dendrites and the solute enriched liquid stops the dendrite growing into this region.



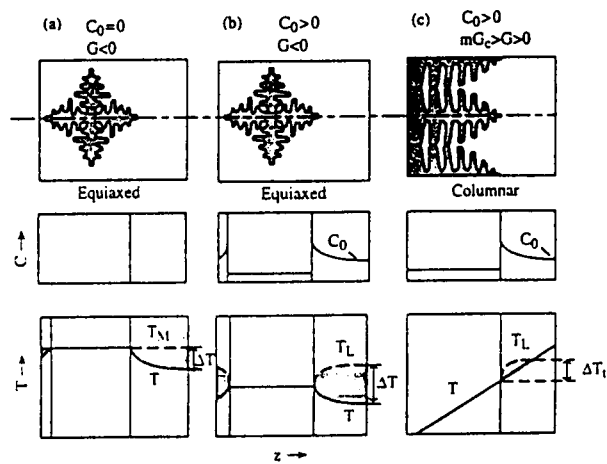
**Figure 3.6**

*Primary, Secondary and Tertiary Dendrite Arms with New Primary Arms  
Growing from Secondary Arms. [56]*

However if the gap between the dendrites is large enough and the temperature gradient produces an area of undercooling, as in figure 3.4, then secondary and tertiary dendrite arms can grow. Figure 3.6 [56] shows secondary and tertiary arms growing from primaries and if the dendrite spacing is very large new primary arms grow from secondary arms.



Another type of dendrite can also be found in castings, equiaxed dendrites. These dendrites, as their name suggests, are of equal dimension along each axis and therefore do not emanate from the die walls.



**Figure 3.7**

*Growing Equiaxed Dendrites; a, b Free Growth and c Constrained Growth  
with Compositions and Temperature Fields Along the Dendrite Axis*

*Trivedi and Kurz [64]*

It is not fully understood how equiaxed dendrites form, as the nucleation points will be in relatively high temperature liquid. Trivedi and Kurz [64], however, say that;

"Growth from an undercooled melt in which generally an equiaxed dendritic crystal structure forms, the latent heat of fusion is dissipated through the cooler liquid ahead of the interface. In this case the temperature gradient in the liquid at the interface is negative, whereas that in the solid is nearly zero". Figure 3.7 a and b shows equiaxed dendritic growth with the relevant composition and temperature conditions.

Other mechanisms, for equiaxed dendrite growth, include; dendrites breaking away from the original nucleation sites and floating into the melt, which then grow before fully remelting and the melting off of secondary dendrite arms which then go on to be nucleation points for growth,[57].

### **3.3.1 Dendritic Coherency.**

As the dendrites grow, from all sides of the casting, eventually they will impinge onto each other which will, not only, restrict their growth but form a lattice structure throughout the casting. The resulting latticework of dendrites gives a degree of structure to the solidifying alloy. As soon as the lattice structure is apparent throughout the casting a state called dendritic coherency is reached [65].

The interdendritic liquid that remains is free to flow through this structure until the temperature falls to allow it to solidify. It is during this time that many of the defects occur in the structure of the casting such as shrinkage porosity, hot tearing and macrosegregation.

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During squeeze casting, however, the virtual lack of any porosity is partly due to the coherent dendritic structure. The application of pressure to the solidifying casting forces the interdendritic liquid to flow through the structure into any casting voids. A major drawback with this forced fluid flow is macrosegregation within the casting. This is covered in depth in the following chapters. No shrinkage porosity forms as the applied load does not allow the solidifying alloy to contract away from the die walls or sections within the casting.

Work has been carried out to determine the fraction solid of material at the coherency point for various aluminium alloys, [61, 66]. The figure given for the alloy A356, which is of the same composition as LM25, the alloy used in the present work, is 13-21% solid.

### **3.4 SOLIDIFICATION UNDER PRESSURE**

The application of pressure to a liquid has the effect of raising its equilibrium freezing temperature. This can be shown by the Clausius-Clapeyron equation [56].

$$\frac{\Delta T_p}{\Delta P} = \frac{T_m \Delta V}{\Delta H} \dots\dots\dots 3.1$$

Where

$T_m$  = Equilibrium melting point.

$\Delta T_p$  = Change in the equilibrium melting point.

$\Delta P$  = Change in pressure.

$\Delta V$  = Change in volume.

$\Delta H$  = Change in enthalpy.

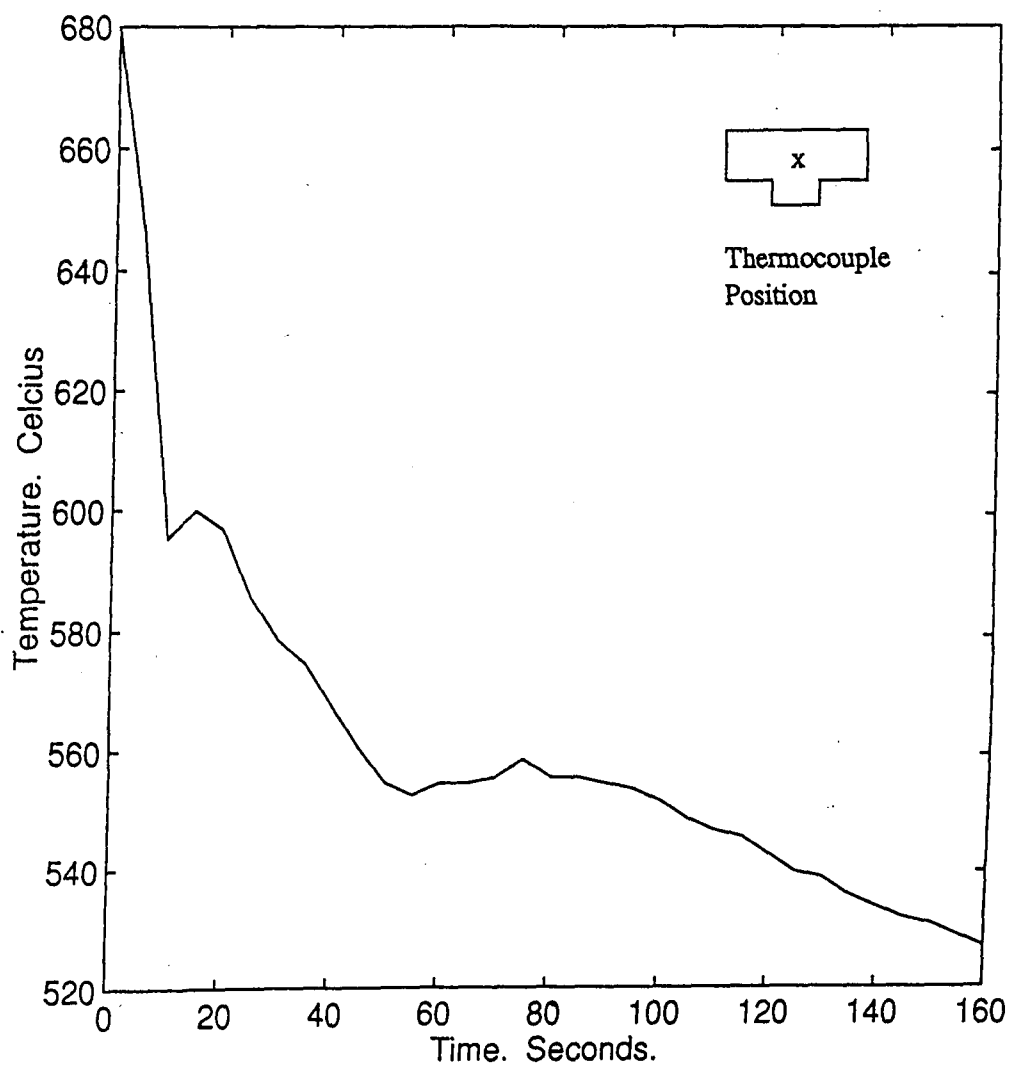
For liquid metals there is a relatively small change in equilibrium melting point of the order of  $10^{-2}$  °C/atm [56]. However for a typical applied pressure between 50 and 150 MPa, as given in the literature, this equates to a temperature rise of between 4.5 and 15 °C. For the present study an applied pressure of 50 MPa was found to be sufficient. A rise in the equilibrium melting point temperature of around 4.5 to 5 °C can therefore be expected.

The cooling rate during solidification affects the microstructure of the final casting. In general the higher the cooling rate the finer the microstructure and the better the mechanical properties. In sand casting the rate of heat removal is slow, figure 3.8, mainly due to the insulating effect of the sand, and a coarse grained structure is produced. A typical microstructure is shown in the photograph, figure 3.9.

Squeeze casting, however, produces a much faster rate of heat removal, figure 3.10, which in turn produces a much finer microstructure, figure 3.11.

The high rate of heat extraction is achieved because the applied load forces the molten alloy into close physical contact with the die thereby creating a very

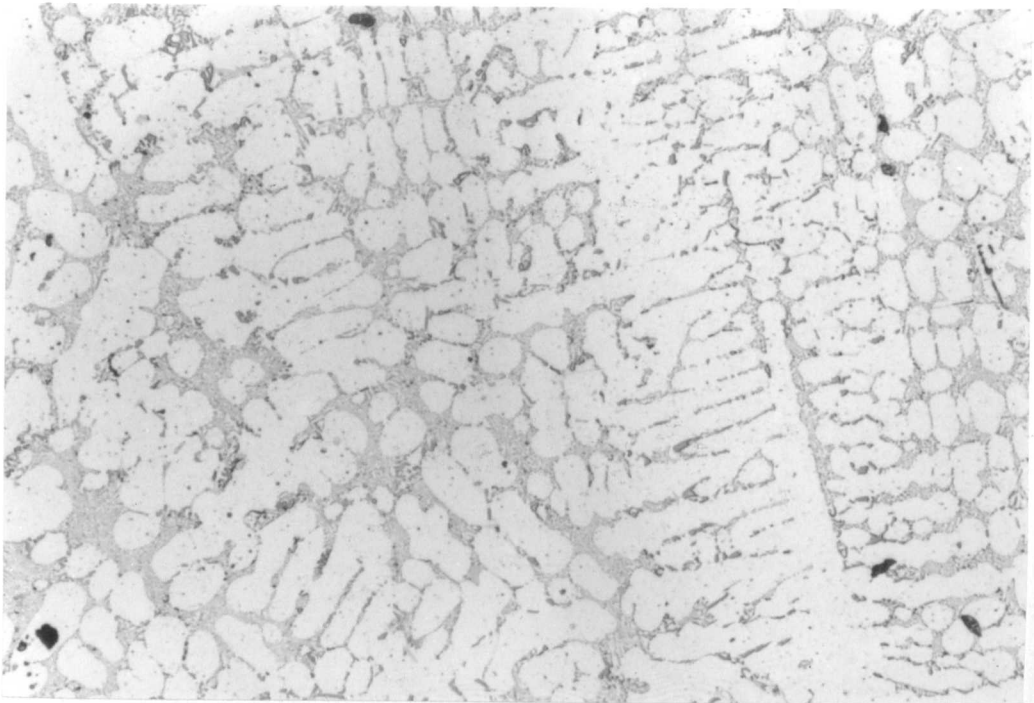
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**Figure. 3.8**

*Typical Cooling Curve for a Sand Cast Test Bar. Material LM25.*

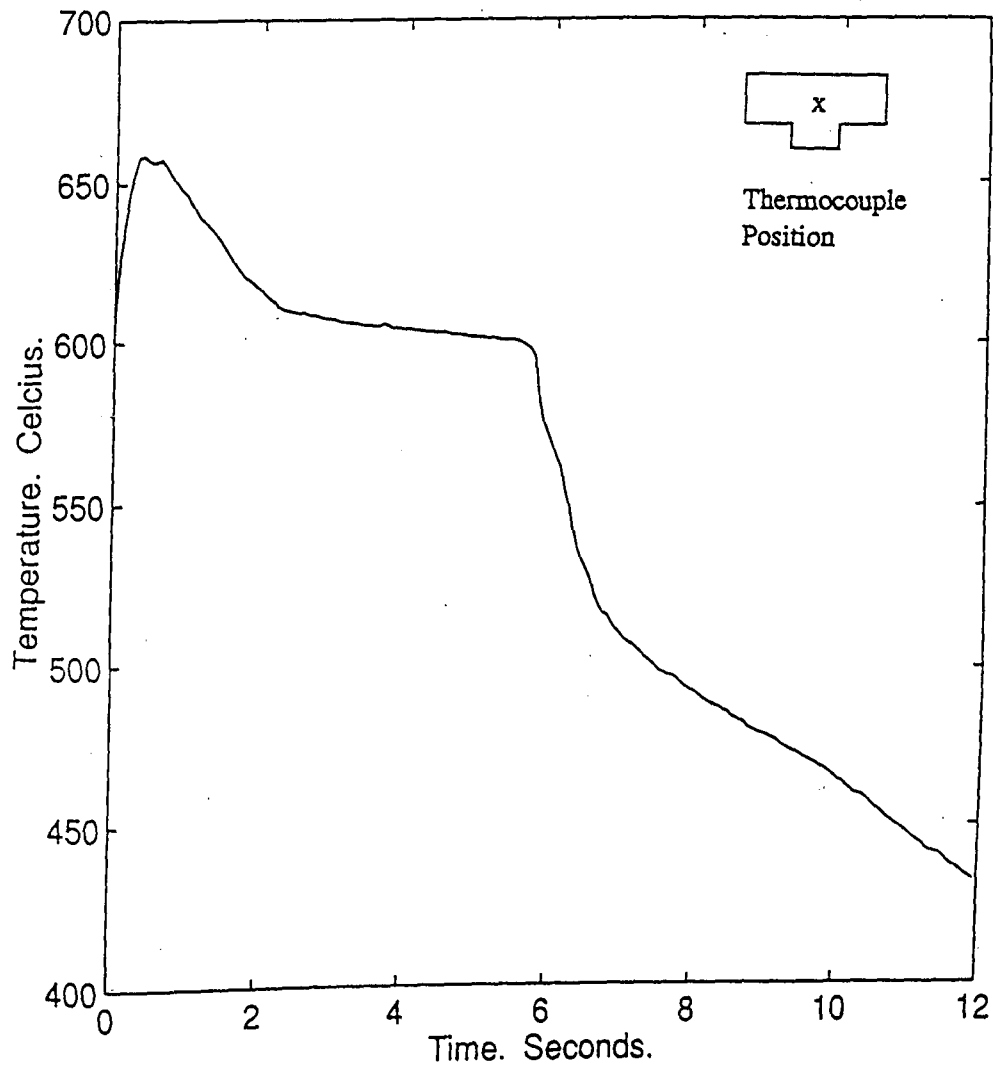
*Thermocouple Placed in the Centre of the Casting*



100  $\mu\text{m}$

**Figure 3.9**

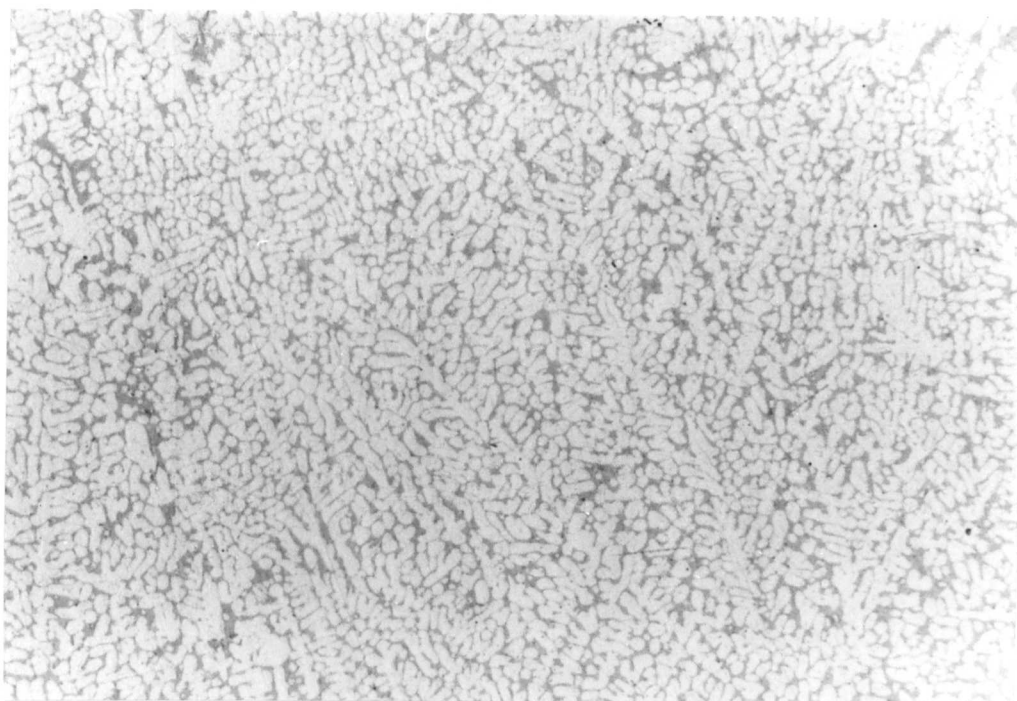
*Typical Microstructure of a Sand Cast Test Bar. Material LM25*



**Figure 3.10**

*Typical Cooling Curve of a Squeeze Cast Test Bar. Material LM25*

*Thermocouple Placed in the Centre of the Casting*



100  $\mu\text{m}$

**Figure 3.11**

*Typical Microstructure of a Squeeze Cast Test Bar. Material LM25*



good thermal contact between the solidifying alloy and the die [67]. With comparatively cold dies, preheated to 250-300 °C, compared to the initial melt temperature, from 620-700 °C for aluminium, there is a significant thermal gradient across the melt/die interface to drive the heat flow. As the load is maintained throughout the whole solidification cycle there is no solidification shrinkage away from the die. Therefore the rate of heat extraction is also maintained throughout the whole solidification cycle.

The isostatic pressure in the liquid alloy is relatively high at 50-150 MPa and this stops the formation of both gas and shrinkage porosity within the casting. Dissolved gasses within the alloy cannot be liberated because this requires a drop in pressure within the casting for the gas bubbles to form, [68]. Likewise shrinkage porosity is suppressed by the large applied load. Any voids which may exist after initial die filling are force filled by the application of the load.

# CHAPTER

## 4

# SEGREGATION

### 4.1 INTRODUCTION

The solidification of an aluminium silicon alloy, as described in chapter 3, was driven by thermal and composition gradients.

Composition gradients can not only drive solidification but they can also be the cause of solute enriched areas within the final casting. The mechanism of segregation will now be discussed.

### 4.2 WHAT IS SEGREGATION ?

Segregation as defined in the Metals Handbook [69] is:

‘A casting defect involving a concentration of alloying elements at specific regions, usually as a result of the primary crystallisation of one phase with the subsequent concentration of other elements in the remaining liquid.’

In general, segregation within the microstructure leads to a deterioration in the mechanical properties of the casting. This is due to the segregated region

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having physical properties which are significantly different from the bulk of the casting.

Once these segregated areas have been formed it can be extremely difficult to remove them by conventional heat treatment techniques. There are two distinct types of segregation; microsegregation and macrosegregation. The control of segregation, both microsegregation and macrosegregation, is therefore of major importance.

### **4.3 MICROSEGREGATION**

Microsegregation refers to segregation on a microscopic scale. That is, on a scale of cells, dendrites and grains.

#### **4.3.1 Cellular Segregation**

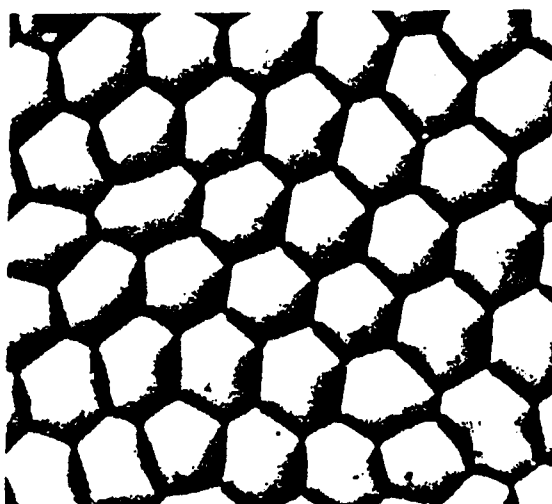
In cellular solidification as the cells grow solute is diffused laterally away from the tip, [63, 70]. This gives rise to a solute enriched region between each cell, on the cell boundary. This cellular structure, in tin, is shown in figure 4.1.

The amount of solute rejected is dependent on the distribution coefficient  $k$ , where  $k$  is the solute composition of the solid,  $C_s$ , divided by the solute composition of the liquid,  $C_l$ .

$C_s$  and  $C_l$  follow the solidus and liquidus lines on the equilibrium phase diagram. If these lines are straight, or can be assumed to be straight, then  $k$  is a

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constant for that particular alloy. When  $k < 1$  the cell boundaries are rich in solute and when  $k > 1$  the cell boundaries are poor in solute [56].



**Figure 4.1**

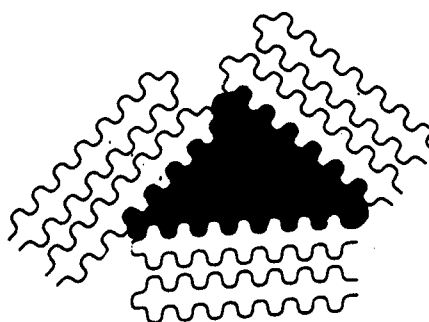
*Growing Cells with a Solute Rich Region Between Each One on the Cell  
Boundary, [71]*

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### 4.3.2 Interdendritic Segregation

Microsegregation, in dendritic structures is also termed coring, because the core of the dendrite is of a different composition to the outside of the dendrite [70]. The same diffusion effects are present as found in the cellular solidification structure as covered above except that here it is between growing dendritic arms as opposed to growing cells.

The solute enriched liquid can also become trapped between the growing dendrite branches, figure 4.2. This can then lead to an even more non uniform distribution of the solute rich liquid as no diffusion in the liquid can occur.



**Figure 4.2**

*Solute Entrapment Between Growing Dendrites*

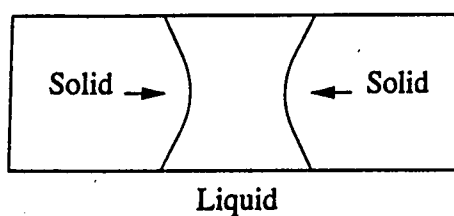
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### 4.3.3 Intergranular Segregation

As the grains solidify so the liquid surrounding the grains becomes enriched with solute. Thus there exists a region which is high in solute at the grain boundaries.

If the grains are growing parallel to each other the solute enriched liquid is pushed ahead of them, i.e. towards the solidification boundary. There will however be some solute enrichment between the grains similar to the cellular structure. It is in the equiaxed zone however that the main feature of inter granular segregation is found, [70].

Here grains growing in an equiaxed fashion produce solute enriched material around the whole grain. As more grains grow and they become larger the remaining liquid becomes ever more enriched with the solute. Figure 4.3 shows two such grain boundaries growing towards each other. The solute enriched material from both grain boundaries is pushed into the region between the two grains. This inter granular region becomes very solute rich, by the combining of the two solute rich layers. In most of these cases the inter granular region is so enriched with solute that the material is usually of a eutectic or peritectic composition, [56, 70, 72].



**Figure 4.3**

*Solute Enriched Area Between Converging Equiaxed Grains, [70]*

#### **4.3.4 Microsegregation During Squeeze Casting**

It has been shown previously that microsegregation is influenced by the growth history of the solid [10, 69, 73]. For equiaxed dendritic solidification an increase in cooling rate leads to an increase in microsegregation, [69]. In unidirectional dendritic solidification, however, similar to that found in squeeze casting an increase in the cooling rate, and hence the solid growth rate, leads to a decrease in microsegregation [59]. Hence the general lack of

microsegregation within squeeze cast components. In a small casting therefore, such as the one used in this work, the very rapid solidification achieved leads to the virtual elimination of all microsegregation.

Microsegregation, in squeeze casting, therefore is not a significant problem when compared to macrosegregation. The next section deals with macrosegregation which is a totally different proposition.

### 4.4 MACROSEGREGATION

Whereas microsegregation refers to segregation over small distances macrosegregation refers to gross differences in composition from one area of a casting to another. The difference in composition is caused by liquid or solid, of a different composition to the mean composition, moving within the mushy zone of the solidifying material [56].

Macrosegregation, in steel ingots, is well known and has been well documented [74, 75, 76, 77]. The effects are similar for all alloys that solidify with a dendritic structure. As such the driving forces for macrosegregation are also similar in castings and ingots of most alloys.

The driving forces for the liquid movement are, [69];

- Contraction of the solid during liquid to solid transformation.

- Gravity effects on density differences caused by phase or compositional changes.

- Formation of gas bubbles.

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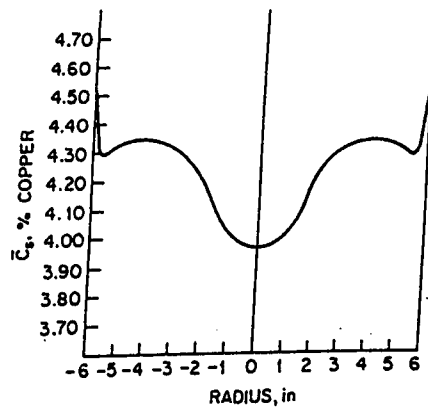
Deformation of the solid phase, from thermal stresses or static pressure.

Capillary forces.

External forces.

#### 4.4.1 Normal (Positive) Segregation

Normal segregation consists of a concentration of alloy constituents that have a lower melting point, than the bulk, in those areas of a casting that solidify last. In the usual course of events the last parts of a casting to solidify are those in the centre, since the cooling starts at the edges adjacent to the die walls and thus the casting solidifies at the walls first. Normal segregation, therefore, is usually found within the centre areas of a casting.



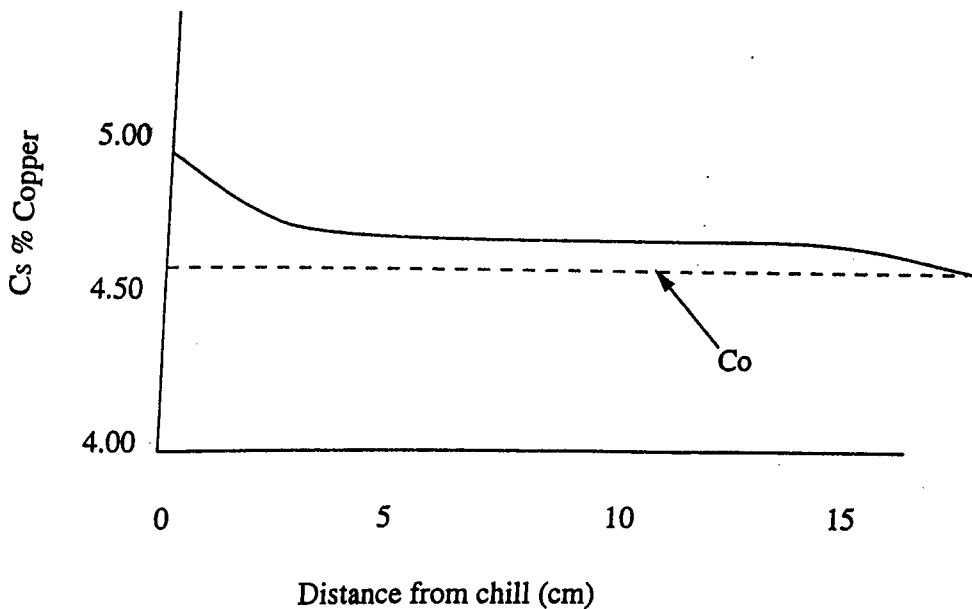
**Figure 4.4**

*Segregation in a Cylindrical Continuously Cast Al-Cu Ingot Produced Under Relatively Poor Casting Conditions. Olins Metals Research Laboratories.[56]*

Figure 4.4 shows the percentage copper across a cylindrical aluminium-copper ingot. The ingot is low in alloy in its centre and high in alloy at the walls.

#### 4.4.2 Inverse (Negative) Segregation

Segregation where an excess of lower melting point constituents is found in areas of a casting that freezes first is termed inverse, or negative, segregation. A solute rich region of this type can be found in the areas of a casting that are close to the die wall in the forms of casting that use metal dies. A chill area is formed when the molten metal hits the much cooler die.



**Figure 4.5**

*Macrosegregation as a Function of Distance in Unidirectionally Solidified*

*Al 4.5 % Cu alloy. [From 78]*

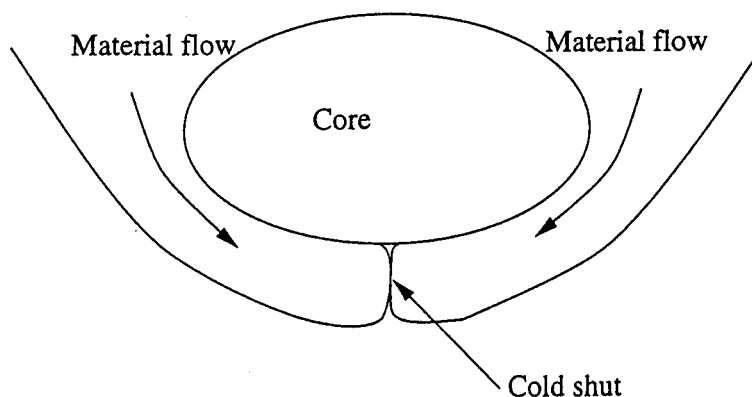
Flemings and Nereo [78] conducted research into unidirectionally solidified Al 4.5 % Cu alloy. The macrosegregation as a function of distance from the chill face was obtained, figure 4.5. Here an alloy with a large freezing range was poured against a chill, with a finite resistance to heat flow. A solute enriched region was obtained adjacent to the chill. Solute enrichment then falls as the distance from the chill face is increased.

#### **4.4.3 Exudation**

A particularly severe form of inverse segregation can occur when a void forms between the mould and the solidifying surface of the alloy, or between two converging flows. The interdendritic liquid can be pushed into the void, driven by solidification shrinkage or gas bubble formation, creating an area which is very rich in solute [56, 69]. The formation of a cold shut, between the two surfaces as shown in figure 4.6, is also a consequence of this problem.

#### **4.4.4 Interdendritic Fluid Flow**

The interdendritic fluid is made to flow, in non pressurised casting processes, by solidification shrinkage, thermal and solute gradients and gravity [69]. In solidification



**Figure 4.6**

*Cold Shut Formed from Converging Flows*

shrinkage as the solid and liquid shrinks a pressure difference is set up between areas of the casting and the interdendritic fluid flows to fill the low pressure area. These same driving forces act in squeeze casting with the additional driving force given by the externally applied load.

The flow of interdendritic fluid in the mushy zone of a casting during solidification is one of the main causes of macrosegregation. The permeability of the mushy zone will have a direct influence on the macrosegregation found within the casting [79]. In the mushy zone, of a solidifying melt, the channels available for the interdendritic fluid to flow are very small. Dendritic arm spacings of 10 to 100  $\mu\text{m}$  are typical [56, 64]. With flow channels this small,

laminar flow may be assumed, [68, 80], especially at low liquid fractions (<0.6)[79]. The mean interdendritic fluid velocity,  $v$ , is governed by Darcy's law

$$v = -\frac{K}{\mu g_l}(\nabla P + \rho_l g_r) \dots\dots\dots 4.1$$

Where

- $v$  = mean interdendritic fluid velocity.
- $K$  = permeability of the medium
- $P$  = pressure
- $g_r$  = acceleration due to gravity
- $\mu$  = viscosity
- $g_l$  = volume fraction of liquid
- $\rho_l$  = density of the liquid

Temperature gradients provide a significant driving force for convection within the liquid even when the temperature gradient is very small. A dimensionless Rayleigh number is used to give a quantitative value to the amount of convective mixing [12].

$$R_a = \frac{g_r \beta R^3 \Delta T}{\alpha \nu} \dots\dots\dots 4.2$$

Where:

$g_r$  = acceleration due to gravity

$\beta'$  = volume coefficient of thermal expansion

$R$  = radius of the fully liquid region

$\Delta T$  = temperature difference in the bulk liquid

$\nu$  = kinematic viscosity

$\alpha$  = thermal diffusivity in the liquid

Rayleigh numbers in excess of  $10^8$  indicate turbulent flow within the interdendritic fluid. Large steel ingots and castings have high Rayleigh numbers during superheat dissipation, [12]. Smaller castings however have a very much smaller  $R$  value and therefore the Rayleigh number will tend to be less than  $10^8$  giving a laminar flow pattern.

In the squeeze casting, of small components, the effect of liquid convection will be to a great extent curtailed because of the speed at which the casting solidifies.

The largest contribution to interdendritic fluid flow in squeeze casting is almost certainly from the externally applied load. This will force fluid to flow into any voids within the die or the solidifying casting even if a skin has formed over the area. It is this phenomenon which is studied further in this particular work..

#### **4.5 MACROSEGREGATION IN SQUEEZE CASTING**

The squeeze casting process is particularly susceptible to macrosegregation. It is the application of a high external pressure, which not only produces many of the inherent advantages of the process but also the macrosegregation.

As the alloy solidifies a dendritic structure begins to grow. The remaining liquid is forced to flow between the dendrites under pressure. This movement of liquid not only produces a very low porosity casting but if the design of the die is such that voids are created during die filling it can produce large segregated areas.

Severe composition gradients can be set up within the casting which can have a significant negative effect on the mechanical properties of the final component. The composition gradients can be so severe that it is not possible to remove them by thermal homogenisation treatments [10].

# **CHAPTER**

## **5**

### **EXPERIMENTAL METHODS**

#### **5.1 INTRODUCTION**

This chapter deals with the experimental approach adopted for this particular work. The first section sets out the experimental philosophy of the project with the remaining sections covering the methods, equipment and materials used.

The results obtained from the experimentation are presented in chapters 6, 7 and 8.

#### **5.2 EXPERIMENTAL PHILOSOPHY**

It became clear from reviewing the literature that there was a lack of understanding of the causes behind the generation of pressure assisted macrosegregation within the squeeze casting process, particularly at features such as webs and corners. The aim of the experimental methods, described in this chapter, is, therefore, to provide information which will give a better understanding of the phenomenon and to provide data which will allow the control or elimination of macrosegregation within small castings.

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It has been established previously, in section 4.4.4, that the flow of the interdendritic fluid during the solidification cycle is one of the main causes of macrosegregation within a casting. Studying the interdendritic fluid within a solidifying squeeze casting will therefore provide the data required. By inserting thermocouples into the alloy, as it solidifies, the temperature profile of the solidifying alloy can be recorded. The temperature profile can then be used to further understand the flow of the interdendritic fluid and thus the causes of macrosegregation in squeeze casting.

Previous researchers have found optimum temperature and pressure ranges for squeeze casting aluminium,[6, 7, 44]. Using this information it was decided to produce test bars using two melt temperatures, 650 °C and 720 °C and two pressures, 50 MPa and 95 MPa. After some initial experimentation it became possible to produce the components in a controlled and consistent way. By using inserts in the die it was possible to produce the component with a web, which ran along the bottom of the component, of either 2, 4 or 8 mm in width, see section 5.4. This then allowed the phenomenon to be further examined.

### 5.3 MATERIAL

The alloys used in the experimentation were supplied in the form of as cast ingots. Two types of material were used.

In the initial stages of the experimentation LM25 alloy supplied by Mil-Ver Metal Company Ltd, Coventry was used. The second material used was an

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aluminium based silicon carbide particulate metal matrix composite. The MMC material was supplied by Hydro Aluminium, Metallurgical R&D Centre, Sunndalsora, Norway. The two alloys were of similar specifications so as to allow a comparison between the base alloy and the metal matrix composite. The manufacturers chemical analysis are given in Table 5.1.

	Si	Mg	Fe	Mn	Cu	Ni	Zn	Pb	Sn	Ti	Al
LM25	7.35	0.53	0.24	0.08	0.01	0.01	0.1	0.02	0.01	0.07	Bal.
MMC	7	0.6									Bal.
Base											

Reinforcement in the metal matrix composite: 20% SiC<sub>p</sub> 20µm

**Table 5.1**

*Manufacturers Chemical Analysis*

## 5.4 THE COMPONENT

The component produced is a simple test bar, figure 5.1. The bar was originally designed to fit a tensile test machine owned by the engineering department [12]. To provide the means for the macrosegregation phenomenon to be studied a further three die inserts were designed and made to enable the production of test

bars with a web, of either 2, 4 or 8 mm width, along the middle of the bottom of the component, figure 5.2.

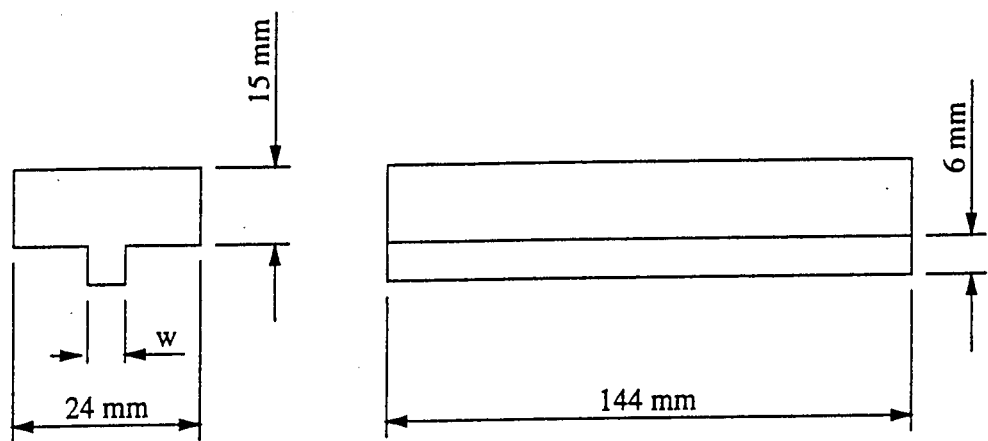
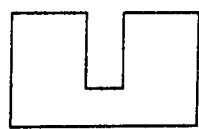
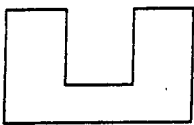


Figure 5.1

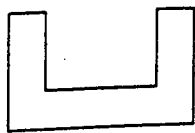
*Component Produced in Squeeze Casting Facility*



2mm wide web



4mm wide web



8mm wide web



Corners with 2mm radii

Figure 5.2

*Inserts Used to Produce Webs*

## **5.5. EQUIPMENT**

### **5.5.1 Furnaces**

Two types of furnace were available. The alloys were initially melted in an induction furnace. If required the molten alloys could be held at constant temperature in a radiant type furnace.

### **5.5.2 Press**

A single action press capable of a maximum applied pressure of 95 MPa, over the plan area of the die, formed the basis of the squeeze casting facility. The majority of the facility was designed and built as part of previous third year projects [12, 13].

Some parts were however redesigned as part of the present work and new parts added. The main new addition for the present work was the introduction of thermocouples into the melt itself. This allowed the production of cooling curves for the materials under research, see section 5.5.3.

The press has heated plattens and to these are fixed the upper and lower heated dies to form the completed die assembly. Thermocouples are installed in the dies and plattens to enable close control of the die temperature. A temperature controller, running dedicated software, keeps the temperature of the dies within plus or minus 10°C of the preset value

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The press functions are also operated by a programmable controller. A complete cycle can be programmed in and run at the touch of a button. The program includes:

Maximum load.

Ramp up time. (Time taken to close the dies and apply maximum load selected.)

Dwell time. (Time component is kept under full load for complete solidification to take place.)

Ramp down time. (Time to open the dies and return ready for next cycle.)

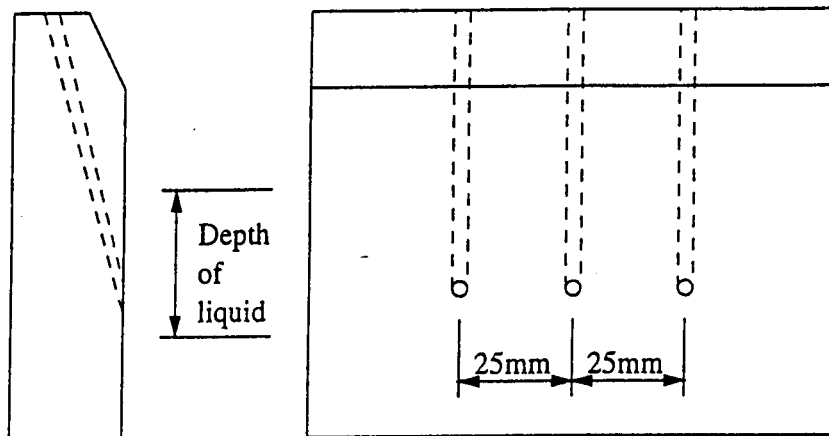
The component is then ejected by hand using a thread driven mechanism.

### **5.5.3 Temperature Measurement.**

One of the die liners was modified to enable the introduction of three thermocouples into the melt. This would allow the temperature of the melt to be recorded during the solidification cycle and enable the production of cooling curves. Three 2 mm diameter holes were formed in one of the die liners by the electro discharge machining process, figure 5.3.

The holes were made just large enough for the sheathed thermocouple to pass through. This close fit prevented any significant amount of molten alloy from being forced into the hole alongside the thermocouple wire. No further sealing

of the holes was therefore required. Type K, nickel-chromium/nickel-aluminium, thermocouples were used.

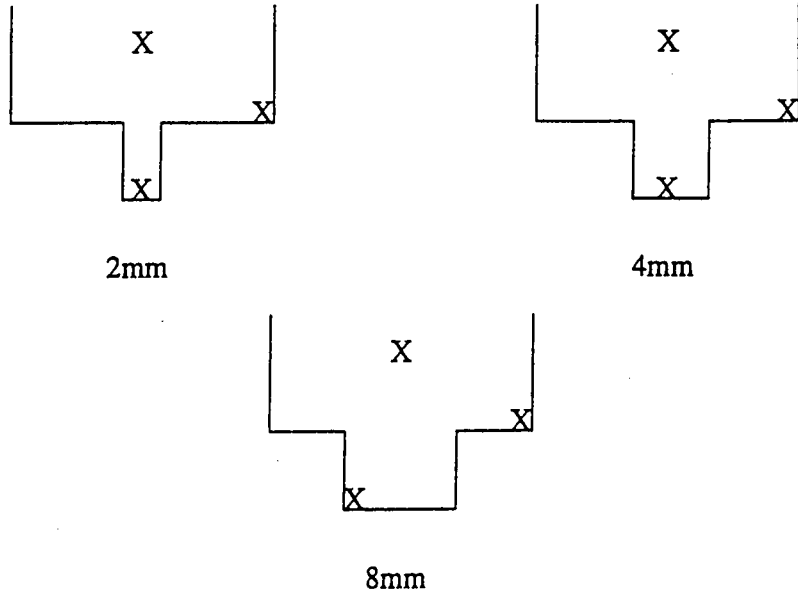


**Figure 5.3**

*Position of Holes in the Die Liner to Enable the Thermocouples to Penetrate the Component During the Solidification Cycle*

#### **5.5.4 Data Capture and Manipulation**

The three thermocouples were placed into position as shown in figure 5.4 and connected to a Comark Compuface 7000 data capture unit. A p.c. running a GW Basic program, written specifically for the purpose, controlled the whole data capture and storage process. A copy of the GW Basic program can be found in Appendix 1.



**Figure 5.4**

*Positions of Thermocouples in the Casting*

Thermocouple one, placed within the bulk of the casting, was used as a trigger to start the data capture. It was set, via the GW Basic program, to sample the temperature at intervals of 200 ms. When the temperature exceeded a preset level, 180° C, the data capture program was triggered to start. This occurred when the molten alloy being poured into the die came into contact with the trigger thermocouple.

The data capture program then set the three thermocouples to sample the temperature, in turn, at regular intervals. The delay between thermocouples was 20 ms giving a gap between readings, for the same thermocouple of 60 ms.

The departmental Unix system running Matlab was used to manipulate, smooth and display the data in a graphical form to produce thermal cooling curves. These can be found in chapter 6.

## **5.6 MELTING AND CASTING PROCEDURE**

Prior to melting the alloy the dies were brought up to their operating temperatures, 250°C for the top die and 270°C for the bottom die. The transfer ladle was preheated to 720°C to cut down on the heat loss from the alloy between the furnace and the squeeze casting facility. The ladle was returned to the heating furnace between each casting cycle. Using a heated ladle, of known size, a measured amount of the molten alloy was transferred to the squeeze casting facility and the casting process started.

There was found to be some heat loss, from the alloy in the ladle, between the furnace and the activation of the temperature recording. A drop in temperature of approximately 30°C was noted. The melt in the furnace therefore was kept at 30°C higher than the temperature being studied. It was found from experimentation that this increase in temperature did not have a detrimental effect on the microstructure or results.

Coverall flux was added to the melt to stop oxidation of the melt surface, see Appendix 2. This was removed and replaced as necessary during the casting operation.



### **5.6.1 Melting and Casting of LM25**

For each casting session 4-5 Kg of LM25 alloy was melted in a silicon carbide crucible in an induction furnace. On reaching a temperature of 650°C the LM 25 alloy was degassed by the addition of a standard degassing tablet, see Appendix 2. After skimming and covering with the coverall flux the alloy was brought to the temperature required, for the particular casting parameters, and held at that temperature for the duration of the session. Two casting temperatures were used; 650 and 720°C.

A preheated ladle of the correct size for the die was then used to transfer the molten alloy to a preheated die mounted on the plattens of a press.

### **5.6.2 Pressing Parameters**

After pouring the alloy into the bottom half of a die the press was started manually. The software, controlling the operation of the press, then took over closing the die. The specified load was then applied, held for a specified time and then released. Two pressures were used, 50 MPa and 95 MPa.

There was an unavoidable time delay between the alloy being poured into the die and the full load being applied. At any one time this was a minimum of 5.5 seconds.

The full load was sustained for 15 seconds by which time the component was solid. Ejection of the component is then achieved manually.

### **5.6.3 Casting with a Particulate Metal Matrix Composite**

The procedure for the particulate MMC was the same as for the LM25 alloy except for the degassing. No degassing was carried out as this can pull the silicon carbide particles out of suspension along with the dissolved gasses, [40].

## **5.7 METALLOGRAPHY**

The components were rough cut into smaller samples by hand hacksaw, final shaping before mounting was done with a diamond slitting wheel. The samples were then mounted in resin using a Prontopress-2 press. Grinding and polishing was carried out on a Pedemax 2 machine. The grinding and polishing procedure followed is shown in Appendix 3.

Optical examination was carried out using a Nikon Optiphot microscope and Seescan image analysis software. Photographs of the microstructure were taken using a Nikon FX-35A camera mounted on the microscope.

# **CHAPTER**

## **6**

### **MACROSEGREGATION WITHIN LM25**

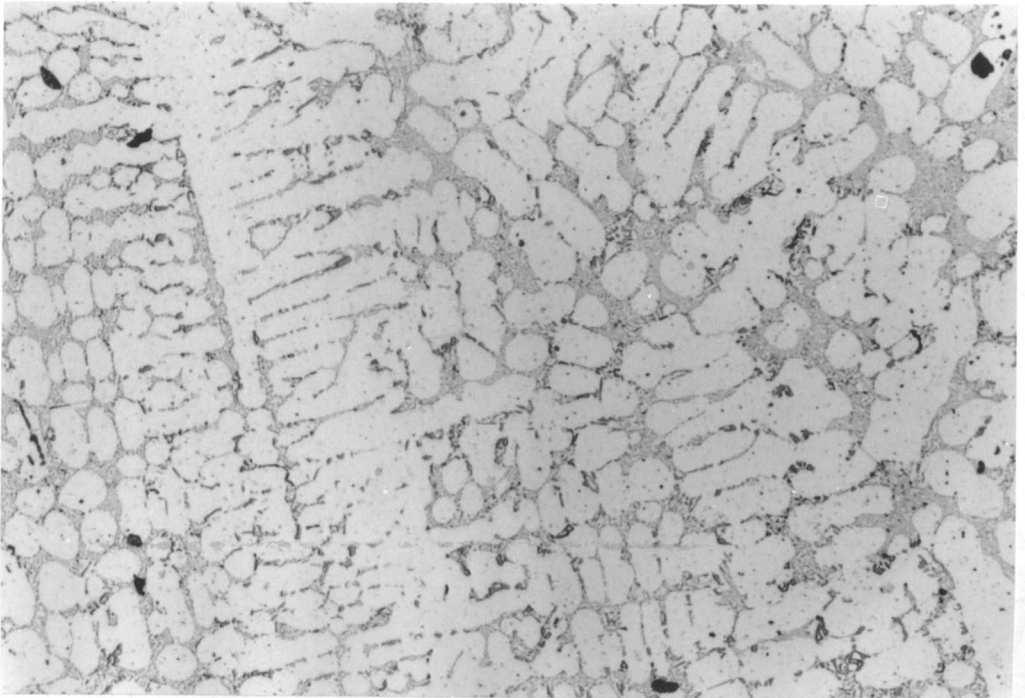
#### **6.1 INTRODUCTION**

This chapter presents the metallographic results obtained after the squeeze casting of LM25 aluminium alloy. The casting parameters for each sample are given along with the metallographic data relevant to each microstructure.

The chapter is opened with a comparison between the microstructures of a sand casting and a squeeze casting. A series of photographs is then presented showing macrosegregation within the microstructure of a small squeeze cast component. The effects of temperature, pressure and geometry of the component on the macrosegregation are covered.

#### **6.2 COMPARISON OF MICROSTRUCTURES**

Figures 6.1 and 6.2 show the typical microstructures, in LM25 aluminium alloy, obtained from a sand casting and a squeeze casting respectively. The relevant thermal cooling curves are in figures 7.1 and 7.2, chapter 7.



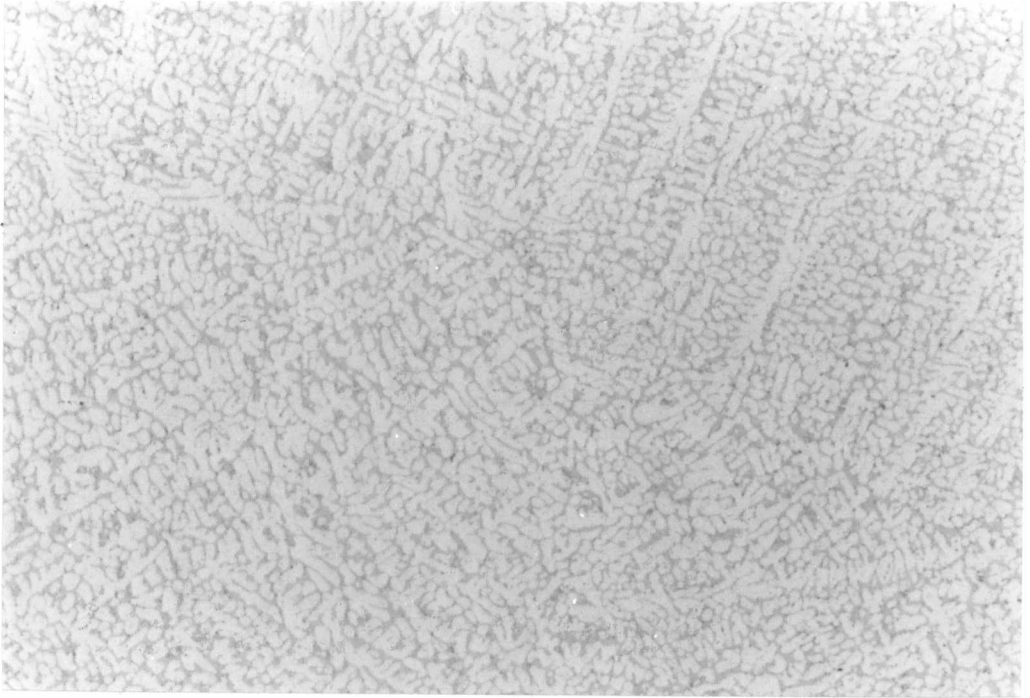
100  $\mu\text{m}$

**Figure 6.1**

*Typical Microstructure of a Sand Casting*

Melt temperature      720°C

Cooling curve Figure 7.1



100  $\mu\text{m}$

**Figure 6.2**

*Typical Microstructure of a Squeeze Casting*

Melt temperature      650

Squeeze pressure      50 MPa

Cooling curve Figure 7.2

The sand cast microstructure, figure 6.1, is, as was expected, of a dendritic nature. The dendrite structure is fairly coarse with the primary dendrites growing in straight lines along the growth directions. Secondary and tertiary dendritic growth is also visible. There is some evidence of porosity within the microstructure, which can sometimes be a problem in sand cast aluminium.

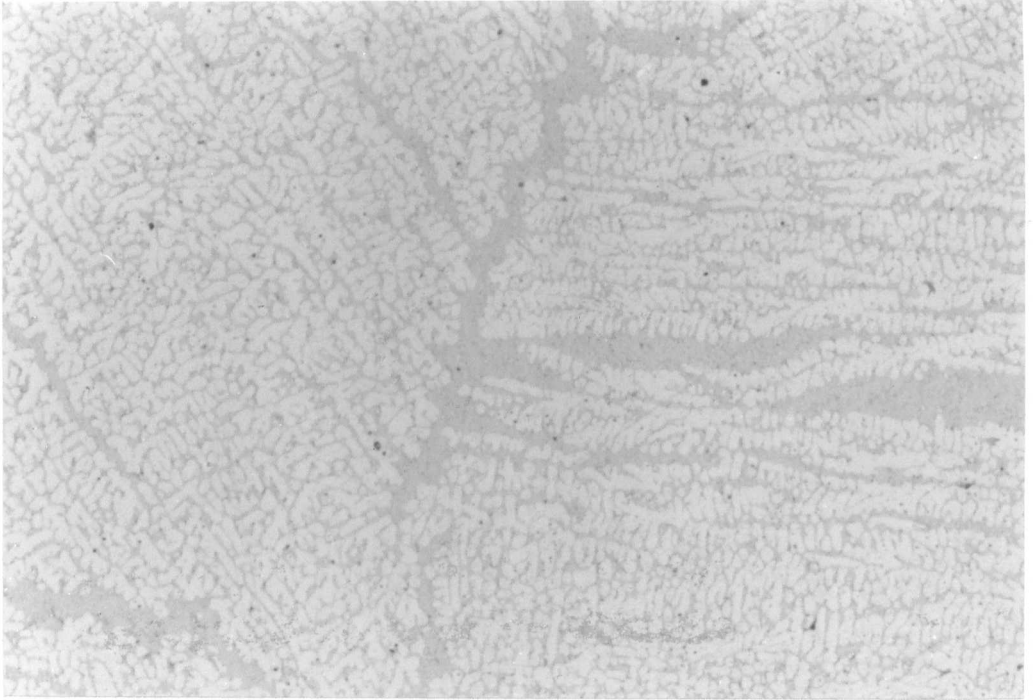
The squeeze cast microstructure, figure 6.2, has a dendritic structure much the same as the sand casting but is very much finer, note the magnification of each is the same (80X). The growth directions are visible as above but because of the fine size of the structure and the growth rate achieved the secondary dendrites are very small and the tertiary dendrites virtually non existent. There is very little porosity evident within the microstructure.

### **6.3 MACROSEGREGATION**

Figures 6.3, 6.4 and 6.5 show macrosegregation within a squeeze cast component. The segregated areas are easily seen as they have little, or in some cases, no aluminium dendrites visible, the area being rich in solute, which in LM25 is mainly eutectic. The segregated area can also be very large. In the thin web case, figure 6.5. the whole web is filled with the solute rich material, the web being 2 mm wide and 6 mm deep.

Figures 6.3 and 6.4 show a number of solute rich strands, or canals. One can be seen to run vertically and the others run horizontally. The largest of the horizontal areas is located at the entrance to a 4 mm wide web on the casting,

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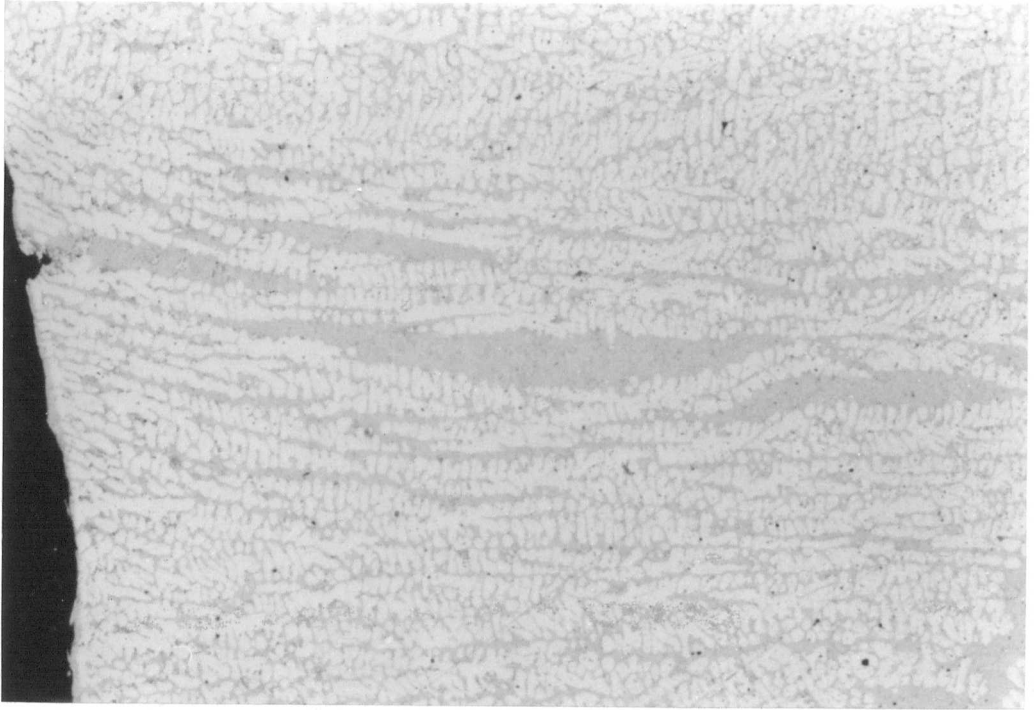


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**Figure 6.3**

*Stands or Canals Within the Microstructure*

Melt temperature	650°C
Squeeze pressure	50 MPa



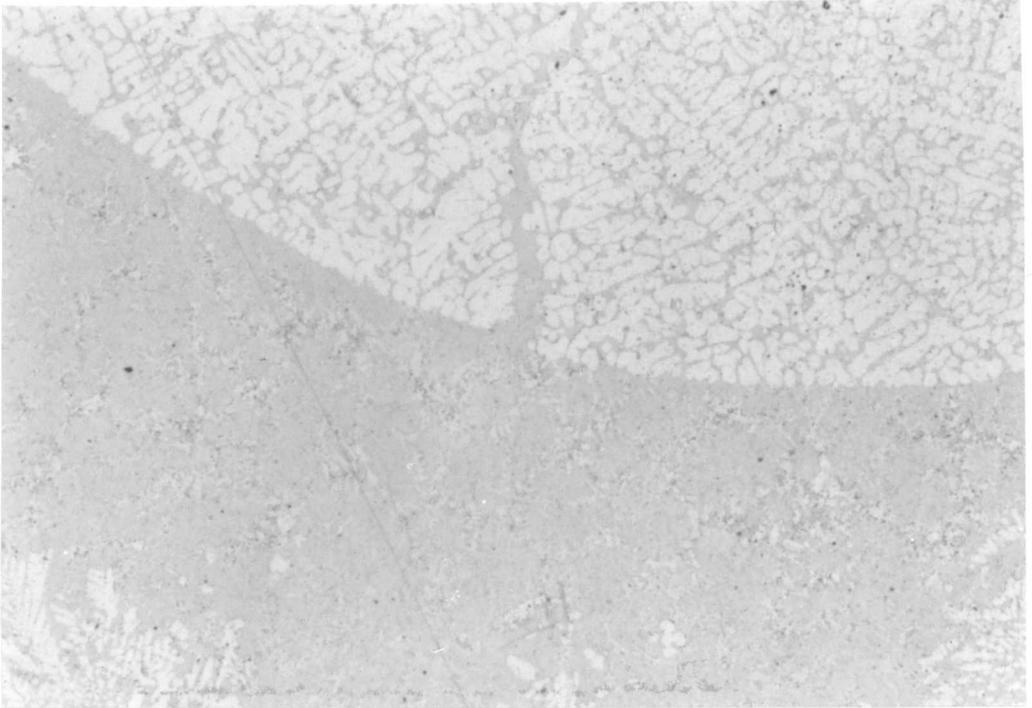
100  $\mu\text{m}$

**Figure 6.4**

*Solute Rich Area Created by a "Hot Tear"*

Melt temperature	650°C
Squeeze pressure	50 MPa





100  $\mu\text{m}$

**Figure 6.5**

*Very Large Solute Rich Area in the Web, Showing the Circular Boundary  
between the Bulk Material and the Segregated Web.*

Melt temperature      650°C  
Squeeze pressure      50 MPa  
Web size (width)      2 mm  
Cooling curve Figure 7.6

the corner of which can be seen in figure 6.4. The area is most likely a "hot tear" that has been filled with solute rich interdendritic material on the application of the external load. It can be seen that the growth direction of the primary dendrites have been deformed. One primary dendrite, in figure 6.4 in particular, has been grossly deformed in comparison to its neighbours. The dendrite starts on the die wall, growing from left to right away from the wall, on the lower part of the solute rich area. It has been pushed down, along with its neighbours, and then it turns upwards across the solute rich area to the top edge of the area. This shows that the area was started by tearing the microstructure apart during solidification, i.e. a "hot tear". The tear is then filled with interdendritic material which is forced into it by the application of the external load. The fluid enters from a point of least resistance and continues to flow until the area is completely full. As the fluid material originates from a hotter, liquid area of the casting it can cause melting of the microstructure in close proximity to the fluid flow which is then carried away by the flowing material. This creates another canal within the microstructure. The vertical canal in the photograph of figure 6.3 is one of these feed canals. This particular canal continues on down to the bottom of the web and feeds a segregated area in a concave corner.

Figure 6.5 shows another feed canal which is, in part, responsible for the vast area of segregated material at the bottom of the photograph. The photograph graphically shows the boundary between the bulk of the material and a fully segregated 2 mm wide web.

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The circular boundary in figure 6.5 is formed when the molten alloy is poured into the die. Surface tension prevents the alloy from filling the web and the interdendritic fluid is forced into the web on the application of the external load in much the same manner as described above for the "hot tear". The boundary has a radius of approximately 2 mm.

#### **6.4 THE EFFECT OF TEMPERATURE ON THE MACROSEGREGATION**

Two temperatures were used to see if an increase in temperature affected the macrosegregated areas. A lower temperature of 650 °C, 40 °C above the liquidus, was chosen. Attempts to squeeze cast below this temperature proved unsuccessful as the alloy solidified in the die before the application of the pressure. A higher temperature of 720 °C was chosen as this is the usual pouring temperature for sand casting LM25. (Note that this temperature is also higher than would normally be used during squeeze casting as one of the benefits of the process is the ability to use low temperatures during casting.)

The microstructure of the low temperature casting was shown in figure 6.5, section 6.3 and the microstructure of the high temperature casting is shown in figure 6.6.

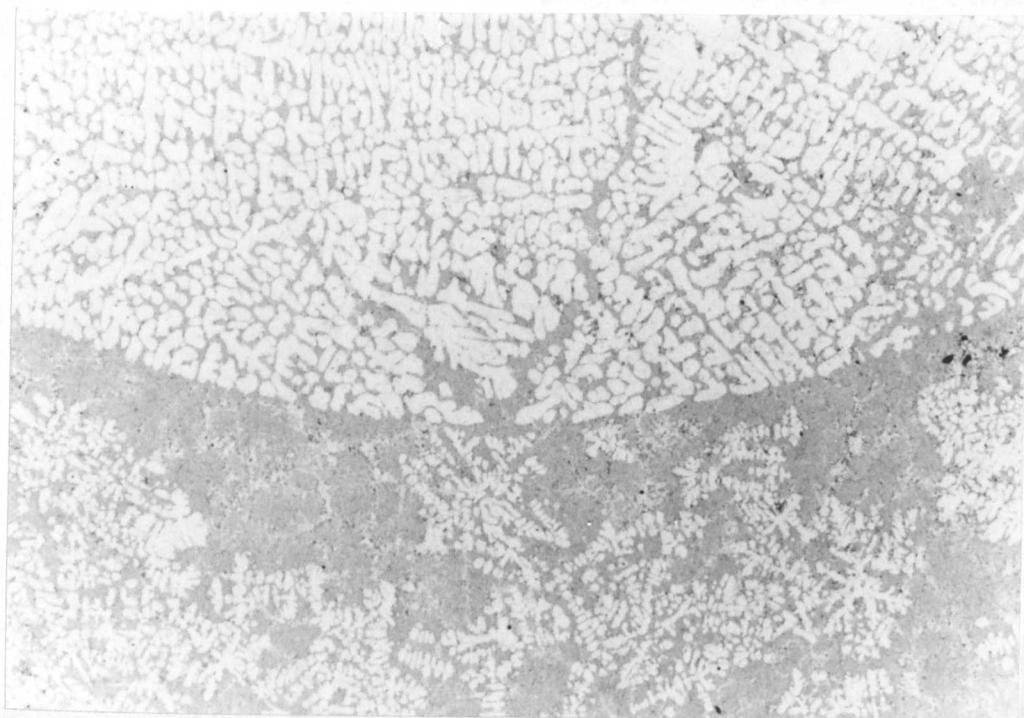
The two microstructures are very similar with both showing a smooth curved boundary between the main body of the casting and the segregated area in the web. The grain size is similar in both as is the amount of segregation. The higher temperature casting appears to have slightly more  $\alpha$  aluminium within the

---

segregated area. However taken over the whole section of the web the ratio of  $\alpha$  aluminium present within the segregated area is similar in both microstructures.

Both microstructures have a vertical channel of segregated material which extends into the main body of the casting. These channels fed the segregated area with interdendritic material from the main body from the time the load was applied to final solidification.

As with the previous case, figure 6.5, the radius of the boundary is approximately 2 mm.



100  $\mu\text{m}$

**Figure 6.6**

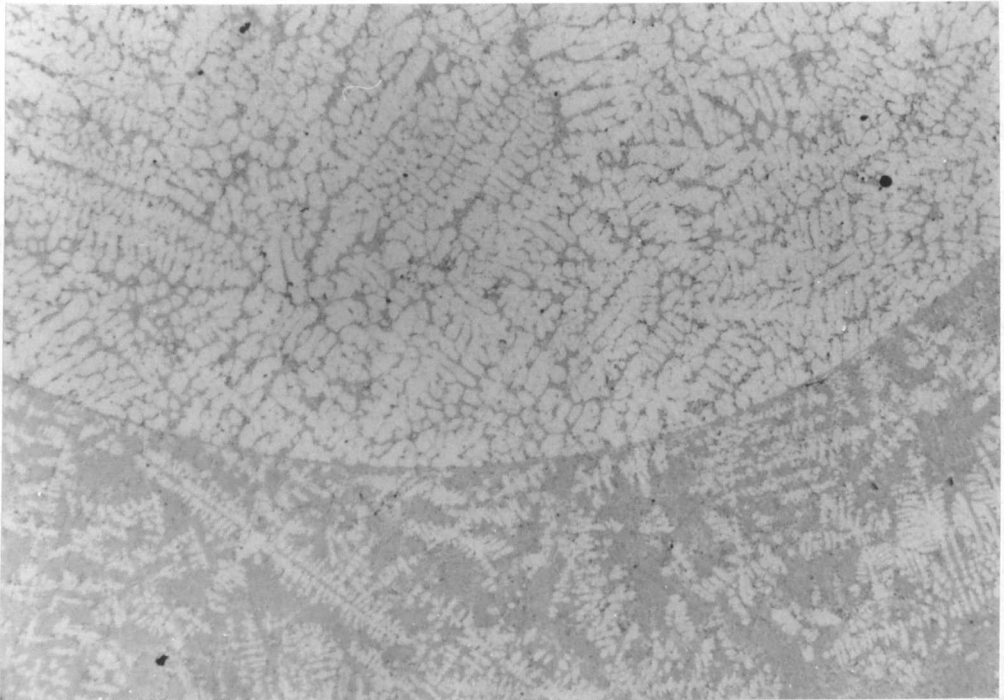
*Solute Rich Area in the Web of a Casting. Casting Temperature 720°C*

Melt temperature	720°C
Squeeze pressure	50 MPa
Web size (width)	2 mm
Cooling curve	Figure 7.7

## 6.5 THE EFFECT OF PRESSURE ON THE MACROSEGREGATION

Two pressures were used to test the effect of pressure on the macrosegregation. A minimum pressure of 50 MPa and a maximum pressure of 95 MPa were chosen. The minimum pressure is the recommended minimum from the literature, chapter 2, and the maximum pressure is the maximum that the particular press used can deliver with the particular die set. The pouring temperature was set at 650 °C as the previous section has shown that the temperature has no significant effect on the segregation. The microstructures are in figures 6.5 and 6.7 for the 50 MPa and 95 MPa pressures respectively.

The microstructure of 6.7 is very similar to 6.5 with the smooth curved boundary very prominent. Once again the grain sizes are similar as is the amount of segregation. The radius of the boundary is also approximately 2 mm as before.



100  $\mu\text{m}$

**Figure 6.7**

*Microstructure of the Web in a High Pressure Casting*

Melt temperature	650°C
Squeeze pressure	95 MPa
Web size (width)	2 mm
Cooling curve	Figure 7.8

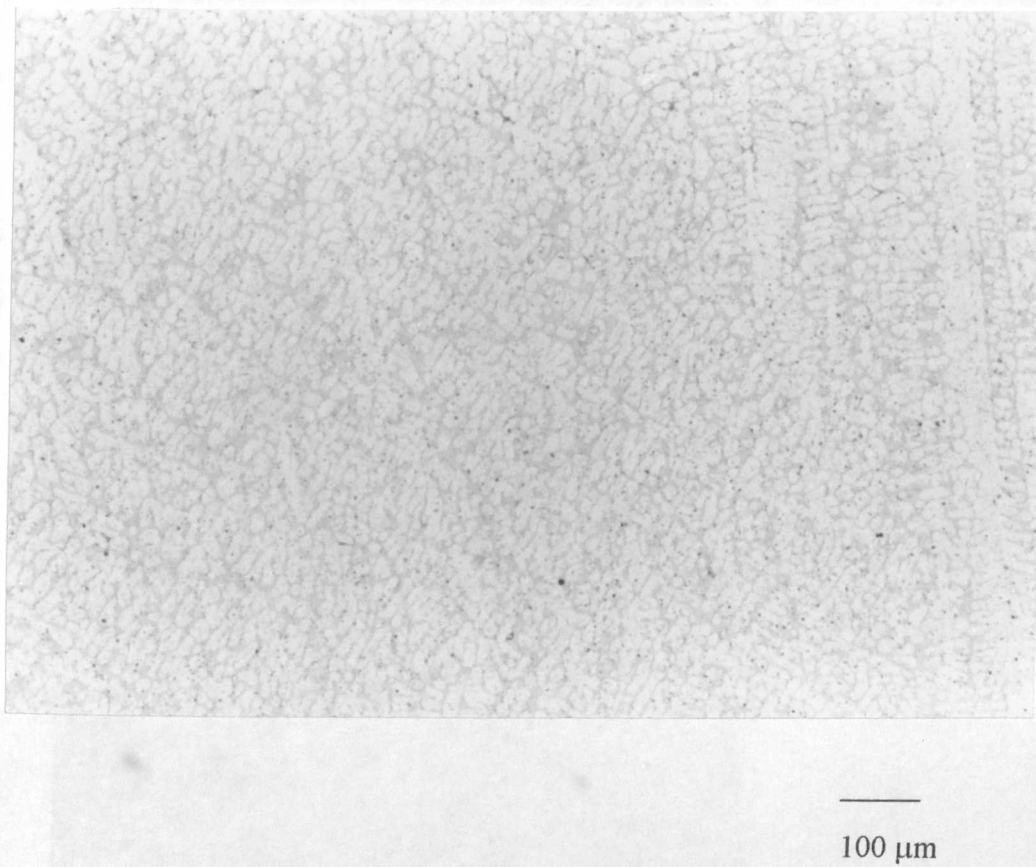
## 6.6 EFFECTS OF USING WIDER WEBS

All of the examples so far, figures 6.2 to 6.7, show the macrosegregation within a 2 mm wide web. The effect, on the macrosegregation, of widening the web is shown in this section. The casting parameters used for this assessment, are a temperature of 650 °C and an applied pressure of 50 MPa. These values were obtained from the work in the previous sections and are the settings that are the most common for use in an industrial process.

Figures 6.8 and 6.9 show the microstructure from a 4 mm wide web. The 4 mm web in figure 6.8 shows no sign of segregation within the main part of the web. The microstructure is of a fine dendritic nature similar to the main body of the casting. Figure 6.9, also from a 4 mm web, shows a small amount of segregation on the wall of the web close to the top corner. Within the segregated area is a very large area of porosity.

The 8 mm wide web, figure 6.10, on the other hand shows no sign of the segregation at all any where within the main body of the web. The segregation within this size of web was only found in the concave corners at the bottom of the web. The corners of the main body of the casting also had some segregation within them. Segregation in concave corners not only of the webs but also the main body of the casting is shown in the next section, 6.7.

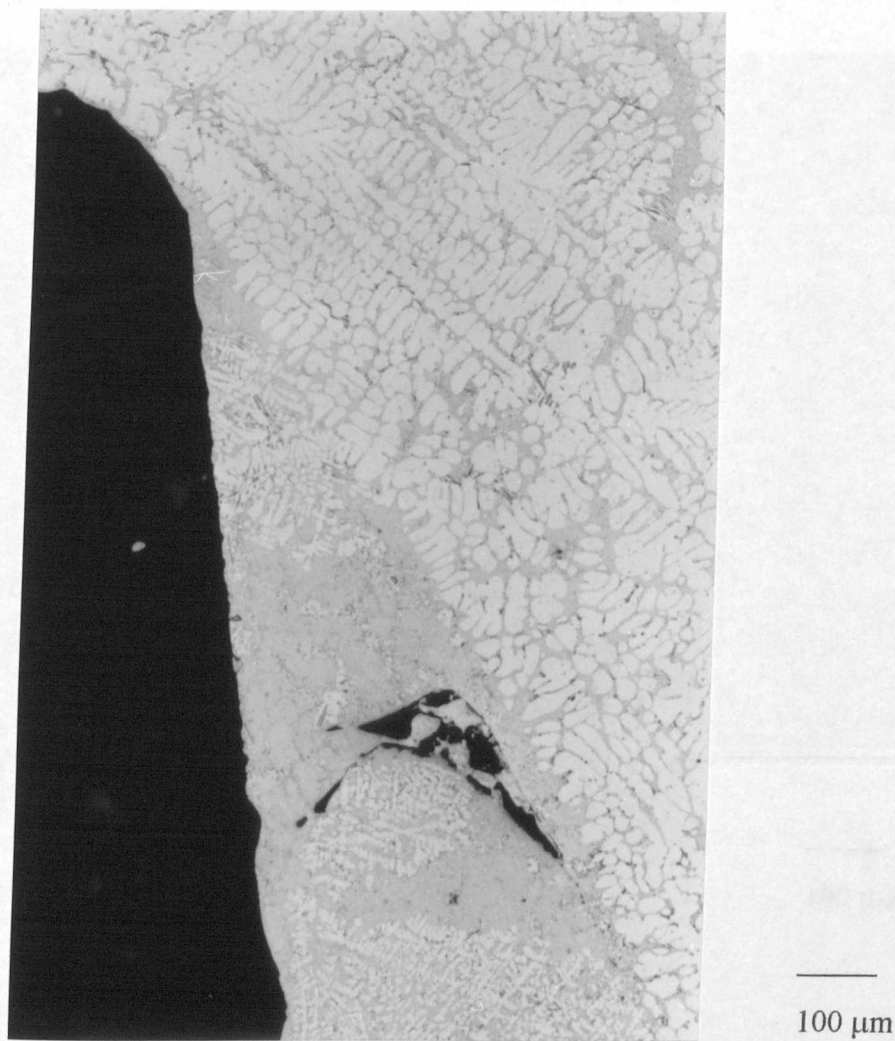




**Figure 6.8**

*Microstructure of 4 mm Wide Web, Showing no Macrosegregation in the Main Part of the Web*

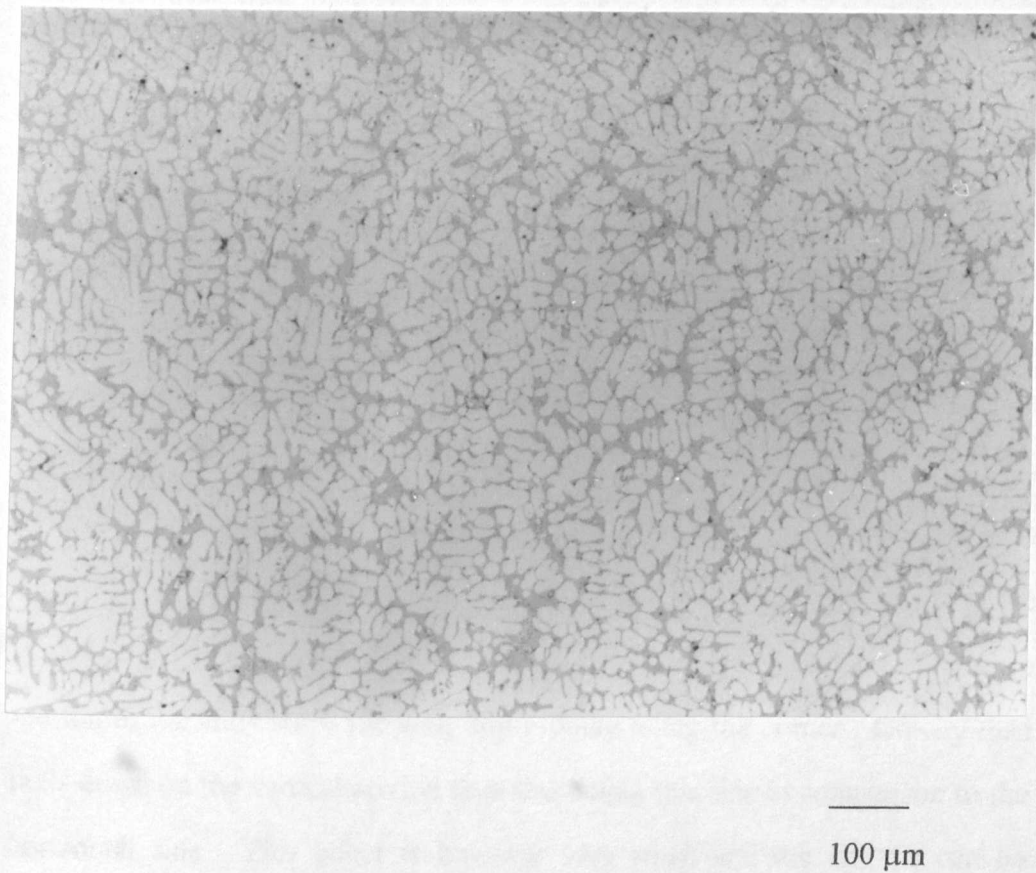
Melt temperature	650 °C
Squeeze pressure	50 MPa
Web size (width)	4 mm



**Figure 6.9**

*Microstructure of 4 mm Wide Web, Showing Some Macrosegregation in the Main Part of the Web*

Melt temperature	650°C
Squeeze pressure	50 MPa
Web size (width)	4 mm



**Figure 6.10**

*Microstructure of 8 mm Wide Web, Showing no Macrosegregation Within the Main Body of the Web*

Melt temperature	650°C
Squeeze pressure	50 MPa
Web size (width)	8 mm

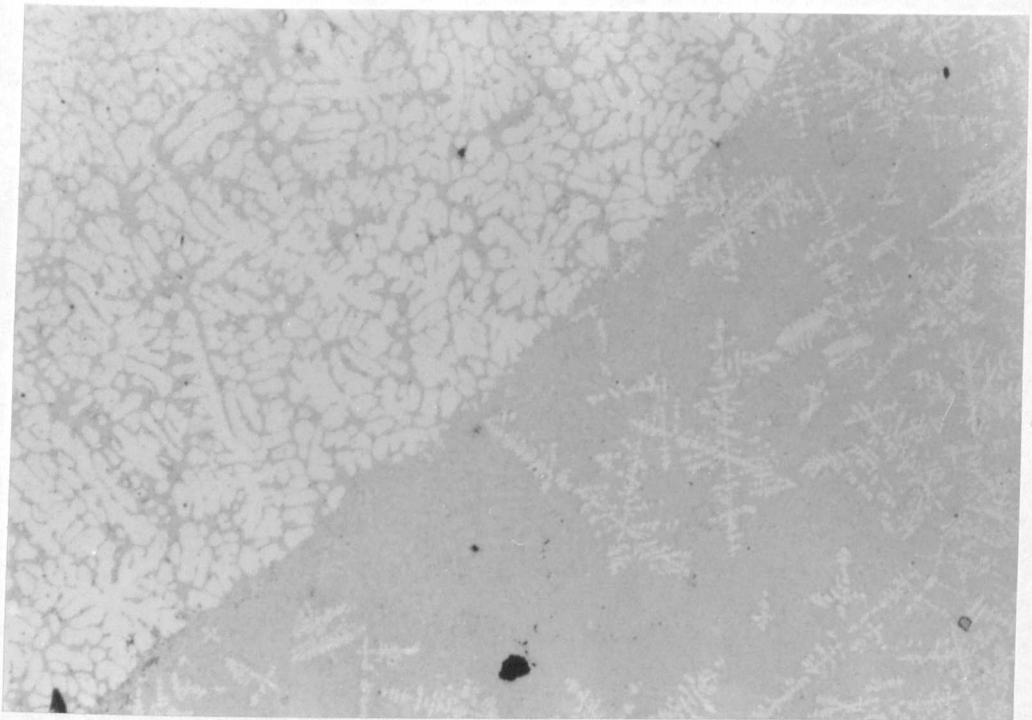
## 6.7 MACROSEGREGATION WITHIN THE CORNERS OF THE CASTING

As well as in the webs, macrosegregation was found to occur in the concave corners of the castings. Figures 6.11, 6.12 and 6.13 show the macrosegregation found in the corners of a casting. The segregation was found to occur in the corners on the main body of the casting and the corners of the larger webs.

The macrosegregation is of the same type as the web segregation and includes the very visible curved boundary between the segregation and the main body microstructure. The boundary in these cases though is not quite as circular as in the webs where each side of the web is at the same level. Here one side of the web is on a horizontal surface and the other is on a vertical surface. The surface tension of the alloy stops the alloy from totally filling the corner. Gravity then pulls down on the vertical section thus shortening this side in comparison to the horizontal side. This effect is however very small and the corners can be regarded as part of a circle.

All of the segregated areas, in the webs, corners and the hot tear, are fully fused to the main body of the casting. The only sign of porosity or other matter on the boundary is in figure 6.11. Here there is a small amount of porosity and what looks like silicon growth.





100 μm

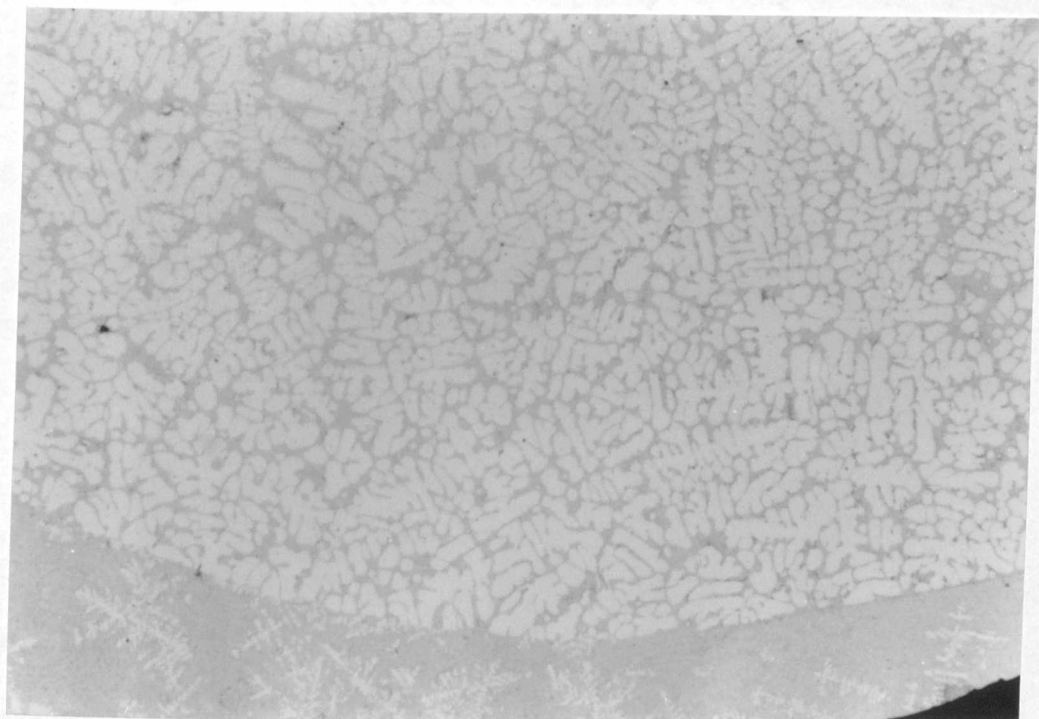
**Figure 6.11**

*Macrosegregation Within the Corner of a Casting. Low Temperature*

Melt temperature      650°C

Squeeze pressure      50 MPa

Cooling curve Figure 7.9

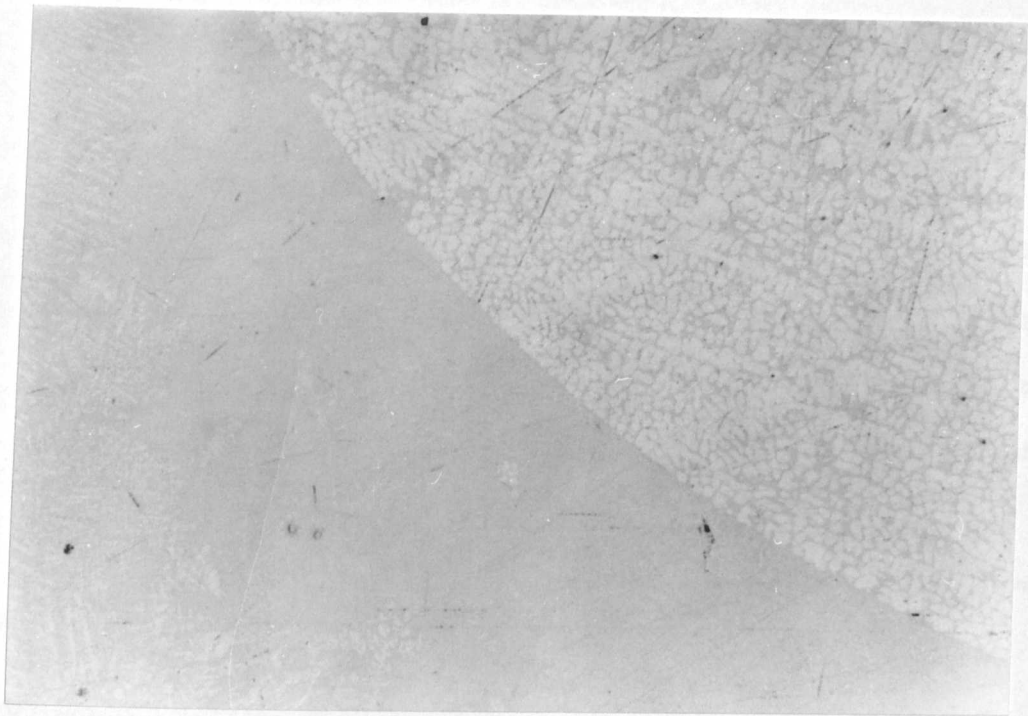


100  $\mu\text{m}$

**Figure 6.12**

*Macrosegregation Within the Corner of a Casting. High Temperature*

Melt temperature      720°C  
Squeeze pressure      50 MPa  
Cooling curve Figure 7.10



100 μm

**Figure 6.13**

*Macrosegregation in a Corner of a Casting. High Pressure*

Melt temperature      650°C

Squeeze pressure      95 MPa

Cooling curve Figure 7.11

## **6.8 SAMPLE WITH NO PRESSURE APPLIED**

Figure 6.14 shows the cross section of a casting which was produced with no pressure being applied to the top surface.

A small curved protrusion is all that exists of the web. The web has not been formed as the alloy has not been able to flow into the narrow web. The radius of this protrusion is between 1.5 and 2 mm. The top of the component has been machined. The two bottom corners are rounded similar to the web. The radius of these corners is also 1.5 to 2 mm.

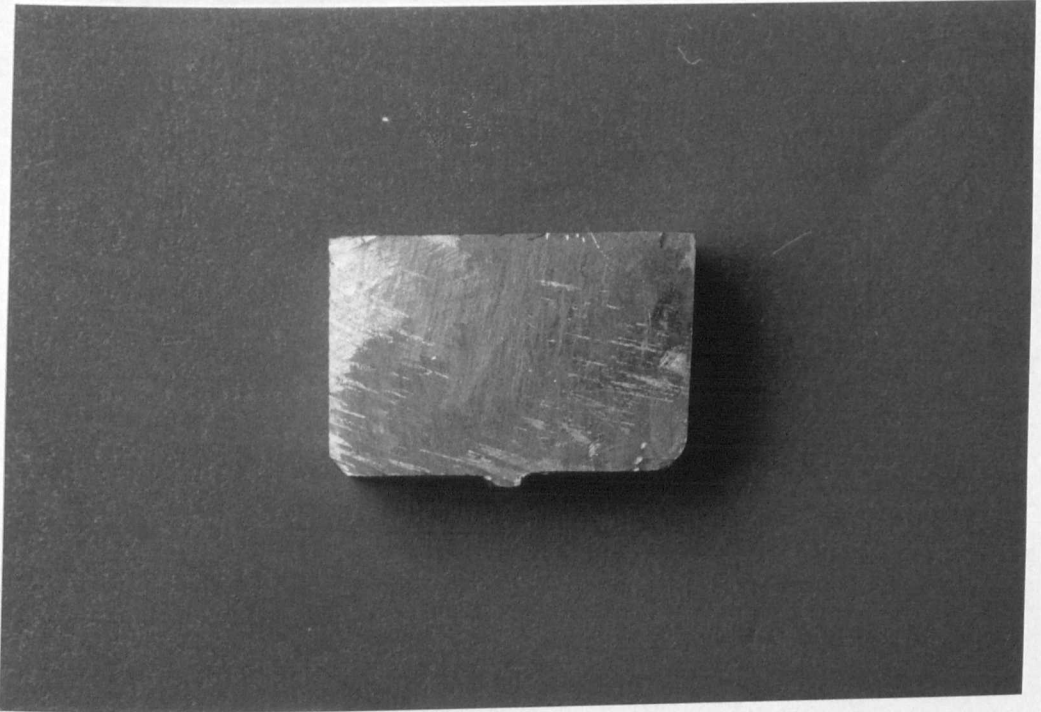
These radii, on the corners and the web protrusion, are the same as those for the boundary line found in the segregated samples.

## **6.9 SAMPLE PRODUCED IN A DIE WITH A RADIUS ON THE CORNER**

The microstructure of figure 6.15 shows the corner of a casting produced with a 2 mm radius on the corner of the die. There is no segregation visible and the microstructure along the curve is the same as found in the main body of the casting.

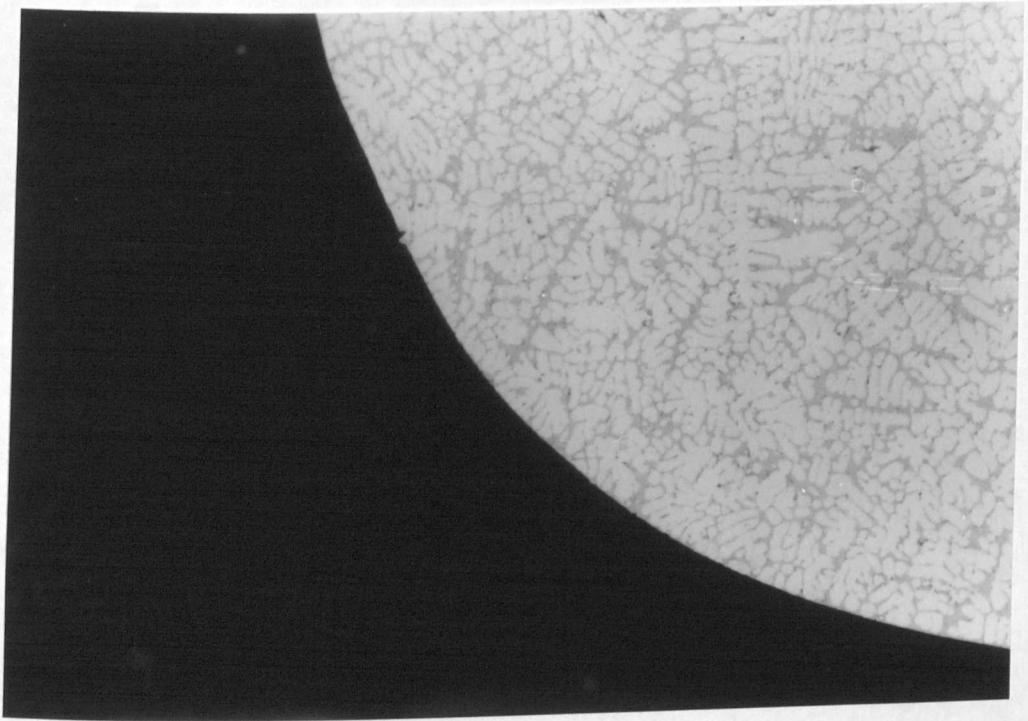
Thus by putting an appropriate radius on the corners of the die the segregation in the corner is completely eliminated.





**Figure 6.14**

*Cross-section of Casting. No Pressure Applied.*



200 μm

**Figure 6.15**

*Microstructure of a Casting Produced with a 2 mm Radius on a Corner*

Melt temperature      650°C

Squeeze pressure      50 MPa

# **CHAPTER**

## **7**

### **COOLING CURVES DURING MACROSEGREGATION**

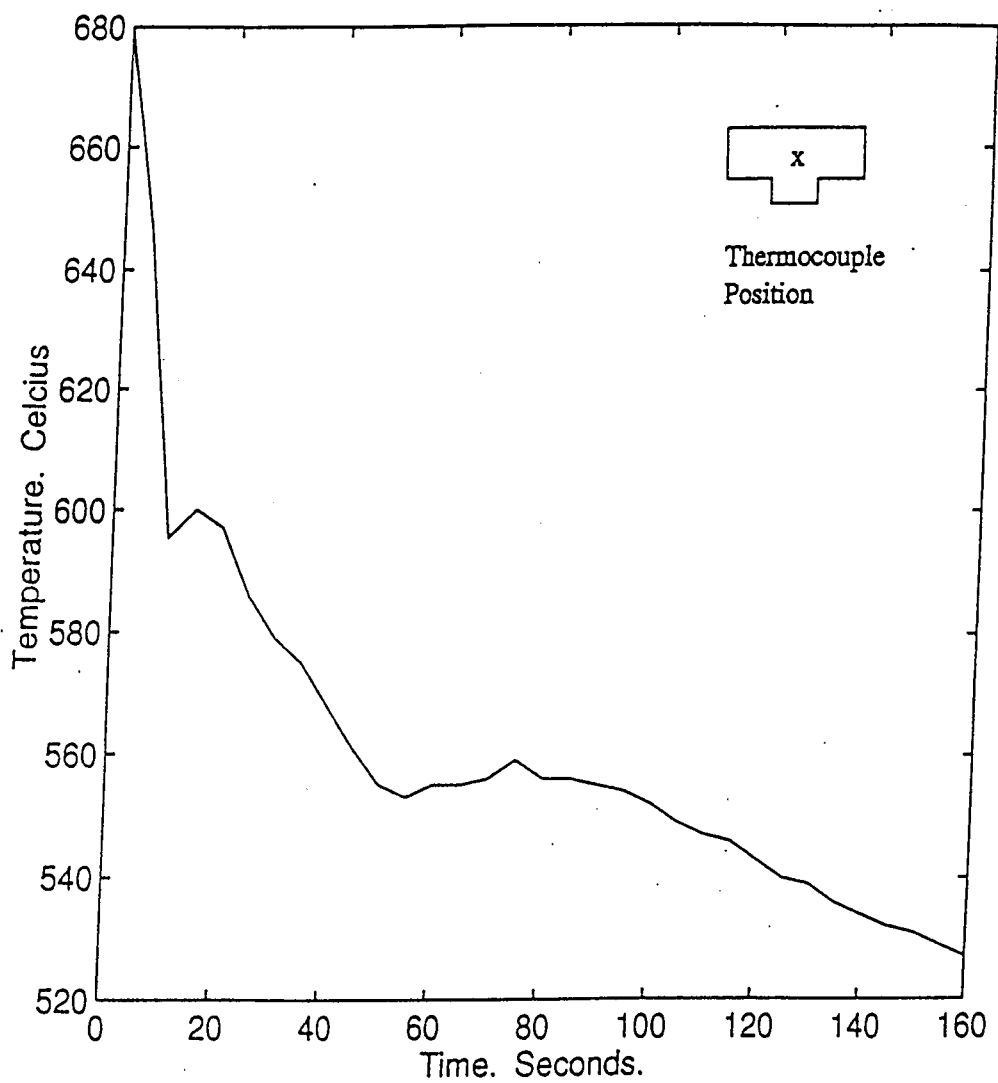
#### **7.1 INTRODUCTION**

This chapter contains the thermal cooling curves for LM25 obtained during the experimentation. The curves were generated from data collected via thermocouples embedded in the casting during solidification, see section 5.5.4. As with chapter 6 a comparison between a sand casting and a squeeze casting is first made. A series of cooling curves are then presented along with the relevant casting data to show the relationship between the temperature profile and the macrosegregation. The cooling curves confirm the findings of chapter 6. Changing the pouring temperature or the applied pressure has no significant effect on the segregation whereas changing the geometry of the die does have a significant effect.

#### **7.2 COMPARISON OF COOLING CURVES**

Figures 7.1 and 7.2 show typical cooling curves for a sand casting and a squeeze casting respectively. The corresponding microstructures are in figures

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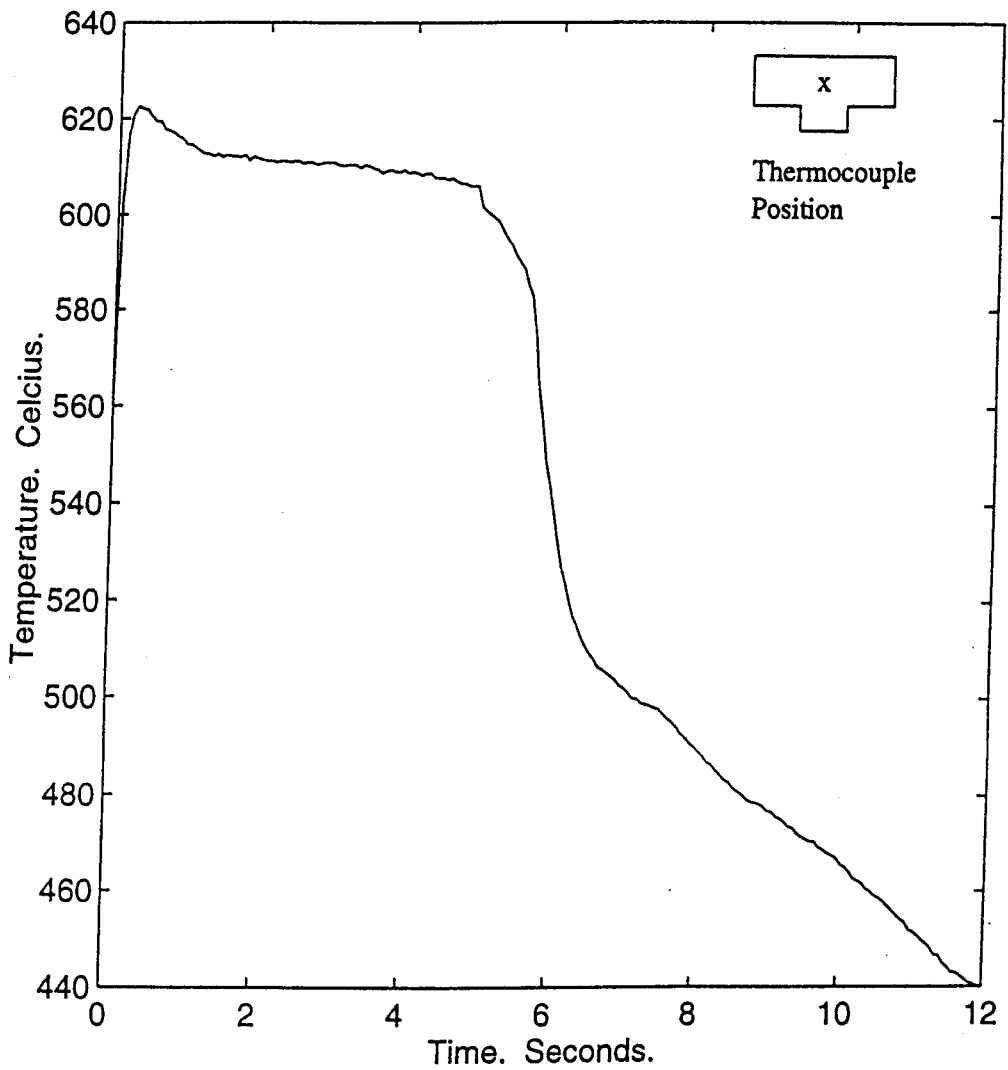


**Figure 7.1**

*Typical Cooling Curve from a Sand Casting*

*Thermocouple Placed in the Centre of the Casting*

Melt temperature     720°C



**Figure 7.2**

*Typical Cooling Curve from a Squeeze Casting*  
*Thermocouple Placed in the Centre of the Casting*

Melt temperature      650°C

Applied pressure      50 MPa

6.1 and 6.2. It can clearly be seen that squeeze casting has a much faster cooling rate compared to sand casting. The faster cooling rate leads to a finer microstructure and greatly improved mechanical properties over sand casting. The curves are consistent with previously published material and form the basis for the curves that follow.

### **7.3 COOLING CURVES AND MACROSEGREGATION**

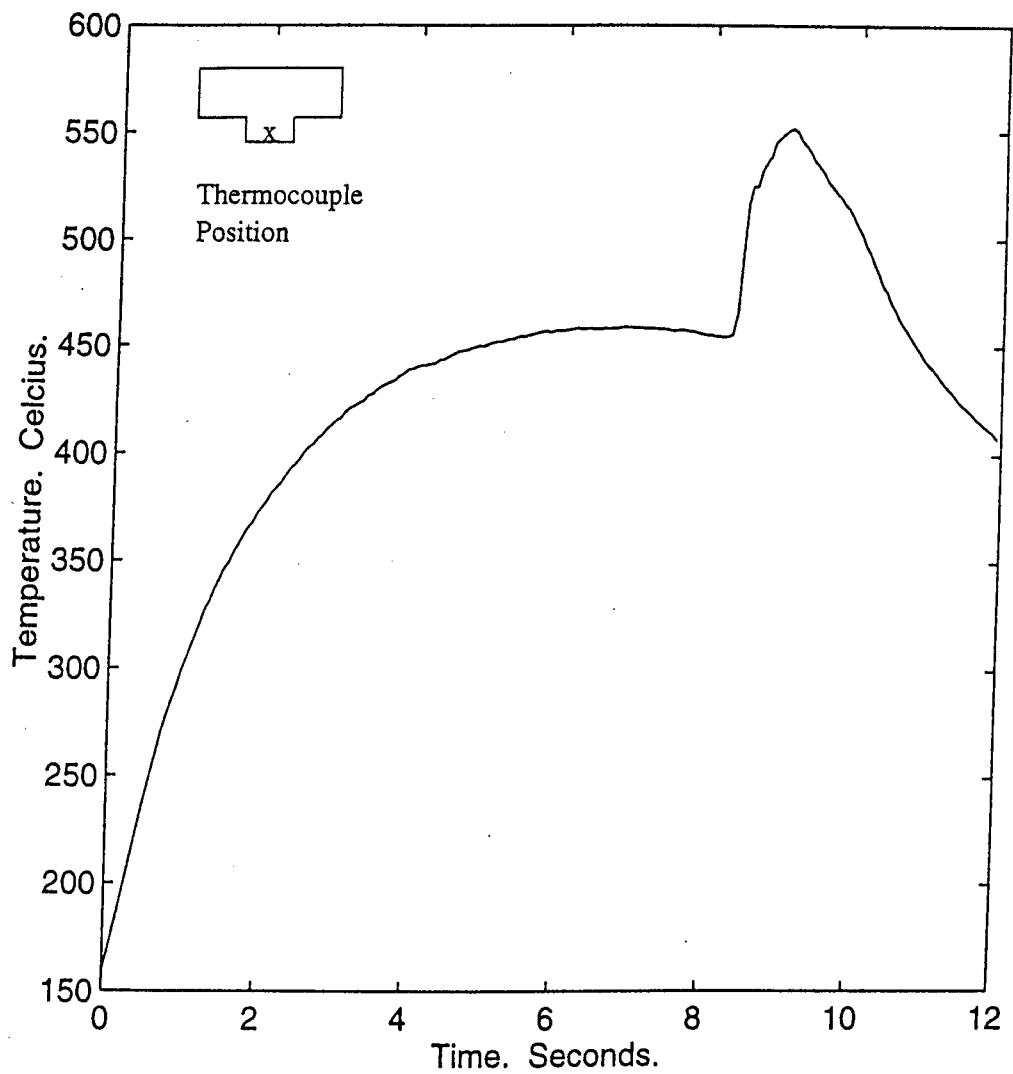
Placing the thermocouples in the areas where the macrosegregation occurred enabled the recording of the local temperature profile. The areas into which the thermocouples were to be placed were found by studying the microstructure of previous samples.

Figures 7.3 and 7.4 show the cooling curves of two such areas, a web and a corner segregated area respectively.

### **7.4 COOLING CURVE WITHIN A WEB**

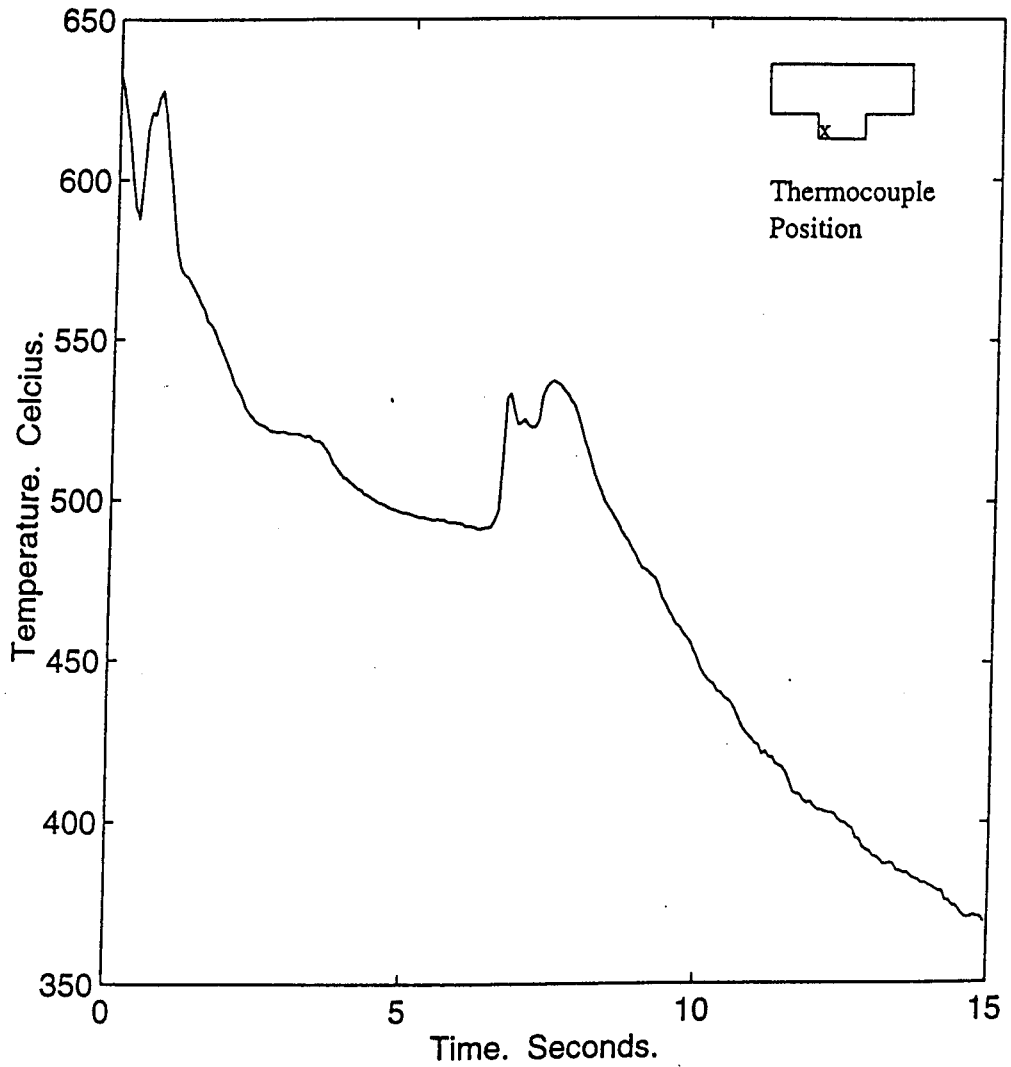
Figure 7.3 shows the thermal trace obtained from a thermocouple placed in the bottom of a 2 mm wide web. The microstructure of the sample is shown in figure 6.5.

The cooling curve, figure 7.3, shows a rise in temperature over a period of 7 seconds, to approximately 450 °C. There is then a very sharp increase in



**Figure 7.3**

*Typical Cooling Curve from a Thermocouple Placed in the Bottom of a 2 mm  
Web*



**Figure 7.4**

*Typical Cooling Curve from a Thermocouple Placed in the Corner of a Casting*

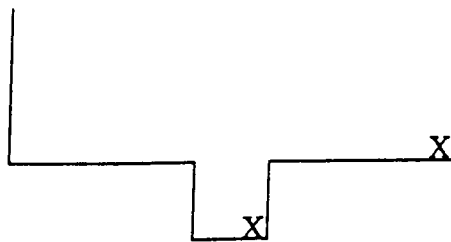


temperature at the time the external load is applied, to approximately 550° C. The temperature then falls off rapidly as the casting solidifies.

### 7.5 COOLING CURVE WITHIN A CORNER

Figure 7.4 shows the thermal trace obtained from a thermocouple placed in the corner of a casting. The corner referred to can be any concave corner on the casting as shown in figure 7.5. This only applies in the web corners if the web is of sufficient width to allow the molten alloy to flow in without pressure being applied. The microstructure of the sample is shown in figure 6.12.

The cooling curve, figure 7.4, shows an immediate rise in temperature. The rise is close to the pouring temperature of the alloy. The temperature then falls over the next 6 seconds until a very sharp increase in temperature is experienced at approximately 7 seconds. The temperature then falls off rapidly as the casting solidifies.



**Figure 7.5**

*Corners on the Casting where Thermocouples were Placed*

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## **7.6 THE EFFECT OF THE MELT TEMPERATURE AND THE APPLIED PRESSURE ON THE COOLING CURVE**

The remaining figures, from 7.6 onwards, show cooling curves produced by thermocouples placed in webs and corners of samples with varying temperatures and pressures. Each figure is accompanied by the melt temperature and the pressure relevant to the sample. Each curve has a corresponding microstructure and a reference is given alongside. The microstructure for each curve can be found in chapter 6.

### **7.6.1 Webs**

Figures 7.6, 7.7 and 7.8 show the cooling curves obtained from a thermocouple within a 2 mm wide web. Each of the curves are very similar in shape. There is an initial steady rise in temperature followed, at around 7 seconds, by a very sharp increase in the temperature to approximately 550 °C. The temperature then falls off rapidly.

### **7.6.2 Corners**

Figures 7.9, 7.10 and to 7.11 show cooling curves obtained from a thermocouple placed within a corner of the casting. Like the curves for the webs the curves for the corners are all very similar.

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The initial temperature of each curve is governed by the pouring temperature of the alloy. This is the only significant difference in the curve. After the initial temperature rise the temperature falls off steadily over time. At around 7 seconds there is a sharp, although not large, increase in temperature. The temperature then falls off rapidly.

The initial temperature, in the segregated corner, is very much higher than is found in the segregated web. This is because the void in the corner is very small in comparison to the web. The end of the thermocouple is therefore very close to the molten alloy, and in some cases may be touching it. Thus the temperature read is close to the actual pouring temperature.

The rise in temperature at approximately 7 seconds is similar in all of the segregated samples. It is this rise in temperature that is the significant part of the curve.

### **7.7 NO PRESSURE APPLIED TO THE CASTING**

The thermal cooling curve, of figure 7.12, is from a web of a die which was filled with alloy but no pressure was applied. A cross section of the component produced is shown in figure 6.15.

The cooling curve shows a rise in temperature to approximately 400 °C which then levels off, over the range of time covered. The time range is the same for all of the cooling curves. The rise in temperature is similar to that found, before

the very sharp increase due to the load application, in the segregated webs, figures 7.6, 7.7 and 7.8.

The cross section, figure 6.15, shows that the alloy did not penetrate the web as the web has not been formed on the component.

Thus the cooling curve of figure 7.12 and the cross section of figure 6.15 show that no alloy penetrates the web before the application of the load in the 2 mm web.

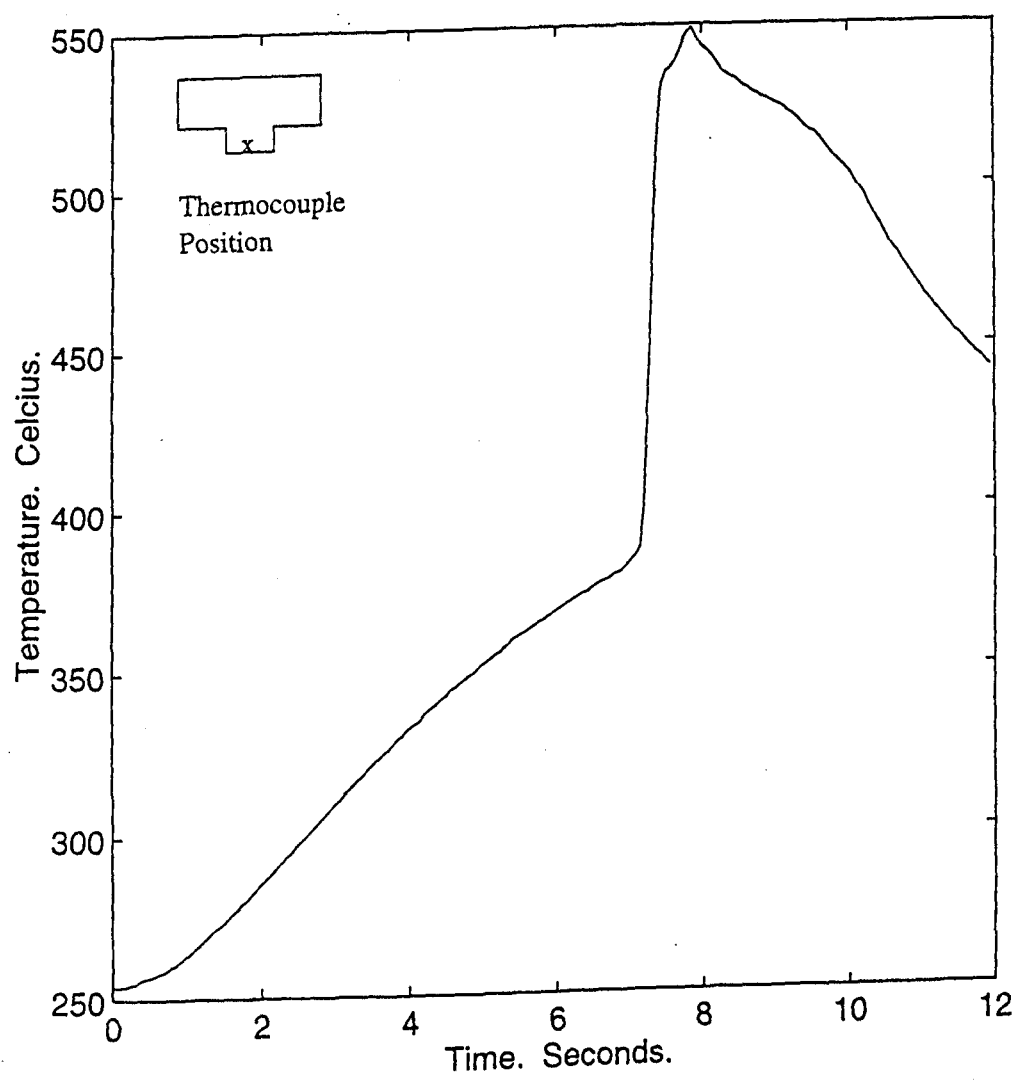
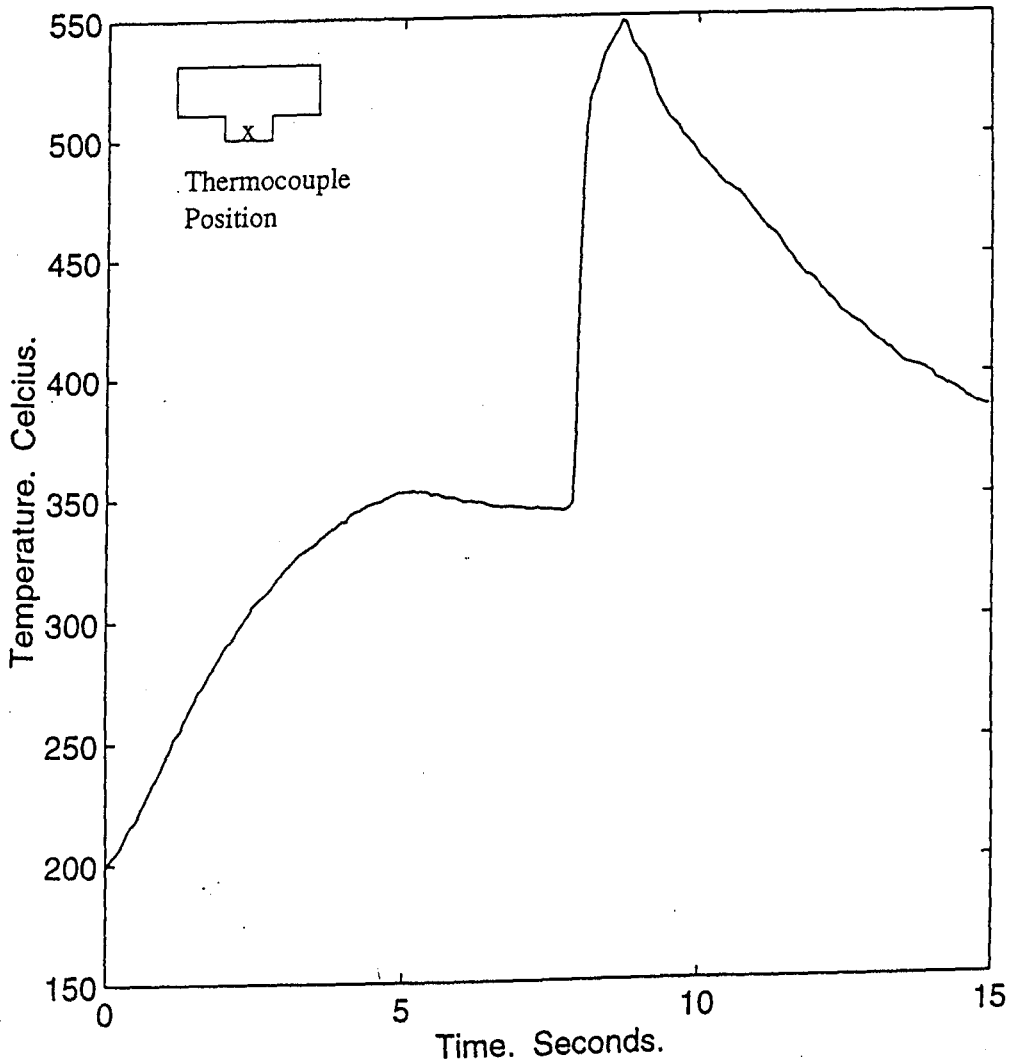


Figure 7.6

*Cooling Curve from a Thermocouple Placed in a Web*

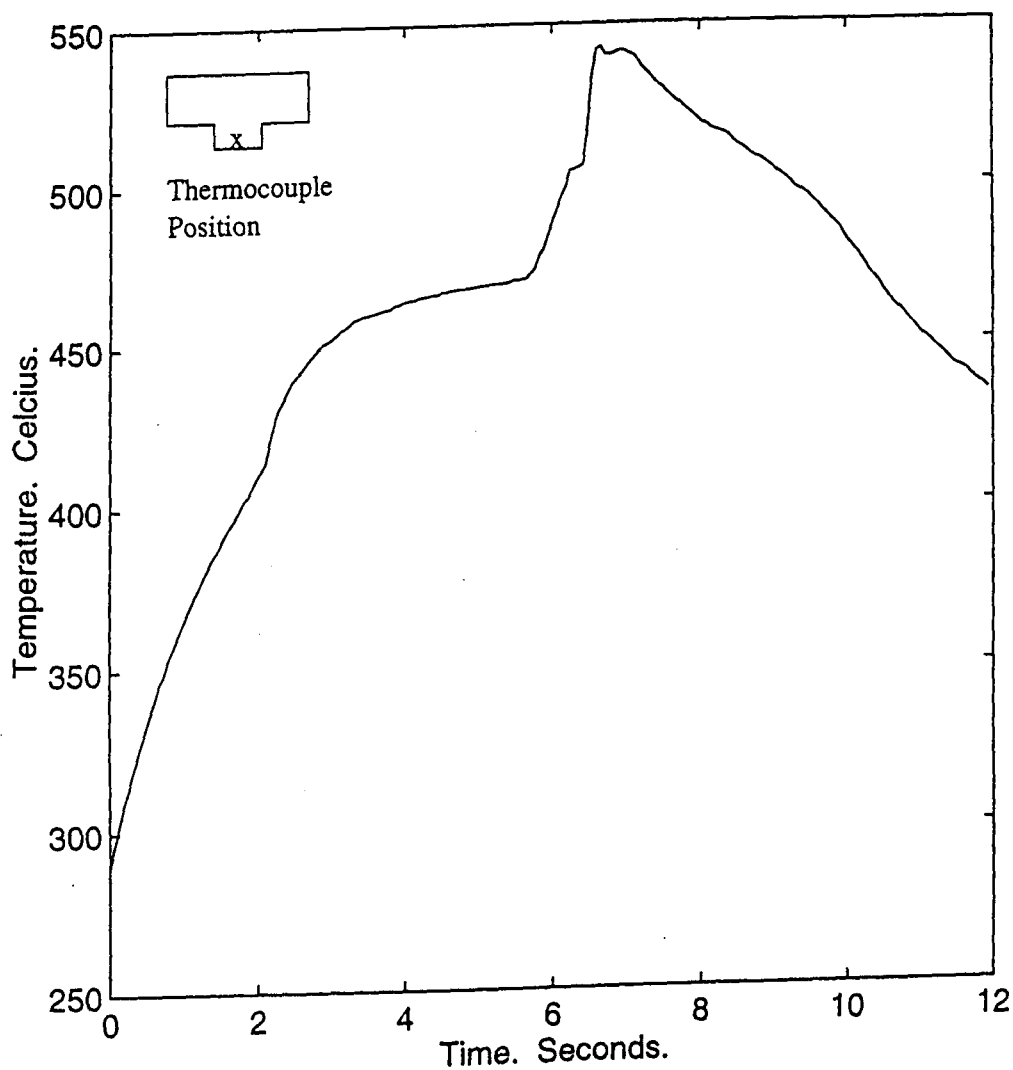
Melt temperature	650°C		
Applied pressure	50 MPa		
Web size	2 mm	Microstructure	Figure 6.5



**Figure 7.7**

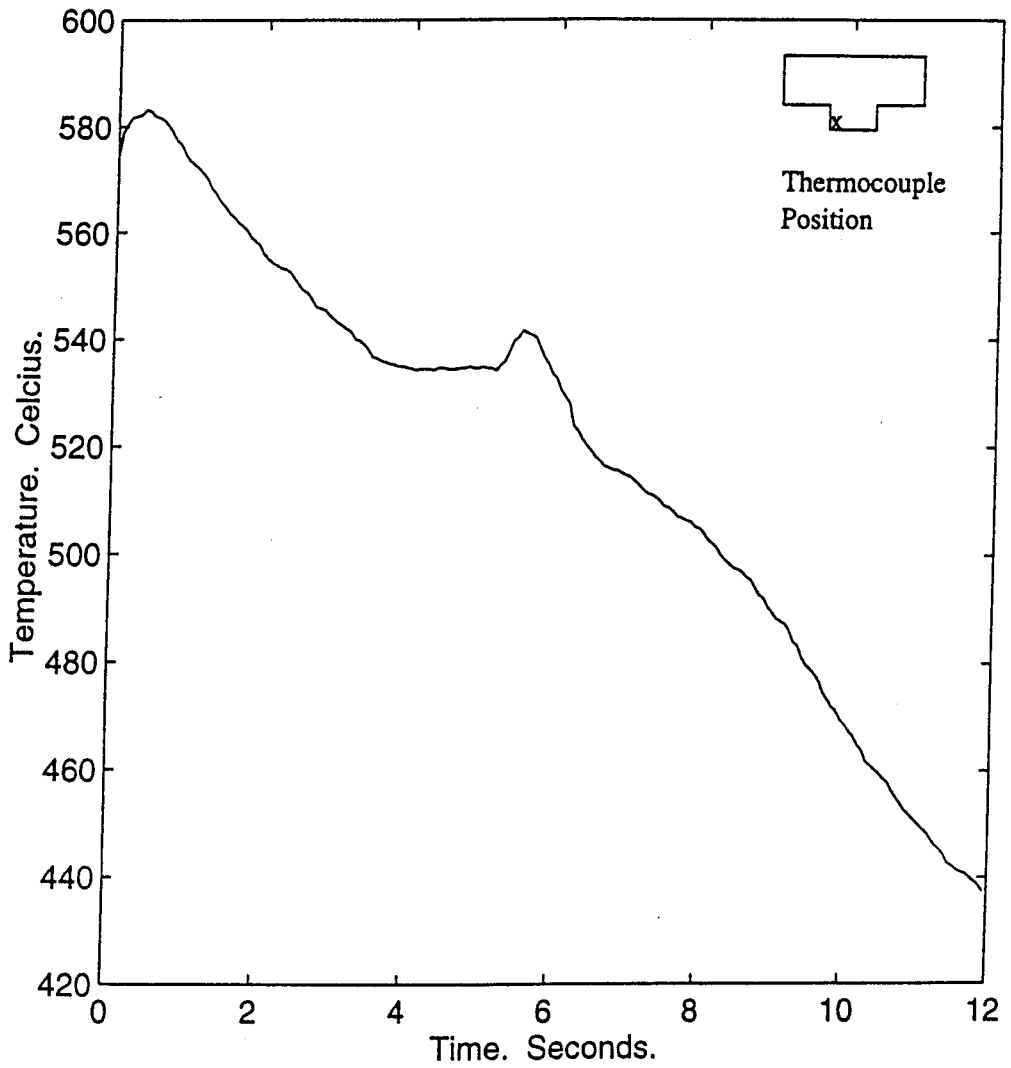
*Cooling Curve from a Thermocouple Placed in a Web*

Melt temperature	720°C		
Applied pressure	50 MPa		
Web size	2 mm	Microstructure	Figure 6.6

**Figure 7.8**

*Cooling Curve from a Thermocouple Placed in a Web*

Melt temperature	650°C		
Applied pressure	95 MPa		
Web size	2 mm	Microstructure	Figure 6.7

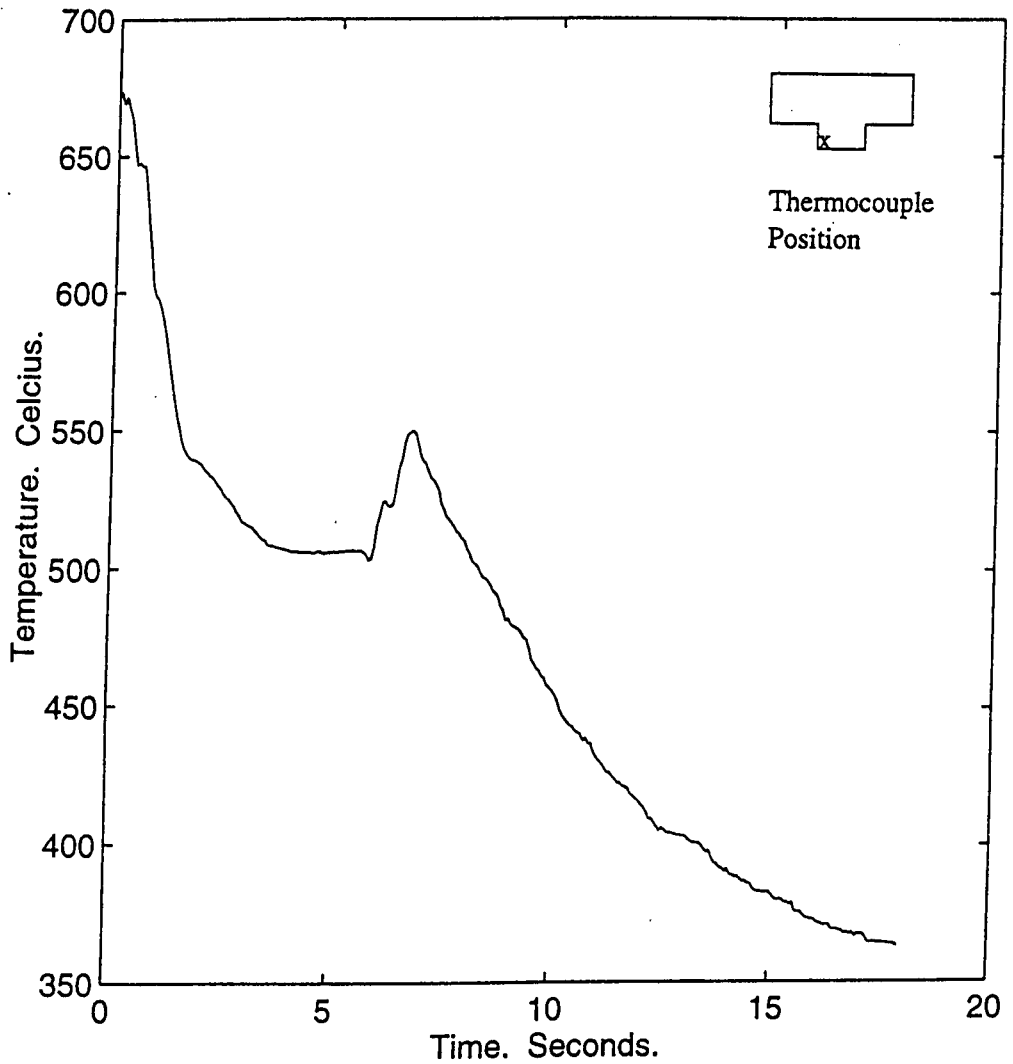


**Figure 7.9**

*Cooling Curve from a Thermocouple Placed in a Corner*

Melt temperature	650°C
Applied pressure	50 MPa
Microstructure	Figure 6.11

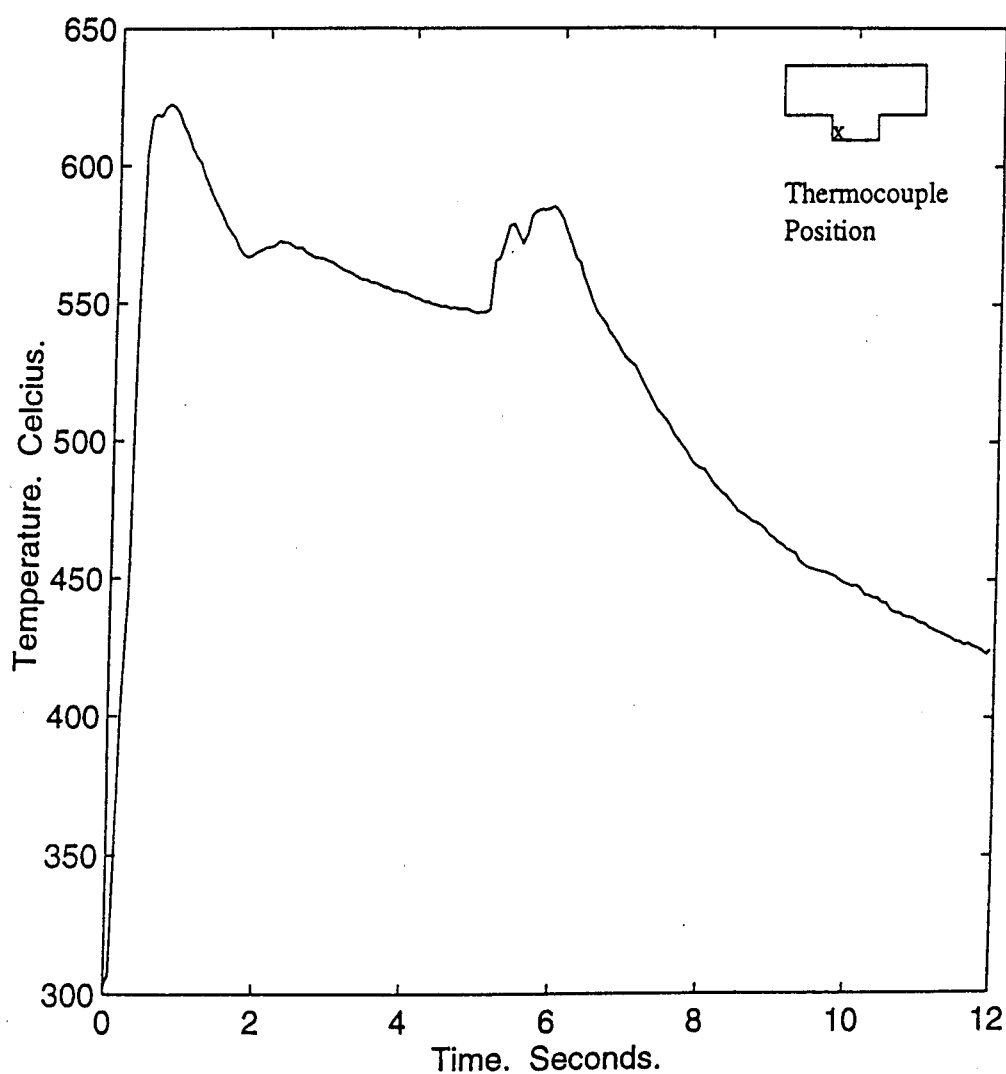




**Figure 7.10**

*Cooling Curve from a Thermocouple Placed in a Corner*

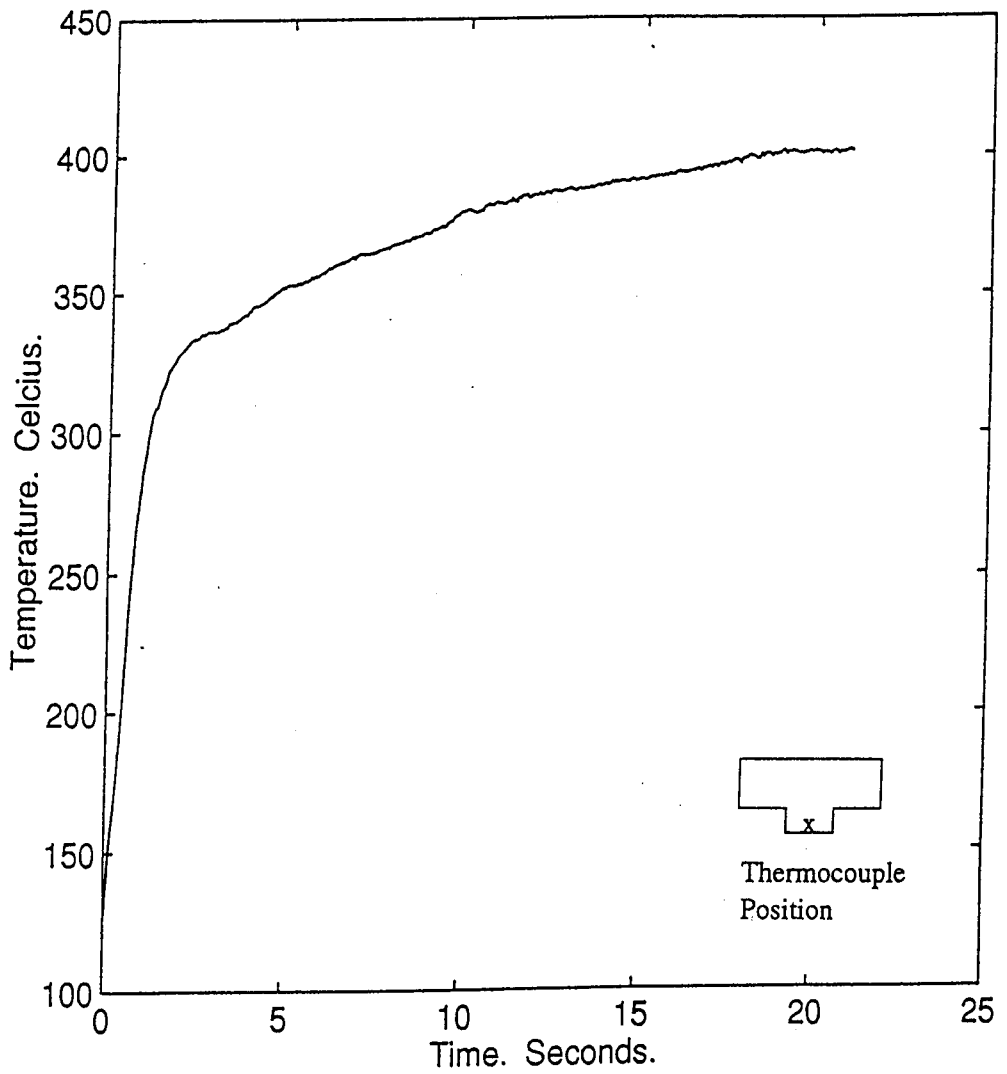
Melt temperature	720°C
Applied pressure	50 MPa
Microstructure	Figure 6.12



**Figure 7.11**

*Cooling Curve from a Thermocouple Placed in a Corner*

Melt temperature	650°C
Applied pressure	95 MPa
Microstructure	Figure 6.13



**Figure 7.12**

*Thermal Trace from a Thermocouple Placed in a Web. No Pressure Applied*

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# **CHAPTER**

## **8**

### **MACROSEGREGATION WITHIN A PARTICULATE METAL MATRIX COMPOSITE**

#### **8.1 INTRODUCTION**

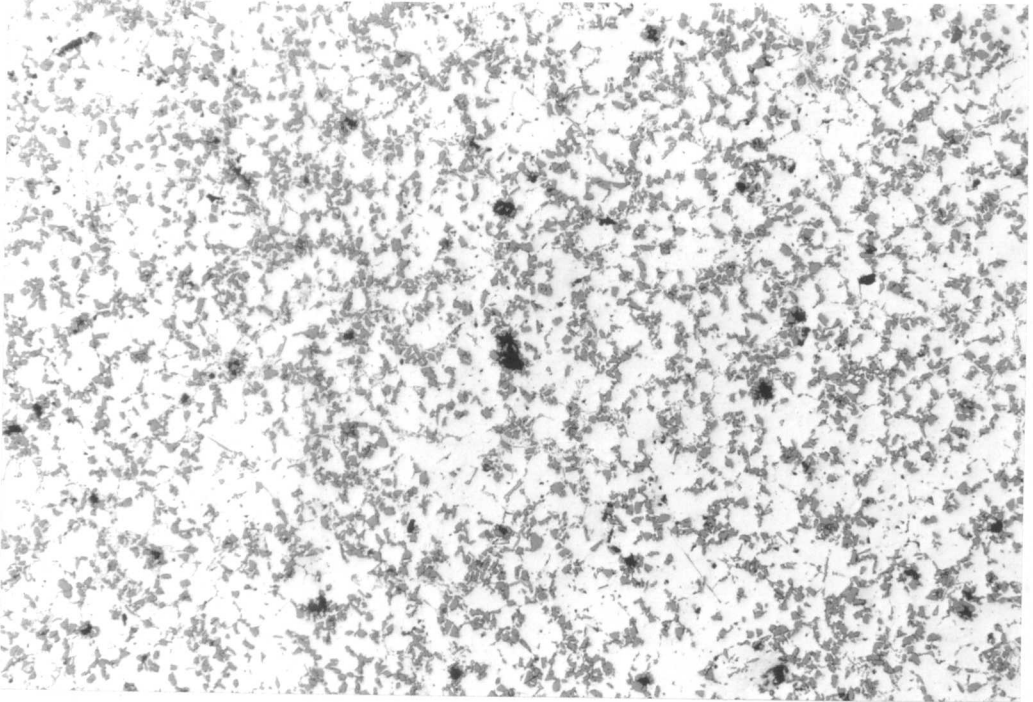
Following on from the results obtained for the LM25 alloy this chapter presents the results gained for a particulate metal matrix composite. Firstly a comparison is made between the microstructure of the as supplied ingot and a squeeze casting of the composite.

Using the same parameters and settings as for the LM25 alloy, castings were made with the composite material. The results are presented in section 8.3.

The metal matrix composite has an Al-Si7-Mg0.6 alloy matrix with 15 % vol. 20  $\mu\text{m}$  SiC particles.

#### **8.2 COMPARISONS OF MICROSTRUCTURES**

A comparison is first made between the as supplied ingot and a squeeze casting to show the effects of squeeze casting on the material. Figure 8.1 shows the microstructure of the as supplied ingot.



200  $\mu\text{m}$

**Figure 8.1**

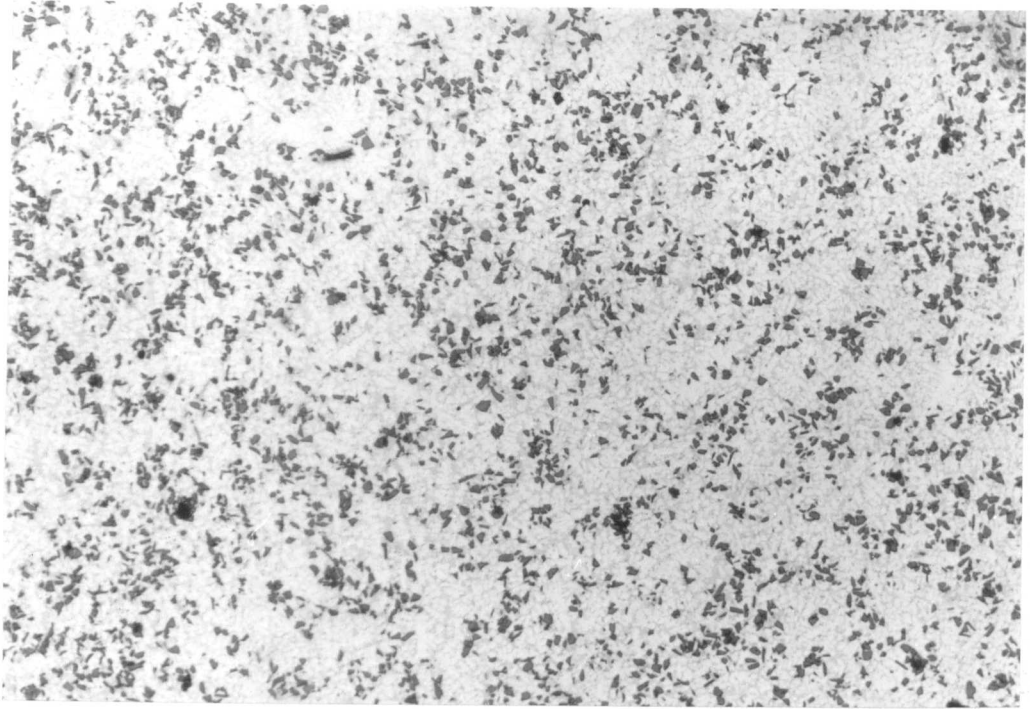
*Microstructure of the As Supplied Metal Matrix Composite Ingot*

The microstructure is a mixture of large aluminium dendrites with the silicon carbide particles fairly uniformly distributed within the interdendritic regions. There are some slightly larger dark areas surrounded by silicon carbide particles. These are areas of interdendritic porosity produced during the solidification of the ingot. The silicon carbide particles are sited in the interdendritic regions and as such they have a tendency to coalesce around the porosity.

Using the data gained for the LM25 alloy, and the literature, chapter 2, a casting temperature of 650°C with an applied pressure of 50 MPa were selected for the squeeze casting of the composite material. Figures 8.2 and 8.3 show the resulting microstructure and a typical cooling curve, respectively.

The microstructure is of a fine dendritic nature with the silicon carbide particles residing in the interdendritic spaces. It is noticeable that there are less silicon carbide particles in the squeeze cast sample than the sample from the as supplied ingot. The squeeze cast sample was cast from a melt that had been molten for some minutes. During this time the silicon carbide particles settled, under the force of gravity, thereby denuding the top of the melt. As the melt is ladled from the top and poured into the die there is a deficit of particles in the final casting.

The silicon carbide particles are not as uniformly distributed as in figure 8.1 and they tend to coalesce in small groups. There is also some interdendritic porosity which is surrounded by silicon carbide particles.

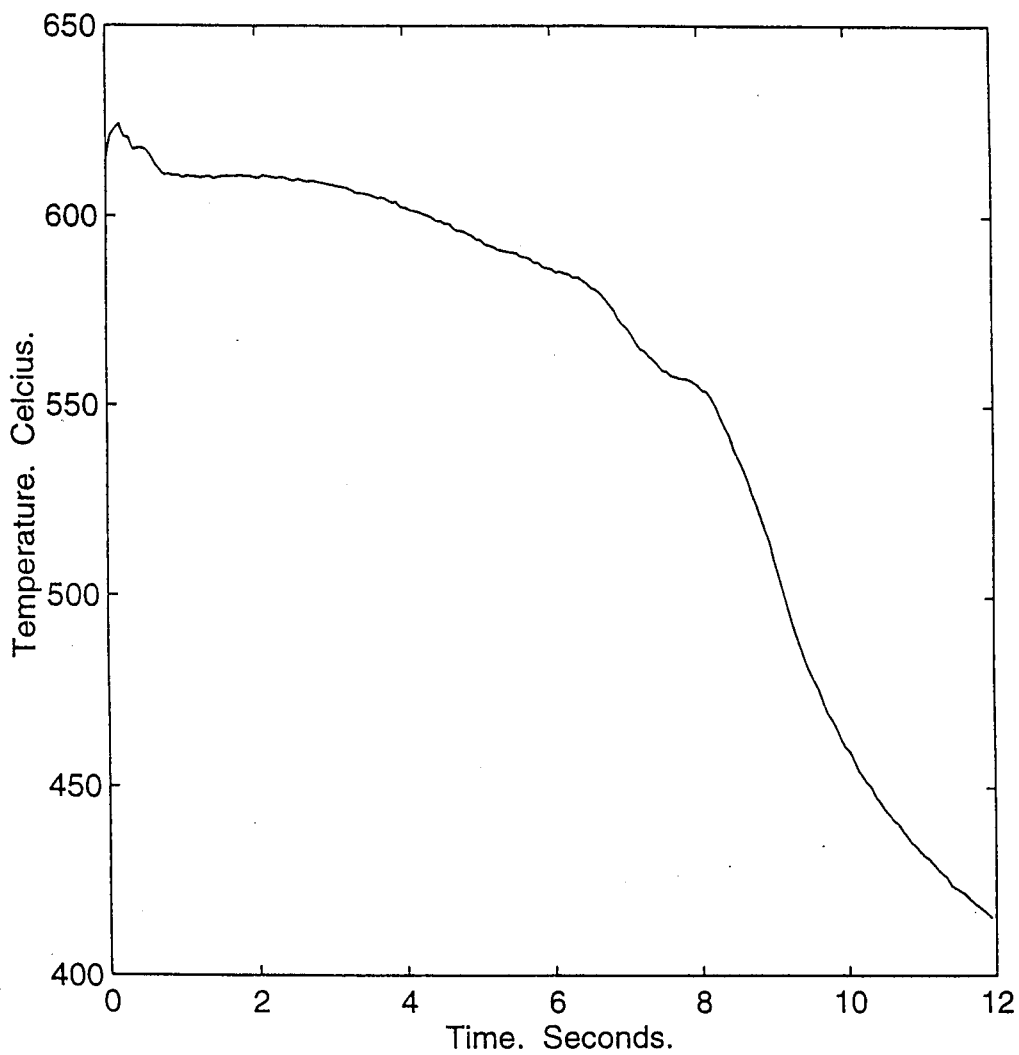


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200  $\mu\text{m}$

**Figure 8.2**

*Typical Microstructure of a Squeeze Cast Particulate Metal Matrix Composite*



**Figure 8.3**

*Typical Cooling Curve for a Squeeze Casting of a Particulate Metal Matrix Composite. Thermocouple Placed in the Centre of the Casting*



### **8.3 MACROSEGREGATION IN WEBS AND CORNERS**

#### **8.3.1 Segregation in Webs**

Figure 8.4 shows the segregation obtained within a 2 mm wide web.

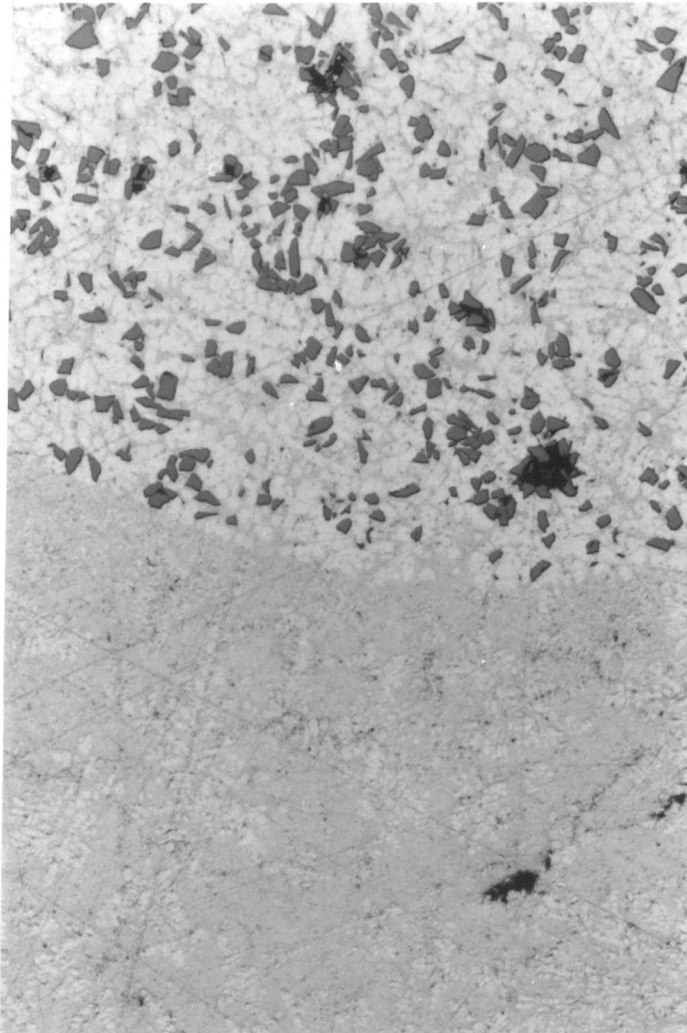
The main body of the casting has a fine structure similar to the LM25 alloy. There is a distinct boundary between the solute rich segregated area and the bulk of the casting. The boundary has the curved appearance found in the LM25 alloy castings.

The aluminium dendrites are approximately the same size as the silicon carbide particles. There are no silicon carbide particles within the segregated area. They are only to be found in the main body of the casting residing in the interdendritic spaces, trapped there because the interdendritic spacings are significantly smaller than the particles.

There is also a significant amount of porosity in both the main body of the casting and the segregated area.

#### **8.3.2 Segregation in Corners**

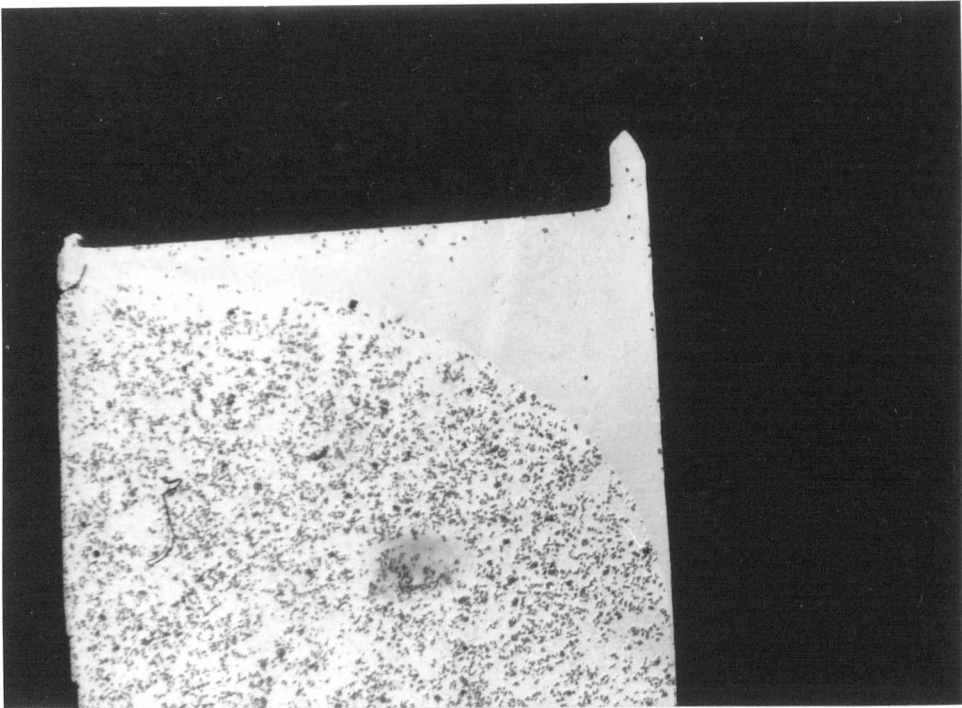
Figure 8.5 shows the segregation within a corner. As described in section 8.3.1, for the web, the microstructure is very similar to that obtained when using LM25. Again the segregated area is totally denuded of silicon carbide particles.



100 μm

**Figure 8.4**

*Segregation within a Web of a Squeeze Cast Particulate Metal Matrix  
Composite*



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1 mm

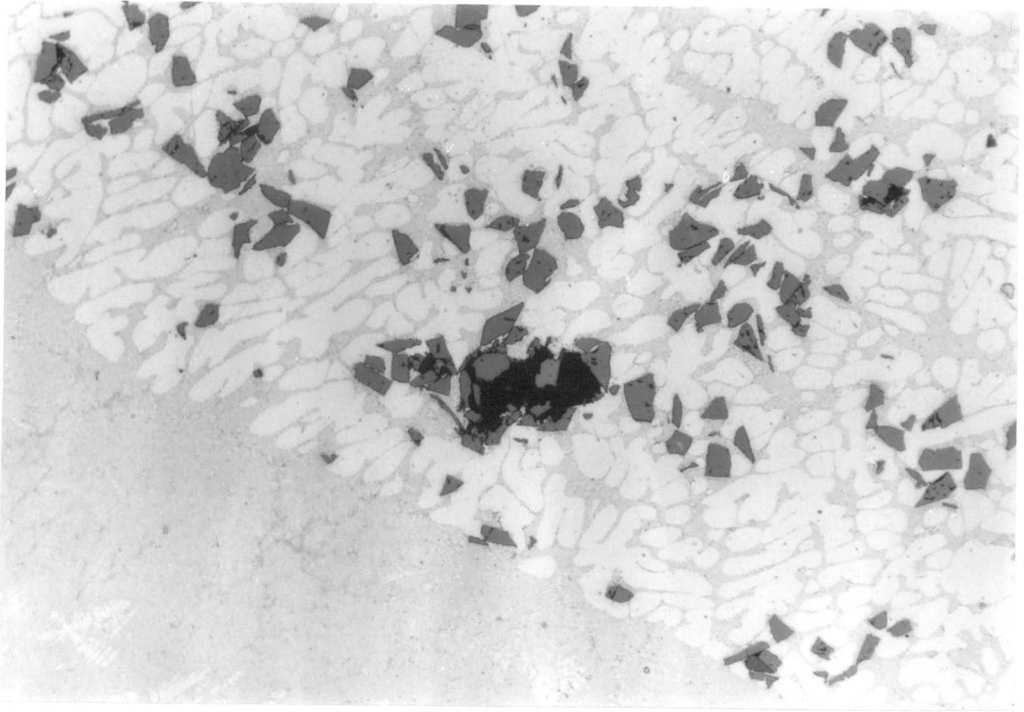
**Figure 8.5**

*Segregation within a Corner of a Squeeze Cast Particulate Metal Matrix Composite. Note this is only one half of the component.*

### **8.3.3 Increase in Porosity**

The close up of the boundary in figure 8.6 clearly shows the division between the segregated area and the main bulk of the casting. The silicon carbide particles are shown to reside in the interdendritic spaces and are clearly not as well dispersed throughout the matrix as in the as supplied ingot, figure 8.1.

There is a significantly large pore in the microstructure that is not found in the LM25 alloy. The pore is surrounded by silicon carbide particles.



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200  $\mu\text{m}$

**Figure 8.6**

*Close up of a Segregated Boundary in a Metal Matrix Composite*

## **8.4 COOLING CURVES**

Cooling curves were produced for the particulate MMC as for the LM25 alloy. The geometry of the curves produced by the MMC are very similar to those obtained for the base alloy.

### **8.4.1 Segregation in Webs**

The cooling curve obtained from a thermocouple placed in a 2 mm wide web is shown in figure 8.7. The microstructure is shown in figures 8.4 and 8.6.

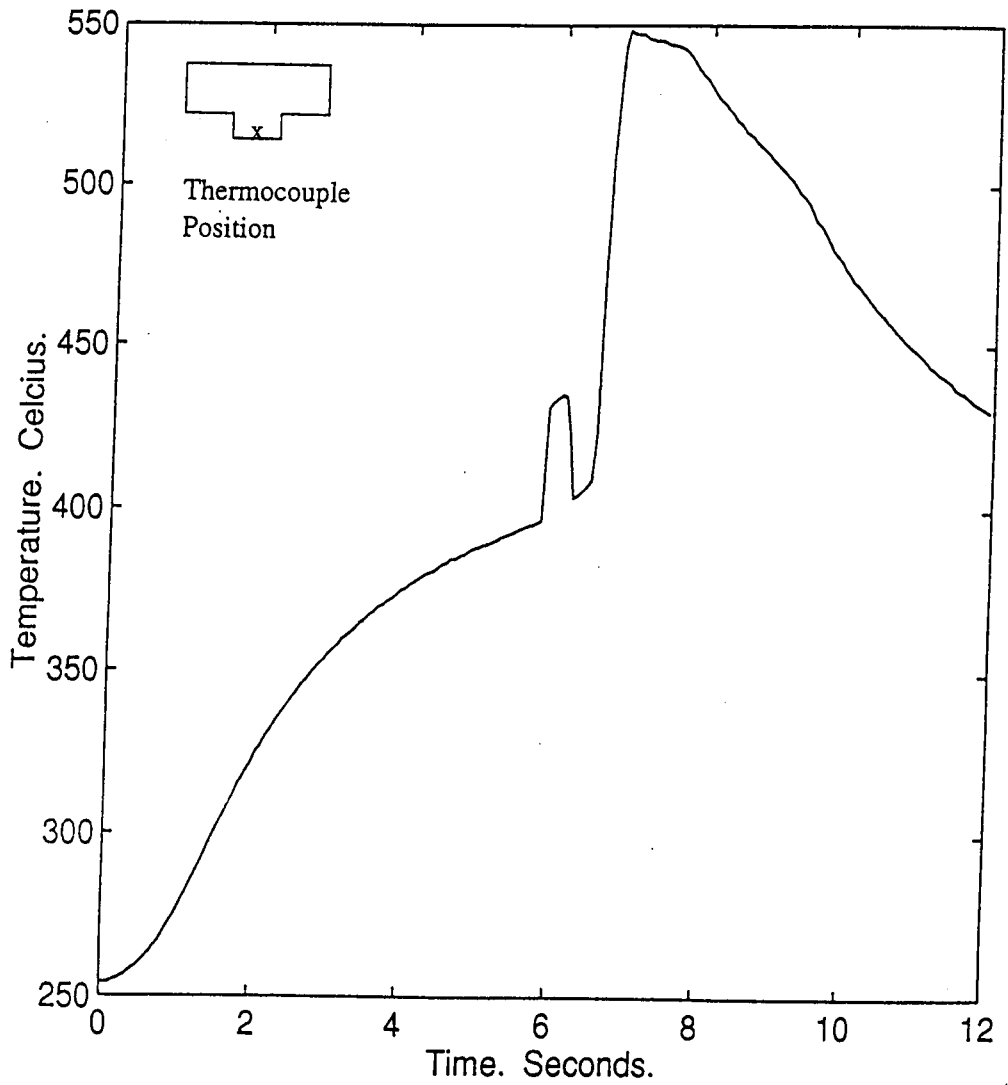
The temperature rises slowly over 5 to 6 seconds to 400° C. There is then a very sharp increase in temperature to approximately 550° C. The temperature then falls off very rapidly as solidification progresses.

### **8.4.2 Segregation in Corners.**

The cooling curve obtained from a thermocouple placed in the corner of a casting is shown in figure 8.8. The microstructure obtained is shown in figure 8.5.

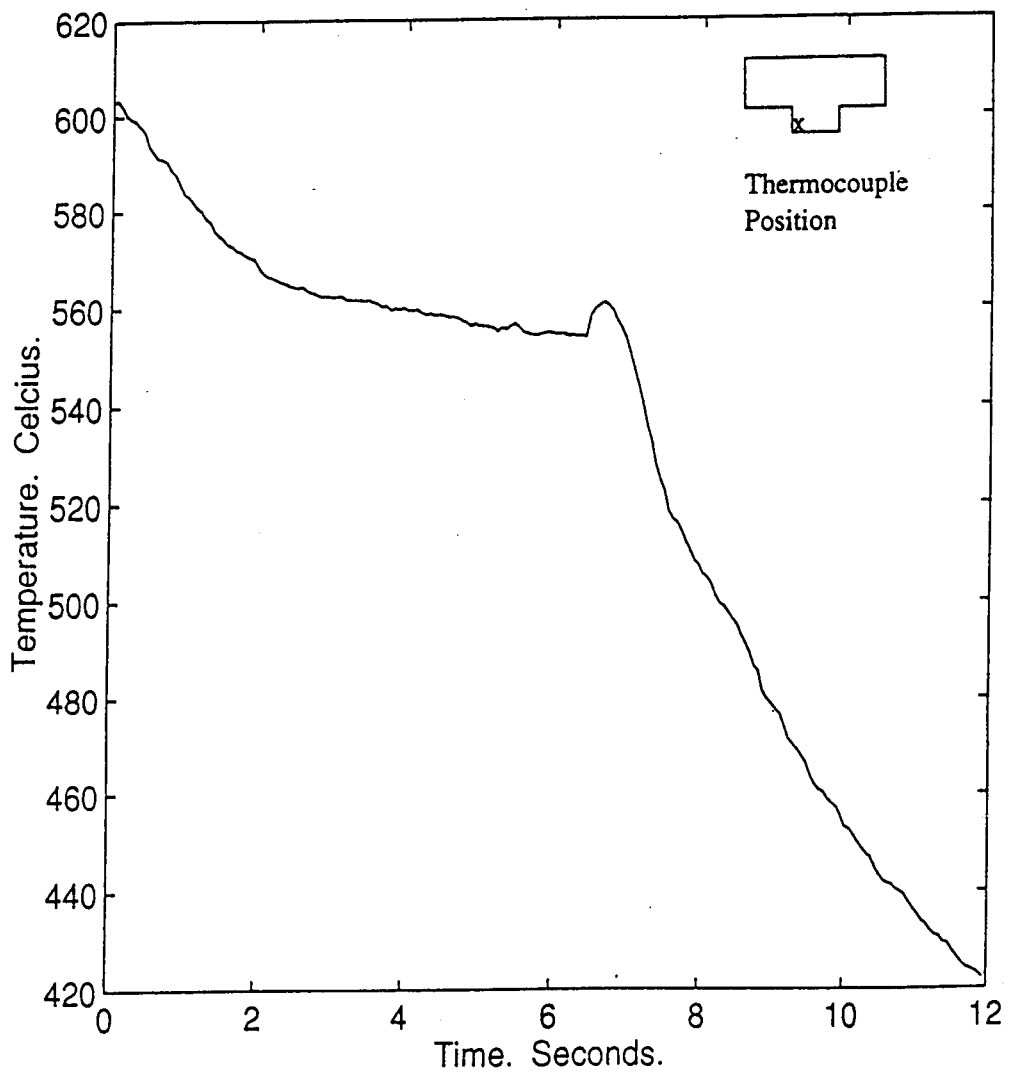
The cooling curve shows an initial increase in temperature, as the alloy is poured into the die. Over the next 5 to 6 seconds the temperature gradually falls before there is a sharp increase in the recorded temperature to around 560° C. The temperature then falls off rapidly as the casting solidifies.

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**Figure 8.7**

*Cooling Curve for a Squeeze Cast Particulate Metal Matrix Composite from a Thermocouple Placed in a Web*



**Figure 8.8**

*Cooling Curve for a Squeeze Cast Particulate Metal Matrix Composite from a Thermocouple Placed in a Corner*



# **CHAPTER**

## **9**

# **ANALYSIS OF RESULTS**

## **9.1 INTRODUCTION**

This chapter analyses the results described in chapters 6, 7 and 8. Some design details, for the component, are arrived at throughout the chapter. These are then presented together as a set of design criteria for squeeze casting in chapter 10 along with a first approximation model for the segregation.

## **9.2 GENERAL OBSERVATIONS ON THE RESULTS**

The effects of temperature, pressure and part geometry on the formation of the segregated material in LM25 and a particulate metal matrix composite are assessed. Segregation occurred in both the webs and the concave corners within the casting. For clarity the following sections assume it is the segregation in the web which is under consideration. Segregation within concave corners is dealt with in section 9.4

### 9.2.1 Effect of Pouring Temperature on the Segregation

The generally accepted pouring temperature, for squeeze casting, is 10 to 80 °C above the liquidus temperature of the particular alloy being cast, section 2.7.2. With the chosen pouring temperatures of 650 & 720 °C, section 6.4, the usual range of temperatures is reasonably well covered.

Figures 6.5 and 6.6 show the microstructures and figures 7.6 and 7.7 show the relevant cooling curves obtained for castings with a pouring temperature of 650 and 720 °C respectively. It is clear that there is no significant difference between the two microstructures. Both webs are filled with the segregated material and both have the same circular boundary between the web and the bulk material.

The higher temperature melt would be expected to have a slightly lower surface tension, in comparison to the lower temperature melt. This would allow the liquid to penetrate the web slightly further and produce a boundary with a smaller radius, however there is no discernible difference in the size of the radii between the two samples, both being approximately 2 mm.

The temperature of the melt, within the normal casting range, has, therefore, little or no effect on the amount or the placing of the segregation in the particular casting under consideration. The choice of casting temperature for any future squeeze casting is therefore not influenced by the segregation phenomenon.

### **9.2.2 Effect of Pressure on the Segregation.**

The literature provides a range of pressures for the production of a sound squeeze casting, section 2.7.4. These range from 50 to 150 MPa. The present study used two pressures 50 MPa and 95 MPa, section 6.5, which covers the lower half of the range.

The microstructures of the castings can be found in figures 6.5 and 6.7 and their relevant cooling curves are in figures 7.6 and 7.8. Once again it is clear that there is no discernible difference between the two microstructures. The same amount of segregation is present in both as is the circular boundary. As with the temperatures this suggests that the amount of pressure applied, in the above range, has little or no effect on the particular casting under consideration. The choice of applied pressure, to produce a sound casting, is therefore dependent on the alloy and the size and complexity of the component and not on the need to control pressure assisted segregation.

### **9.2.3 Effect of Geometry on the Segregation.**

The previous two sections show that the segregation is not controlled by either the pouring temperature or the applied pressure, i.e. the main manufacturing parameters of squeeze casting. Changing the component geometry whilst keeping the pouring temperature constant at 650 °C, and the applied pressure constant at 50 MPa provided the clue to the formation and the control, of the

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segregation within the casting. The values for the temperature and pressure were arrived at from the previous two sections.

With a web 2 mm wide a fully segregated web resulted, figure 6.5. By increasing the web width to 8 mm, segregation within the main body of the web was completely eliminated, figure 6.10. A half way increase, to 4 mm, produced a non-segregated web in the vast majority of cases. Where segregation did occur in the 4 mm web it was confined to the side wall close to the web entrance, figure 6.9. Here it is caused by the alloy flowing over the sharp edge producing a waterfall effect which creates an air pocket. The air pocket is then back filled with the interdendritic fluid on the application of the pressure.

The corners of the casting, be they in the main body of the casting or in large webs, were also areas where segregation occurred, figures 6.11, 6.12 and 6.13. Each corner had a common detail, the geometry of the curve between the segregated area and the main body. The geometry of a corner from a non squeeze cast sample, i.e. a sample cast in the dies but with no squeezing pressure applied, figure 6.14, is also very similar to the previous cases. In all cases the corner had a radius of approximately 2 mm. Thus by designing the corner to have a radius slightly in excess of 2 mm from the outset the segregation in the corner can be eliminated, figure 6.16.

Rajagopal and Altergott, in their work [10], suggest that a reduction in macrosegregation defects, resulting from melt extrusion, can be reduced by switching to a casting grade alloy. This suggests that this form of

macrosegregation is a manufacturing problem. The alloy used in this work, however, LM25 is a high grade casting alloy and the segregation is still present. It has been shown, however, that by changing the design of the component the segregation can be eliminated. This makes it a design problem and not a manufacturing problem as it is the geometry of the component that is the critical parameter in controlling the segregation. Therefore aluminium alloys of all types from a low alloy content to a high alloy content can be used for squeeze casting.

### **9.3 SEGREGATION IN A WEB**

Figure 7.3 shows the thermal trace obtained from a thermocouple placed in the bottom of a 2 mm wide web when macrosegregation occurs during solidification. The microstructure is shown in figure 6.5.

The thermal trace shows that the temperature initially rises, over a period of 7 seconds, to approximately 450 °C. As the molten alloy temperature is around 610 °C no alloy has penetrated the web. The rise in temperature is due to the air trapped in the web being heated by the molten alloy in the main body of the die. This is confirmed by the thermal trace in figure 7.12 and the photograph in figure 6.15. Molten alloy, at a temperature of 650 °C, was poured into the die but no pressure was applied. The first part of the thermal trace is the same shape as that in figure 7.3 rising to a maximum temperature of approximately

350 °C. Figure 6.15 shows that no alloy entered the web as the web has not been formed.

At 8 seconds, figure 7.3, the temperature rises very sharply to approximately 550° C before rapidly falling off as solidification occurs. The temperature rise occurs at the time that the load is applied to the surface of the casting. The temperature increases as the interdendritic fluid is forced into the web. The maximum temperature recorded is around 550 °C, which is slightly lower than would be expected for the normal solidification temperature of the alloy. This is most likely due to a small error within the temperature recording equipment. The temperature then falls off rapidly as solidification is completed, as the pressure stops the solidifying alloy from shrinking away from the die wall.

The circular boundary, in the microstructure of figure 6.5, is very pronounced and smooth. The boundary has an approximate radius of 2 mm. There is no sign of any oxides or joins along the boundary path. This would suggest that no substantial oxide layer had formed prior to the application of the external load. This type of boundary is also found on the other samples of segregated webs and corners.

### **9.3.1 Partial Segregation in a 4 mm Web**

Although the 4 mm wide webs were not segregated, as shown in section 9.2.3, it was found, in some cases, that there were some areas of segregation within the web. Figure 6.9 shows one of these areas. It is on the wall of the web close to the web entrance.

The void was formed by the alloy not flowing easily into the web because of the corners on the web entrance. These corners are very sharp, and do not provide a funnel into the web. It would seem that the molten alloy flowed over the edge of the web entrance rather like a waterfall. This effect is well known in other forms of casting. On entering the web an air bubble was formed on the web wall. The bubble is then filled with interdendritic material when the load is applied.

The large porosity is present because of the way the void is filled. Two areas of segregated material converged, one from the top and one from the bottom, to trap the porosity between them. The segregated area is fully fused to the web and the two areas are fused together close to the die wall.

### **9.4 SEGREGATION IN A CORNER**

The microstructure of a segregated corner is shown in figure 6.12, with the corresponding thermal trace in figure 7.4.

The microstructure is very similar to that found in the web, section 9.3, as is the thermal trace. The main difference is found on the thermal trace. The initial rise, figure 7.4, is much higher than is found in the web trace, figure 7.3. This is because the void in the corner is very much smaller than the void in the web. The end of the thermocouple is thus very close to the molten alloy. Heat is also conducted along the thermocouple wire, because the molten alloy is so close to the end of the wire. After the initial rise the temperature then begins to gradually fall as the alloy begins to solidify from the die wall to the centre.

At approximately 7 seconds, as the external load is applied by the press, the temperature rapidly rises to a value similar to the temperature rise in the web, figure 7.3. This is due to the interdendritic fluid being forced to flow and fill the void in the corner. The temperature then falls away as the casting continues to solidify.

The circular boundary is very similar to that found in the microstructure of the segregated web, figure 6.5. It has a radius of approximately 2 mm. There is no sign of any oxides or joins and the segregated area is fully fused to the main body.

## **9.5 SEGREGATION IN A PARTICULATE COMPOSITE**

The results using a particulate MMC, chapter 8, show many similarities to those obtained for the LM25 alloy. As the base alloy for the MMC is a 7 % Si

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aluminium alloy the segregation behaviour of the base alloy would be expected to be similar to the LM25 alloy. Indeed this was found to be the case.

Both the webs and the corners exhibited the same type of segregation found in the LM25 alloy. The one extra feature of note however is the total lack of silicon carbide particles within the segregated areas. The particles are only found in the interdendritic spaces in the main body of the casting and do not cross the circular boundary.

McCoy and Wawner, [29], say that the particles are pushed along by the growing dendrites into the last-frozen regions during solidification. Hence they end up trapped in the interdendritic spaces.

In this particular case the interdendritic spaces are smaller than the particles thus when the load is applied the particles remain trapped while the interdendritic fluid can flow between the dendrites and the particles into the voids. It should be noted however that if the particles were smaller than the interdendritic spaces, either by using smaller particles or producing larger spaces, then the voids would contain not only interdendritic fluid but also particles that had been carried in with the flow.

An important point to note, when casting a particulate MMC, is that the alloy cannot be totally liquid. The MMC used, in this study, has 15 % by volume of silicon carbide particulates. Thus even when the matrix alloy is liquid there are still 15 % solids in the alloy as a whole. This may have an effect on the viscosity of the alloy, and therefore an effect on the filling of webs and corners during the initial stages of die fill when the alloy is being poured. No discernible

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differences were found however, between the MMC and the base alloy, in the present work. Further work on this aspect of squeeze casting MMC's would verify whether this is a consistent finding.

## 9.6 CONCLUSION

The work in this chapter, and the previous chapters, has shown that pressure assisted segregation in squeeze casting is a design problem and not as is widely believed a manufacturing problem. With good casting design coupled with good casting practice the production of sound squeeze castings is possible from a wide range of aluminium alloys.

Chapter 10 contains a modelling of the segregation and the design recommendations and casting parameters required to produce a sound squeeze casting.

# **CHAPTER 10**

## **DISCUSSION**

### **10.1 INTRODUCTION**

At present most components that are squeeze cast are not specifically designed to be manufactured by the squeeze casting process. They tend to be adaptations of existing components that were manufactured by a different route, usually another casting process. It has been shown, in chapter 9, by specifically designing with squeeze casting in mind these components will not only have the benefits of squeeze castings but will also be produced without many of the drawbacks described in chapter 2 section 2.8..

A model, to a first approximation, of the pressure assisted segregation mechanism is presented. As with any model, assumptions and simplifications are made and these are clearly defined in the text. The model was tested by producing squeeze cast samples from pure zinc and pure aluminium.

The use of the model in design is discussed and graphical representations of the model, in the form of constant material parameters and constant alloy depth graphs are presented.

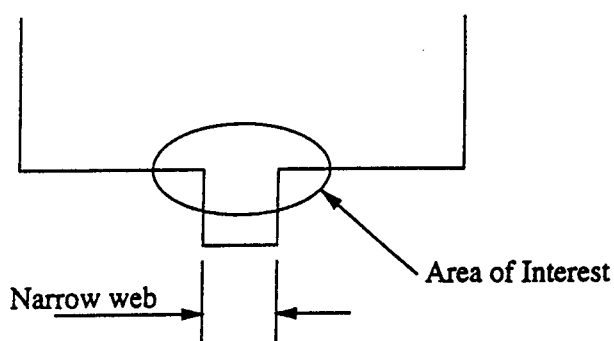
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## 10.2 THE SEGREGATION MECHANISM

Squeeze casting is particularly prone to the segregation phenomenon under investigation. A similar form of segregation is, however, found in many other casting processes. This is called exudation [81]. It occurs when voids are formed, usually between the casting and the die wall because of solidification shrinkage, which are then filled with interdendritic liquid. The interdendritic liquid is forced into the voids under pressure, the pressure being supplied by the contraction of the solid during the solidification cycle.

In squeeze casting, however, it is the application of the external load that provides the driving force for the interdendritic fluid. It can therefore be termed, pressure assisted segregation. The applied load can also, in some circumstances, plastically deform a semi-solid region to fill an unfilled section of the die. It is the ability of the process to make the interdendritic fluid flow that produces the large areas of solute rich material in pressure assisted segregation. It is this same ability, however, that allows the process to produce virtually porous free castings. A clearer understanding of the mechanisms behind the segregation phenomenon will, therefore, provide information to enable a better design of components for squeeze casting.

For clarity of discussion, for this section, it is assumed that the area of the casting of interest is the entrance to a narrow web, figure 10.1. However, the mechanisms involved are in fact very similar for the corner segregation.

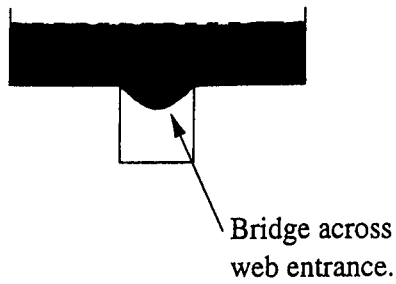


**Figure 10.1**

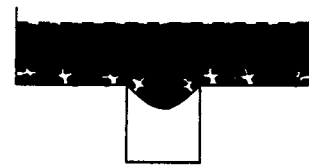
*Area of Interest*

The molten alloy is poured into the bottom half of a preheated die and the alloy flows to fill most of the die. The surface tension of the alloy, however, stops it flowing in to the narrow web. There now exists a bridge across the web entrance, figure 10.2a, held there by surface tension.

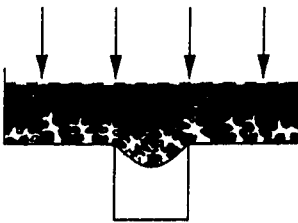
As the molten alloy is poured at a temperature of  $650^{\circ}\text{C}$  and with the die preheated to  $250^{\circ}\text{C}$  a large temperature difference exists. This temperature difference causes the molten alloy to begin solidifying almost as soon as it touches the die. Before the load is applied aluminium dendrites begin to solidify out from the die walls, figure 10.2b.



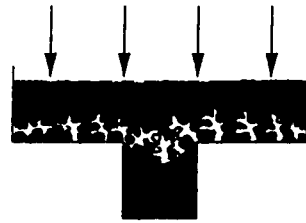
- a. Surface tension stops the alloy from filling the web.



- b. Aluminium dendrites solidifying before the external load is applied.



- c. A coherent structure of dendrites now exists. The external load is applied and the semi-solid casting is subjected to an isostatic pressure.



- d. The interdendritic fluid is forced into the web. The dendritic structure remaining in place.

**Figure 10.2**

*The Segregation Mechanism in Aluminium Alloys*

A coherent structure, (see section 3.3.1), of aluminium dendrites is formed across the web entrance. The coherent structure, bridging the web entrance, is completed before the load is fully applied. However as the load is applied gradually an isostatic pressure is built up in the liquid surrounding the structure. The isostatic pressure holds the structure in place as the load is applied. The pressure increases in the liquid, reaching its maximum when the full load has been applied, figure 10.2c.

As the majority of the dendritic structure is surrounded by interdendritic fluid it will not, therefore, collapse under the influence of the load. This is because the interdendritic fluid exerts an isostatic pressure on the structure. The isostatic pressure is high enough, however, to force the interdendritic fluid to burst through the thin skin that has formed across the web entrance.

Once the skin has been breached the fluid is forced to flow between the dendrites into the web. The web thus becomes full of solute rich material, figure 10.2d.

### **10.3 RELATIONSHIP BETWEEN SURFACE TENSION AND WIDTH OF WEB**

It is the relationship between surface tension and the width of the web that is fundamental to the segregation process. It is this that first causes the voids to be formed within the web. Once the void has been created and a coherent structure, of dendrites has formed over the web entrance, the forcing of the remaining liquid into the web, by the application of the external load, is almost

certain. An exception occurs, however, if the time delay, before the application of the load, is sufficiently long. This would allow more solidification to take place but would of course lead to a mostly non-squeeze cast structure. It is therefore assumed for the following explanation that this does not happen.

### **10.3.1 Definition of Surface Tension**

Surface tension is a property possessed by liquid surfaces whereby they appear to be covered by a thin elastic membrane in a state of tension. The surface tension is a measure of the force acting across a unit length of the surface. The units of measurement are  $\text{Nm}^{-1}$ , [82].

### **10.3.2 Surface Tension of Liquid Aluminium Alloy.**

An alloy with a high surface tension will not fill as intricate a die as one with a lower surface tension, for the same pouring temperature. Temperature affects the surface tension of an alloy as does the alloy composition. This section deals with temperature and composition in respect of the LM25 alloy used in the present study.

The surface tension,  $\gamma$ , of pure aluminium is given as

$$914 \text{ mNm}^{-1} \text{ [83].}$$



The addition of alloying elements, to pure aluminium, however, has a dramatic effect on the surface tension.

Figure 10.3 is a copy, from Metals Handbook [84], of a graph showing alloying element against surface tension for 99.99 % aluminium in an inert atmosphere, argon, at 700 to 740 °C.

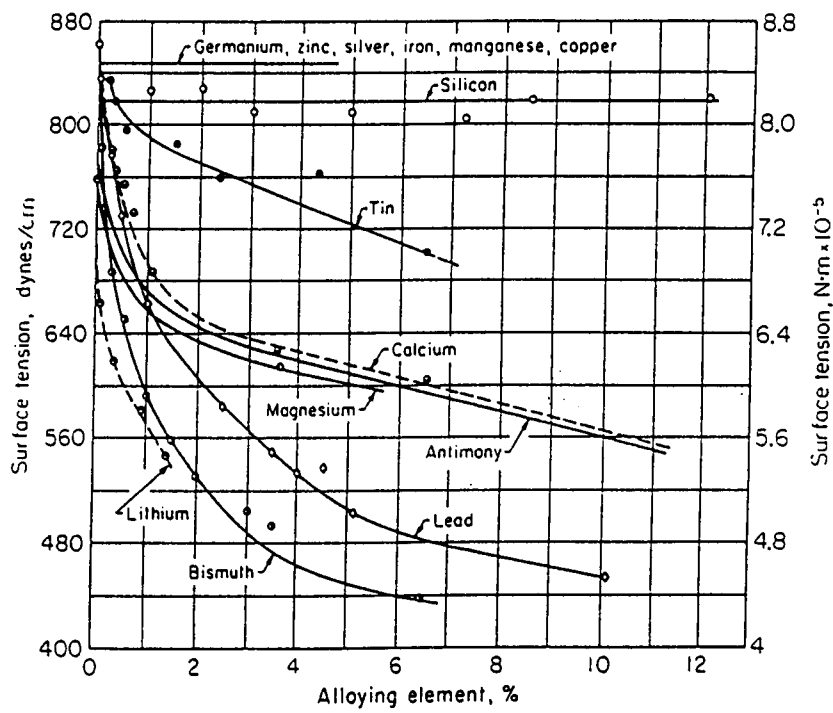


Figure 10.3

*Effect of Various Elements on Surface Tension of 99.99 % aluminium in Argon  
at 700 to 740 °C, [84]*

The graph shows that the addition of 7.35 % silicon, as found in LM25, has the effect of lowering the surface tension to approximately  $820 \text{ mNm}^{-1}$ .

However the addition of 0.5 to 0.6 % magnesium has a more dramatic effect. It lowers the surface tension to approximately  $680 \text{ mNm}^{-1}$ .

The surface tension of metals is generally dominated by the alloying element with the lower surface tension value as this element tends to concentrate at the surface, [85]. Thus the value for the surface tension used in the analysis is  $680 \text{ mNm}^{-1}$  at  $720^\circ\text{C}$ .

The temperature of the liquid also has an effect on the surface tension with an increase in temperature leading to a decrease in surface tension. The surface tension of most liquids can be found over the usual temperature ranges by the linear equation

$$\gamma = \gamma_0 + (t - t_0) \frac{d\gamma}{dt} \quad [481] \dots\dots\dots 10.1$$

Where

$\gamma$  = surface tension (at the temperature being considered).

$\gamma_0$  = surface tension at the reference temperature. ( $0.680 \text{ Nm}^{-1}$  at  $720^\circ\text{C}$ )

$\frac{d\gamma}{dt}$  = rate of change of surface tension (approx.  $-0.35 \text{ mNm}^{-1}\text{K}^{-1}$ )

$t$  = temperature under consideration ( $^\circ\text{C}$ )

$t_0$  = reference temperature ( $^\circ\text{C}$ )

For the two temperatures in this work therefore the surface tension of the LM25 alloy is, from equation 10.1;

$$\text{at } 650\text{ }^{\circ}\text{C} \quad \gamma = 0.701\text{ Nm}^{-1}$$

$$\text{at } 720\text{ }^{\circ}\text{C} \quad \gamma = 0.680\text{ Nm}^{-1}$$

Thus it is clear that temperature has little effect on the surface tension in comparison to the alloy composition. The choice of alloy is therefore a major consideration in the segregation mechanism.

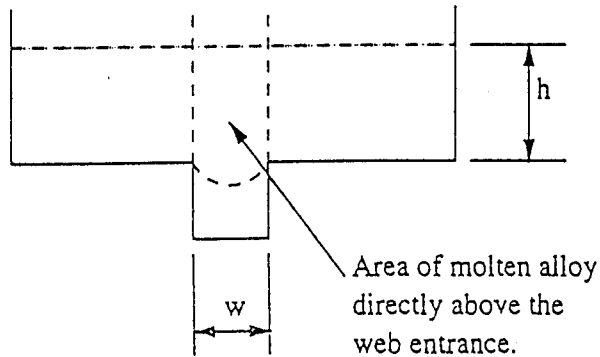
#### **10.4 Modelling Segregation in a Web**

The model will focus, initially, on the creation of voids within the web section of the die. The area of the casting immediately above the web, the area of interest, is shown in cross section in figure 10.4.

In figure 10.4 the molten alloy has been poured into the die and is assumed to be at rest prior to the application of the external load. The surface tension of the alloy is stopping the alloy from flowing into the web section.

As long as the surface tension of the alloy can support the mass of liquid above it the web will not be filled. For the web to be filled with molten alloy an increase in the static pressure head above the web is required. This can be achieved by either widening the web, increasing the depth,  $h$ , of the liquid or

using a different alloy with greater density or lower surface tension. Applying an external load or giving the molten alloy kinetic energy, by pouring into the web from a ladle, will also tend to fill the web with molten alloy. However neither of these happen at the crucial stage being studied in the practical process.



**Figure 10.4**

*Cross Section of Molten Alloy in the Die*

By considering the pressure difference across the thin film of the curved surface in figure 10.4 a model can be built by using the Young and Laplace equation, equation 10.2. This gives the pressure difference across a curved surface as

$$\Delta P = \gamma \left( \frac{1}{r_1} + \frac{1}{r_2} \right) \dots\dots\dots 10.2$$

Where

$\Delta P$  = change in pressure across the surface.

$\gamma$  = surface tension

$r_1 \& r_2$  = radii of curvature of the curved surface under consideration.

$r_1$  is at 90° to  $r_2$ .

The pressure difference across the surface  $\Delta P$  = pressure inside the bubble - pressure outside the bubble, i.e.

$$\Delta P = (P_{atm} + \rho gh) - P_{atm}$$

As both of the surfaces under consideration are exposed to the atmosphere the above equation reduces to

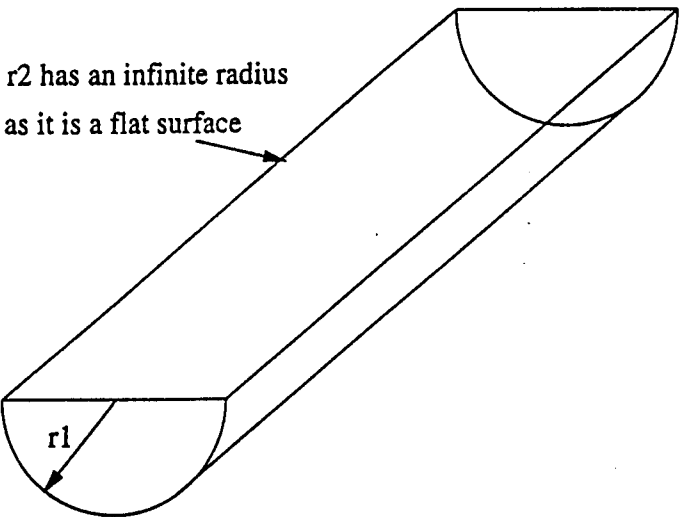
$$\Delta P = \rho gh$$

Just before the alloy enters the web the surface of the liquid at the entrance to the web is in the shape of a cylinder cut length ways,  $r_2$  therefore has an infinite radius. See figure 10.5. Equation 10.2 therefore reduces to

---

$$\rho gh = \frac{\gamma}{r_1} \dots\dots\dots 10.3$$

There is now a simple relationship between h and  $r_1$ . Thus if the depth of the liquid is known, the minimum width of web for full penetration with molten alloy, for a specific alloy, can be calculated.



**Figure 10.5**  
*Cylinder Cut Lengthways Showing  $r_1$  and  $r_2$*

Note; It has already been shown that the surface tension changes in response to temperature, section 10.3.2. It should also be noted that the density of a liquid metal also changes with temperature. The variation of the density can be found by the equation

$$\rho = \rho_o + (t - t_o)(d\rho/dt) \dots 10.4$$

Where

$\rho$  = density (at the temperature being considered) ( $\text{kg m}^{-3}$ )

$\rho_o$  = density (at its melting point) ( $\text{kg m}^{-3}$ )

$t$  = temperature being considered ( $^{\circ}\text{C}$ )

$t_o$  = temperature of melting point ( $^{\circ}\text{C}$ )

$d\rho/dt$  = rate of change of density ( $\text{mg cm}^{-3} \text{ K}$ )

Thus for the LM25 aluminium alloy, under consideration the parameters are

$$\rho = 2393 \text{ kg m}^{-3} @ 650 ^{\circ}\text{C} \text{ and } 2373 \text{ kg m}^{-3} @ 720 ^{\circ}\text{C}, [83].$$

$$\gamma = 0.701 \text{ Nm}^{-1} @ 650 ^{\circ}\text{C} \text{ and } 0.680 \text{ Nm}^{-1} @ 720 ^{\circ}\text{C}, [83].$$

$$h = 0.015 \text{ m}$$

$$g = 9.81 \text{ ms}^{-2}$$

The minimum web width can now be calculated. Thus the minimum width of web, from equation 10.3, is

---

Temperature ( °C)	r <sub>1</sub> (mm)	Web width (mm)
650	2.0	4
720	1.9	4

The figures above are not given to a greater degree of precision as there are so many other factors that can influence the result. Also for most practical applications there is no need for a greater degree of precision. The figures given do, however, present a good approximation to the results found in the experimental work. Practical considerations in the casting procedures also swamp any further precision.

The minimum web width, for full alloy penetration before load application, for this particular component and composition of alloy is 4 mm. It should be noted that this web size is for a particular size of component and composition of alloy. If the component size or the composition of the alloy is changed the minimum web width will also change.

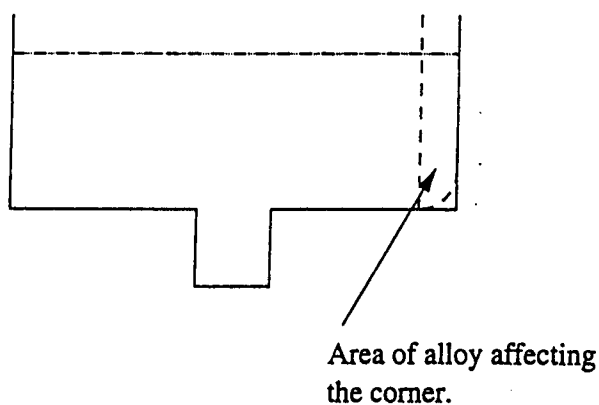
#### 10.4.1 Segregation in the Corners

The process behind the creation of a void in a corner is very similar to that for a web. It is the surface tension that initially stops the molten alloy from completely filling the corner and the application of the load which then forces the interdendritic fluid into the void.



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The model used above, for the creation of voids within a web, can also be used for the corners. In fact the model of the corner is a half section of the model for the web, figure 10.6.



**Figure 10.6**  
*Cross Section of the Die Corner*

In the experimental work the radius of the boundary of a segregated corner was found to be virtually the same as that for the boundary of the segregation in a unfilled web. The radius found for an unfilled web can therefore also be used for the radius required on the corner, assuming the web and the corner are at the same relative depth.

Intuitively the larger the casting the smaller this radius will be as the static pressure head will be greater forcing more material into the corner. The model follows this as more depth of material will produce a higher pressure which leads to a smaller radius of corner.

#### **10.4.2 Development of the Segregation Model**

The segregation occurs because a void has been created between the molten alloy and the die. Once the void has been created the rest of the segregation process follows naturally as described in section 10.2.

To further improve the model work is required on the initial rate of solidification of the alloy close to the die walls, during the time period between the filling of the die and the application of the load. The study of solidification within this time interval will establish exactly when the coherent structure, across the web, has been realised. This could then possibly lead to a smaller web being used by either controlling the solidification or by designing the die so that it can be closed much faster.

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## 10.5 GRAPHICAL REPRESENTATION OF THE MODEL

Figure 10.7 shows  $r$ , that is half the web width or the radius of a corner, plotted against  $\gamma/\rho gh$ , the material parameters of surface tension and density and the component parameter of depth.

The solid line on the graph represents the theoretical minimum web width or corner that will be filled, with no external assistance, for a given combination of  $\gamma/\rho gh$ . Combinations of  $\gamma/\rho gh$  above the line should fill the web or corner whereas combinations below the line should not.

The numbered points on the graph relate the positions of some of the castings produced from LM25 aluminium alloy used in the experimental part of the present study. They verify the model in that those above the line had filled webs whereas those below the line did not. To further verify the validity of the model more tests were conducted this time using pure zinc and pure aluminium. The parameters were entered into the model, see section 10.5.1 for zinc and section 10.5.2 for aluminium. These points are also plotted on the graph of figure 10.7, numbers 5, 6 and 7. Photographs of the components produced are shown in figures 10.8, for zinc, and 10.9, for aluminium.

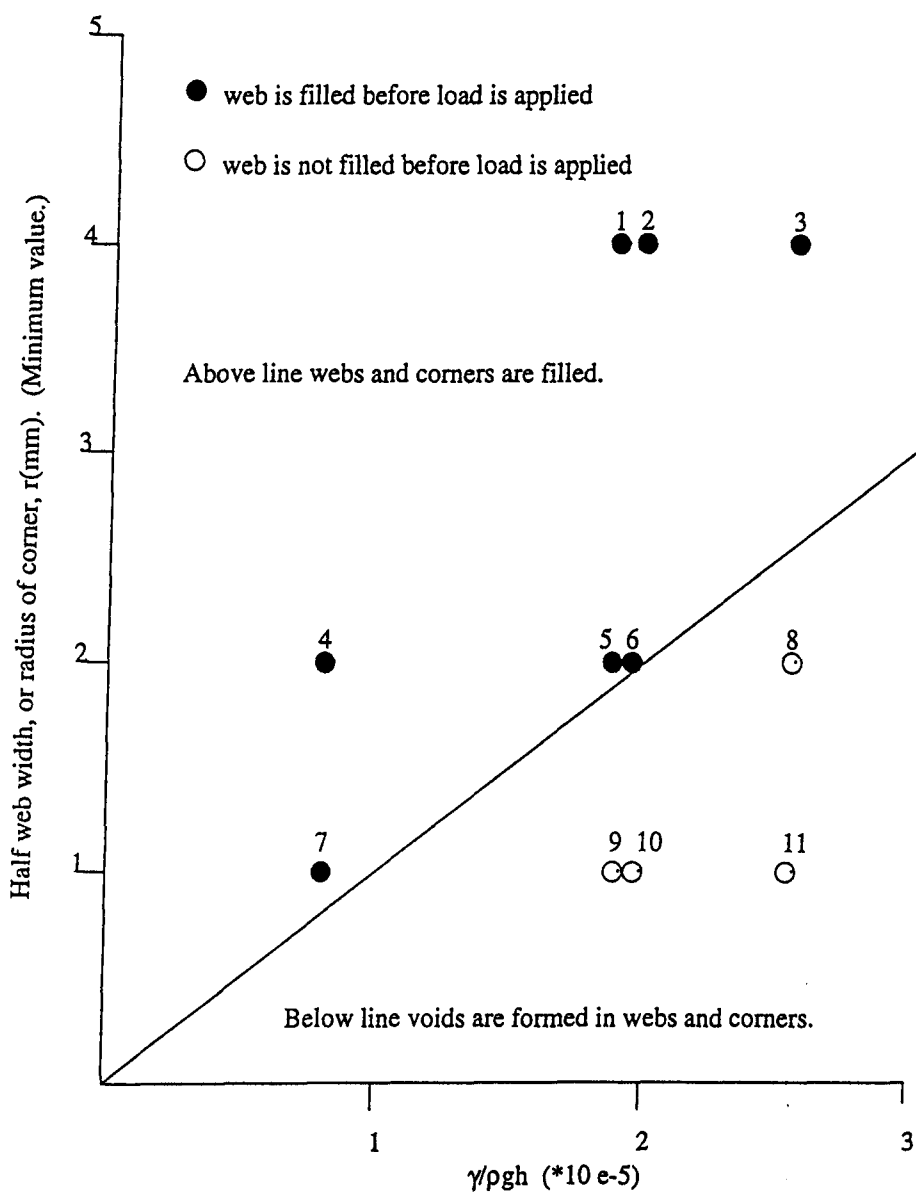


Figure 10.7

*Half Minimum Web Width, or Corner Radius Plotted Against Material Parameters of Surface Tension and Density and Component Parameter of Depth of Alloy*

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Point No.	Material	$\gamma$ (N m <sup>-1</sup> )	$\rho$ (kg m <sup>-3</sup> )	Temp.(°C)	Web Filled
1	LM25	0.680	2373	720	Yes
2	LM25	7.701	2393	650	Yes
3	Pure Al.	0.893	2368	720	Yes
4	Pure Zinc	0.777	6541	450	Yes
5	LM25	0.680	2373	720	Yes
6	LM25	0.701	2393	650	Yes
7	Pure Zinc	0.777	6541	450	Yes
8	Pure Al.	0.893	2368	720	No
9	LM25	0.680	2373	720	No
10	LM25	0.701	2393	650	No
11	Pure Al.	0.893	2368	720	No

**Table 10.1**

Points on figure 10.7

**10.5.1 Zinc**

Pouring temperature = 450 °C

 $\gamma = 0.777$  N/m @ 450 °C, [83]. $\rho = 6541$  kg/m<sup>3</sup> @ 450 °C, [83]. $h = 0.015$  m

Putting the above parameters into equation 10.3 gives

$$r_1 = 0.8 \text{ mm} \quad \text{i.e. a minimum web width of 1.6 mm}$$

The model states that the pure zinc will fill the 2 mm wide web with no pressure being applied.

The subsequent experimentation proved this to be the case. In figure 10.7 the experimental point, number 5, is clearly above the theoretical minimum line. Figure 10.8 shows a photograph of a casting produced from zinc, with no pressure being applied.

The web along the bottom edge is complete. The corner radii, on the casting were measured, and also found to be in agreement with the model. The radius of the corners is approximately 0.8 mm.

### 10.5.2 Pure Aluminium

$$\text{Pouring temperature} = 720 \text{ }^{\circ}\text{C}$$

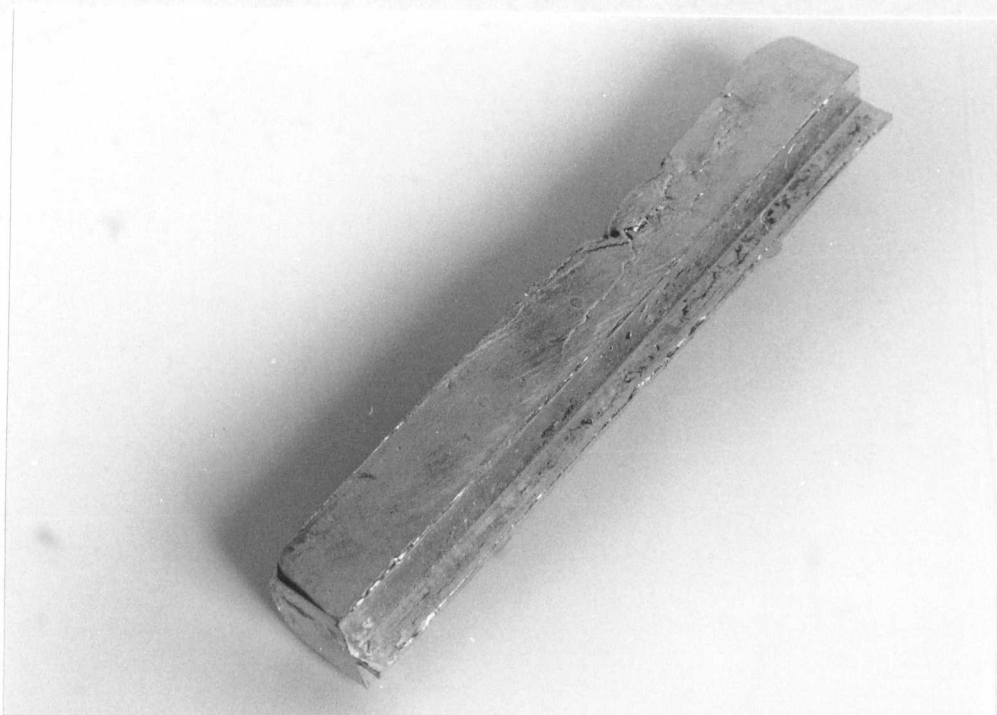
$$\gamma = 0.893 \text{ N/m @ } 720 \text{ }^{\circ}\text{C}$$

$$\rho = 2368 \text{ kg/m}^3 \text{ @ } 720 \text{ }^{\circ}\text{C}$$

$$h = 0.015 \text{ m}$$

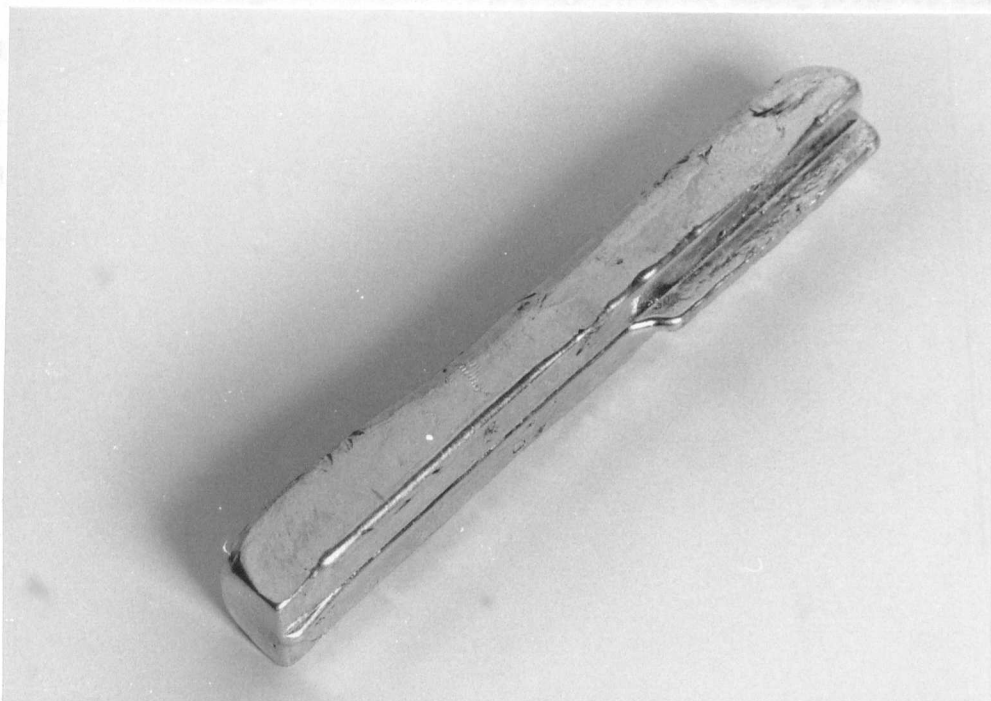
Putting the above parameters into equation 10.3 gives

$$r_1 = 2.5 \text{ mm i.e. a minimum web width of 5 mm}$$



**Figure 10.8**

*Casting Using Pure Zinc Showing Complete Filling of 2 mm web*



**Figure 10.9**

*Casting Using Pure Aluminium. The 4 mm Web is Only Filled Directly Below the Pouring Point*



The model predicts that the aluminium will not fill the 4 mm wide web as it requires a web of at least 5 mm in width. The experimental results confirm this. In figure 10.7 the point for the 4 mm web is well below the line thus the web will not be filled. Figure 10.9 shows a photograph of the casting produced from aluminium with no pressure being applied. The web is incomplete with most of its length only having a small protrusion into the web at the web entrance.

The protrusion into the web, along the remaining length of the component, has a radius of approximately 2.5 mm as do the corners, confirming the model predictions. Part of the web has, however, been filled. This is directly below the point, in the die, into which the molten alloy was poured. The aluminium on pouring has a certain amount of energy because of the pouring head and it is this that forces the alloy into the web.

A casting made using the 8 mm web was completely filled, as expected. Figure 10.7, number 7, shows the point is well into the area above the line. The radius found on the corners, of the casting, was the same as measured above, approximately 2.5 mm. A radius of approximately 2.5 mm confirms that a 5mm web is the minimum size required.

#### **10.6 POINTS THAT LIE CLOSE TO THE LINE**

There are some points that lie very close to the line and it is not very clear from the graph whether they will have a filled web or not. A closer look at the variables provide some answers.

The variables  $\rho$  and  $h$  are known to a relatively high degree of accuracy and can therefore be used with confidence. Surface tension on the other hand is notoriously difficult to measure as even a small amount of surface oxidation can have a significant effect on the result. Surface tension is also affected by the composition of the alloy and the pouring temperature of the alloy.

A particular alloy is not, in general, made up from one precise chemical composition. Take for example LM25 the alloy used in the main for this study. According to the chemical composition, BS 1490:1988 alloys, LM25 can have the composition (weight percentage)

0.20 Cu, 0.20-0.6 Mg, 6.5-7.5 Si, 0.5 Fe, 0.3 Mn, 0.1 Ni, 0.1 Zn, 0.1 Pb, 0.05 Sn, 0.2 Ti, Balance Al.

The element of most interest here is Mg. From the specification it can range from 0.2 to 0.6 % by weight. As has been shown in section 10.3.2 this can give a range of surface tensions from 0.670 to 0.705  $\text{Nm}^{-1}$ . The variation in surface tension is approximately  $\pm 2.5$  % from the Mg alone.

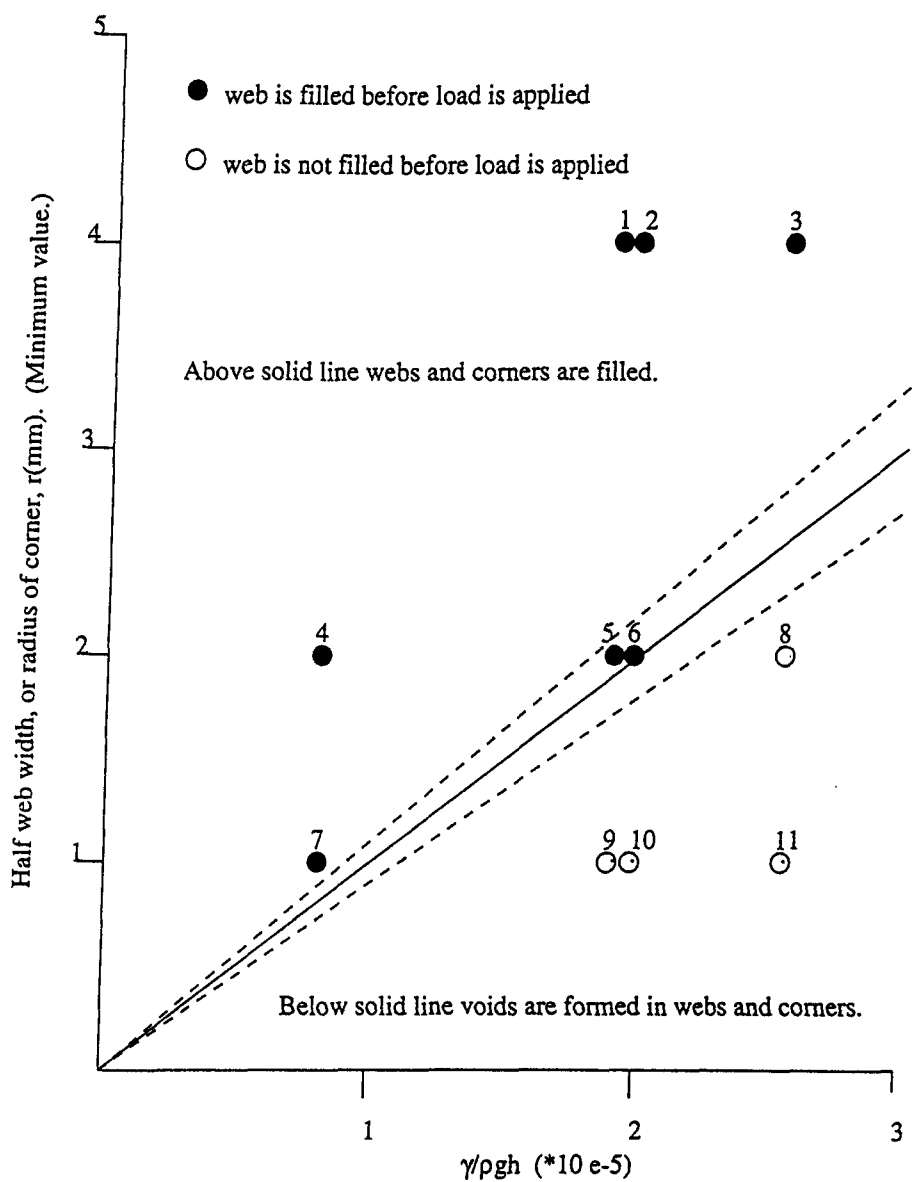
The values given in the literature for surface tension of alloys are to be treated with some caution also as the measurements were carried out in very controlled conditions. For the subsequent casting of the alloy the conditions, especially in considering the oxidation of the surface of the alloy, will not be as controlled. It is almost impossible to be accurate about the effect that this will have on the

surface tension values. An error band of  $\pm 5\%$ , however, is a reasonable estimate for this effect.

The theoretical minimum line as shown in figure 10.7 will therefore have a range of values over which it is possible for the minimum radius to exist for a particular alloy. The value for the minimum web width will also be influenced by fluctuations in melt pouring temperature, thickness of the die coating and over time die wear.

Thus it would be safe to assume an error band of at least  $\pm 10\%$  to include errors in melt temperature, die coat thickness and die wear. This is probably a conservative estimate but for a first approximation this is reasonably good.

Figure 10.10 shows the theoretical line complete with the error bands of  $\pm 10\%$ . It is clear that the points close to the line in figure 10.7 will have filled webs as they lie in the top half of the error band. In practice the webs were found to be filled. The actual amount of Mg in the batch of LM25 used is close to the maximum allowed at 0.53 %, see appendix 2, thus the surface tension is comparatively low.



**Figure 10.10**

*Half Minimum Web Width, or Corner Radius Plotted Against Material Parameters of Surface Tension and Density and Component Parameter of Depth of Alloy Complete with Error Bands of  $\pm 10\%$*

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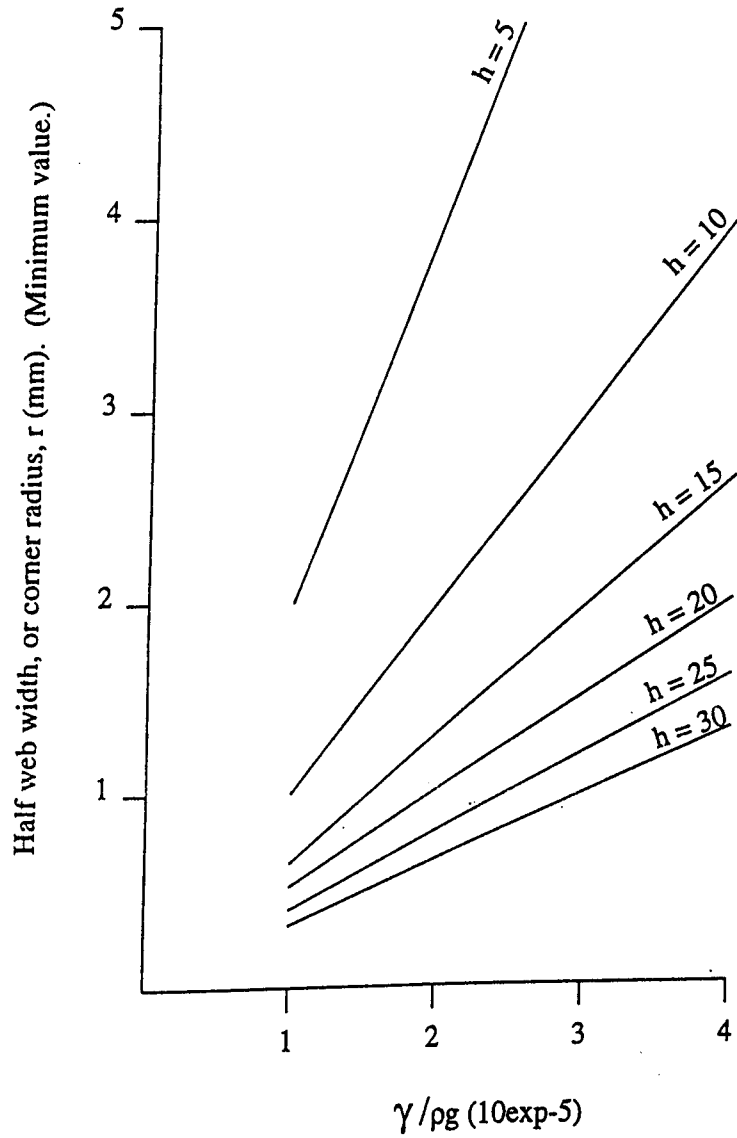
## 10.7 USING THE MODEL

Although figure 10.7 shows the model in graphical form it is not easy to relate the web size to a specific parameter. A more useful representation is achieved by plotting lines of known constant parameters on slightly altered graphs as shown in figures 10.11 and 10.12. These graphs provide a good guide for the designer but some allowance for compositional changes must be made as shown in figure 10.10.

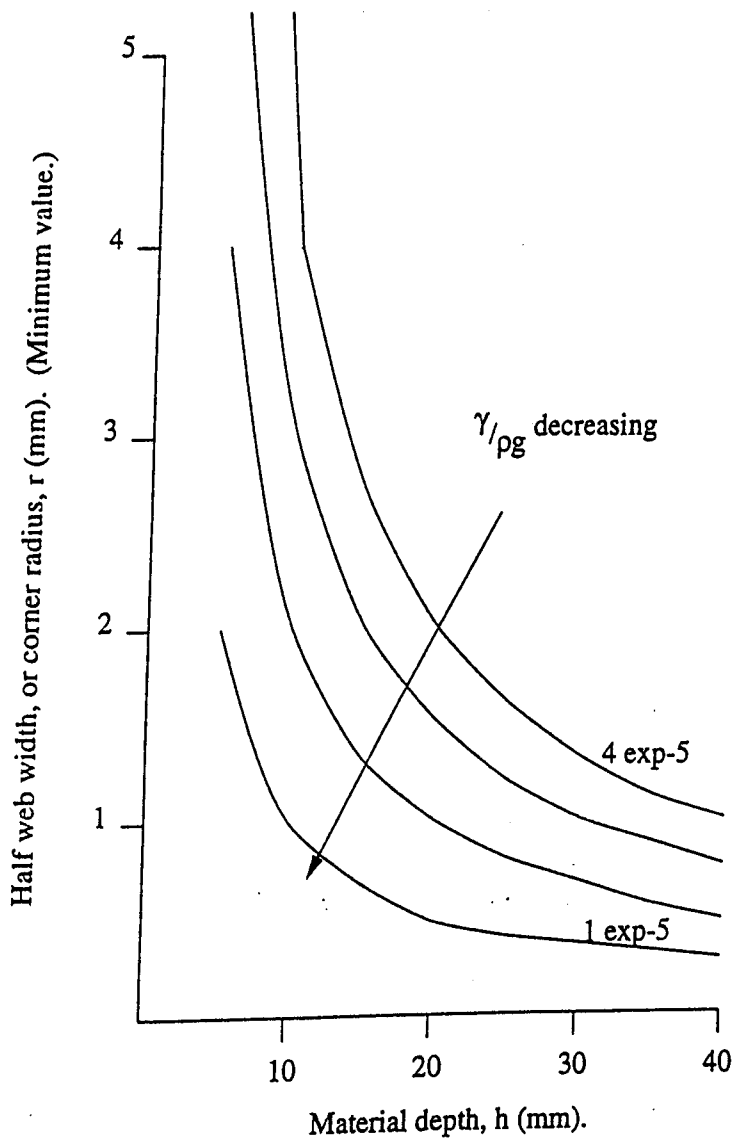
### 10.7.1 Constant Depth

Figure 10.11 shows  $r$ ,  $1/2$  minimum web width, plotted against  $\gamma/\rho g$ . The lines on the graph are for constant depths of alloy,  $h$ .

Thus for a chosen material the minimum web width or corner radius can be found for various depths of molten alloy. This is achieved by finding the material parameter,  $\gamma/\rho g$ , along the  $x$  axis and then tracing a line vertically upwards to the line which represents the depth of alloy in the die. The half web width  $r$  can then be read direct from the  $y$  axis.

**Figure 10.11**

*Half Web Width Plotted Against Material Parameters with Constant Depth Lines*

**Figure 10.12**

*Half Web Width Plotted Against Alloy Depth with Constant Material*

*Parameter Lines*

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### 10.7.2 Constant Material Parameter

Figure 10.12 shows  $r$  plotted against  $h$ . Here the lines are of the constant material parameters  $\gamma/\rho g$ , i.e. different materials.

Thus for a known size of component, i.e. a known depth of molten alloy, the minimum web width, or corner radius, can be found for a variety of materials.

## 10.8 MANUFACTURING AND DESIGN CONSIDERATIONS

Some manufacturing and design considerations can now be given for the production of squeeze castings where the presence of pressure assisted segregation is minimal or even eliminated. Although these parameters are specific to the component being produced they do provide guidance for other squeeze cast components.

### 10.8.1 Manufacturing Considerations

#### Pouring Temperature

The pouring temperature, over the range studied, has no significant effect on the segregation, section 9.2.1.

The reason for this is evident from the change in surface tension and the density of the metals at different temperatures,



$\gamma$  changes by approximately  $-0.00035 \text{ Nm}^{-1} \text{ K}^{-1}$

and

$\rho$  changes by approximately, for aluminium  $-0.28 \text{ kg m}^{-3} \text{ K}^{-1}$ , and for zinc  $-1.1 \text{ kg m}^{-3} \text{ K}^{-1}$ .

These are very small changes, which also tend to cancel each other out in equation 10.3. However as has already been stated the alloy composition can have a significant effect and as such needs to be carefully considered.

### Squeezing Pressure

The squeezing pressure, over the range studied, has no significant effect on the segregation, section 9.2.2. This is because the applied pressure is not a primary cause of the segregation but provides the driving force that forces the interdendritic fluid into a void caused by the surface tension of the alloy and the geometry of the die.

### Load Application Time

The delay in applying the load should be kept to a minimum. In the direct squeeze casting process however this time lag is quite significant. A time of up to 9 seconds is quoted for an industrial application, [8]. The best possible for the equipment used in this work was 6 seconds

thus the working practices adopted and presented in this work are a valid interpretation of the process.

### **10.8.2 Design Considerations**

#### **Webs**

The minimum width of web is dependent, in the main on the surface tension of the alloy, the density of the alloy and the depth of the molten alloy in the die. Equation 10.3 will give, to a first approximation, the size of the web that will allow the alloy to flow in freely with no external force being applied. However webs placed directly under the pouring head may be filled because of the pouring head pressure.

#### **Corners**

The same equation 10.3 can be used to find the radius that is required, on the concave corners, to produce a casting with no pressure assisted segregation in them. The larger the casting the smaller the radius of corner required.

The convex corner at the entrance to the web also requires a radius as the segregation in figure 6.9 shows. This work does not specifically cover segregation made by the waterfall phenomenon. As such a size of radius for the corner to stop this form of segregation cannot be suggested here.

# CHAPTER 11

## CONCLUSIONS

The objectives for the project, as set out in chapter 1, have been realised. The conclusions of the work are set out below following the same order as the objectives. These conclusions draw to an end the stated objectives. However there is a need for further work in some areas. Some suggestions for further work are given.

- (i) Reviewing the literature it became clear that there was a general lack of knowledge about segregation within the squeeze casting process. Only passing references were made, especially about the form of extrusion segregation that was studied in this work.
  
- (ii) The fundamental cause, of the segregation studied, was identified as the relationship between the surface tension, of the alloy, and the design of certain parts of the die. Incomplete filling of the die due to surface tension causes the formation of voids which are then filled with interdendritic material.

(iii) The application of the external load provides the driving force for the interdendritic fluid flow. This gave rise to the term pressure assisted segregation. An important point to note here is, the driving force for pressure assisted segregation, i.e. the application of an external load causing fluid flow, is also the fundamental process involved in squeeze casting. It is true to say that the process relies on the movement of liquid to produce the virtually pore free structures found in the final casting.

(iv) When squeeze casting an aluminium based particulate metal matrix composite it was found that the segregated areas were completely devoid of particulates. The particles were found to be trapped in the interdendritic spaces in the main body of the casting. The interdendritic spaces were smaller than the particles, in the particular alloy used, thus the particles become trapped between the growing dendrites while the interdendritic fluid can still flow between the dendrites and particles. The particulates were however fairly homogeneously spread throughout the main body of the casting though not to the same extent as in the as received alloy.

(v) A theoretical explanation behind pressure assisted segregation has been proposed. It is clear from this explanation that the pressure assisted macrosegregation is due, to a large extent, to the design of the

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component. Not, as is generally thought, because of a fundamental weakness with the process itself. The original design of the component plays an important role in the initial stages of the segregation mechanism.

Poor die design can allow the surface tension, of the molten alloy, to stop the die being completely filled. Voids are then formed where the molten alloy has not been able to flow. Between the pouring of the alloy and the application of the load solidification begins in regions close to the die wall. A state of coherency is then reached when the solidifying dendrites form an interlocking lattice that bridges any voids. On applying the load the interdendritic fluid is forced to flow through the lattice and into the voids which then solidifies. Thus the voids are filled with a solute rich material.

By designing the component, with squeeze casting as the chosen method of manufacture, pressure assisted segregation can be greatly reduced and in some cases eliminated. Thus components can be made which have webs with a minimum width and corners with a minimum radius.

(vi) A model, to a first approximation, has been proposed for obtaining the size of webs and corners so that no segregation is present. The parameters of surface tension, density and depth of the alloy are required.

By using

$$\rho gh = \gamma/r$$

Where

$\rho$  = density of the alloy

$g$  = gravity

$h$  = molten alloy depth

$\gamma$  = surface tension of the molten alloy

$r$  = half the web width or the corner radius

The above equation can be rearranged to give  $r$ , the minimum radius for a corner

$$r = \frac{\gamma}{\rho gh}$$

and for the webs the minimum web width,  $w = 2r$ .

(vii) The model was tested by using pure zinc and pure aluminium. From the model zinc required a web 1.6 mm wide and aluminium a web 5 mm wide. Subsequent experimentation confirmed these predictions.

(viii) Manufacturing and Design criteria were discussed for the production of sound castings. The pouring temperature, squeezing pressure and pressure duration were all shown to have a negligible effect on the segregation. The physical dimensions of the die and the alloy composition both were found to have a major effect on the segregation.

(ix) To enable a casting to be produced, using LM25 aluminium alloy, with no segregation the webs on the die were required to have a minimum width of 4 mm and the convex corners required a minimum radius of 2 mm. This is for LM25 only. For another alloy the model should be used to find the required dimensions of web and corner radii.

(x) Further refinement of the graphical interpretation of the model allowed two further graphs to be produced. The first shows half the minimum web width, or corner radius,  $r$ , plotted against material parameters for constant depths of alloy figure 10.11. And the second shows  $r$  plotted against alloy depth for constant material parameters figure 10.12.

Knowing the alloy and the size of component these graphs help the designer to produce sound squeeze castings with no segregation in the corners or webs.

## **SUGGESTIONS FOR FURTHER WORK**

### **Work on the model**

The model proposed in this work is to a first approximation. More work is required to produce a higher specification model.

What happens to the alloy in the immediate region of the die wall during pouring and in the time between pouring and the load application may have a significant effect on the model. Research into when this region actually begins to solidify, how fast and how far the solidification front advances, would help to determine what refinements the model requires.

### **Particulate MMC's**

Particulate metal matrix composites are not completely liquid, even at elevated temperatures, as the particulates are usually a ceramic. The coherency point given for the base aluminium alloy may not therefore apply to the MMC. An investigation into the effects that the particulates have, if any, on the coherency point of the alloy would provide the answer. It should be noted however that in the present study no significant difference was found in the radius, of the boundary, in either the corner or web segregated areas between the MMC and the LM25 alloy.



An interesting effect is possible if an alloy could be produced containing particles that were smaller than the interdendritic spaces. The particles would then be able to flow with the interdendritic fluid into any voids that were formed. By purposely designing the component so that a series of voids are produced along a face it may be possible on the application of the pressure to force the silicon carbide particles along with the interdendritic fluid into the voids. Thus a surface layer full of very hard silicon carbide particles would be formed on the casting.

### **Centrally segregated areas**

The centrally segregated areas are extremely difficult to model. There is no base point that can be used, as in the web for instance, as the segregated area can be of any shape and in any place. A better understanding of why these areas are prone to segregation may lead to the ability to model them and if possible design the component so as to eliminate them as far as is possible.

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## **APPENDIX ONE.**

### **Data Capture Program.**

**Data Capture program.**

Data capture program program written in GWBasic.

```

10  REM Cooling curve program by D. J. Britnell. For 3 thermocouples
20  REM
30  REM Ask for a valid data storage filename
40  REM
50  FILES
60  REM
70  INPUT "Please enter Data Storage Filename. 1 to 8 Characters:
      ",FILENAME$
80  REM
90  REM Check That Filename is Valid
100 FILELEN=LEN(FILENAME$)
110 IF FILELEN < 1 OR FILENAME > 8 THEN GOTO 40
120 REM
130 BEEP
140 INPUT " Please Enter Filename for Unix System: ",UNIXNAME$
150 REM
160 REM Check That Filename is Valid
170 UNIXLEN=LEN(UNIXNAME$)
180 IF UNIXLEN < 1 OR UNIXLEN > 8 THEN GOTO 130

```

---

```
190  REM
200  REM Open com channel 1
210  OPEN " com1:9600,N,8,2,cs,ds,cd"AS#1
220  REM
230  REM Set Defaulttemp*****
240  DEFAULTTEMP = 300
250  REM
260  REM Dimension array for data storage*****
270  N = 200
280  M = 3
290  DIM A(N,M)
300  REM
310  REM Set values for data capture from Comark*****
320  PRINT#1,"Q1110000000000000"
330  PRINT#1,"S100"
340  PRINT#1,"N200"
350  PRINT#1,"G2"
360  REM
370  REM Start of Data Capture Program
380  INPUT " Press Any Key When Ready To Start",W$
390  BEEP
400  PRINT " Program Waiting for Temperature To Be >
      ",DEFAULTTEMP," Degrees"
```

---



```
410 PRINT#1,"TO1"
420 INPUT#1, TEMPERATURE
430 PRINT "Current Temperature: ";TEMPERATURE
440 IF TEMPERATURE < DEFAULTTEMP THEN GOTO 410
450 CLS
460 REM
470 REM Start of Data Capture Via Comark
480 PRINT#1,"E"
490 REM
500 REM Upload Data from Comark
510 PRINT#1,"U"
520 FOR I=1 TO N
530 FOR J=1 TO M
540 INPUT#1, A(I,J)
550 NEXT J
560 NEXT I
570 REM
580 REM Convert Output Voltage to Temperature for Display
590 FOR I=1 TO N
600 FOR J=1 TO M
610 A(I,J) = A(I,J) * 24 + 3.75
620 NEXT J
630 NEXT I
```

---

```
640  REM
650  REM Close Data Link
660  CLOSE #1
670  REM
680  REM Save Data into Named File
690  OPEN FILENAME$ FOR APPEND AS#1
700  FOR I=1 TO N
710  FOR J=1 TO M
720  WRITE #1 ,A(I,J)
730  PRINT A(I,J)
740  NEXT J
750  NEXT I
760  CLOSE #1
770  REM
780  REM Save Another Data Set of Data for Use on Unix System
790  OPEN UNIXNAME$ FOR APPEND AS#1
800  Z = 1
810  FOR J = 1 TO M
820  WRITE #1, " Thermocouple:",Z
830  PRINT " Thermocouple",Z
840  FOR I = 1 TO N
850  WRITE #1,A(I,J)
860  PRINT A(I,J)
```

---

```
870  NEXT I
880  Z = Z + 1
890  NEXT J
900  CLOSE #1
910  REM
920  PRINT " Data Saved To File: ";FILENAME$
930  REM
940  PRINT " Data also Saved to Unix File: ";UNIXNAME$
950  REM
960  END
```

## **APPENDIX TWO**

### **Consumable Materials Used in the Melting of Alloys**

## **Materials Used in the Melting of Alloys**

### **Specification of alloys**

Attached are a copy of the manufacturers specification sheets; for LM25 aluminium alloy and a 7% Si aluminium alloy containing 15 % by volume of silicon carbide particles. These alloys were used in the present study.

### **Degassing and grain refiner:**

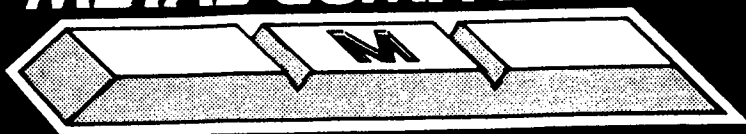
Foseco Degasser 190

### **Cover flux:**

Foseco Coverall 11

# MIL-VER METAL COMPANY LTD.

CORONEL AVENUE  
ROWLEYS GREEN  
COVENTRY CV6 6AP  
Telephone (0203) 667098



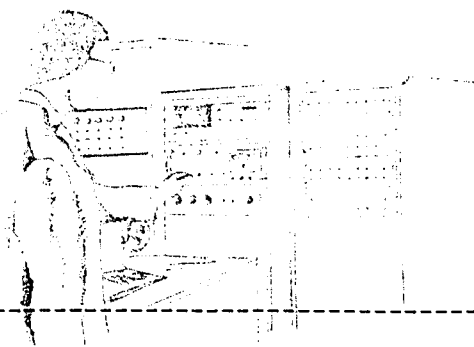
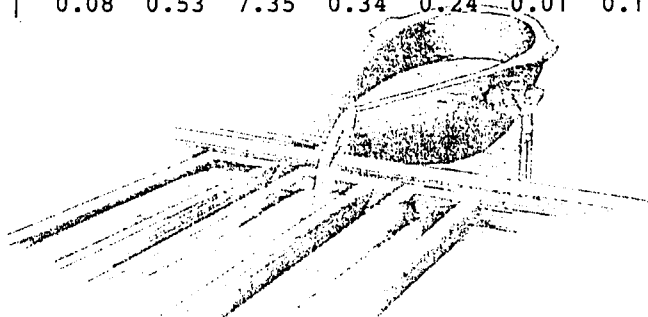
MANUFACTURERS OF ALUMINIUM INGOTS

TO:  
UNIVERSITY OF WARWICK.  
COVENTRY,  
WEST MIDLANDS.

DATE: 19.6.89  
SALES CONTRACT NO: 1995  
ALLOY: LM25  
COLOUR CODE: YELLOW/BLUE  
NO.OF BUNDLES: 2  
WEIGHT: 1129 KG

## CERTIFICATE OF ANALYSIS

CHARGE NUMBER	CU	MG	SI	FE	MN	NI	ZN	PB	SN	TI	SR
127	0.08	0.53	7.35	0.34	0.24	0.01	0.10	0.02	0.01	0.07	0.00



For and on behalf of  
MIL-VER METAL COMPANY LTD.

CHEMIST

**Postadresse / Postal address**

Hydro Aluminium a.s  
Metallurgical R&D Centre  
P.O. Box 219  
N-6601 Sunndalsøra  
Norway

Telephone + 47 71 69 30 00  
Telex + 56 15 222 sual n  
Telefax + 47 71 69 36 02  
+ 47 71 69 36 60 Casthouse Dept.

Til / To	Navn / Name	Telefax nr. / No.
	G.Key	095-44-21-482-3702
	Adresse / Address	
Rover Group LTD, POB 41, Longbridge, MIRMINGHAM B312TB, UK		

Fra / From	Dato / Date	Antall sider / No. pages
Olle Johan Sørheim	9-Jun-94	1

**Kommentarer / Comments****Re. MMC MATERIAL SUPPLY to postgraduate student David Britnell**

cc: Carl Lewin, R&D Centre, Karmøy (fax 52854380) and O Vorren, HA Årdal, Norway (fax 57649550).

Ref. to Your contact with Mr. Carl Lewin, HA R and D Centre, Karmøy (telefax dated 060694), where You ask for 60-100 kg MMC material AlSi-7/LM 25/BS1490 + 10-15 % SiC. As You may know, we have a pilot plant for production of MMC here at our R and D Centre at Sunndalsøra, Norway. At the moment I'm afraid we don't have the requested material in Ø 50 mm small billet/bar.

The nearest we can offer You is:

1. Remelt ingot of ca size (80 mm x 80 mm x 700 mm). Weight ca 12 kg each.

Alloy: AlSi7Mg.6 + 15 vol% SiC (ca 20 µm). As casted.

2. Small billet/round bar of dimension Ø 95 mm). Various length.

Alloy: AlSi7Mg.3 + 10 vol% SiC (ca 20 µm). As casted.

3. Small billet/round bar of dimension Ø 95 mm). Various length.

Alloy: AlSi7Mg.6 + 15 vol% SiC (ca 20 µm). As casted.

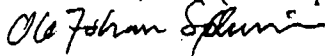
4. Small billet/round bar of dimension Ø 78 mm). Various length.

Alloy: AlSi7Mg.6 + 15 vol% SiC (ca 20 µm). As casted.

We have this material on storage, and can supply You this within a few days. Material cost will be ca 40 NOK/kg. Hope this is sufficient information at the time being and please don't hesitate to contact me if You need some additional information.

Best regards from R and D Centre, Sunndalsøra, Norway.

Ole Johan Sørheim (PMMC project leader)



## **APPENDIX THREE**

### **Metallography**



### **Mounting of Samples.**

Samples were cut and mounted in an epoxy hot mounting resin with filler for best edge retention and planeness. Material supplied by Struers.

Code: RESIF

40100024

4325-4451

The backing resin.

Black bakelite resin

Code: RESIK

**Grinding and Polishing Procedures**

**Al-Si cast alloys.**

Grinding	1	2	3	4
Abrasive	SiC	SiC	SiC	SiC
Grit/grain size, grit	220	500	1200	4000
Disc/cloth	Paper	Paper	Paper	Paper
Lubricant	Water	Water	Water	Water
Speed, rpm	300	300	300	300
Pressure setting	5	5	5	5
Time, seconds	30	30	30	60

Polishing	1	2
Abrasive	Diamond	OP-U
Grit/grain size, $\mu\text{m}$	3	0.04
Disc/cloth	DP-Dur	OP-Nap
Lubricant	Blue	Water (Drip)
Speed, rpm	150	150
Pressure setting	5	3
Time, seconds	120	60-120

**Al-SiC MMC.**

Grinding	1	2	3
Disc/support	Metal bonded	Resin bonded	Resin bonded
Abrasive	Diamond	Diamond	Diamond
Grit/grain size, $\mu\text{m}$	40	30	10
Lubricant	Water	Water	Water
Pressure setting	4	4	4
Speed, rpm	300	300	300
Time, seconds	Until plane	120	120

Polishing	1	2	3	4
Polishing cloth	Pan W	Pan W	Pan W	OP-U
Abrasive	Diamond	Diamond	Diamond	OP-Chem
Grit/grain size, $\mu\text{m}$	15	6	1	0.04
Lubricant	Blue	Blue	Blue	Water
Pressure setting	3	3	3	3
Speed, rpm	150	150	150	150
Time, seconds	120	120	60	60