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Grain growth behaviour on reheating Al-Nb containing steel in the homogenised condition

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Grain size development during reheating is important to the mechanical properties of steels, and non-uniform grain growth including abnormal and bimodal grain growth is undesirable for steel manufacturing. In this paper, reheating treatments for Al-Nb containing steel (0.057 wt% Al and 0.019 wt% Nb) were selected based on Thermo-Calc predictions for the dissolution temperatures of precipitates in the steel for a fully homogenised condition. Abnormal grain growth occurred in the homogenised Al-Nb sample during reheating, associated with random dissolution of the grain boundary pinning AlN precipitates; whilst bimodal grain growth was not observed. So material in a homogenised condition would not be expected to provide an fully uniform grain structure during reheating treatment.

Key words: HSLA steel, Homogenisation, Reheating treatment, Abnormal grain growth, Bimodal grain growth

Introduction

High strength low alloy (HSLA) steels are widely used in structural applications due to their greater combination of strength and toughness than conventional C-Mn steels [1-5]. These properties are derived from grain refinement and precipitate strengthening linked to minor addition of V, Ti and Nb (less than 0.1 wt%) and thermo-mechanically controlled rolled (TMCR) processing. To achieve the desired fine, uniform grain size the steel compositions and processing parameters are designed to minimise grain growth during reheating and to refine the grain size through recrystallisation during hot deformation. However, undesirably large grains can form during reheating, such as abnormal or bimodal grain structures observed in the HSLA steels, which result in a non-uniform final recrystallised grain size and increased

scatter in toughness properties [6, 7]. The conditions under which these non-uniform grains form need to be investigated and determined so that the process window for optimum grain size development can be predicted.

Abnormally large grain growth, which results in a significantly large grain surrounded by a matrix of much smaller grains, has been studied by many researchers [8-15] who identified the phenomenon as being associated with reheating temperatures at/ or close to the microalloying particle's dissolution temperature resulting in the local unpinning of some grain boundaries, but not others so that growth rates are unequal. The large grains with concave boundaries are expected to grow at expense of the adjacent small grains when local unpinning occurs on the boundaries of large grains. Abnormal grain growth was reported to occur at the austenite coarsening temperature which was determined as below the precipitate dissolution temperature by 125K in a Nb containing steel [16], or slightly less by 40-70K in a Nb-Al containing steel [17]. The occurrence of this abnormal grain growth (AGG) has been defined by the relative difference (*RD*) [18] in grain sizes in the grain size distribution, as shown below:

$$RD = [\frac{(GS_{abnormal} - GS_{normal})}{(GS_{normal})}] \tag{1}$$

Where $GS_{abnormal}$ is the abnormally large grain size, and GS_{normal} is the mode grain size of the distribution (dominated by the smaller grains). It was reported that the abnormal grain growth occurred when the *RD* exceeded 0.9 [18]; specifically, abnormally large grains were deemed to be present if their sizes were greater than approximately 2 times that of the mode grain size [19, 20].

Several studies have reported bimodal grain size development in steels with segregated compositions which is linked to the inhomogeneous precipitate distribution [21-26]. Bimodal distributions differ from abnormal grain growth in that the

3

coarse and fine grains alternate in a banded appearance rather than being isolated large grains surrounded by a matrix of finer ones. The inhomogenous pinning force is expected at critical reheating temperatures where the precipitates dissolve in the solute-depleted (usually dendritic) regions but still present in the solute-enriched (usually interdendritic) regions, which results in non-uniform grain growth where the bands of coarse and fine grains have been observed. In rolled material these would be associated with elongation of solidification segregation regions during hot deformation.

The studies of these inhomogeneous grain size distributions have been carried out on cast or cast and hot rolled forms of the steels with few reports of homogenisation. Those homogenisation steps that have been carried out are either insufficient ([12] which used 2 hours at 1200 °C when 50 hours is predicted for the full diffusion of [Nb]) or have not been carried out on steels not containing slow diffusing elements such as Nb, e.g. where the microalloying element is Al [14]. These studies have also concentrated on banded bimodal structures rather than abnormal grain growth and so the question remains as to whether segregation is essential for these inhomogeneous grain structures to develop, in which case reduced segregation levels should decrease the risk of their formation, or whether they can form in homogenised material, in which case different strategies are needed for their avoidance. Thus the aim of this study is to characterise the austenite grain growth behaviour in an Al-Nb containing HSLA steel during reheating treatment after complete homogenisation using the criteria above to assess the development of abnormal / bimodal grain distributions and thus establish whether segregation has an integral part to play in development of these heterogeneous grain distributions.

4

Experimental

A laboratory as-cast ingot of Al-Nb containing HSLA steel measuring 340 mm long with a 130×130 mm cross-section was supplied by TATA Steel, UK; the chemical composition of which is listed on Table 1.

Table 1 Chemical composition (wt %) of the as-cast Al-Nb containing HSLA steel

C	Si	Mn	P	S	Cr	Mo	Ni	Al	N	Nb	Ti	V
0.1	0.29	1.42	0.018	0.004	0.01	0.005	0.32	0.057	0.008	0.019	0.001	0.052

Specimens 10×20×60 mm in size were taken from the quarter-width and depth position of cross-sections of the as-cast ingot steel and encapsulated in silica tubing under a partial pressure of argon. These were subjected to an homogenisation treatment at 1300 ºC (temperature needed for complete dissolution of all precipitates predicted by Thermo-Calc 4.0 using TCFE-7 database and bulk steel composition) for 24 hours (calculated time for Nb to diffuse a distance corresponding to half of the SDAS ~ 75 μm, according to previous studies by Kundu [27]). As shown in Figure 1, reheating trials with a heating rate of 3.8 ºC/s (measured by K-type thermo-couple) were carried out for one hour over the temperatures range of 1075 ºC – 1200 ºC determined from Thermo-Calc predictions for various precipitate dissolution temperatures in the homogenised (bulk composition) condition. All reheated samples were quenched to give martensite, sectioned, mounted, polished to a 1 μm diamond paste finish and etched in 2%-nital (2 ml HNO_3 in 98 ml ethanol) to reveal the microstructure or in hot (60 ºC) saturated aqueous picric acid (20g picric acid, 1000ml H_2O, 14 drops HCl and 5 ml TEEPOL) with magnetic stirring, to reveal the prior austenite grain structure. If required, further light polishing (1 μm finish) was carried out to improve the clarity of the boundaries in contrast to the matrix.

The samples were examined using a Zeiss Axioskop-2 optical microscope equipped with AxioVison image capture software. ImageJ software (windows version) was used to measure and analyse the proportion of ferrite in the ferrite and pearlite microstructure, and the prior-austenite grain size (measured manually at least 1000 grains based on the ECD (equivalent circle diameter) method) in the martensitic structure. The grain size distribution (in grain size classes) was used in the present study to characterise the different grain growth behaviour, such as uniform and non-uniform (leading to abnormal and / or bimodal grain size distributions).

A JEOL 7000 field emission gun scanning electron microscope (FEG-SEM) operated at 20 kV and equipped with an Oxford INCA energy dispersive X-ray spectroscopy (EDS) system was used to characterise the microalloying precipitates' composition in the homogenised and re-heated samples. EDS mapping was also used to characterise the microalloying precipitates' spatial distribution. Moreover, secondary-electron (SE) imaging for morphology analysis and back-scattered electron (BSE) imaging for compositional contrast (due to different atomic numbers) were used to identify the Al-rich (generally dark) and Nb-rich (normally bright) particles.
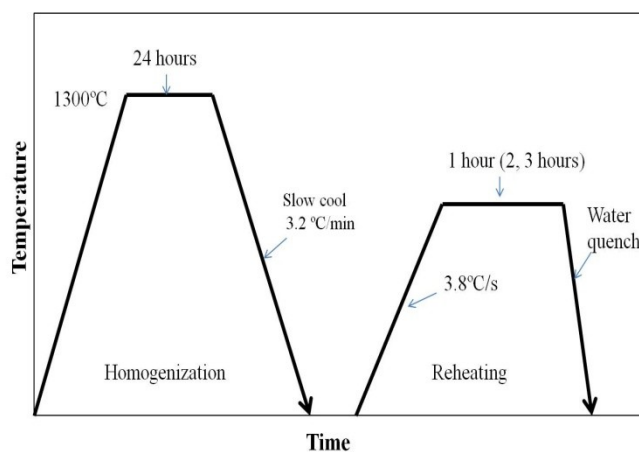

Figure 1 Schedule of homogenisation treatment and reheating treatment in the Al-Nb containing steel.

Results and discussion

Initial microstructure for Al-Nb containing steel in homogenised condition

The microstructure after homogenisation and slow cooling, Figure 2, is largely ferritic with pearlite present mostly along the ferrite / ferrite grain boundaries. The volume fraction of ferrite was of around 95% close to the equilibrium volume fraction of 98% predicted by Thermo-Calc, and EDS revealed a relatively uniform content of Mn (approximately 1.3 – 1.5 wt%, as seen in Figure 3) which is close to the bulk composition of Mn in Table 1. Some variation in [Mn] is expected across pearlite (and observed) due to the diffusional nature of the transformation from austenite during slow cooling. However, similar bulk Mn levels would be expected to give segregated ranges between 1.0 and 1.8 wt% in the as-cast condition. No dendritic structure was observed in the homogenised Al-Nb steel whilst the pearlitic structure was distributed uniformly in the matrix of ferrite.
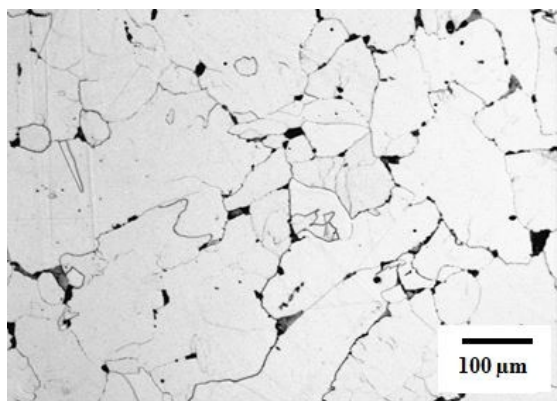

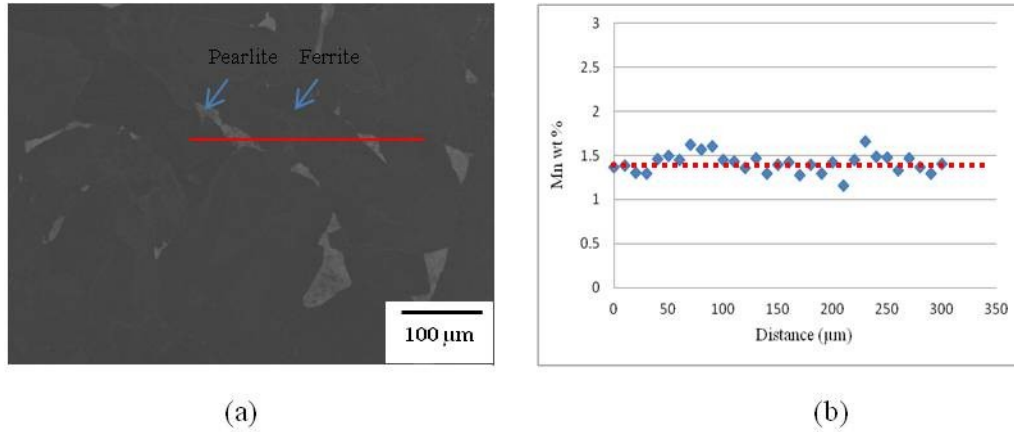Figure 2 Optical micrograph of the homogenised Al-Nb containing steel

Figure 3 (a) SEM result shows the line-scan EDS analysis crossing ferrite (dark phase) and pearlite (bright phase) in the homogenised sample along the selected line (red line); (b) contents of Mn against distance (bulk Mn content of 1.42 wt% is indicated as red dotted line)

Precipitate characterisation in the homogenised sample was carried out using SEM, with SE imaging (Figure 4(a)) for morphology analysis, and BSE imaging was used to detect the particle type since the Al-rich particle appeared dark and Nb-rich particles appeared bright, as shown in Figure 4 (b). EDS analysis was used to further confirm the type of selected particles indicating peaks of Al and Nb, as seen in Figure 4 (c) and (d) respectively. The microalloying particles were observed randomly in the matrix with no regions having noticeably higher or lower concentrations of particles (e.g. precipitate-rich or precipitate-depleted regions) appearing in the homogenised sample. Figure 5 shows that a larger number of Nb-rich particles in the size range of 150-200 nm are present than that of Al-rich particle, but the maximum size of Al-rich particles (400 nm) appears larger than that of the Nb-rich particle (300 nm), which would indicate greater growth of Al-rich particles than Nb-rich particles during cooling after the homogenisation treatment, but a greater nucleation rate for the latter.
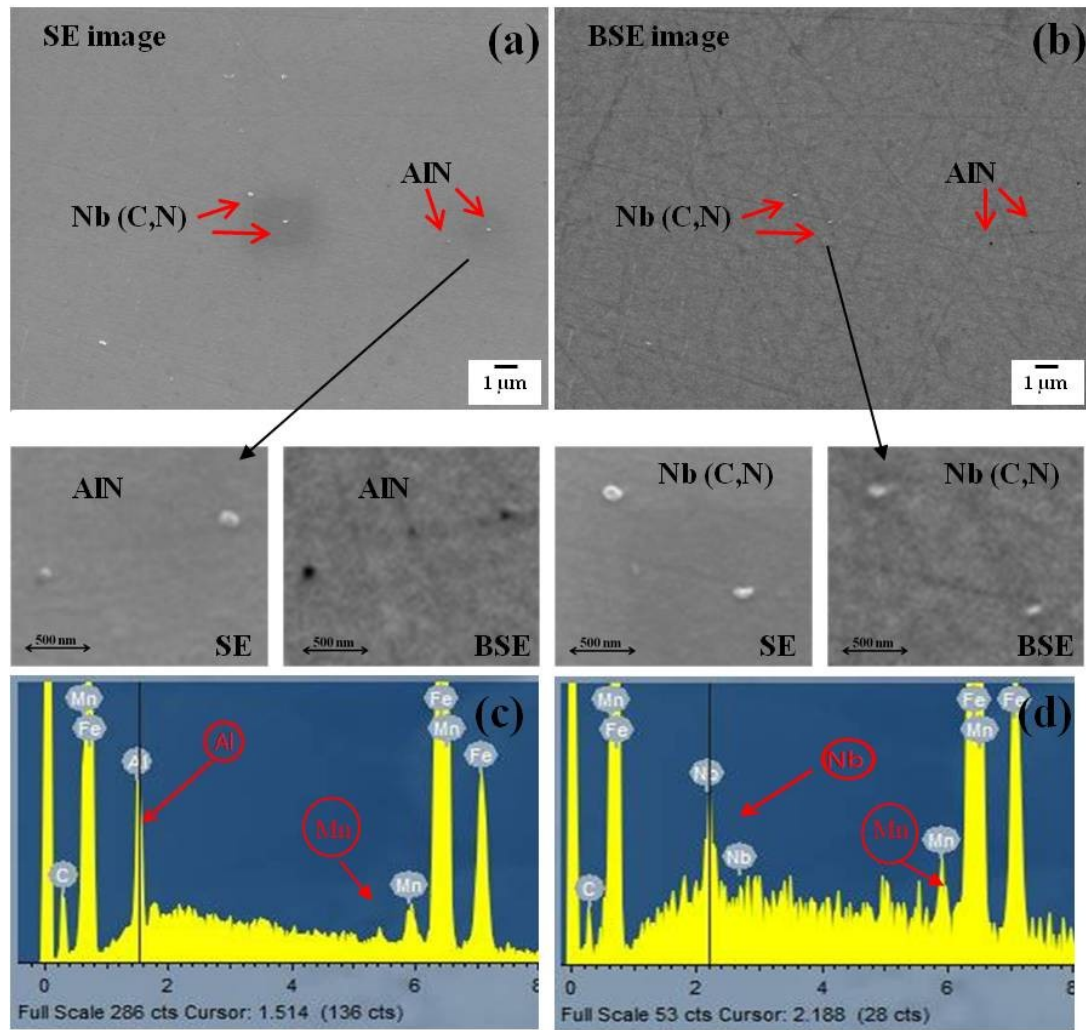
8

Figure 4 Characterisation of Al-rich and Nb-rich precipitates in (a) morphology analysis under SE image; (b) dark Al-rich particles and bright Nb-rich particles in BSE image; (c) and (d) EDS trace indicating the Al and Nb peak in the spectrum taken from the investigated particles respectively.
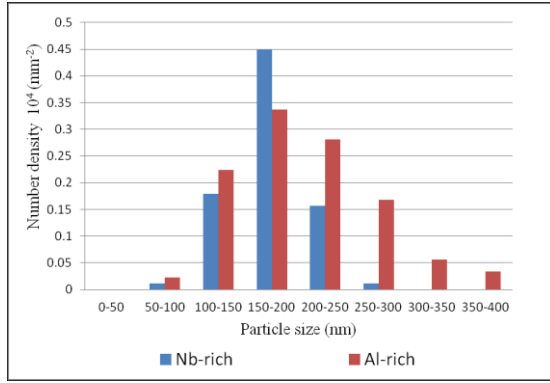
Figure 5 Distribution of Al-rich and Nb-rich particle with number density in the homogenised Al-Nb steel.

Thermo-Calc prediction of microalloying precipitates' thermal-stability

Grain growth during reheating is strongly controlled by the stability of the boundary-pinning particles, i.e. determined by precipitates' dissolution temperature, which can be predicted by Thermo-Calc software using the chemical composition of the Al-Nb steel listed in Table 1 as input. The predicted precipitate volume fractions as a function of temperature in the homogenised condition are summarised in Figure 6. It indicates that the predicted dissolution temperature of Nb(C,N) is lower than that of AlN which is mainly due to a lower amount of Nb in the bulk composition compared to Al in this steel.

The published criteria for non-uniform grain growth would therefore indicate that abnormal growth could occur at temperatures close to 1090 ºC and 1170 ºC, where dissolution of each of the potential boundary-pinning particles would take place and so the kinetics of dissolution might result in variation in local pinning forces and so differential grain growth. Abnormal grain growth is more likely at temperatures around 1170 ºC as the reduction in volume fraction with temperature is more rapid for AlN than for Nb(C, N) at the lower temperature giving greater risk of variation in

10

local pinning forces. Bimodal grain distributions, if they occur, would form between these regions, i.e. in the temperature range 1100 – 1150 ºC.
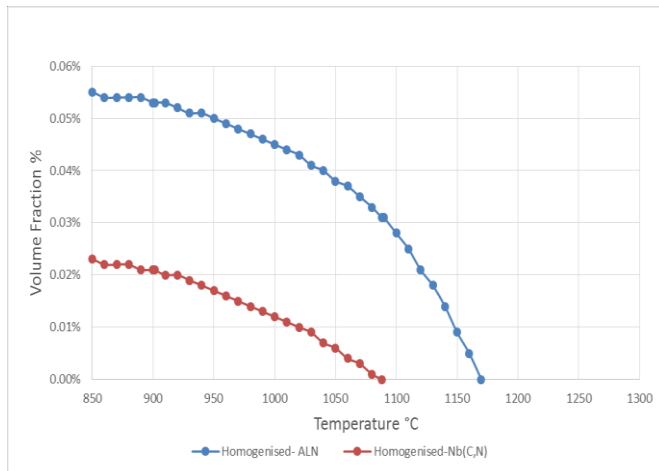

Figure 6 Dissolution temperature of precipitates for homogenised condition

Abnormal grain growth in homogenised Al-Nb containing steel

The prior austenite grain sizes were initially characterised in terms of the mode grain size and the 95% grain size (representative of the upper extreme of grain size in the accumulated area fraction from size distribution). The variation in these two parameters for the reheating trials is shown in Figure 7. The variation in both mode and 95% grain size is slight for reheating (1 hour) at temperatures up to 1130 ºC, despite the expected full dissolution of Nb(C, N). The behaviour shown in Figure 7 for this temperature range is consistent with a gradual reduction in boundary pinning forces (and rise in boundary mobility), but in a uniform manner so that the mode and 95% grain size increase in parallel.

A uniform prior austenite grain structure was observed after one hour reheating at 1100 ºC, as shown in Figure 8 (a), which was over the dissolution temperature of Nb(C,N) at 1090 ºC, predicted by Thermo-Calc. A uniform size distribution plotted in Figure 8 (b) indicates a small mode grain size of around 27µm and confirms that the

11

bimodal grain size distribution has not occurred at this temperature. This is probably due to the significant pinning effect provided by the large amount of Al-rich particles in the matrix of small grains, as shown in Figure 9(a, b), when Nb-rich particles have dissolved at this temperature, e.g. no Nb-rich precipitates are observed in the EDS mapping result shown in Figure 9 (c) after quenching to room temperature.

The levels of AlN (the fraction is approximately 0.0002 from Figure 6) and their sizes, coupled with any remaining Nb solute drag are sufficient to effectively pin the prior austenite grain boundaries. The parallelism of the mode and 95% grain size trend lines would also indicate that bimodal grain distributions are not developing to any major extent.
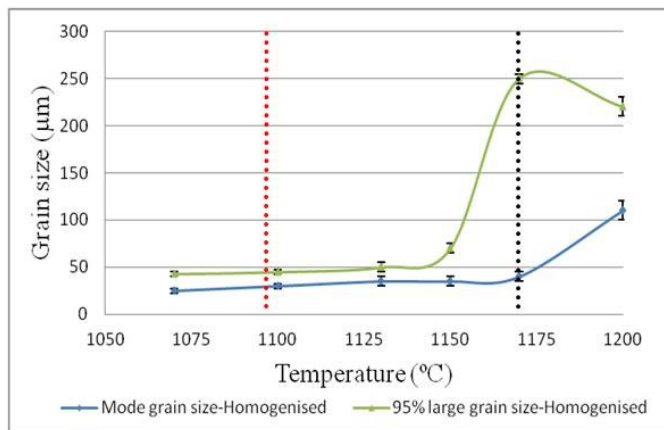

Figure 7 Variation in prior austenite grain size for reheating homogenised steel for 1 hour at temperatures between 1070 ºC and 1200 ºC. The red and black dashed lines indicate the dissolution temperature of Nb(C,N) and AlN respectively.
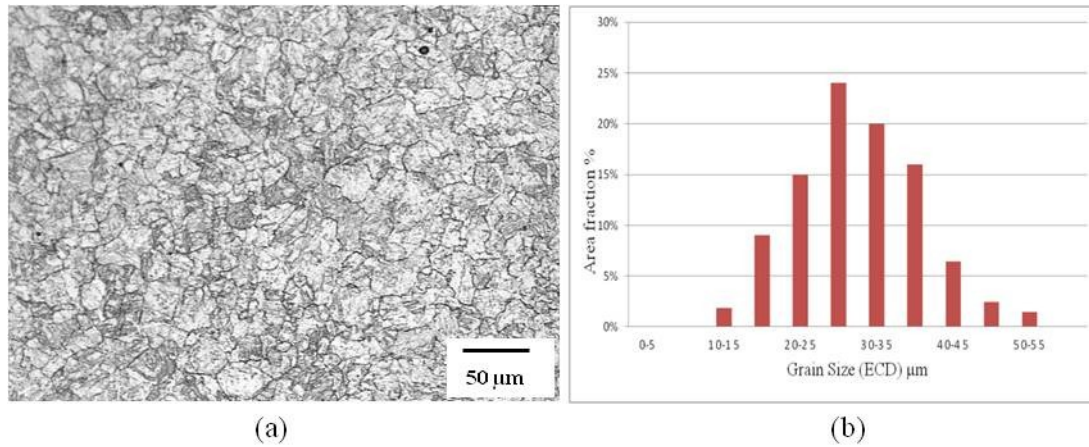
Figure 8 Prior austenite grain structure and grain size distribution for the homogenised steel after reheating for one hour at 1100 ºC.
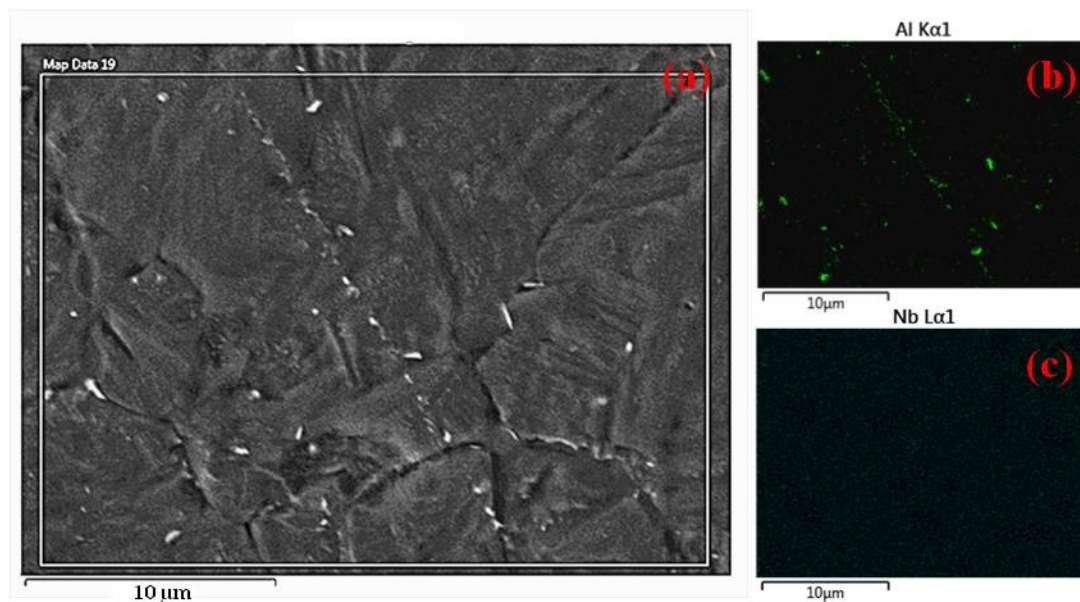

Figure 9 (a) Characterisation of precipitates in the homogenised Al-Nb steel after reheating at 1100 ºC corresponding to the EDS mapping result of (b) Al and (c) Nb.

Increasing the reheat temperature to 1150 ºC sees a larger increase in the 95% grain size with no similar increase in mode grain size, consistent with some grains increasing markedly in size and consuming finer grains around them to offset the effect of the uniform smaller growth of these grains on the mode grain size. This temperature is expected to be associated with a reduction in the volume fraction of AlN to below 0.01 %. This process is most marked for the next reheating temperature

(1170 ºC), where full AlN dissolution is predicted. At this temperature the mode grain size remains below 50μm, but the 95% grain size has increased to 250μm. The abnormal nature of grain growth is confirmed by visual inspection and the grain size distribution in Figure 10. An isolated, abnormally large grain surrounding by finer grains is shown in the Figure 10 (a), and the accompanying grain size distribution in Figure 10 (b) shows a largely normal distribution around the mode grain size (indicative of normal grain growth – NGG), but isolated numbers of abnormally large grains (abnormal grain growth – AGG) rather than a smoother skewed large grain size distribution (in addition to the NGG distribution) indicative of bimodal structures. It would appear, therefore, that segregation is required for bimodal grain distributions. The mode grain size of 35 μm is observed as normal grain size whilst the 'abnormal grains' are identified when the grain size is above around 70 μm with a low area fraction in an extensive size range (from 70 μm to 290 μm) being observed. A large grain size (in 95% accumulated area fraction) to average grain size ratio of 6 – 7 has been found during abnormal grain growth which is significantly greater than the abnormal grain growth criterion of "2". Abnormally large grains occurred randomly either individually or in low number clusters because grain growth in most regions was expected to be prevented by Al-rich particles that were still dissolving or Al and N from dissolved particles that had not diffused away from the boundary and so provided continued solute drag.
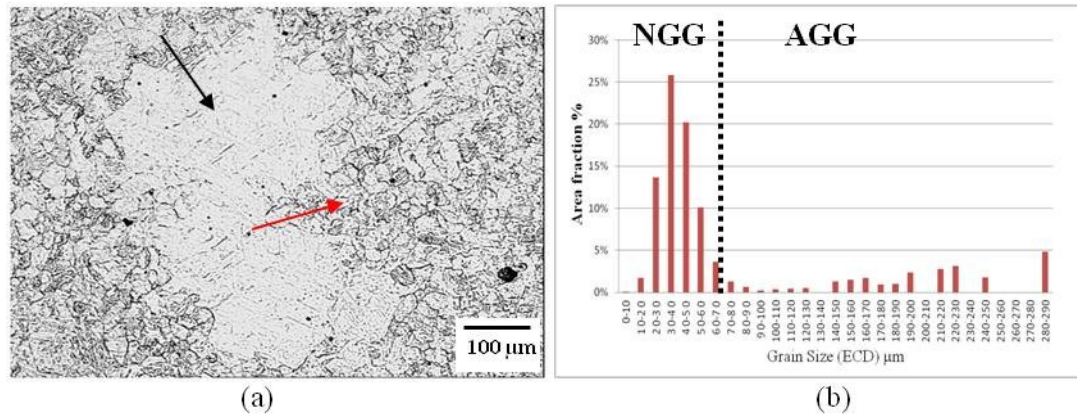
Figure 10 Prior austenite grain structure and grain size distribution for the abnormal grain growth after reheating homogenised Al-Nb containing steel at 1170 ºC

The SEM results at 1170 ºC, as seen in Figure 11, indicate the typical abnormally large grains surrounded by small grains, where EDS mapping was carried out and identified Al-rich particles on the boundaries of the large abnormal grains and the adjacent small grains. Nb-rich particles have not been observed which indicate the complete dissolution of Nb-rich precipitates at this high temperature 1170 ºC; higher than the predicted dissolution temperature of Nb(C, N) at 1090 ºC. The evidence of abnormal grain growth linking to precipitates' distribution behaviour is achieved from SEM result (one example shown in Figure 11 (b, c)), which indicates that Al-rich particles decorate the boundary of the abnormally large grain, with a small number density is only a third that of the AlN present on the small grains' boundaries (as seen in Figure 11 (d, e)), meaning that the precipitates on the boundaries of abnormal grain region have been reduced. This result agrees with literature reports which have discussed that random dissolution of local particles can result in large grains growing readily. Small grained regions, in the size range of 30 – 40 µm (as seen in Figure 10) are associated with the finer grains whose growth is restricted due to the large numbers of precipitates presenting on their boundaries, for example a cluster Al-rich

15

particles have been observed on the boundaries of small grains in Figure 11 (f, g); however, an obvious decrease of Al-rich particles in the small grained matrix can be observed at 1170 ºC (Figure 11 (d, e)) compared to the precipitate distribution at 1100 ºC (Figure 9 (b)), which has confirmed the dissolution of AlN being consistent with Thermo-Calc prediction. The differences in grain growth would appear to be related to the differing kinetics of dissolution of the particles and diffusion of the constituent elements into the bulk of the grains.

Increasing the reheating temperature (1 hour) to 1200 ºC still gives a 95% grain size that is twice the mode grain size, but the extremeness of the abnormal grains compared with the finer grains seen at 1170 ºC has been reduced. This would be consistent with more complete and faster dissolution of AlN so that more grains grow more rapidly leading to the larger mode grain size. As these grains develop they will reduce the extent to which the abnormally large grains grow before impinging with the mode grains and so the 95% grain size decreases slightly.
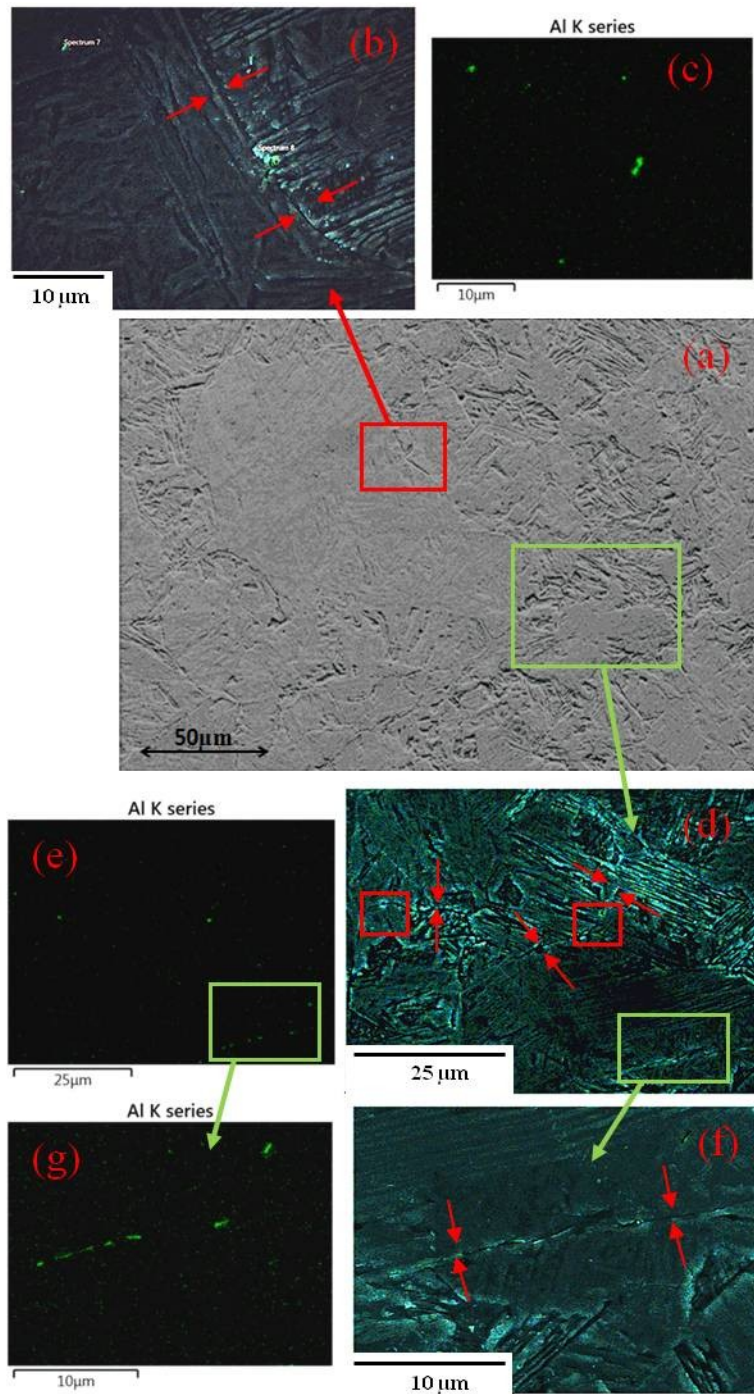
Figure 11 SEM images and EDS mapping analysis for the precipitates in homogenised Al-Nb steel reheated at 1170 ºC for one hour; (b,c) Al-rich precipitates on the boundary of abnormally large grains; and (d, e, f, g) shows the Al-rich particles on the boundaries of small grained regions. The grain boundaries are indicated by red arrows.

The SEM results support the abnormal grain growth behaviour expected during reheating treatment, which also agrees with the prediction stated previously correlating to the thermal stability of the microalloying particle predicted by Thermo-Calc.

Further investigations have been carried out to characterise the development of the abnormally large grains for longer soaking times during reheating treatments. The grain size distributions in Figure 12(a) indicate the obvious continued faster growth of large grains compared with the bulk. The grain size development in Figure 12(b) demonstrates that the abnormally large grain size (95% accumulated area fraction) has changed from around 250 μm (1 hour), to 270 μm (2 hours) and 320 μm (3 hours), whilst the mode grain size is stable at around 40 − 60 μm. It means that the abnormal grains are continuing to coarsen as holding times at the critical dissolution temperature increase. It has reported that the development of abnormally large grains is expected to be limited after certain holding hours [18] probably due to the exhaustion of driving force for those abnormal grain growth. Flores [10] also found that abnormal grains were not observed when steel was reheated at the dissolution temperature (1273K) of the Nb-rich particles for a short holding times (5 minutes) but appeared for longer heating times (in 30 minutes), which indicated that the onset of abnormal grain growth also depended on the soaking time. So, in future, the time and temperature aspects of grain growth behaviour need to be considered when devising schedules to reduce the risk of inhomogeneous grain size distributions.

The result of abnormal grain growth occurred in the homogenised steel indicate that the material in a homogenised condition can not guarantee a fully uniform grain structure as expected during reheating treatment. To minimise the risk of non-uniform grain structure during reheating, more efforts should be paid on the investigations of

18

elemental segregation behaviour and composition design in a HSLA steel, which would be also beneficial for the energy saving during the steelmaking process.
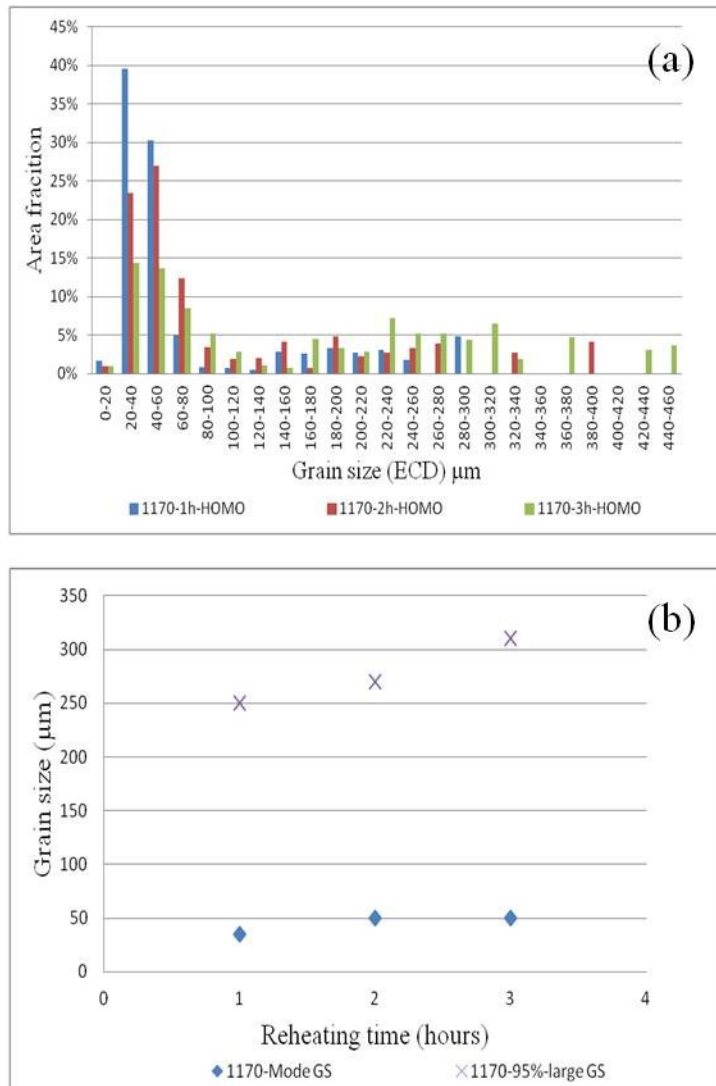

Figure 12 (a) Grain size distribution and (b) grain size development as a function of holding time after reheating at 1170 ºC for one, two and three hours. Mode grain size from area fraction distribution (Mode GS); and large grain size represented by the accumulated 95% area fraction (95% - large GS)

Conclusions

The grain growth behaviour during reheating in an homogenised Al-Nb steel (containing 0.019 wt% Nb and 0.057 wt% Al) has been investigated. In particular the

19

role of microalloying precipitates on boundary pinning during reheating has been considered, which correlates to non-uniform grain growth behaviour such as abnormally large grain growth and bimodal grain size distributions. The main conclusions from this work are:

1. Non-uniform grain growth occurs in the homogenised Al-Nb containing steel for some reheating treatments. Abnormal grain growth has been observed as a localised phenomenon with large grains isolated by the small grained region and a largely skewed grain size distribution, but no bimodal grain size distributions developed in the any of the heat treated, homogenised samples. Thus AGG can occur in homogenised samples (but may be accentuated by segregation), whereas bimodal grain size distributions appear to rely on segregation for their occurrence.

2. Abnormally large grains originate randomly from the reheated microstructure due to local unpinning, which is mainly determined by the most stable microalloying precipitates dissolving during reheating and occurs around the predicted equilibrium dissolution temperature of those particles (in this steel AlN).

3. Abnormal grain growth in homogenised Al-Nb steel occurred at 1170 ºC; there were no abnormal grains when the reheating temperature was below or more than 30 ºC above the AlN dissolution temperature. The size distribution and volume fractions of boundary pinning particles have been related to grain growth behaviour as a function of reheating time and temperature (and thus particle dissolution).

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20

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