Effect of Equalisation Time on the Austenite Grain Size of Simulated Thin Slab Direct Charged (TSDC) Vanadium Microalloyed Steels

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The thin slab direct charging process (TSDC) as applied to microalloyed steels has had a considerable development due to its relatively low production costs and ability to produce steels with good mechanical and toughness properties, comparable to those manufactured by the conventional cold charging process (CCC). This research is concerned with the effect of equalisation time on the microstructures. Three equalisation times (53 min, 318 min, and 1333 min) at 1150°C were investigated for a vanadium microalloyed steel, which contained a low carbon content (0.073 wt%) and a high nitrogen content (0.021 wt%). The experimental results show that MnS precipitates provided the main pinning forces during casting due to the high solubility temperature, but MnS particles did not provide a sufficient pinning force to prevent the austenite grains from growing. The austenite grain growth appears more likely to be abnormal in this stage. AlN precipitates were only identified at the longest equalisation time due to a low precipitation rate. The AlN pinning effect was evident at 1333 min equalisation time, and was able to prevent further austenite grain growth by providing an additional pinning force. The study shows that vanadium does not have any effect on austenite grain growth at 1150°C because of its low solubility temperature in austenite. When the steel was water-quenched, vanadium remained supersaturated in the solution but tempering in the ferrite phase field (650°C) can promote VN or VCN precipitation often using AlN as the nuclei.

KEY WORDS: microalloyed steel; austenite grain size; abnormal grain growth; equalisation time.

1. Introduction

Thin slab direct rolling of microalloyed steels is becoming a more widely used process because of the low energy consumption and good mechanical properties that can be achieved. A number of studies have been reported on the effects of precipitation in steels containing two or more of the common microalloying additions which are used for both grain size control and precipitation strengthening.1–4 It is well accepted that there are many factors which can determine the final mechanical properties of microalloyed steels. During the processing, equalisation, which occurs when the continuously cast steel is fed directly into a furnace and held for 15–40 min, that is without the temperature falling to ambient, is one of the important aspects which can influence the microstructures of steels during a direct charging process before they enter the rolling mill. Some publications have considered the influence of equalisation temperature on austenite grain growth.5–7 They found that for some lower nitrogen steels, an equalisation temperature at about 1050°C could bring about grain growth and coarsening, and affect the final mechanical properties after rolling. Priestner6 pointed out that the final grain size after rolling will depend on the austenite grain size, and that large austenite grains could lead to large ferrite-pearlite grains.

The equalisation time is another factor which can determine the austenite grain size. A literature search has not highlighted any papers on this subject. The present work is concerned with a study of the effect on microstructure of the time the as cast steel was held in the equalisation furnace. This would provide back ground information in the event of a breakdown in the process, for example during rolling or coiling, which resulted in the stock receiving a longer equalisation holding time than the normal 15–40 min.

2. Experimental Procedure

The composition (wt%) of the steel used in this study is given in Table 1.

Three 18kg ingots of the experimental steel were cast and solidified, stripped, and then directly charged to an equalisation furnace. The ingots were held for different times and then water-quenched. This simulated TSDC process was carried out at Swinden Technology Centre, Corus Group, Rotherham. Figure 1 shows a time-temperature graph of the simulated direct charged processing pro-
The ingots were stripped from the mould at $T_1 = 1450–1500°C$. During stripping, the temperature of the ingots dropped to $T_2 = 1000–1100°C$. These ingots were equalised at $T_3 = 1150°C$ for three times, 53 min, 318 min, or 1333 min respectively and then water-quenched. Specimens 10 mm were taken from the equiaxed as-cast austenite zones. Half were tempered at 650°C ($T_4$) for 30 min. All the specimens, before or after tempering, were metallographically prepared and etched in 2% nital. The length of 200 intercepts was measured to obtain the austenite grain size. The grain microstructures were examined by optical microscopy. Precipitate morphology and general structural effects were studied on carbon extraction replicas in a Philips EM-400T transmission electron microscope (TEM) at 80 or 100 kV. Precipitate sizes and distributions were obtained by measuring 200 particles and these were collated using Image-Pro Plus, a software package for image analysis.

3. Experimental Results

3.1. Austenite Grain Growth

Figure 2 shows the bainite microstructure of the steel after 53 min equalisation and water-quenching, in which precipitation occurs in the ferrite laths. The average packet size was 0.57 mm and there was no significant difference in the average packet size with equalisation time. Figure 3 shows the as-cast austenite grain size as a function of equalisation time. The grain size distributions are given in Fig. 4. The as-cast austenite grain size increased with an increase in equalisation time. Initially, the grain growth rates are high, and then they decrease as the equalisation time increases. Analysis of the standard deviation of as-cast austenite grain size gives more details about the grain growth. The longer the equalisation time, the greater the deviation in the austenite grain sizes. Comparing the data in Fig. 4, it is obvious that there are more large grains (>0.9 mm) and fewer small grains (<0.3 mm) after 318 min equalisation time than after 53 min equalisation time.

Qualitatively, this behaviour can be considered as abnormal grain growth. Some research has attempted to establish a criterion to distinguish between the normal and abnormal grain growth. The method devised by Ralph,9) used a bimodal distribution, while other authors10) provided the evidence of abnormal grains by multiplying the frequency by the grain size, to obtain a more obvious second modal in the coarse grain area of the histogram. Following this procedure for the present experimental results, it can be found that there is an obvious second modal in the grain size distribution histogram shown in Fig. 4. Using the above criterion, the grain growth in the present experiment can be considered as abnormal grain growth.
3.2. Particles

3.2.1. Untempered Steel

After equalisation at 1150°C and water-quenching, several different kinds of particles were found in the microstructure. A few particles with a size over 250 nm were almost all MnS. They were often found along the prior austenite grain boundaries. The average particle size and distribution of MnS particles are shown in Figs. 5 and 6. Some particles with a size less than 80 nm were observed by TEM in the microstructure of the steels. An analysis of these particles showed that most were cementite, which are considered to form during quenching. When the steel was equalised for 1333 min, AlN particles were identified. The distribution and average diameter of AlN particles are shown in Fig. 7. Some very fine (about 20–30 nm) cuboid particles were seen occasionally in the bainitic laths following equalisation for 53 min and 318 min (Fig. 8). These were shown by EDX analysis to contain Ti and V (Fig. 9). The cuboid shape is a well known form of TiN, but due to overlapping of Ti L and NK lines in the EDX spectrum, and the close matching of the lattice parameters of the carbides and nitrides of titanium and vanadium carbonitrides, the presence of nitrogen was not confirmed by EDX. However, it was confirmed in a parallel study of the same steel using EELS.11,12) Following the 1333 min equalisation, no vanadium was detected in precipitates and a few AlN particles (average size 104 nm) and some AlN–MnS particles were found (Fig. 10). A diagram is used to show the appearance of precipitates in austenite after the equalisation for three different times (Fig. 11).

3.2.2. After Tempering

There are no obvious differences in the average size and the distribution of the austenite grains, before and after tempering. As the steels were treated at 650°C, an increase...
in austenite grain size would not be expected. After temper-
ing, VN particles were observed associated with AlN pre-
cipitates (Fig. 12(a)). The unusual reflections found in the
[101] zone axis of VN are considered to be due to the par-
ticular bend contours in the complex particle. The electron
diffraction pattern (Fig. 12(b)) shows that the orientation
relationship from the indexed pattern of VN and AlN (Fig.
12(c)) is: (111–)VN//(011)AIN, [101]VN//[311–]AIN. The calcu-
lated interplanar spacings of these two planes are very
close: 

\[ d_{111\text{VN}} = 0.2361 \text{ nm} \quad \text{and} \quad d_{011\text{AIN}} = 0.2371 \text{ nm} \]

and as no large lattice deformation is required for matching these
planes, their relationship should be epitaxial.

4. Discussion

The effect of temperature and time has an influence on
the austenite grain size developed during the equalisation
process prior to rolling. In a previous paper, the effect of
three equalisation temperatures, 1050°C, 1100°C and
1200°C was studied13) for an equalisation time of 30–45
min. However, as an investigation of the evolution of mi-
crostructure of a series of vanadium microalloyed steels
was the objective, the effect of equalisation time was con-
fined to one temperature, 1150°C, and this has been studied
in the present paper.

4.1. Precipitation during Processing

To avoid big temperature reductions, the ingots in the
simulated hot charging process of the microalloyed steels in
several present study entered the equalisation furnace with only a
short delay after they were stripped. The temperature of \( T_\gamma \),
Fig. 1, was above \( A_c_3 \), a temperature which can be calcu-
lated using the equation derived by Andrews14):

\[ A_c_3 = 910 - 203\sqrt{C} - 15.2\text{Ni} + 44.7\text{Si} + 104\text{V} + 31.5\text{Mo} + 13.1\text{W} - (30\text{Mn} + 11\text{Cr} + 20\text{Cu} - 700\text{P} - 400\text{Al} - 120\text{As} - 400\text{Ti}) \]

where the steel composition is given in weight percentage.
Using Eq. (1), the calculated \( A_c_3 \) temperature for the com-
position of the steel given in Table 1 is 869°C, which is
much lower than the lowest possible temperature (about
1000°C) of the ingots before they entered the equalisation
furnace. In this circumstance, no \( \gamma \rightarrow \alpha \) or \( \alpha \rightarrow \gamma \) transfor-
mations will be expected during the stripping and equalisa-
tion. The austenite grains which grow during equalisation
are those which formed and grew during casting and strip-

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ping. Therefore, in the present work, the possible precipitation of MnS, AlN, and VN is expected to occur only in austenite during solidification following casting, stripping or equalisation. These phases are unlikely to precipitate during water quenching following equalisation.

There are several binary equations used to calculate the equilibrium solution temperatures in austenite of the precipitates found in this study. All these equations were considered according to the limitation of the processes and steel compositions employed in the experiments used in their derivation. For the steel composition given in Table 1, four equations given in the literature quoted in the papers,15–18 for respectively TiN, MnS, AlN, and VN, were chosen because of the similar chemical composition of steels (Table 2). Although Ti was not a deliberate addition to the steel, it did appear in some of the particles. Therefore, the equation for TiN is also listed here and the highest Ti content, 0.005%, was used in the calculation. These equations and the calculated solubility temperatures are given in Table 2 and show that the predicted equilibrium solubility temperatures of these precipitates in austenite are in turn 1506°C for TiN, 1390°C for MnS, 1262°C for AlN, and 1094°C for VN. However, these temperatures are modified when inter-element effects are taken into account using the ChemSage software package. According to the composition of the steel in the present work, the solubility temperatures calculated by using the ChemSage software are 1440°C for MnS, 1425°C for (Ti,V)N, and 1275°C for AlN.

Titanium nitrides, such as TiN and (Ti,V)N, are normally considered to precipitate at the very beginning of the solidification process and are the main category of pinning particle in the early formation of austenite because of its high solubility temperature. However, due to the low content of Ti in the steel used in present research, the effect of Ti may not play an important role in the austenite grain growth control even as titanium vanadium nitride. MnS is also an important pinning particle in the early stage of the solidification because of its high solubility temperature in austenite. AlN is another compound which may precipitate during the solidification. It is known that most AlN will dissolve in austenite at 1500°C and under equilibrium conditions most AlN will precipitate by 700°C.19 However, it has been stated that the precipitation of AlN is slow, and would take more than 6 h at about 1100°C.20 Above 1100°C, the precipitation is extremely slow due to the decreasing degree of austenite supersaturation.21 AlN will be expected to precipitate gradually during the equalisation treatment. AlN precipitates appear in the present work only in the ingots equalised at 1150°C for 1333 min, while MnS precipitates appear in the steel ingots for all three equalisation times. In fact, the EDX spectrum analysis shows that some AlN can precipitate with MnS, which may act as a nucleus for the AlN (Fig. 10).

Vanadium is not expected to precipitate directly in the form of VN due to its low solubility temperature, even if the temperature of the ingot dropped to as low as 1000°C (lower than solubility temperature of 1094°C) for a short time. However, it was found that although simple VN cannot precipitate, an unexpected trace of titanium did promote the precipitation of VN in the formation of a more complex vanadium titanium nitride. In their previous work,22 Prikryl et al. clarified that the mixed nitride, (Ti,V)N formed on casting, is a metastable phase that evolves to form a stable TiN. On equalisation, the (Ti,V)N particles diminish in size due to the dissolution of vanadium. The large difference in the equilibrium solubility of TiN and VN in austenite caused the partitioning of V to austenite, leaving TiN as the stable phase. In the present work, EDX spectra containing Ti and V, such as Fig. 9, were associated with small particles (about 20–30 nm), but which were generally segregated. These particles are not considered to play a major role in austenite grain size control.

### 4.2. Effect of Equalisation Time

A number of publications have discussed the equalisation or reheating of microalloyed steels.5,11,13 It is mentioned that austenite grain growth is not only a function of holding temperature but also holding time, and therefore the kinetics of grain growth can be described in terms of the Arrhenius equation and an estimate derived for the apparent activation energy for the growth process.23 It is well accepted that austenite grain growth may follow the parabolic law,

\[ d^2 - d_o^2 = Kt \]  

where \( d \) is the mean grain diameter at time \( t \), \( d_o \) is the initial mean grain diameter and \( K \) is a rate constant. The present experimental result taken from Fig. 3 shows that the austenite grain growth does not follow a parabolic law. Following the relatively fast austenite grain growth period observed in the 53 min and 318 min equalisation times (Fig. 3), the average austenite diameter equalised for 1333 min at 1150°C is much lower than the value predicted by Eq. (2). This result indicates that an extra pinning force occurs in the longer equalisation time specimens. Moreover, as the
parabolic law uses the average grain diameters, it is unable to be used to identify abnormal grain growth using this approach.

During the stripping of the ingot after casting, the temperature decreased from 1450–1500°C to 1000–1100°C. At this stage, MnS could precipitate in austenite and act as a barrier to the austenite grain growth (Fig. 13). In the early times of equalisation of the present steel, MnS might be the only precipitate which could be effective in prohibiting austenite grain growth due to insufficient (Ti,V)N precipitates in the austenite matrix to provide a similar effect. There have been many attempts to model the effect of a particle pinning force on the restriction of grain growth. Zener’s classic equation was modified by many others including Gladman and Rios. Previous research by Gao and Baker showed that the Gladman and Rios equations have the best agreement for their Al–V–Ti–V microalloyed steels which contained several precipitate types. Therefore, their equations are used in the present work to estimate the diameters of MnS particles and to calculate the volume fraction of MnS particles. Gladman’s equation is given as

\[ D = \frac{\pi d}{6f} \left( \frac{3}{2} - \frac{2}{Z} \right) \] ..........................(3)

where \( D \) is the diameter of austenite grains, \( d \) is the diameter of particles, \( f \) is the volume fraction of particles, and \( Z \) is a parameter related to the ratio of diameters of the maximum austenite grains and the average austenite grains and lies between \( \sqrt{2} \) and 2. An equation derived by Rios to predict grain growth in systems in which particles are coarsening and dissolving where

\[ D = \frac{d}{6f} \] ..........................(4)

If the sulphur in the composition is considered to combine completely with manganese, the volume fraction, \( f \), of the MnS is \( 1.039 \times 10^{-4} \). The calculated and measured austenite grain sizes are listed in Table 3. Comparing the calculated austenite grain diameters by using Gladman equation, Eq. (3) and measured average austenite grain diameters, when \( Z \) is 1.6, the measured average austenite grain sizes fit the calculated results quite well in 53 min and 318 min equalisation times (Fig. 14). However, the increase of the measured austenite grain size is lower than the calculated results at 1333 min. This result shows that in the present work, MnS is the most likely pinning particles during the equalisation for shorter times up to 318 min at 1150°C.

On the other hand, the precipitation of AlN is quite slow and takes a long time, and it was only found in the steel which was equalised for the longest time (1333 min) at 1150°C. The coarsening of MnS particles in the equalisation furnace would reduce the pinning effect on the austenite grain boundaries and bring about the sudden growth of some austenite grains. From the point of view expressed by Ralph, this type of austenite grain growth must be regarded as abnormal. Some huge austenite grains (>2 mm) were found (Fig. 4). Unlike normal grain growth, where many grains grow and collide, thereby reducing the driving force for grain growth and slowing the growth rate, the abnormally large grains grow into the unchanged fine-grained matrix and maintain a rapid growth rate, unless they collide with other abnormal grains. Gladman mentioned that this grain coarsening effect could be observed at approximately 1050°C in aluminium grain controlled steels. Also it was pointed out by Priestner and Zhou that in Al–V–N microalloyed steels, the grain coarsening temperatures are about 950–1150°C, depending on the Al and N contents. However, in present experiments, these randomly distrib-

| MnS particle diameter, \( \mu m \) | Calculated austenite diameter using Gladman Equation, mm | Calculated austenite diameter using Rios Equation, mm | Measured austenite diameter, mm |
|---------------------------|-----------------------------|-----------------------------|-----------------------------|
| 0.39                      | 0.17                        | 0.49                        | 0.98                        | 0.63                        | 0.49                        |
| 0.56                      | 0.24                        | 0.69                        | 1.41                        | 0.90                        | 0.67                        |
| 0.75                      | 0.32                        | 0.94                        | 1.89                        | 1.20                        | 0.71                        |

Fig. 13. MnS on austenite grain boundary when equalised at 1150°C for 53 min.

Fig. 14. The calculated and measured austenite grain size at different equalisation time by using Gladman and Rios equations.
uted fine AlN particles (about 100 nm in size) pin the grain boundaries and prevent grains from growing further because of their gradual precipitation during the equalisation period. It is shown in Fig. 14 that the measured average austenite grain size is smaller than the calculated average austenite grain size during 1333 min equalisation. The austenite grain coarsening may commence during the solidification and austenite grains grow in the shorter equalisation times of 53 min and 318 min. The occurrence of AlN after more than 318 min equalisation time will pin the smallest austenite boundaries and prevent these austenite grains from the further significant growth.

In order to identify the effect of the different MnS particle sizes on the different austenite grains, an attempt has been made in which the MnS particles and austenite grains were divided into the different groups according to the distribution (Figs. 4 and 6). The Gladman equation was then modified as the following

\[ D = \frac{\pi \alpha_{\text{MnS}} d}{6 \alpha_{\text{MnS}}} \left( \frac{3}{2} \frac{2}{Z} \right) \]  

(5)

where \( \alpha_{\text{MnS}} \) is the volume percentage of MnS particles in each particle diameter group, \( \alpha_{\text{MnS}} \) is the volume percentage of austenite grains in each austenite grain diameter group.

It was pointed out\(^{(26,29)}\) that when the diameters are greater than 0.8 \( \mu \text{m} \), the particles may stimulate the nucleation of new grains. Therefore, considering that MnS particles with a greater diameter of 0.8 \( \mu \text{m} \) do not play a role in pinning the austenite grain boundaries, the comparison between the calculated austenite grain sizes from these MnS particles and the measured austenite grains is not listed in Table 4. However, the calculated austenite diameters compare well with the measured austenite grain diameters only for an equalisation time of 53 min and when the MnS particle size is less than 0.6 \( \mu \text{m} \) in diameter. The reason could be due to the abnormal grain growth.

Comparing the distribution of the austenite grain size (Fig. 4) and MnS particle size (Fig. 5), it can be found that when the steel was equalised for 53 min, the amount of coarse MnS particles with a size greater than 0.8 \( \mu \text{m} \) is only 2%. When the steel was equalised for 318 min and 1333 min, larger numbers of coarse MnS particles (>0.8 \( \mu \text{m} \)) were observed. The percentages are 16% and 30%, respectively. In this circumstance, these particles will not play a role in pinning the austenite grains. The above analysis could be used to explain why the fitting extent in 53 min equalisation time is much better than those in 318 min and 1333 min equalisation times.

Although the long time equalisation (more than 318 min) does not lead to a significant increase of austenite grain diameters, it affects the precipitation of AlN. When the equalisation time is 1333 min, AlN will be able to completely precipitate from austenite and grow. However, when the equalisation time has only reached 318 min, Al and N remain supersaturated in austenite. During the complete TSDD processing, where rolling and coiling follow equalisation, the hot deformation results in a marked increase in the kinetics of precipitation of AlN.\(^{(21)}\) The precipitation of the fine AlN particles during rolling may promote the formation of fine ferrite grains in the final stage and affect the properties of final products.

4.3. Water-quenching and Tempering

After the equalisation at 1150°C for different times, the ingots were water-quenched to room temperature. Unlike a laboratory experiment, the ingots were processed in a simulated commercial condition. After water-quenching, a bainitic microstructure was found instead of martensite. In this microstructure, most of particles were cementite which were considered to form during quenching and not during stripping or equalisation. These cementite particles are dependent on the nucleus forming conditions after the \( \gamma \rightarrow \alpha \) transformation and did not play any role in austenite grain growth.

When the steel was water-quenched to room temperature, vanadium remained supersaturated in the ferrite matrix. However, the solute atoms at room temperature have a relatively low activity and do not have enough energy to overcome the nucleation and diffusion energy barrier. In this case, it is very difficult for VN or VCN to precipitate. Therefore, no vanadium were detected in water-quenched bainite microstructure except in the small amount of (Ti, V)N particles.

The higher temperature (650°C) during tempering provides sufficient activation energy the precipitation of solute atoms, such as vanadium and nitrogen, from the ferrite matrix. When some precipitated particles can act as the heterogeneous nuclei for VN, the precipitation of VN will be much easier. As shown in Fig. 12, VN precipitates with AlN. When the steel was equalised at 1150°C for 1333 min and tempered, AlN has sufficient time to precipitate fully. These AlN particles can therefore become the potential nuclei of VN.

5. Conclusions

(1) At shorter equalisation times of 53 min and 318 min at 1150°C, the austenite grain growth in V-microalloyed steels in the TSDD process is controlled by the pinning effect of MnS particles precipitated during the stripping. The grain growth is usually abnormal because MnS does not have a strong pinning effect on the austenite grain growth.

(2) The slight increase of the austenite grain growth of V-microalloyed steels occurs in the TSDD process when the equalisation time increases from 53 to 1333 min. The gradual precipitation of AlN particles during the equalisation is the main factor which prevents the growth of the smallest austenite grains at 1333 min.

(3) VN does not precipitate during equalisation at 1150°C and it will remain supersaturated in bainite after
water-quenching. Tempering can promote the precipitation of VN and also AlN acts as a nucleus for VN precipitation.

(4) From a practical perspective, in the present work, it can be concluded that a delay in the rolling or coiling, which results in significantly longer equalisation time, has no major effect on the austenite grain size in this V-microalloyed steel but will lead to the precipitation of AlN prior to rolling. The latter factor may affect the properties of the final products.

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