The Effects of Heating Rate on Austenite Grain Growth in a Ti-modified SAE 8620 Steel with Controlled Niobium Additions

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The Effects of Heating Rate on Austenite Grain Growth and precipitate distribution in Ti-modified SAE 8620 steels with Nb additions of 0.02, 0.06 and 0.1 wt% were evaluated with pseudo-carburizing, i.e. without a carburizing gas, heat treatments characteristic of high temperature vacuum carburizing. Laboratory plates were produced to simulate conventional hot-rolling and controlled-rolling processes. Specimens were heated at rates between 10 and 145°C min⁻¹ to 1 050 and 1 100°C, held at the desired austenitizing (carburizing) temperature for 60 min, and immediately quenched in iced-water. Austenite grain structures developed at the austenitizing temperatures were evaluated with light optical metallography, and precipitate dispersions were evaluated using extraction replicas in the transmission electron microscope. Abnormal grain growth was observed in all samples processed at the highest heating rate to 1 050°C, but was suppressed at the lower heating rates with additions of 0.06 and 0.1Nb. Suppression of abnormal grain growth was correlated with the development of a critical distribution of fine NbC precipitates, stable at the austenitizing temperature. The importance to industrial carburizing practice of heating rate effects on precipitates and austenite grain size evolution are discussed.

KEY WORDS: niobium microalloying; carburizing; precipitation; abnormal grain growth.

1. Introduction

Grain growth control in microalloyed steels during thermomechanical processing, including heat treating, critically depends on second phase particles. With the increased interest in higher temperature vacuum carburizing, Laboratory plates were produced to simulate conventional hot-rolling and controlled-rolling processes. Specimens were heat treated at 1 100°C, new steel alloys based on microalloy additions of Ti and Nb are being developed to produce specific precipitate dispersions that are effective in minimizing austenite grain growth.1–3) In carburizing gear steels, the importance of minimizing austenite grain coarsening in order to maximize fatigue performance has been clearly demonstrated.4,5) Austenite grain growth in microalloyed steels is influenced by numerous factors, including austenitizing time and temperature, alloy composition, thermomechanical history, initial grain size distribution, and rate of heating to the austenitizing temperature. Of these variables, the effect of heating rate has received the least attention and previous studies have reported mixed observations as summarized below.

Previous studies on plain carbon steels have shown that an increase in heating rate has no effect on austenite grain growth,6,7) increases austenite grain growth,8,9) or results in a decrease in the rate of austenite growth.8) Some studies have reported different responses in a single steel. For example, Sheard and Nutting,9) in a study on a high purity Fe–C alloy with 0.47 wt% carbon heated to 795°C, found that increasing the heating rate reduced austenite grain size, whereas when this material was austenitized at a relatively higher temperature (830°C), the opposite response was observed.

The response to heating rate in plain carbon steels depends also on alloy purity and starting microstructure. Rosenberg and Digges10) found that the effects of heating rate through the transformation region on grain growth in 0.5 wt% carbon steels depended on alloy purity. Specifically, in a high purity alloy austenitized at 800 to 870°C, austenite grain size increased with a decrease in heating rate, while the austenite grain size in a commercial steel increased with an increase in heating rate.9) Whiteley11) found for medium carbon steels that a fine initial microstructure was more sensitive to heating rate than a coarse initial microstructure, and in either case grain refinement resulted from an increase in heating rate. Furthermore, the effect of the starting microstructure on the sensitivity to heating rate was completely eliminated at high austenitizing temperatures.11)
Austenite grain size evolution in steels with controlled precipitate dispersions has also been reported to exhibit mixed responses to heating, and thus reflect the effects of heating rate on precipitate dispersions present during grain growth annealing. For example, Militzer et al. found that austenitizing an Al-killed plain carbon steel at 1 100°C after heating at 300°C min⁻¹ produced a substantially larger austenite grain size than obtained after an extremely rapid heating rate (6 000°C min⁻¹) for similar holding times. The effect of the slow heating rate was believed to result from AlN dissolution on heating, leading to a precipitate dispersion not capable of suppressing grain growth. Decreases in heating rate also resulted in increased austenite grain sizes in V-microalloyed steels, an observation that was correlated with an increased mean VC particle size, potentially due to particle growth and/or dissolution during heating. Rossi et al. showed that austenite grain growth in a Nb-containing 0.36%C steel exhibited a stronger dependence on heating rate than a similar steel alloyed with Ti, and a finer initial particle dispersion was more effective in suppressing grain growth.

The experimental results summarized here suggest that grain size control in steels with second phase particles depends on initial austenite grain size at the end of the α to γ transformation, as well as the volume fraction of second phase particles, the particle size distribution, and the particle stability or coarsening resistance. Starting with the initial work of Zener, as quoted in Smith, Gladman has developed analyses that predict the interrelationships between initial grain size, particle size, and grain size on the response of steel microstructures to grain growth, and these analyses predict different critical conditions that must be met to suppress grain growth. While it is beyond the scope of this paper to review in detail the theoretical models that have been developed, several general observations are important to the understanding of the effects of heating rate on grain growth. For example, for a given particle size and volume fraction, if the initial austenite grain size is smaller than the critical value described by Gladman, then the particles will be ineffective in preventing grain growth. At high heating rates, the initial austenite grain size after transformation has been found to decrease with an increase in heating rate, and thus for a given material a critical heating rate may exist, above which unstable grain growth occurs. Correspondingly, in a steel with a given initial austenite grain size and particle density, if the particle size is less than a predicted critical particle radius, then the particles are ineffective and grain growth occurs. The effects of heating rate on the precipitate distribution at the onset of grain growth will depend on the initial precipitate size, amount of solute in the matrix, and solute diffusivities, along with the time-temperature history controlled by heating rate. It is conceivable that significant modifications to the initial precipitate distribution may occur on heating, and may contribute to the mixed effects of heating rate in various steel alloys reported above.

The previous studies have shown the potential effect of heating rate on austenite grain growth behavior in selected alloy systems. In complex component geometries that include both thick and thin sections, heating to austenitizing temperatures will result in variations in heating rate in a single part. With the development of new Nb-microalloyed carburizing steels designed to suppress grain growth in specific heat treated components processed at higher carburizing temperatures, a complete understanding of the interrelationships between microalloy additions and heating rate is required to optimize processing. Therefore, the present work was undertaken to experimentally investigate the influence of heating rate on austenite grain coarsening and precipitate distributions in a Ti-modified SAE 8620 steel containing different Nb levels, designed for use in forged and carburized gears. The specific steels evaluated here were the subject of a recent study on the effects of Nb additions on austenite grain growth and the experimental program was designed to complement the previous study.

2. Experimental Materials and Procedures

Three microalloyed steels having Nb additions to a reference Ti-modified SAE 8620 steel were produced as laboratory heats with the compositions shown in Table 1. These Ti–Nb-modified steels contained nominally constant levels of carbon, nitrogen, and titanium, with different niobium contents (0.02Nb, 0.06Nb and 0.1Nb, wt%). The laboratory melted alloys were processed to simulate conventional hot-rolling (HR; finish rolling temperature of 1 100°C) or controlled-rolling (CR; finish rolling temperature of 850°C) and details of the alloy design and processing history are summarized elsewhere. Data are also presented on a Nb-free steel examined previously.

The effects of heating rate on austenite grain coarsening behavior and precipitate distributions were evaluated on 20×15×10 mm³ samples heated at rates of 10, 20, 40, and 145°C min⁻¹ to 1 050°C and 1 100°C, held at the desired pseudo-carburizing (i.e. without the presence of a carburizing gas) temperature for 60 min, and immediately quenched in iced-water. These pseudo-carburizing heat treating conditions were chosen based on results of a previous study of grain growth in the same alloy series processed at a single heating rate. Heat treat coupons were placed in a controllable cylindrical high temperature furnace connected to a programmable temperature controller and heated from room temperature at the indicated heating rates. One control thermocouple and two monitoring thermocouples were imbedded in holes drilled into the surface of each coupon. After heat treatment, samples were mounted, mechanically ground to remove at least 1.5 mm from the exposed surface, polished, and etched with a saturated aqueous picric acid.

Table 1. Chemical compositions of experimental steels (in wt%), N and O in ppm.

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>Al</th>
<th>Nb</th>
<th>Ti</th>
<th>N</th>
<th>O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base</td>
<td>0.204</td>
<td>0.85</td>
<td>0.015</td>
<td>0.012</td>
<td>0.25</td>
<td>0.59</td>
<td>0.42</td>
<td>0.20</td>
<td>NA</td>
<td>0.027</td>
<td>0.0</td>
<td>0.034</td>
<td>77</td>
<td>NA</td>
</tr>
<tr>
<td>0.02Nb</td>
<td>0.21</td>
<td>0.82</td>
<td>0.016</td>
<td>0.002</td>
<td>0.24</td>
<td>0.60</td>
<td>0.42</td>
<td>0.20</td>
<td>0.01</td>
<td>0.023</td>
<td>0.022</td>
<td>0.030</td>
<td>95</td>
<td>24</td>
</tr>
<tr>
<td>0.06Nb</td>
<td>0.22</td>
<td>0.80</td>
<td>0.017</td>
<td>0.003</td>
<td>0.24</td>
<td>0.60</td>
<td>0.43</td>
<td>0.21</td>
<td>0.01</td>
<td>0.022</td>
<td>0.054</td>
<td>0.034</td>
<td>92</td>
<td>28</td>
</tr>
<tr>
<td>0.1Nb</td>
<td>0.22</td>
<td>0.85</td>
<td>0.019</td>
<td>0.003</td>
<td>0.25</td>
<td>0.59</td>
<td>0.43</td>
<td>0.20</td>
<td>0.01</td>
<td>0.024</td>
<td>0.109</td>
<td>0.032</td>
<td>87</td>
<td>24</td>
</tr>
</tbody>
</table>
solution containing a wetting agent (teepol) and HCl. Details on the etching procedure to reveal the prior austenite grain boundaries are presented elsewhere.22)

Prior austenite grain sizes were measured on printed light optical micrographs by the line intercept method according to ASTM E112. Grain boundary intercepts with a 500 mm line were counted on 10 fields per specimen. Carbon extraction replicas were used to examine precipitate characteristics, including size, number density, morphology, and distribution using a scanning transmission electron microscope (STEM) operating at an accelerating voltage of 120 kV. For each sample evaluated in the STEM, at least three replicas were examined, and for each replica at least seven different fields of view were imaged. At least 200 precipitates, excluding precipitates larger than 100 nm, were measured to characterize the precipitate size distribution. Precipitate number densities were estimated by counting observed particles in a nominal total area of approximately 37 μm² (multiple fields of view), and results are expressed as number of particles per square micron.

3. Results

Figure 1 shows selected light optical micrographs that
reveal the effects of heating rate (20, 40, and 145°C min\(^{-1}\)) and Nb-additions on prior austenite grain structures for samples austenitized at 1050°C for 60 min. Abnormal grain growth (AGG) is observed at all heating rates for the Nb-free alloy (Figs. 1(a) to 1(c)). In samples heated at the two lower rates, the development of fine/uniform austenite grain structures is promoted in the 0.06Nb (Figs. 1(g) and 1(h)) and 0.1Nb (Figs. 1(j) and 1(k)) steels. At the highest heating rate for all Nb steels, and at all heating rates for the 0.02Nb steel, a duplex structure (DS) indicative of abnormal grain growth is evident. The response summarized in Fig. 1 is representative of both hot rolled (HR) and controlled rolled (CR) steels.\(^1,2\)

Figure 2 shows prior austenite grains for samples austenitized 60 min at 1100°C for the 0.02Nb HR steel and the 0.1Nb CR steel after heating at 10, 20, and 145°C min\(^{-1}\). These selected images show a duplex grain structure for the 0.1Nb steel with a higher fraction of fine grains at the lower heating rates. In the 0.02Nb steel at all heating rates, uniform coarse grain structures characteristic of normal grain growth (NGG) are evident.

Figures 3 (1050°C) and 4 (1100°C) summarize quantitative grain size measurements as a function of heating rate and Nb addition for all pseudo-carburized samples. Data are presented for both HR (Figs. 3(a) and 4(a)) and CR (Figs. 3(b) and 4(b)) processing histories. Also shown in Fig. 3(a) are data for the HR Nb-free steel from a related study.\(^2,3\) Each point is also characterized utilizing terminology\(^1,2,2\) to describe the grain size distributions evident in the light optical micrographs. In each figure, the effects of heating rate and Nb content are similar in both HR and CR steels, but the final austenite grain size for a specific condition is smaller in the CR steels.

The precipitate distributions in selected samples exhibiting significant differences in grain growth response were evaluated using extraction replicas and the results are summarized in transmission electron micrographs in Figs. 5 and 6. Figure 5 shows precipitates in specimens that correspond to the light optical micrographs in Figs. 1(g) and 1(i) for the HR 0.06Nb steel annealed for 60 min at 1050°C after heating at 20 and 145°C min\(^{-1}\) where the lower heating rate clearly resulted in a finer prior austenite structure. Figure 6 shows results for the CR 0.06Nb steel annealed for 60 min at 1050°C after heating at 20, 40, and 145°C min\(^{-1}\); as shown in Fig. 3(b), the CR samples exhibited a transition in grain structure from fine and uniform at the lower heating rates to a duplex structure characteristic of abnormal grain growth at the higher heating rate. Well developed precipitates are evident in each image and the number of fine particles is higher in the samples subjected to the lower heating rates consistent with their finer observed prior austenite grain sizes.

Precipitate size distributions, average precipitate sizes, and precipitate number densities were quantified using the TEM images. Figure 7 shows the precipitate size distributions for the HR 0.06Nb steel and demonstrates that there is a higher density of fine precipitates in the sample heated at the slower rate. Similar behavior was obtained for the CR 0.06Nb steel.\(^2,2\) Table 2 summarizes the particle number densities and average particle sizes obtained from multiple areas examined in the TEM replicas. The data in Table 2 confirm that the average precipitate size increases and the precipitate number density decreases with an increase in heating rate, and the precipitates are finer in CR samples. For example, the average precipitate size in the pseudo-carburized HR 0.06Nb steel after heating at 20°C min\(^{-1}\) was one-half that of produced after heating at 145°C min\(^{-1}\). Furthermore, the precipitate number density after heating at
20°C min⁻¹ was about three times greater than observed after heating at 145°C min⁻¹. As shown in Table 2, similar comparisons were evident for the CR 0.06Nb steel.

4. Discussion

Increased susceptibility to abnormal grain growth during high temperature carburizing is observed to occur with an increase in heating rate in the Nb alloyed steels considered here. This behavior reflects the multiple factors that control grain growth during high temperature isothermal heat treating. Grain growth during austenitizing depends on the initial grain size and grain size distribution and the precipitate size distribution present at the annealing temperature. The effects of heating rate on the initial grain size distribution at the annealing temperature was examined in a related study on the same steels considered here, by evaluating microstructures in samples immediately quenched from specified annealing temperatures after heating at rates from 10 to 40°C min⁻¹. The results of this study are illustrated in Fig. 8 that shows light optical micrographs of the HR 0.06Nb steel heated at 10°C min⁻¹ (Fig. 8(a)) and 40°C min⁻¹ (Fig 8(b)) to 950°C, and quenched without annealing (i.e. a zero-time anneal). This figure illustrates that

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Fig. 3. Effects of Nb content and heating rate on the mean austenite grain diameter for samples pseudo-carburized for 60 min at 1 050°C: (a) Hot Rolled (HR) steels and (b) Controlled Rolled (CR). The following nomenclature is used to identify grain structures: F/U= fine and uniform; IAGG=initial abnormal grain growth; DS=duplex structure; and NGG=normal grain growth.

Fig. 4. Effects of Nb content and heating rate on the mean austenite grain diameter for samples pseudo-carburized for 60 min at 1100°C: (a) Hot Rolled (HR) steels and (b) Controlled Rolled (CR). The following nomenclature is used to identify grain structures: F/U= fine and uniform; IAGG=initial abnormal grain growth; DS=duplex structure; and NGG=normal grain growth.

Fig. 5. Electron micrographs from extraction replicas showing precipitate structures in HR 0.06Nb specimens annealed for 60 min at 1 050°C after different heating rates. (a) 20°C min⁻¹; (b) 145°C min⁻¹.
the population of small austenite grains present at the onset of the austenitization heat treatment increased with an increase in heating rate through the intercritical region, and as a result the finer starting microstructure exhibits a higher driving force for grain growth.22) The rate of heating to the austenitizing temperature also strongly influenced the average size and distribution of Nb precipitates. For example, as shown for the CR 0.06Nb steel in Table 2, a decrease in heating rate from 145 to 20°C min⁻¹ resulted in a decrease in the mean particle size from 30 to 17 nm and an increase in number density from 7.2 to 16.4 nm⁻². These observations provide a basis to interpret the effects of alloying, heating rate, and annealing temperature presented in Figs. 1 to 7.

The precipitate refinement associated with a decrease in heating rate is perhaps counterintuitive based on conventional models of precipitate growth and coarsening. The influence of heating rate through the intercritical region on precipitation, dissolution, and coarsening kinetics of Nb-rich precipitates was evaluated in a related study by AlOgab,22) and the results of that study are represented in plots of hardness versus aging time as well as quantitative precipitate distributions. Figure 9 summarizes the effects of

![Figure 8](image1)

![Figure 9](image2)

**Table 2.** Precipitate number densities and average precipitate sizes for selected pseudo-carburizing conditions.

<table>
<thead>
<tr>
<th>Steel</th>
<th>Pseudo-carburizing conditions</th>
<th>Precipitate number density (nm⁻²)</th>
<th>Average precipitate size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T (°C) t_e (min) Heating rate (°C min⁻¹)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>HR-0.06Nb</td>
<td>1050 60 20</td>
<td>6.3</td>
<td>22</td>
</tr>
<tr>
<td></td>
<td>1050 60 40</td>
<td>2.1</td>
<td>44</td>
</tr>
<tr>
<td>CR-0.06Nb</td>
<td>1050 60 145</td>
<td>16.4</td>
<td>17</td>
</tr>
<tr>
<td></td>
<td>1050 60 145</td>
<td>9</td>
<td>26</td>
</tr>
<tr>
<td></td>
<td>1050 60 145</td>
<td>7.2</td>
<td>30</td>
</tr>
</tbody>
</table>

Fig. 6. Electron micrographs from extraction replicas showing precipitate structures in CR 0.06Nb specimens annealed for 60 min at 1 050°C after different heating rates. (a) 20°C min⁻¹; (b) 40°C min⁻¹; and (c) 145°C min⁻¹.

Fig. 7. Precipitate size distributions in HR 0.06Nb specimens annealed for 60 min at 1 050°C after different heating rates. (a) 20°C min⁻¹; (b) 145°C min⁻¹.

Fig. 8. Light optical micrographs showing austenite grain structures for steels heated at 10°C min⁻¹ (a) and 40°C min⁻¹ (b) to 950°C and immediately quenched to reveal the initial austenite grain structure at the annealing temperature.
aging for 2 h at temperatures between 400 and 700°C on the hardness of the HR 0.02Nb, 0.06Nb, and 0.1Nb steels. The data illustrate a secondary hardening peak at approximately 600°C. The appearance of the secondary hardening peak suggests that solute Nb was present in the as rolled steels. Similar results were shown for the controlled rolled steels, although the magnitude of the hardness increase associated with the secondary hardening peak was lower suggesting more complete precipitation and lower free Nb after initial cooling to room temperature. Since all samples for a given composition were assumed to have the same precipitate distribution after a given rolling process, then the differences between precipitate structures shown in Figs. 6 and 7 must reflect variations in time-temperature histories associated with the different heating rates during pseudo-carburizing. Specifically, for samples austenitized at 1 050°C, the slower heating rates produced a higher density of fine precipitates upon heating to the austenitization temperature resulting in a structure that was less susceptible to coarsening in the 0.06Nb and 0.1Nb steels. In contrast it is suggested that heating at the higher rate delayed additional precipitation to higher temperatures and led to coarse precipitates that were less able to suppress austenite grain coarsening. For the slower heating rates, the finer precipitate structures suppressed grain growth, and uniform fine grained structures developed as shown in Figs. 1(g), 1(h), 1(j), and 1(k). In contrast to the results on the higher Nb steels, the results on the 0.02Nb steel suggest that with the lower Nb content there was an insufficient precipitate density for all processing conditions, as the development of a duplex structure and large austenite grains was observed for all heating rates. Furthermore, the absence of fine/uniform austenite grain structures in the lowest Nb-containing steel at 1 050°C illustrates the ineffectiveness of the TiN particles (due to their initial large sizes) in limiting austenite grain growth at this temperature in these steels. The TiN distribution is believed to have been established during plate manufacture and remained stable during the heat treatments considered here; thus, heating rate interactions with the TiN dispersion are not considered to play an important role.

Previous calculations suggest that all of the Nb would be in solution at 1 100°C for the 0.02Nb steel, and extensive precipitate dissolution would also be expected in the 0.06 and 0.1Nb steels. It is reasonable that dissolution and coarsening at 1 100°C would render the remaining precipitate structure less effective in preventing grain growth, leading to more extensive abnormal grain growth and large grain sizes at 1 100°C, as shown in Figs. 2 and 4.

In the absence of Nb precipitates, Figs. 1(a) to 1(c) illustrate the susceptibility of Ti-modified 8620 steel to abnormal grain growth in the base (Nb-free) steel exhibited only a small dependence on heating rate as compared to grain growth in the Nb-containing steels. Consistent with some previous studies, this small dependence on heating rate is attributed to the influence of heating rate on the initial austenite grain-size distributions at the pseudo-carburizing temperature. In this context, the decrease in rate of heating through the intercritical region increases the matrix mean grain diameter and, therefore, reduces the driving force for grain growth.

Results of this study clearly demonstrate the important role of heating rate on Nb-rich precipitate distributions (e.g., number density and size) and austenite grain coarsening behavior. It is anticipated that control of heating rate during carburizing will offer an additional process parameter to assist in producing heat treated carburized components with optimized properties. To assess the applicability of utilizing heating rate as a process control parameter, the effects of heating rate to a vacuum carburizing temperature of 1 050°C on microstructures and fatigue properties of the same experimental steels considered here is the subject of related work.

5. Conclusions

(1) The susceptibility of Ti-modified 8620 steel to abnormal grain growth during pseudo-carburizing heat treatments at 1 050°C increases with an increase in heating rate and a decrease in Nb content. In steels with 0.06 or 0.1 wt% Nb, higher densities of fine precipitates capable of suppressing abnormal grain growth develop at lower heating rates (10 to 20°C min⁻¹) while at 145°C min⁻¹ a coarser precipitate distribution develops that is less able to suppress abnormal grain growth.

(2) At the higher austenitizing temperatures considered here, the effects of heating rate are more limited in steels without Nb additions or with lower (e.g. 0.02 wt%) Nb contents. These effects are primarily associated with differences in the initial austenite grain size distributions.

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