Heterogeneous Austenite Grain Growth in ASTM A213–T91 Steel

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Heterogeneous grain growth during austenitization in ASTM A213–T91 steel has been studied using a Gleeble 3500 thermomechanical simulator. Starting from a uniform, fine austenite grain size distribution after 1 min austenite holding time, a heterogeneous austenite grain size distribution was observed after 15 min austenite holding time at 1060°C and 1080°C. The state of precipitation of second phase particles was studied in detail as a function of austenitization time. The particle size distribution changed towards a bimodal feature as a function of time whereas the count frequency of the coarse precipitates increased and the count frequency of fine precipitates diminished with time. V-rich precipitates were dissolved during austenitization and only Nb-rich particles remained after 15 min austenitizing time. The experimental observations on the evolution of the austenite grain size distribution are rationalized on the basis of the Gladman’s approach to abnormal grain growth.

KEY WORDS: austenitization; abnormal grain growth; precipitation; dissolution.

1. Introduction

Ferritic-martensitic steels of the 9%Cr1%Mo type have been extensively used in power plant components, heat exchangers, piping and tubing, etc., due to an excellent combination of properties such as creep resistance, toughness and resistance to oxidation at high temperatures. The continuous improvement of the properties of 9% Cr materials in the last decades has allowed a substantial increment of their benefits: increase of the service temperatures—with the consequent increase in efficiency—and increasingly important values of resistance to rupture. From the environmental point of view, the increase of the efficiency also implies a reduction of CO2 emissions.

Heterogeneous or abnormal grain growth in austenite may be a non-wished result of manufacturing processes implying austenitization. The mechanical properties of materials displaying a bimodal austenite grain size distribution could be degraded, and some examples of such a situation have been already reported.1) The study of the HAZ to this respect is an important issue in welding.2) Thus, it is important to control austenite annealing conditions so as to avoid heterogeneities in grain size. In this sense, the influence of the heating rate to austenite in the austenitic grain size distribution—particularly, in the formation of a heterogeneous grain size structure—has been reported in the literature referred to carbon steels,3) microalloyed steels containing Ti and V4) or Nb5) and ferritic-martensitic steels 9% Cr.6,7) In some cases, a critical value for the heating rate has been found, above which a heterogeneous austenite grain size distribution is developed for fixed conditions of austenite temperature and holding time.6,7)

In steels stabilized by addition of alloying elements—that refine the austenitic grains by precipitation of second phases and subsequent pinning of grain boundaries—empirical relationships have been found between the austenitic mean grain size, the volume fraction of the pinning particles and the critical diameter of those particles, given by expressions of the type

\[ D_{\text{crit}} = \phi \frac{r}{f_n} \]

where \( D_{\text{crit}} \) is the critical mean (three-dimensional) austenitic grain size in presence of a volume fraction \( f \) of particles that have a mean radius \( r \). \( \phi \) is a constant for a given steel, depending on the geometry and coherency of matrix and precipitates. Thus, the heating rate \( V_h \) and the austenitization temperature \( T_h \) would play a role on heterogeneous grain growth through their influence \( f \) or \( r \), which would lead to local variations of \( D_{\text{crit}} \). At the same time, it should be kept in mind that though the action of pinning of the austenitic grain boundaries is exerted by carbides, nitrides or carbonitrides of the form MX (M= Nb, V; X=C, N), the major precipitated phase in T91 or P91 materials in the as-received condition is constituted by carbides of the form M23C6, whose dissolution during heating or austenite holding could also influence the kinetics of precipitation/dissolution of the MX via the increment of the C content in solid solution.

The present investigation deals with the influence of the austenite holding time and the heating rate to austenite on the development of a heterogeneous austenite grain size distribution at temperatures normally used in industrial practice, taking also into account the behavior of the size distribution of second phase pinning particles.
Table 1. Chemical composition of the V&M steel ASTM A213–T91 (in wt%).

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Mo</th>
<th>Si</th>
<th>V</th>
<th>Nb</th>
<th>N</th>
<th>Mn</th>
<th>Ni</th>
<th>Al</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.11</td>
<td>8.28</td>
<td>0.93</td>
<td>0.28</td>
<td>0.21</td>
<td>0.07</td>
<td>0.045</td>
<td>0.48</td>
<td>0.11</td>
<td>0.015</td>
<td>0.004</td>
</tr>
</tbody>
</table>

2. Experimental Procedure

The samples utilized in the present work were machined from an 8 mm-thickness wall tube provided by Vallourec & Mannesmann (France). The material was received in its standard condition, that is, normalized at 1 060°C and tempered 40 min at 780°C. The certified chemical composition for this material is shown in Table 1.

The thermal cycles were carried out in a Gleeble 3500 thermomechanical simulator as follows: heating at a rate of 1 and 50°C/s, austenite holding at 1 060°C and 1 080°C for 1, 15 and 30 min and quenching at 50°C/s. The values 1 and 50°C/s for the heating rate were selected for they were supposed to be below and above, respectively, than the critical heating rate value according to the results of Ref. 6. Samples were cylinders 80 mm long and 6 mm in diameter, and were cut following the rolling direction. Under the conditions of a Gleeble test, only a thin slab in the central part of the cylindrical sample (approximately 1 mm in thickness) was submitted to the mentioned thermal program. After thermal cycles, the central slab of each of the test specimens (containing the welded spot of the thermocouple) was cut with a very thin diamond saw, embedded in a resin, polished to 1 µm cloth and etched with the Vilella reagent for its observation. Metallographic observations were performed in an optical microscope Olympus BX60M and a scanning electron microscope Phillips SEM 515, operated at 25 kV.

Automatic image analysis was performed with a commercial software package on the images acquired in the optical microscope. The recognition of prior austenite grain boundaries in the as-quenched martensite matrix was not evident and a double etching-polishing procedure was applied. Equivalent grain diameters were obtained averaging grain radii taken from the centroid of each grain at intervals of 2 degrees. Grain areas and ASTM grain sizes were measured and classified as well.

Thin foils of the material in the as-received condition were prepared in a double jet apparatus Fischione 120, using a 50% methanol and 10% perchloric acid reagent at a temperature of −35°C and a current of 38 mA. Extraction replicas of the heat-treated samples were prepared by electron beam evaporation of C and etching with the Vilella reagent as well. The size of precipitated phases was measured on these replicas in a Phillips CM200 TEM, operated at 200 kV and attached with an EDAX-DX4 system for energy-dispersive analysis of X-rays (EDS). More than 500 precipitates per sample were measured. The chemical composition of the precipitated phases was studied by EDS in a subset of 50 to 60 particles, in each sample.

3. Results

3.1. Characterization of the As-received Metallurgical State

Figure 1 shows micrographs of a sample in its standard, as received condition. Observations were carried out by optical microscopy and TEM. The material in the as-received state displays a typical morphology of martensite in laths, as shown in Fig. 1(a). Prior austenite grain boundary etching revealed an homogeneous austenite grain structure, with an ASTM 9.8 grain size. Figures 1(b) to 1(d) show images obtained from extractive replicas and thin foils in which precipitated carbides are observed at prior austenite grain boundaries, lath boundaries and interior of laths; zones with dense dislocations arrays are also apparent (Fig. 1(d)).

Figure 2 shows the morphology and chemical composition of the precipitates present in the as-received material. The major precipitated phase corresponded to the M₂₃C₆ carbide, which was observed at prior austenite grain boundaries and lath boundaries (Fig. 1). The size of M₂₃C₆ carbides ranged from 30 to 300 nm and their morphology was diverse. Figure 2(b) shows the results of EDS analyses (n=35) plotted in a ternary diagram of the main metallic constituents Fe–Mo–Cr.

The next precipitated phase as for the volume fraction corresponds to MX (M=Nb, V, Ti; X=C, N) particles. MX precipitates are found at lath boundaries and interior of laths, Fig. 1(c), and can be classified in three types according to their morphology and chemical composition (Fig. 2(a)). Type I MX corresponds to spherical, Nb-rich precipitates with a 21 nm average size. Type II MX designs V-rich precipitates, with cuboid or rod-like morphology. Type III precipitates display a specific morphology, known in the literature as "wing", with an average size of 89 nm. This classification follows the one already stated by other authors in ferritic–martensitic 9% Cr steels. Fig. 2(c) shows the chemical composition of the MX precipitates (n=118) plotted in a ternary diagram Nb–V–Cr.

It is important to mention in this classification the presence of complex MX precipitates with a chemical composi-
tion Nb–V–Ti, a minor volume fraction and a size higher than 40 nm (Fig. 3).

Marginally, very few particles identified on the basis of the hexagonal M₂X structure were also detected (Fig. 4).

A qualitative ranking of the count frequency of the different MX precipitates gives, in descending order, type II, type III, type I, complex Nb–V–Ti and M₂X.

3.2. Growth Behavior of Austenite Grains

Figure 5 exhibits the metallographic aspect of the samples annealed in the austenite phase field at 1 060 and 1 080°C for different times and heating rates to austenite. It is readily apparent that heterogeneous grain growth has been triggered between 1 min annealing and 15 min annealing.

Previous work\textsuperscript{10–12} indicates that the austenite grain size distribution follows a lognormal law. The lognormal distribution is given by

\begin{equation}
    f(d,s) = \frac{1}{(2\pi s^2)^{1/2}} \exp \left[ -\left( \frac{\ln(d/d_\text{g})^2}{2s^2} \right) \right] \tag{1}
\end{equation}

where \( s \) is the standard deviation, \( d \) is the grain diameter and \( d_g \) is the peak grain diameter. The mean grain size, \( d_b \) is given by

\begin{equation}
    d_b = \sum_{i=1}^{k} f_i d_i = d_g \exp \left[ \frac{(\ln s)^2}{2} \right] \tag{2}
\end{equation}

Figure 6 shows the fitting of Eq. (1) to the experimentally determined austenite grain size distributions for the heating rates to austenite of 1 and 50°C/s and different holding times at 1 060°C.

The grain size histogram approaches indeed to a lognormal distribution, and the eventual “abnormal” grains, result of the exaggerated growth, remain integrated at the right tail of the distribution. Several procedures have been proposed to separate the “normal” and “abnormal” grain populations in a grain size distribution.\textsuperscript{13,14} In Ref. 13) the crite-
Fig. 5. Optical micrographs of samples annealed in austenite at: 1°C/s–1060°C, (a) 1 min, (b) 15 min, (c) 30 min; 50°C/s–1060°C, (d) 1 min, (e) 15 min, (f) 30 min; and 50°C/s–1080°C, (g) 1 min, (h) 15 min, (i) 30 min.

Fig. 6. Prior austenite grain size distributions obtained from samples submitted to different austenitizing conditions. The number of measured grains is indicated in each case.
rion is proposed of considering “abnormal” the grains whose logarithmic size is higher than the mean plus two times the standard deviation of the distribution. In this work, the criterion used to “split” the austenite grain size distribution in two populations was to consider as “abnormal” grains those with an ASTM size lower than 5, that is, with an equivalent diameter higher than 65 μm. This criterion originates as follows: first, a preliminary definition of “normal” population was formulated on the basis of logarithmic diameters according the method in Ref. 13); then, an average size for this “normal” population was calculated, giving values in the range ASTM 8–9 (16 to 22 μm) in all of the studied samples. The value ASTM 5, although somewhat arbitrary, was then selected as a safe bound to define “large” grains in accordance with microstructural observations and taking into account the above outlined “normal” average grain size. Furthermore, if we consider the size advantage $Z$ defined by Gladman\(^{14}\) and define an “effective $Z$” as $R_{ab}/R_{av}$, where $R_{ab}$ is the size of a grain with ASTM number equal or lower than 5 and $R_{av}$ is the calculated average grain size (ASTM number 8–9) we get an “effective $Z$” of 3, which enters within the range for abnormal grain growth. As explained, the area percentages of all the measured grains were also classified according to the ASTM size. Thus, even if this restrictive criterion leaves only a few “abnormal” grains, they cover a considerable area percentage. For instance, from the 3 188 grains evaluated for the sample heated at 1°C/s and held 30 min at 1 060°C only 19 turned out to have an ASTM size equal or lower than 5, but their total area percentage was 25.6%. Figure 7 shows the area percentage of “abnormal” grains in the tested austenitization conditions and according to the above definitions.

In all cases, abnormal grains were already present after 15 min austenite annealing. This fact suggests that abnormal or heterogeneous grain growth is triggered at holding times comprised between 1 min and 15 min, since the 1 min austenite grain size distribution shows no large grains (Figs. 5 and 6). Another aspect noticeable from Fig. 7 is that the temperature increment enhances the grain size heterogeneity, whereas the heating rate increment seems not to affect markedly the grain size distribution behavior, at least, to the extent of the error of our methods of image analysis.

3.3. Precipitates in the Austenitized and Quenched Condition

Figure 8 shows TEM micrographs obtained from extraction replicas of samples austenitized at 1060 and 1080°C for different times. Precipitates are observed to display a heterogeneous spatial and size distribution; their morphology in the as-quenched martensite was spherical.

The chemical composition included Nb, V and Cr as the main metallic components. Complex Ti-rich precipitates were also detected in some cases; their occurrence is discussed in the next section. The TEM-EDS analyses of the precipitates are shown in ternary composition diagrams in Fig. 9; these analyses determined that precipitates are of the MX type and, very likely, carbonitrides.

For the 1 min austenite annealing, precipitates were found to be Nb-rich MX (major) and V-rich MX (minor), these last with sizes higher than 50 nm. Very few M\(_2\)C\(_6\) particles were also detected. For the 15’ and 30’ austenite annealing times V-rich precipitates were not found, and the chemical composition of the Nb-rich precipitates displayed values slightly higher than those measured in the as-received state. The chemical composition of the Nb-rich precipitates did not change with austenite annealing time (see Table 2).

Another precipitated phase systematically observed by SEM and TEM-EDS techniques in all of the examined replicas was the M\(_3\)C carbide. According to 9% Cr steel lit-

![Fig. 7.](image_url) Area percentage occupied by the “abnormal” austenite grains for the studied austenitizing conditions.

![Fig. 8.](image_url) TEM micrographs of extractive replicas prepared from samples austenitized in different conditions. (a) 1°C/s–1 min/1 060°C, (b) 1°C/s–15 min/1 060°C, (c) 1°C/s–30 min/1 060°C, (d) 50°C/s–1 min/1 080°C, (e) 50°C/s–15 min/1 080°C and (f) 50°C/s–30 min/1 080°C.
Literature, this carbide is not stable at the tested austenite temperatures, which means that its appearance probably occurs by autotempering during cooling, even at the fast rates used in this work. M3C precipitates are Fe-rich and display a needle-like morphology (Fig. 10).

M3C precipitation was not observed to be related to MX precipitation, as proposed in Ref. 15); on the contrary, zones with MX presence and no M3C particles were observed as shown in Fig. 8. On the other hand, M3C precipitates are mainly found in coarse martensite laths (Fig. 10), a similar observation has already been reported in Ref. 16) for the T92 steel.

3.4. Particle Size Distribution of MX Precipitates

Figure 11 shows the size distribution histograms of the MX precipitates at 1060°C for different austenite holding times, after heating to austenite at 1 and 50°C/s. Particle counts have been normalized dividing by the maximum count. In some cases, the size distribution strongly suggests a bimodal feature, in view of which we have fitted 'all' the distribution histograms using 'two' lognormal equations in every case. Under this assumption, the observed trend indicates a diminution of the frequency count of the "small"

| Chemical composition of the Nb-rich MX precipitates in the as-received and austenitized metallurgical states. |
| --- | --- | --- | --- |
| As received | Nb-rich MX | % Nb | % V | % Cr |
| Nb-rich MX | 80.6 ± 4.1 | 11.9 ± 2.5 | 7.5 ± 2.7 |
| MX type 'wing' nuclei | 76.3 ± 10.6 | 16.4 ± 7.8 | 7.3 ± 2.9 |
| Austenitized 1°C/sec – 1060°C | 1 min. | 83.9 ± 8.3 | 12.9 ± 5.1 | 32 ± 12 |
| 15 min. | 88.0 ± 4.1 | 9.1 ± 3.6 | 2.9 ± 12 |
| 30 min. | 88.1 ± 4.7 | 9.1 ± 3.5 | 2.8 ± 16 |
| Austenitized 50°C/sec – 1060°C | 1 min. | 85.7 ± 5.1 | 10.7 ± 4.1 | 3.6 ± 1.7 |
| 15 min. | 87.3 ± 4.6 | 9.5 ± 3.8 | 3.2 ± 1.5 |
| 30 min. | 88.0 ± 3.8 | 8.8 ± 2.8 | 3.2 ± 1.6 |
| Austenitized 50°C/sec – 1080°C | 1 min. | 84.8 ± 4.6 | 12.1 ± 3.6 | 31 ± 12 |
| 15 min. | 88.1 ± 4.3 | 9.1 ± 3.4 | 2.8 ± 12 |
| 30 min. | 87.4 ± 5.7 | 9.5 ± 4.6 | 31 ± 14 |

* Corresponds to Nb-rich MX precipitates with sizes higher than 50 nm.
(described by the first lognormal curve) precipitates, whereas the frequency count and size of the “large” (described by the second lognormal curve) precipitates increases, especially from 15 to 30 min austenite holding time. This trend seems to be more severe for the 1°C/s heating rate. A similar behavior was observed for the 1080°C austenitizing temperature and the 50°C/s heating rate.

In Fig. 12 we have combined the chemical information given in the preceding section with the size information given in the present section; a relationship is shown between the size and the Ti content of the MX precipitates obtained after 1 min austenite holding time.

It is observed that the highest Ti contents correspond to particles larger than 40 nm. Considering that the austenitiz-
ing time is too short in this case for particles to grow to these sizes and in view of the high thermodynamic stability of Ti-containing precipitates, it is likely that the Ti-containing particles come from the as-received state.

4. Discussion

Austenite grain growth in presence of pinning particles is a complex phenomenon that has deserved extensive research. Just to mention a few examples, studies have been done about the role of the Nb carbonitride precipitation on abnormal grain growth in Nb added case hardening steels, the interaction between second-phase particle dissolution and abnormal grain growth in austenitic stainless steels and the effect of a non-uniform distribution of carbonitride particles on prior austenite grain size in the simulated coarse-grained HAZ of thermomechanical processed steels.

The first key result of the present work is to show a “window” of laboratory austenitizing conditions under which heterogeneous austenite grain growth can be developed at temperatures as low as 1060°C—usual in industrial practice—in the ASTM A213 T91 steel. This heterogeneous growth is probably related to the evolution of MX precipitates during the austenite treatment, as explained below. Assuming that austenite grain growth proceeds once a pinning relationship favorable to boundary motion has been attained, the interpretation of these results is not straightforward, because we deal, in principle, with three types of potential pinning precipitates. Thus, the question arises of what population/s of second phases exerts the pinning action, and what is the cause that triggers the exaggerated growth of a few austenite grains observed from 15 min annealing time on.

In what follows, we will assume that the applied cooling rate (50°C/s) is fast enough to prevent MX precipitation during quenching, i.e., the precipitation state observed at room temperature for MX second phases corresponds to the one produced at high temperature in all cases.

4.1. Precipitation State during Austenitization

Type III particles have been reported to be formed by a two-step precipitation mechanism, by which two V-rich “wings” nucleate and grow during tempering at 760°C on a preexisting Nb-rich spherical particle coming from normalizing at 1060°C in a 9% Cr Nb–V–N steel. Other reports point out that V-wings may suffer substantial growth on tempering at temperatures above 600°C. Our 1 annealing time results suggest that it is possible to discard pinning effects due to Type III precipitates. Indeed, these precipitates have not been observed, that is, either their “duplex” nature is changed or they are dissolved. This result indicates that type III MX configuration is not stable at austenite temperatures, even for short time. To this respect, it is worth to mention that we have already observed the destabilization of type III precipitates at high tempering temperatures (780°C).

Figures 2 and 9 show the TEM-EDS chemical analyses of the precipitates in the as-received and austenitized (in different conditions) states, located in ternary composition diagrams. In particular, Figs. 9 and 12 show that only Nb-rich MX precipitates and Ti-containing complex precipitates remain after 15 min. austenite holding time. This result indicates that the M23C6 carbide (major phase in the as-received state) and the cuboid or rod-like shaped, V-rich MX carbide or carbonitride (one of the MX phases present in the as-received state) are dissolved during austenitizing, with a dissolution rate fast enough to completely disappear after 15 min austenite holding time. It is also interesting to notice that the chemical composition of the Nb-rich precipitates remains almost constant from the 1 min austenite holding time on (Fig. 9 and Table 2). Some controversy seems to exist about the chemical nature of the particles present at austenite temperatures in the range of 1050–1060°C. As shown, in this work only very high-Nb precipitated particles are detected after 15 min austenite holding time; other reports agree with the only presence of Nb-rich precipitates. However, experimental evidence has also been presented as for the presence of V-containing precipitates (wt% V ~50% and higher) in T91 steel samples normalized 10 min at 1050 and 1100°C, and V-rich precipitates (wt% V ~80%) in experimental Fe–Nb–V–C–N alloys austenitized at 1000°C or higher temperatures for several hundred hours.

Tamura and coworkers have studied the precipitation behavior of the NbC, VN and TaC phases during austenitization in different martensitic steels, starting from the martensite state (air cooled) after a solution treatment. They concluded that second phase precipitation is completed after ~30 min austenite holding time.

Precipitation would be promoted by a fast α→γ transformation, because the transformation provides nucleation sites inherited from martensite. These observations provide a basis to suggest that a high volume fraction of Nb-rich particles (with respect to the equilibrium one) could be attained in a short time in our case, since the selected heating rates to austenite were fast. Besides, there are some Nb-rich precipitates in the as-received state, which could play the role of nucleation centers. However, Nb-rich precipitation kinetics could also depend on the Nb and C (and/or N) availability, which in turn is controlled by the dissolution of M23C6 carbides and by the release to the matrix of the Nb bound in the V-rich precipitates—present only in the as-received state and after 1 min austenite holding time—and “wing” precipitates.

Besides the high-Nb precipitates, Fe-rich, needle-like precipitates were observed in the austenitized and quenched samples within the coarse martensite laths (Fig. 10). These precipitates are probably formed during cooling by an autotempering process. Another important microstructural feature is the presence, in all of the austenitized samples, of Ti-containing MX precipitates, that had already been observed in the as received material. The measured range of sizes in the austenitized samples was similar to that in the as-received sample, i.e., higher than 40 nm (Fig. 12). As these precipitates are known by their high thermodynamic stability at the tested temperatures, it is reasonable to suppose that they will join the “large precipitate” population, that is, they won’t be dissolved.

The observed bimodality in the particle size distribution of MX precipitates allows to identify roughly two populations, i.e., “fine” and “coarse” precipitates. Fine MX precipi-
incites are found with a higher count frequency at the beginning of austenitization (Fig. 11); they form by nucleation and growth as a consequence of austenite supersaturation in Nb and C. On the other hand, coarser MX particles come probably from the as-received state (type I MX particles or type III, “wings” particles; it is also important to remind that the V-rich precipitates observed at 1 min austenite annealing have sizes higher than 50 nm). Composition data in Table 2 are in accordance with this hypothesis, showing that 1 min austenitizing leaves almost unchanged the Nb content of Nb-rich precipitates with sizes higher than 50 nm with respect to the as-received material. In turn, the Nb content of the Nb-rich precipitates remained almost constant with further increments of the austenitization time. The mechanism by which the particle size distribution is shifted towards higher values with austenitizing time is not still clear, but it could be assigned to an Ostwald ripening process once the final volume fraction of precipitates is attained. According to the histograms in Fig. 11, the coarsening process seems to be less severe in the sample heated at 50°C/s.

4.2. Precipitate Size and Austenite Grain Size

For the analysis of abnormal grain growth in presence of second phase particles, Gladman(2) has developed a well-known approach that takes into account the interplay between the parameters of the pinning particle size distribution and the grain size distribution. Gladman’s equation for grain growth inhibition is given by

\[ r_{\text{crit}} = 6 \frac{R_s f}{\pi} \left[ \frac{3}{2} - \frac{2}{Z} \right]^{-1} \]

where \( r_{\text{crit}} \) is the critical radius for grain unpinning, \( R_s \) is the matrix grain size, \( f \) is the volume fraction of precipitates and \( Z \) is the size advantage (\( R_{\text{max}}/R_{\text{average}} \)). Grain growth can proceed for particle radii higher than \( r_{\text{crit}} \); in turn, this quantity increases with the increment of the matrix grain size, the precipitate volume fraction and the diminution of \( Z \). Thus, an increment in \( r_{\text{crit}} \) gives rise to a homogeneous and fine distribution of matrix grains, because precipitates must coarsen to attain the critical radius value before abnormal grain growth can start; on the other hand, for a material including high-Z grains even fine particles may be ineffective to pin grain boundaries because \( r_{\text{crit}} \) diminishes with the increment of \( Z \).

On the basis of the above considerations, we propose that the heterogeneity in size and distribution of the precipitates could be the cause of the observed heterogeneity in austenite grain growth. Considering that the austenite “normal” grain size remained almost constant with the austenitization time (Fig. 6) and that a high volume fraction of precipitates (with respect to the equilibrium one) is probably attained in a short time, abnormal grain growth would depend on the interplay between the \( Z \) value and the particle size evolution. In a first stage, austenite grain size remains fine and uniform due to the high density of fine precipitated particles (Fig. 11) which effectively pin grain boundaries. However, as austenitization proceeds, the shift of the particle size distribution to higher sizes favors the unpinning of selected grains (those with higher \( Z \) value) and triggers the heterogeneous growth process. Selected grains with a suitable \( Z \) value will grow in regions with a major presence of coarse precipitates as a consequence of the low pinning ability of those precipitates; these growing grains will increase further their size advantage over their neighbors, which will in turn diminish the critical radius for pinning. As the particle size of precipitates evolves towards higher values with austenitizing time, it is less probable for such grains to find particles fine enough to stop the boundary motion.

4.3. Austenite Grain Size Behavior and Heating Rate: Comparison with Previous Results

In previous work on T91 and other 9% Cr steels(6) it has been shown that the heating rate to austenite has a critical influence on the development of a heterogeneous austenite grain size distribution. In particular, for the austenitizing temperature of 1060°C and 30 min holding time, a critical value for the heating rate should be expected, above which a heterogeneous distribution is developed during austenitization.

The results obtained do not agree with the previous findings. A 50°C/s heating rate to austenite produced—as expected—a heterogeneous grain size distribution after 15 min austenite holding time, but the same result was obtained for a 1°C/s heating rate. In this last case, a homogeneous fine grained distribution was expected according to Ref. 6).

The reasons for such a disagreement are not still clear, but it is worth to notice that, even if the austenitizing conditions were the same in the previous and present work, the heating methods and sample sizes were different. Indeed, in Ref. 6) experiments were performed in a high speed dilatometer, in which small samples (12×2.5 mm cylinders) are heated in a radiant dual elliptical heater. In the Gleeble machine, on the contrary, larger samples are heated by current passage and temperature gradients are generated both in the axial and radial directions of the sample. As the critical heating rate to produce a heterogeneous austenite grain size distribution would be a sharp decreasing function of the austenitizing temperature(4) a 20°C gradient in the temperature of the sample would be enough to drastically change (lower) the value of the critical heating rate. We did not find metallographic evidence of such a gradient in the radial direction, but we still think that the explanation should be sought on heat conduction through the sample grounds.

5. Conclusions

(1) Heterogeneous austenite grain growth in ASTM A213 T91 steel was observed in the conditions of the Gleeble test at 1060°C and 1080°C for austenite holding times from 15 min on, and for the 1°C/s and 50°C/s heating rates to austenite.

(2) The precipitate behavior during austenitization evidenced:
   i) a change in the particle size distribution towards a bimodal feature as a function of the austenite holding time.
   ii) the dissolution of V-rich precipitates (type II and type
III), observed at 15 min austenite holding time. iii) the increment of the count frequency of the coarse precipitates and the diminution of the fine precipitates one with the austenite holding time.

(3) Heterogeneous austenite grain growth was relatively insensitive to the change in the heating rate to austenite, in contrast with previous results (radiant heating of small samples); on the other hand, the austenite temperature played a marked role on grain size heterogeneity.

(4) Gladman’s approach provides a framework to rationalize the heterogeneous austenite grain growth on the basis of the behavior of the second phase particle size distribution during austenitization.

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REFERENCES