Effect of Nb on Recrystallization After Hot Deformation in Austenitic Fe–Ni–C

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In high-strength low-alloy steels, the deformation, restoration and precipitation effects which occur during finish rolling in the austenitic condition are difficult to study because of the transformation of austenite to ferrite and/or martensite on cooling. To overcome this difficulty a series of austenitic Fe–Ni–C alloy were prepared with and without niobium to allow a more detailed study of the phenomena occurring during finish rolling. The effect of niobium on the static recrystallization of austenite after single-pass hot compression has been studied for a strain range of 0.25–0.9 at temperatures in the range of 850–1 000°C.

The results obtained by optical microscopy and transmission electron microscopy indicated that the retardation of static recrystallization was caused by either niobium in solution and/or strain-induced precipitates. Static recrystallization in the niobium steel was retarded when niobium was in solution by several times compared with the niobium-free steel. However, much stronger retardation in the niobium steel was observed when strain-induced precipitates were formed. It was observed that the dislocations, subgrain boundaries, and grain boundaries are preferential sites for strain-induced NbC precipitation. Small precipitates (<3 nm) were responsible for the retardation of recrystallization by the pinning of subgrain boundaries and grain boundaries.

KEY WORDS: recrystallization; austenite; transmission electron microscopy; niobium carbide; grain boundaries.

Table 1.

<table>
<thead>
<tr>
<th>Materials</th>
<th>C</th>
<th>Ni</th>
<th>Nb</th>
<th>Mn</th>
<th>Si</th>
<th>S</th>
<th>P</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nb-free steel</td>
<td>0.15</td>
<td>30.50</td>
<td>---</td>
<td>0.01</td>
<td>0.03</td>
<td>0.001</td>
<td>0.002</td>
<td>0.0005</td>
</tr>
<tr>
<td>Nb steel</td>
<td>0.15</td>
<td>30.70</td>
<td>0.02</td>
<td>0.001</td>
<td>0.01</td>
<td>0.001</td>
<td>0.003</td>
<td>0.0005</td>
</tr>
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</table>
and at 1 175°C for 15 min, respectively, and then cooled to the test temperature. The different reheating treatments allowed the starting grain size to be maintained at 310 μm for both steels. Transmission electron microscopy, combined with observations of the grain growth behaviour at different temperatures, indicated that most or all of the niobium was in solution after the reheating treatment. This conclusion is also consistent with estimates of a solution temperature for 0.02% Nb HSLA steels.8)

Single pass axial compression tests were carried out to strains of 0.25 and 0.5 at a constant strain rate of 0.7/sec. The compressed specimens were isothermally held at deformation temperatures of 850, 900, 950 and 1 000°C. Both optical and transmission electron microscopy were used to study the progress of recovery and recrystallization. The volume fraction of recrystallization was determined by a point counting technique.

To investigate the kinetics of niobium carbide precipitation in work-hardened and recrystallized austenite, thin foil and extraction replica techniques were used. The formation of Nb-rich precipitates was confirmed by the EDX technique. Furthermore, the analysis of diffraction patterns indicated that the particles were NbC precipitates.

3. Results

Transmission electron microscopy studies of thin foils and extraction replicas of niobium steel were performed to investigate the precipitation behaviour of NbC after hot deformation of austenite. In particular, the interaction between precipitation, dislocation structure and recrystallization was investigated.

Figure 1 shows the extracted strain-induced precipitates of NbC in a specimen deformed to a strain of 0.25 at 850°C, and held at this temperature for various holding times. No precipitates were observed in the sample held for less than 22 sec. With holding times ≥22 sec, precipitates were observed in austenite and Fig. 1 indicates the coarsening behaviour of particles with increasing holding times in the range from 22 to 780 sec. These results were later confirmed by TEM observation.

The results obtained from thin foil electron microscopy indicated that the strain-induced precipitates form at grain boundaries, dislocations and subgrain boundaries. Figure 2 presents electron micrographs for a specimen deformed to a strain of 0.25 and isothermally held at 850°C and shows precipitation of NbC at boundaries and in the interior of work-hardened grains. It is clear from this figure that the mean sizes of the particles in grain and subgrain boundaries are larger than those in the interior of grains.

An example of the pinning effect of NbC particles on a subgrain boundaries is presented in Fig. 3. This figure is a thin foil electron micrograph of a specimen which was deformed to a strain of 0.25 and isothermally held at 850°C and shows precipitation of NbC at boundaries and in the interior of work-hardened grains. It is clear from this figure that the mean sizes of the particles in grain and subgrain boundaries are larger than those in the interior of grains.

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The pinning effect of strain-induced NbC particles at the interface between recrystallized and unrecrystallized grains is presented in Fig. 4. It was observed that there was a higher density of small NbC particles in the unrecrystallized regions, and a lower density of coarser particles in the recrystallized regions, see Fig. 5. A thin foil micrograph showing the bright field and dark field images of coarsened and widely spaced particles in the recrystallized region is also presented in Figs. 6(a) and 6(b). Figures 6(c) and 6(d) are the selected area diffraction pattern and the indexed pattern of Fig. 6(a), respectively. The electron micrographs shown in Figs. 4 to 6 correspond to the specimen pretrained to 0.5 and then held for 450 sec at 850°C. This treatment resulted in 60% recrystallization.

4. Discussion

The kinetics of NbC precipitation and the time to start precipitation for the steel investigated were theoretically and experimentally determined. A kinetic model for precipitation in niobium microalloyed steels has been detailed by Dutta and Sellars.9) Since this model was based on thermodynamic considerations and diffusion controlled nucleation theory for niobium in austenite, it was adapted for NbC precipitation in the current work with consideration of the relationship between free energy and composition used for Fe–30% Ni steel.10) By assuming that the shapes of the C-
Fig. 2. TEM micrographs showing the presence of NbC precipitates, (a) at boundaries in deformed austenite, (b) and (c) on dislocations within deformed (unrecrystallized) austenite grains. The specimen was deformed to a strain of 0.25 and isothermally held 40 sec at 850°C.

Fig. 3. TEM micrograph of a thin foil specimen, indicating the pinning effect of NbC precipitates on the movement of a subgrain boundary. The specimen was deformed to a strain of 0.25 and isothermally held 120 sec at 850°C.

Fig. 4. TEM micrographs showing the pinning effect of strain-induced NbC precipitates in the recrystallizing interface. The specimen was deformed to a strain of 0.5 and isothermally held 450 sec at 850°C.

Fig. 5. TEM micrographs showing the recrystallizing interface in a specimen deformed to a strain of 0.5 and isothermally held 450 sec at 850°C. Niobium carbide precipitates are observed to be fine in the unrecrystallized region and coarse in the recrystallized region.
curves for the strain-induced precipitation and for precipitation in undeformed austenite are the same; and using the time to the start of precipitation at the C-curve nose from the experimental data, the C-curve for strain-induced precipitation of NbC was estimated, as shown in Fig. 7.

Sellars\(^1\) collected the results from several workers on the time to the start of NbC and Nb(C,N) precipitation in low alloy austenite. These results indicate that the agreement among the published data is poor. However, most studies have shown that overall precipitation behaviour appears to conform to C-curve behaviour with the nose of the kinetic curve being between 850 and 950°C, and commonly taken as 900°C. The discrepancies among the results of different workers may be caused by the wide variety of techniques which have been used to study the precipitation kinetics, such as hardness testing,\(^{12,15}\) X-ray diffraction,\(^{14}\) quantitative electron microscopy,\(^{15}\) and flow curve analysis.\(^{15,16}\) The lower position of the nose of the kinetic curve for the present case (850°C) compared to that for the microalloyed steels (an average temperature of 900°C) may indicate that the high nickel content in the steel investigated has shifted the nose to the lower temperature.

It was found in the present investigation that the strain-induced precipitation is sensitive to the amount of prior strain. The time to start precipitation at the kinetic nose (850°C) was 22 sec for a strain of 0.25 and 5 sec after a strain of 0.5. As the prestrain increased, the time to the start of precipitation decreased. This behaviour is due to the higher driving force for precipitation in deformed austenite and is consistent with the results of others.\(^{1,12,17,18}\) The experimental results are also consistent with a comparison of precipitation-start curves for strain-induced precipitation and for precipitation in undeformed austenite which indicates the difference of at least two orders in the time to start precipitation at a given temperature.\(^{11}\)

It was observed that the distribution of NbC precipitates in the work-hardened regions was localized (e.g., Fig. 2). This effect might be due to the large initial grain size of the samples and the associated formation of localized work-hardened regions. The localized sites of precipitation were grain boundaries, tangled dislocations, and subgrain boundaries, all of which provide preferential nucleation sites for NbC precipitation.

It was observed that strain-induced NbC particles formed at austenite grain boundaries at the initial stage of precipitation. Intragranular precipitation of NbC particles then took place on dislocations and subboundaries. Since grain boundaries have high energies, they would be preferential.
nucleation sites for NbC precipitation. Subgrain boundaries are also potent sites for nucleation compared to single dislocations and tangled dislocations within the subgrains. This difference might lead to the presence of larger precipitates on grain and subgrain boundaries, and smaller precipitates within the matrix (Figs. 1(a) and 3). The formation of strain-induced precipitates at grain boundaries at the initial stage of precipitation has also been reported by Hansen et al. and Pereloma et al.\textsuperscript{19)}

Strain-induced precipitation can interact with the recrystallization process, causing retardation of recrystallization. The recrystallization start ($R_{\text{str}}$) diagrams for the niobium-free and niobium steels are presented in Fig. 7 for a strain of 0.25. Times to 50% recrystallization ($t_{0.5}$) under certain deformation condition (strain and temperature) for the niobium-free and niobium steels are shown in Fig. 8. In some cases (e.g. a prestrain of 0.5 at 1000°C), the percentage of recrystallization was more than 50% within the quenching time ($t_{q}=2$ sec). Hence, it was not possible to detect the time to 50% recrystallization directly by experiments. In these cases, $t_{0.5}$ was calculated using the Avrami relationship, by extrapolating the fractional recrystallization versus logarithm time curve to shorter times. As shown in Fig. 8, the slopes of the lines for the niobium steel change below a particular temperature (900°C), indicating that recrystallization is more sluggish below this temperature. This effect would be attributed to strain-induced precipitation of NbC in deformed austenite in this temperature regime.

Figure 7 indicates a retardation effect on the start of recrystallization at temperatures above about 900°C for the niobium steel compared to the niobium-free steel. Since no precipitates were observed by transmission electron microscopy before the start of recrystallization in this regime, and the initial grain sizes for two steels were the same, the retardation of recrystallization in this temperature region results from the solute drag effect of niobium atoms. Figure 8 indicates that static recrystallization in the niobium steel was retarded when niobium was in solution by 2 to 3 times compared to the niobium-free steel. The retardation effect of solute atoms of niobium on the onset of recrystallization observed in the present study is consistent with the observations made by Yamamoto et al.\textsuperscript{21} for HSLA steels. Some workers\textsuperscript{11,18} proposed that the degree of solute drag can be described by the differences in atomic size and electron structure. A significant retardation of the start of recrystallization for the 0.02% Nb steel compared to the niobium-free steel (Fig. 7) below 900°C indicates a major role of strain-induced precipitates of NbC on the austenite recrystallization. It was observed that dislocations, subgrain boundaries and grain boundaries are the preferential sites for nucleation of strain-induced NbC precipitates (Figs. 2–5). The very small precipitates (1–3 nm) pin subgrains and grain boundaries, resulting in the retardation of recrystallization. The effects of NbC precipitates on the progress of recrystallization in the present work are shown in Fig. 8, where a comparison is made of the times to 50% recrystallization for the 0.02% Nb steel and the niobium-free steel. It is clear in this figure that a considerable retardation of recrystallization has occurred in the niobium steel, for the temperatures at which strain-induced NbC precipitates were formed (<900°C).

Observations by TEM of thin foils indicated the presence of NbC in contact with the migrating recrystallization interfaces, as shown in Fig. 4. The amount of recrystallization which had occurred in the specimen shown in Fig. 4 was about 60%. It is evident that the interface between recrystallized and unrecrystallized regions has been pinned by the precipitates. Furthermore, the boundary has a curvature between the pinning precipitates towards the unrecrystallized region (Fig. 4(a)).

Comparison between the number density of precipitates present in the recrystallized and unrecrystallized grains indicates a decrease in the number density of the precipitates in the recrystallized grains (Fig. 5). However, the volume fractions of precipitates in the two regions were found to be similar. The dispersion of the NbC precipitates in the recrystallized grains was observed to be much less homogeneous compared to those in the unrecrystallized regions (Figs. 5 and 6). Similar observations were made by Jones and Ralph\textsuperscript{21} and Dunne,\textsuperscript{22} who studied the role of precipitates on recrystallization in an austenitic stainless steel and an Fe–V–C steel, respectively.

The observations made for the steel investigated indicate that many of the fine NbC precipitates dissolve and facilitate the coarsening process of larger particles contained in the recrystallization interface. It has been shown\textsuperscript{21,23} that the critical size for stable precipitates in the recrystallization interface depends on factors such as temperature, the structure of the interface, the energy of the interface at the precipitate and in the vicinity of the precipitate, and the size and spatial distribution of neighbouring particles in contact with the interface. The effective duration of the pinning process will be determined by the coarsening kinetics of the interfacial particles and will therefore depend on time, temperature and diffusion coefficient of Nb in grain boundaries.

Measurements of the mean particle size in the unrecrystallized and recrystallized regions indicated a considerable difference in the mean diameters of particles in these two regions. For instance, for the specimen deformed to a strain of 0.5 and isothermally held at 850°C, precipitates in the
unrecrystallized regions had a mean diameter of ~3 nm. For the same conditions, precipitates incorporated into recrystallized regions had a mean diameter of ~11 nm. The larger particle size indicates marked coarsening by diffusion of Nb in grain boundaries between the unrecrystallized and recrystallized grains.

When the precipitates in contact with interfaces between recrystallized and unrecrystallized regions have increased to a size and spacing which exceed the critical values, they permit the unpinning of the recrystallization interface, allowing its migration into the unrecrystallized region. Jones and Ralph\textsuperscript{21} proposed that this migration occurs under the driving force provided by the stored energy difference and the high local boundary curvature produced around the particles.

5. Conclusions

(1) Direct observation of the interaction of the NbC precipitation with recrystallization of deformed austenite has been carried out using an Nb bearing Fe–Ni–C.

(2) The preferential sites for precipitation of strain-induced NbC particles were confirmed to be grain boundaries, subgrain boundaries and dislocations. However, grain boundaries were found to be the most potential nucleation sites for precipitation because of their high surface energies.

(3) Fine strain-induec precipitates (<3 nm) suppressed static recrystallization by the pinning of subgrain boundaries and grain boundaries. Formation of strain-induced precipitates was significantly accelerated by increasing strain.

(4) Coarsened precipitates were observed in the recrystallized regions as a result of the migration of the recrystallization interface. Precipitates pin the interface between recrystallized and unrecrystallized regions until coarsening by preferential dissolution of fine particles permits the unpinning of the recrystallization front, allowing its migration into the unrecrystallized region. Coarsened and more widely spaced particles were therefore observed in recrystallized regions.

REFERENCES