Inhibition of Abnormal Grain Growth during Isothermal Holding after Heavy Deformation in Nb Steel

Seung Chan HONG, Sung Hwan LIM, Kyung Jong LEE, Dong Hyuk SHIN and Kyung Sub LEE
Division of Materials Science & Engineering, Hanyang University, Seoul, Korea. E-mail: hongsc@hanyang.ac.kr
1) Department of Metallurgy and Materials Science, Hanyang University, Ansan, Kyunggi-Do, Korea.
E-mail: dhshin@hanyang.ac.kr
(Received on May 24, 2002; accepted in final form on August 26, 2002)

Inhibition of Abnormal Grain Growth during Isothermal Holding after Heavy Deformation in Nb Steel

The microstructural evolution during isothermal holding at 590–750°C after heavy deformation was examined in Nb steel. The strain induced ferrite transformation of Nb steel was significantly retarded compared with that of plain C–Mn steel, when Nb was mostly dissolved. When the grain boundary ferrite was present before deformation and strain free ferrite was formed during the following isothermal holding, abnormal grain growth occurred at the regions near the deformed ferrite by strain-induced boundary migration (SIBM). The SIBM was caused by the energy unbalance at the boundaries between deformed and strain free ferrite grains transformed from the deformed austenite. This rapid growth was not inhibited by the strain-induced NbC precipitates, which means the driving force for the abnormal growth was greater than the pinning force by the precipitates. However, the abnormal grain growth could be prevented by isothermal heat treatment in either fully transformed or untransformed structure. The ultrafine and polygonal ferrite grains were obtained by the recrystallization of ferrite and the grain growth was inhibited by strain-induced NbC precipitates during isothermal annealing at 650°C after deformation.

KEY WORDS: grain refinement; strain induced ferrite transformation; strain induced boundary migration; abnormal grain growth.

1. Introduction
Recently, strain induced ferrite transformation through heavy deformation has received much attention because it is very effective to refine ferrite. However, ultrafine grained materials manufactured by heavy deformation exhibited more enhanced grain growth than conventional fine grained materials, since the grain boundary was in a non-equilibrium high energy state. Besides, heavy deformation accumulates extensive internal energy inside materials so that a considerable residual stress would still remain even though a large portion of internal energy is dissipated by grain refinement. Accordingly, heat treatment for stress relief is necessary for their practical application. In order not to lose the advantage of fine grained structure during the heat treatment, the microstructural stability should be maintained. Several previous investigations revealed that the growth rate of ferrite was reduced by microalloying elements such as Mo, Nb and V. These substitutional alloying elements influence γ/α interface motion through a solute drag-like effect. Especially, Nb affects the growth rate of ferrite considerably by solute drag of Nb as well as pinning of the precipitated alloy carbonitrides. Abnormal grain growth can occur during annealing after deformation even in Nb steel. It could be a result of the fact that the driving force for abnormal grain growth is greater than the drag force responsible for the normal grain growth inhibition and that the mobility of their boundaries increased. The strain induced boundary migration (SIBM) during annealing after deformation leads to bimodal distribution of ferrite grain size. The SIBM occurs by the inhomogeneity of strain energy between the neighboring grains. The purpose of the present work is to investigate the abnormal grain growth behavior of ferrite during isothermal holding after heavy deformation at various temperatures and to prevent the rapid growth by process control in Nb steel.

2. Experimental Procedures

2.1. Specimen Preparation
The alloy was prepared by Vacuum Induction Melting. The chemical composition of the steel used is shown in Table 1. The Nb of 0.05 wt% was added to a low carbon steel. The ingot (30 kg) was reheated at 1250°C for 2 h and hot forged and then air cooled to achieve homogeneous microstructure. Cylindrical sample with diameter of 10 mm

Table 1. Chemical composition of the steel used.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Nb</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(wt%)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.14</td>
<td>1.48</td>
<td>0.78</td>
<td>0.052</td>
<td>0.006</td>
<td>0.004</td>
<td></td>
</tr>
</tbody>
</table>
and length of 12 mm, which had an aspect ratio of 1.2, was machined from the hot forged bar. The hot compression test was carried out by Gleeble 1500. A nickel compound was used between WC (Tungsten carbide) anvil and the specimen as a lubricant at high temperature. The WC anvil increased electric resistance at the interface of the specimen so that the uniform temperature along the specimen axis was quickly obtained.

2.2 Determination of Ar$_3$ Temperature

In order to determine the range of deformation temperature, the Ar$_3$ temperature was measured during continuous cooling at a rate of 2°C/s using dilatometry. The sample was reheated at 1250°C for 5 min to dissolve NbC sufficiently. The dissolution temperature of NbC calculated by Thermo-Calc was 1185°C so that it was assumed that Nb was fully dissolved during the reheating. The measured austenite grain size was 230 μm. Two Ar$_3$ temperatures were defined. One is the starting temperature for ferrite formation at grain boundary marked as Ar$_3$ (g.f). Another one is for acicular ferrite formed austenite grain interiors marked as Ar$_3$ (a.f). The Ar$_3$ temperatures were determined by both the observation of the deviation from the tangential line in dilatation curve and the microstructural examination in the quenched sample.

2.3. Hot Compression Test Followed by Isothermal Annealing

The specimens compressed 80% at a strain rate of 5/s at various temperatures between 590°C and 750°C were held isothermally for 10 min and then water quenched. When the specimen after deformation was not fully recrystallized after holding for 10 min at 590°C, it was reheated to 650°C.

![Fig. 1. Dilatation curve during continuous cooling at a rate of 2°C/s from 1250°C.](image)

![Fig. 2. Microstructures quenched at various temperatures during continuous cooling.](image)

(a) 750°C, (b) 700°C, (c) 680°C, (d) 645°C, (e) 610°C, (f) 590°C.
to complete the recrystallization. Another set was that specimen compressed 80% reduction at 590°C was immediately reheated to 650°C without isothermal holding for 10 min. The microstructure was observed by OM and SEM.

3. Results and Discussion

3.1. Determination of the Transformation Temperature (Ar₃)

The Ar₃ (g.f) and Ar₃ (a.f) were determined as about 720°C and 635°C respectively by both the deviation point from the tangential line in dilatation curve (Fig. 1) and the quenched microstructures (Fig. 2). The transformation was finished at 540°C. The grain boundary ferrite was formed first between 720°C and 635°C, but the amount of ferrite was small, and then acicular ferrite was formed between 635°C and 540°C. The difference between transformation starting (720°C) and finishing temperature (540°C) was about 180°C, which was 50°C wider than that of C–Mn steel under the same condition. This means the ferrite transformation of Nb steel was delayed owing to the soluble Nb. The effect of soluble Nb on γ/α transformation has been well established. Lee et al. have suggested that solute Nb strongly segregates to γ/α interphase boundary and reduces ferrite growth kinetics due to solute drag effect.

3.2. Microstructural Evolution during Isothermal Holding after Heavy Deformation

The strain induced ferrite transformation during 80% reduction was significantly retarded by solute Nb so that most ferrite grains were formed during the following isothermal holding. Figure 3 represents the microstructures after 80% reduction and after subsequent isothermal holding for 10 min above and below the Ar₃ temperature. At 750°C just above the Ar₃ (g.f), few ferrite grains were formed during hot deformation and the polygonal ferrite grains were de-

---

Fig. 3. Microstructural change during isothermal holding at various deformation temperatures.
(a) As deformed (750°C), (b) 10 min holding (750°C), (c) as deformed (680°C), (d) 10 min holding (680°C), (e) as deformed (645°C), (f) 10 min holding (645°C), (g) as deformed (590°C), (h) 10 min holding (590°C).
veloped along the austenite grain boundaries after holding for 10 min, as shown in Fig. 3(b). At 680°C, which is between the Ar3 (g.f) and Ar3 (a.f), grain boundary ferrite was deformed and then an inhomogeneous microstructure was generated near the deformed ferrite grains as shown by arrow head in Fig. 3(d). The evolution of inhomogeneous microstructure could be explained by strain-induced boundary migration. Further, at 645°C, which is also the temperature region between Ar3 (g.f) and Ar3 (a.f), more elongated ferrite grains indicated the increase of grain boundary ferrite grains before deformation. As a result, quite inhomogeneous microstructure was found after holding for 10 min as shown in Fig. 3(f). On the other hand, when the hot compression followed by isothermal holding was carried out at 590°C (below the Ar3 (a.f)), the acicular ferrite was observed within the austenite grains as well as the grain boundary ferrite before the deformation as shown in Fig. 2(f). The deformed ferrite in grain boundary and the deformed acicular ferrite coexisted after deformation at 590°C as shown in Fig. 3(g). However, the abnormal grain growth was not observed during isothermal holding, since the temperature was too low to move the grain boundary as shown in Fig. 3(h).

3.3. Abnormal Grain Growth by Strain-induced Boundary Migration (SIBM)

When the deformed and strain-free ferrite grains coexisted during deformation in the region between the Ar3 (g.f) and Ar3 (a.f), such as at 645°C, abnormal grain growth of ferrite occurred during the subsequent isothermal holding. Figure 4 shows the abnormal grain growth behavior with isothermal holding. It was shown that the ferrite formed at grain boundaries during continuous cooling was deformed by 80% reduction in Fig. 4(b). The new polygonal ferrite was observed at the boundary between deformed ferrite and austenite after 20 s. With increasing the holding time, new ferrite grains were grown rapidly at the deformed ferrite boundaries. After holding for 30 min, very coarse and fine grains were exhibited, leading to bimodal grain size distribution. Zwaag et al. reported the similar phenomena after intercritical deformation as a result of the combination of transformation of deformed austenite into undeformed ferrite, followed by rapid ferrite grain growth into the deformed ferrite grains and extensive recovery of deformed ferrite in 0.1wt% C–0.49wt% Mn steel. The abnormal grain growth could result from inhomogeneity of strain distribution. Figure 5 shows the schematic illustration of the ab-

![Fig. 4. Microstructural evolution with isothermal holding after 80% reduction at 645°C. (a) Before deformation, (b) as deformed, (c) 20 s, (d) 1 min, (e) 30 min, (f) 60 min.](image-url)
normal grain growth by strain induced boundary migration (SIBM). As the strain-free ferrite grain was formed near the highly deformed ferrite grain, the boundary moved to the deformed ferrite grain by stored energy difference between the two grains. SIBM was explained by ‘bulge nucleation model’.19) The boundary will tend to bulge if the stored energy difference between the two grains is greater than the grain boundary energy. As the region bulging out grows, the dihedral angle of bulge, $\alpha$, increases. Figure 6 shows TEM micrograph showing bulged grain due to difference of dislocation density between the two grains induced by the strain inhomogeneity during isothermal holding for 1 min at 645°C. Therefore, SIBM was caused by strain inhomogeneity, which induce the difference of dislocation density between the two grains. This rapid growth was not inhibited by strain induced NbC precipitate, which means the driving force for the abnormal growth was greater than the pinning force by the precipitate. Figure 7 reveals the formation of the strain-induced NbC, which was not observed before hot deformation.

### 3.4. Inhibition of Abnormal Grain Growth

In the case of 590°C (below the $\text{Ar}_1$), the inhomogeneous structure was not exhibited due to low temperature and the deformed austenite was transformed to ferrite during isothermal holding as shown in Fig. 8. In addition, the cementite particles of 0.3 $\mu$m in diameter were dispersed at
subgrain boundaries. However, the ferrite grains were still elongated. The isothermal annealing was performed at 650℃ as the schematic process marked as a bold line in Fig. 9(a). During annealing, the morphology of ferrite was changed to a polygonal shape with an average size of about 2 μm by the recrystallization and then the fine structure was maintained even after 30 min as shown in Figs. 10(a) and 10(b). When the process was performed without isothermal holding at 590℃ marked as a dot line in Fig. 9(b), the ferrite transformation occurred during the isothermal annealing. While, the recrystallization of ferrite was not much preceded due to the competition between the transformation and recrystallization. The deformed ferrite formed during the intercritical deformation at 590℃ was still observed as shown in Fig. 11(a). This result indicates that the ferrite transformation during annealing retarded the recrystallization resulting in microstructure of the deformed ferrite and cementite. On the other hand, when deformation was carried out above the Ar3 (g.f), austenite was fully presented before isothermal annealing. The transformation of deformed austenite into undeformed ferrite occurred during the isothermal annealing as represented in Fig. 11(b). Therefore, the fully transformed ferrite or fully austenite structure is desirable before the annealing to prevent abnormal growth.

4. Conclusions

(1) When heavy deformation was performed after the formation of grain boundary ferrite the deformed and strain-free ferrite grains coexisted during the subsequent isothermal annealing. The abnormal grain growth occurred by the energy unbalance at the boundary between the deformed and strain-free ferrite grains. This rapid growth was not inhibited by strain induced NbC precipitate, which means the driving force for the abnormal growth was greater than the pinning force by the precipitate.

(2) The abnormal grain growth could be inhibited by imposing the isothermal heat treatment after formation of the fully transformed or fully untransformed structure. From fully transformed ferrite structure, ultrafine and polygonal ferrite grains were formed by recrystallization of ferrite during isothermal annealing at 650℃ and were maintained after 30 min.

![Fig. 9. Schematic process for prevention of abnormal grain growth.](image)

![Fig. 10. Microstructures showing the ferrite grains with isothermal annealing time at 650℃.](image)

(a) 1 min, (b) 30 min.

![Fig. 11. Microstructures after subsequent isothermal annealing at 650℃ for 30 min.](image)

(a) After 80% reduction at 590℃, (b) after 80% reduction at 750℃.
REFERENCES