Effect of initial microstructures prior to cold-rolling and intercritical annealing on ferrite recrystallization and ferrite-to-austenite phase transformation in Nb bearing low-carbon steels

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Abstract. This study investigates the relationship between the initial microstructures of Nb bearing low-carbon steels and their microstructural evolution during subsequent intercritical annealing. The initial microstructures of specimens P, B, and M comprised ferrite/pearlite, bainite, and martensite, respectively. In the specimen lacking of Nb, the time for ferrite recrystallization during annealing decreased in the order of M > P > B. Recrystallized ferrite grains in specimen M are fine and equiaxed. The ferrite-to-austenite phase transformation during intercritical annealing occurred more rapidly in specimen M than in specimens P and B. The high austenite fraction in specimen M is presumably caused by the large number of austenite nucleation sites due to the ferrite grain refinement. In the Nb bearing specimens, the time for ferrite recrystallization decreased in the order of M > B > P. The Nb addition retarded the ferrite recrystallization and accelerated the ferrite-to-austenite phase transformation. The high austenite fraction in the Nb bearing specimen is presumably caused by the large number of austenite nucleation sites owing not only to the ferrite grain refinement, but also to the increased number of non-recrystallized ferrite grains.

1. Introduction
Dual phase (DP) steels have good ductility and have been used in various automobile parts [1]. DP steels generally consist of ferrite and martensite, and their mechanical properties mainly depend on the volume fraction, size, morphology, and distribution of martensite [2-5]. Therefore, to ensure high ductility of DP steels, a method to control the above-mentioned microstructural features of martensite is demanded.

DP steels are typically manufactured by continuous casting, hot-rolling, cold-rolling, and subsequent annealing. Previously, we found that the volume fraction and distribution of martensite in low-carbon steels strongly depend on the initial microstructures prior to cold-rolling [6,7]. Therefore, recrystallized ferrite grains are fine and equiaxed when the initial microstructure consists of martensite, which leads to the acceleration and homogenization of austenite formation at the early stage of intercritical annealing [6]. The austenite at intercritical temperature transforms into martensite during the cooling process. This implies an important role for the morphology of recrystallized ferrite grains in martensite formation in low-carbon steel. During intercritical annealing, the microstructure is highly...
dependent on the interaction between ferrite recrystallization and the ferrite-to-austenite phase transformation.

We also investigated the interaction between ferrite recrystallization and the ferrite-to-austenite phase transformation during intercritical annealing in low-carbon steels [8,9]. For instance, we confirmed that ferrite recrystallization is remarkably retarded when it accompanies a ferrite-to-austenite phase transformation during intercritical annealing [8]. In Nb bearing low-carbon steels, the Nb addition retards the ferrite recrystallization and accelerates the ferrite-to-austenite phase transformation during intercritical annealing [9]. Clearly, the Nb addition results complicated interaction of the ferrite recrystallization and the ferrite-to-austenite phase transformation during intercritical annealing. However, the relationship between the initial microstructures of microalloyed low-carbon steels and their microstructural evolution during annealing remains unknown. Designers of low-carbon steel materials would desire to understand the roles of the initial microstructures and microalloying elements on the microstructural evolution during annealing.

The present study aims to relate the initial microstructures of Nb bearing low-carbon steels to their microstructural evolution during subsequent intercritical annealing. In particular, it is dedicated to metallurgical phenomena (ferrite recrystallization and the ferrite-to-austenite phase transformation) during cold-rolling and intercritical annealing.

2. Experimental procedures

The chemical compositions of two steels, one Nb-less specimen and one Nb bearing specimen, are listed in Table 1. The results of the Nb-less specimen were reported in our previous articles [6,7]. The vacuum-melted ingots were rough-rolled to a thickness of 30 mm. The rough-rolled steels were hot-rolled to 3.0 mm at a finishing temperature of 900 °C in the austenite region, and subsequently water-cooled to 650 °C (the ferrite/pearlite specimen P), 500 °C (the bainitic specimen B), and below 100 °C (the fully martensite specimen M), and then cooled to room temperature in air. The hot-rolled sheets were cold-rolled to a thickness of 1.0 mm (reduction: 67%).

After cold-rolling, the specimens were heated to 500, 660, and 700 °C at 0.5 °C/s, and then water-quenched to room temperature within 2 s of removal from the furnace. The rapid quenching avoided the austenite-to-ferrite phase transformation. The austenite phase in these steels, present during intercritical annealing, was transformed to martensite during cooling. The A1 and A3 temperatures of the tested steels were approximately 670 °C and 820 °C, respectively.

The microstructure was observed by a scanning electron microscope (SEM). The microtexture of the annealed specimens was analyzed at the quarter-thickness position on the rolling plane by an electron backscatter diffraction/field emission scanning electron microscopy (EBSD/FEGSEM) system employing OIM software. The step size of the EBSD measurements was 0.2 μm. In the EBSD data analysis, the fraction of recrystallized ferrite grains was analyzed by kernel average misorientation (KAM) analysis. Regions with misorientation results below 1° in the KAM analysis were assumed as recrystallized ferrite grains. In our previous report [8], the area fractions of the recrystallized ferrite grains evaluated by KAM analysis and the point-counting method showed good agreement. The martensite fraction was estimated by the point-count method (ASTM E 562), as mentioned in our previous studies [6,7]. The martensite fraction was estimated from 2475 counting points.

| Table 1. Chemical compositions of the steels (in mass%). |
|----------------|---|---|---|---|---|---|---|---|
|               | C  | Si | Mn | P  | S  | Nb | Al | N  | O  |
| Nb-less specimen | 0.10 | < 0.003 | 2.03 | 0.010 | 0.0029 | < 0.003 | 0.027 | 0.0030 | < 0.001 |
| Nb bearing specimen | 0.10 | < 0.003 | 2.02 | 0.010 | 0.0030 | 0.050 | 0.027 | 0.0031 | < 0.001 |

3. Results and discussion

Figure 1 shows the image quality (IQ) and KAM maps of the Nb bearing specimens heated to 660 °C (immediately below the Ac1 temperature). After heating, non-recrystallized ferrite grains remained in the specimens P (Figure 1(a)) and B (Figure 1(c)), whereas ferrite recrystallization was almost completed in specimen M (Figure 1(e)). Previously, we reported that ferrite recrystallization in the Nb-
less specimen was completed at 660 °C in specimens of all compositions [6]. Therefore, the Nb addition clearly retarded the ferrite recrystallization. Furthermore, the recrystallized fraction was larger in specimen B than in specimen P (Figure 1(b) and (d)). These results suggest that the ferrite recrystallization time during annealing decreased in the order of M > B > P. In the Nb-less specimen, we previously demonstrated that the ferrite recrystallization time during annealing decreased in the order of M > P > B [6]. It appears that ferrite recrystallization was significantly retarded by Nb addition in specimen P.

![Figure 1](image-url)

**Figure 1.** Image quality and kernel average misorientation maps of the Nb bearing specimens (a, b) P, (c, d) B and (e, f) M heated to 660 °C (αR: recrystallized ferrite, αNR: non-recrystallized ferrite).

Figure 2 shows the Vickers hardness changes in the Nb bearing specimens during isothermal holding at 500 °C. In specimen M, the hardness gradually decreased with holding time. Meanwhile, the hardness of specimen P slightly decreased after isothermal holding for 2000 s, while that of specimen B hardly changed. These results indicate that the recovery time of the specimens during annealing decreased in the order of M > P > B. We previously demonstrated that ferrite recovery and recrystallization occurs more rapidly in martensite than in bainite or pearlite, because the dislocation density and its heterogeneity after cold-rolling was larger in specimen M than in specimens P and B [6]. Furthermore, as more solute Nb increases prior to cold-rolling, the recovery progress becomes more retarded [10]. The amount of precipitates containing microalloying elements such as Nb and Ti increases with increasing cooling stop temperature after hot-rolling [11]. Therefore, the amount of solute Nb prior to cold-rolling was probably smaller in specimen P than in specimen B. This would explain the stronger retardation of recovery in specimen B than in specimen P.

Although the Nb bearing specimen B exhibited stronger retardation to recovery than specimen P, its ferrite recrystallized more quickly than in specimen P. The interaction between recovery and recrystallization in steels has been extensively studied [12-14]. Belyakov et al. [12] demonstrated that
Recrystallization is retarded when the recovery is rapid because the driving force of recrystallization decreases as the recovery accelerates. Therefore, the driving force of recrystallization is probably larger in specimen B than in specimen P. These results suggest that Nb addition can vary the effect of the initial microstructures on the recovery and recrystallization behaviors of ferrite.

**Figure 2.** Changes in Vickers hardness of the Nb bearing specimens during isothermal holding at 500 °C.

Figure 3 shows the microstructures of the Nb bearing specimens heated to 700 °C (above the $A_{c1}$ temperature). In specimens P and B, non-recrystallized ferrite grains and martensite connected along the rolling direction were observed (Figure 3(a) and (b)). In contrast, fine and equiaxed recrystallized ferrite grains were observed in specimen M, and the martensite was distributed rather homogeneously in this sample (Figure 3(c)). Figure 4 shows the fraction of martensite in the specimens heated to 700 °C. The martensite fractions in the Nb-less specimen were below 10%, irrespective to their initial microstructures. In contrast, martensite fractions in all Nb bearing specimens exceeded 10%. We previously demonstrated that the nucleation sites of austenite during intercritical annealing are mainly at the recrystallized ferrite grain boundaries and the subgrain boundaries in non-recrystallized ferrite grains [6]. In addition, we reported that increasing the number of austenite nucleation sites increases the fraction of austenite during intercritical annealing [7]. Accordingly, increasing the number of recrystallized ferrite grain boundaries and non-recrystallized ferrite grains also increases the fraction of austenite during intercritical annealing. In specimens P and B, the Nb addition increased the numbers of recrystallized ferrite grain boundaries and non-recrystallized ferrite grains (Figure 3(a) and (b)), but in specimen M, it increased only the recrystallized ferrite grain boundaries. The enhancement of recrystallized ferrite grain boundaries and non-recrystallized ferrite grains after Nb addition probably increased the fraction of austenite during intercritical annealing in all specimens. In particular, the austenite fraction was probably largest in specimen P, because the fraction of non-recrystallized ferrite grains (i.e., austenite nucleation sites) was largest in the specimen.

**Figure 3.** Microstructures of the Nb bearing specimens (a) P, (b) B and (c) M heated to 700 °C ($\alpha_R$: recrystallized ferrite, $\alpha_{NR}$: non-recrystallized ferrite, $\alpha'$: martensite).
Figure 4. Fraction of martensite in the specimens heated to 700 °C (Data of Nb-less specimen were taken from a previous report [6]).

Figure 5 illustrates the effects of Nb addition and initial microstructures on the interaction between ferrite recrystallization and the ferrite-to-austenite phase transformation behaviors during heating of each specimen. The Nb and initial microstructures strongly influenced the ferrite recrystallization; in turn, the ferrite recrystallization behavior strongly affected the austenite formation behavior during intercritical annealing.

| Specimen P | Specimen B | Specimen M |
|------------|------------|------------|
| Nb-less | Nb-less | Nb-less |
| immediately below $A_{C1}$ temperature | | |
| Nb bearing | Nb bearing | Nb bearing |
| austenite nucleation | austenite nucleation | austenite nucleation |
| | | |

Figure 5. Schematics showing the microstructural evolution during heating of each specimen ($\alpha_{NR}$: non-recrystallized ferrite, $\alpha_R$: recrystallized ferrite, $\theta$: spheroidal cementite, $\gamma$: austenite, SG: subgrain).
4. Conclusions
We investigated the effect of initial microstructures prior to cold-rolling and intercritical annealing on ferrite recrystallization and the ferrite-to-austenite phase transformation in Nb bearing low-carbon steels. The time of ferrite recrystallization during annealing decreased in the order of \( M > P > B \) in the Nb-less specimens, and in the order of \( M > B > P \) in the Nb bearing specimens. The Nb addition retarded the ferrite recrystallization during annealing and accelerated the ferrite-to-austenite phase transformation. This trend, which occurred in all specimens, was attributed to the increase in the number of recrystallized ferrite grain boundaries and non-recrystallized ferrite grains after the Nb addition, and the consequent increase in the austenite fraction during intercritical annealing.

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