Conditions for Grain Boundary Bulging during Tempering of Lath Martensite in Ultra-low Carbon Steel

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1. Introduction

Lath martensite in steel contains a high density of dislocations around 10 15/m2. This dislocation density corresponds to that in 80% cold-rolled ferrite.1) Due to the large stored energy by dislocations, as-quenched martensitic steel undergoes recrystallization during tempering even without pre-working when the carbon content is reduced to an ultra-low level.2,3) In addition, it was demonstrated that the recrystallization in lath martensite occurs through bulging of the pre-existing grain boundaries (bulge nucleation 4,5 and growth mechanism 6). Considering that lath martensite consists of fine substructures, namely, packets, blocks and laths, a very fine-grained recrystallization structure would be formed just by quenching and tempering if all the grain boundaries could cause bulging. However, the grain boundary bulging during tempering does not frequently occur in spite of the high density of grain boundaries in martensitic structure, resulting in a formation of coarse grained structure after the completion of recrystallization.4,5) This fact suggests that there is some necessary condition for grain boundary to migrate and cause bulging in lath martensite and that only the limited boundaries satisfy the condition could become a preferential nucleation site for recrystallized grains. In order to understand the unique recrystallization behavior of lath martensite, various unknown factors such as the grain boundary character of bulging boundaries, the substructural change which triggers grain boundary migration, the growing behavior of recrystallized grains etc. must be clarified. In this study, EBSD analysis was performed to examine the characteristics of bulging boundaries and matrix structure adjacent to the boundary. The conditions for grain boundary bulging was then discussed in terms of the mobility of grain boundary and the driving force for grain boundary migration.

2. Experimental Procedure

An ultra-low carbon steel with a chemical composition of Fe–1.55Mn–0.002Si–0.01P–0.015S–0.009C–0.0029N–0.0015B (mass%) was used in this study. The addition of manganese (Mn) and boron (B) aimed at obtaining fully lath martensitic structure by enhancing the quench hardenability. The ingot produced by induction melting in an Ar gas atmosphere was homogenized at 1473 K for 18 ks and then hot-rolled to 50% reduction in thickness at 1273 K. The steel plates were subjected to solution treatment in the austenite single phase region up to 1473 K. The specimens with lath martensitic structure were then annealed at 823 to 973 K for 0.18 to 172.8 ks. The microstructure was observed with an optical microscope (OM). The distribution of the crystallographic orientation in the microstructure was examined by the electron back scattering diffraction (EBSD) method with a field-emission scanning electron microscope (Hitachi S-4300). Specimens for EBSD measurements were prepared by using electrical polishing method in a solution of 10% perchloric acid and 90% acetic acid. The data obtained by the EBSD method was analyzed with the Tex SEM Laboratories, Inc. orientation imaging microscopy system. Choi et al. 7) reported that the image quality (IQ) obtained by EBSD is related to the number of lattice defect such as dislocation, and thus, it is reasonable that the IQ can be used as a measure of stored energy in deformed microstructure. In accordance with his report, the IQ map was used to evaluate the dislocation density distribution in this study.

3. Results

3.1. Nucleation of Recrystallized Grains through Grain Boundary Bulging in Tempered Ultra-low Carbon Lath Martensite

The low-magnified optical micrograph (a) and its tracing figure (b) of a partially recrystallized specimen are represented in Fig. 1 to reveal the distribution of recrystallized grains. Austenitization was performed at 1473 K for several hours to coarsen the grains sufficiently so as to make the nucleation sites clear. The gray region and the white region in the tracing figure correspond to tempered lath martensite and recrystallized grains, respectively. The broken lines indicate prior austenite grain boundaries. It is found that almost all the recrystallized grains are formed along the prior austenite grain boundaries although there are many other high angle boundaries such as packet boundaries and block boundaries. In addition, it should be noted that the formation of recrystallized grains is localized at limited prior austenite grain boundaries: none of the recrystallized grain exists at the arrowed grain boundaries. This indicates that some condition must be satisfied for grain boundary to cause bulging, as discussed later. Due to the limitation of bulging grain boundaries, the recrystallization of lath martensite results in a formation of coarse-grained ferritic structure although the initial densities of dislocations and grain boundaries are very high.4,5) The nucleation behavior of a recrystallized grain is focused in Fig. 2. This represents an optical micrograph of a recrystallized grain (a) and three kinds of EBSD maps obtained from the area surrounded by the white frame in the optical micrograph: orientation imaging map (b), grain boundary map (c) and image quality (IQ) map (d). The red lines in (c) denote high angle grain boundaries with misori-
entation angles between 15° and 45°, while the blue lines show the other high angle boundaries with higher misorientation angles (>45°). This classification of grain boundaries is based on the difference in mobility mentioned later. The IQ value roughly corresponds to dislocation density, and thus, the color indicated in (d) gradationally changes from red to blue with decreasing dislocation density. From the orientation imaging map (b), it is found that the recrystallized grain formed at the high angle grain boundary grows to the lower right region and that the martensite block structure has been removed in the region swept by the boundary. In addition, the orientation of the recrystallized grain is almost identical to that of the adjacent tempered martensite in the upper region, which demonstrates this ultra-low carbon lath martensite has recrystallized through bulging of the pre-existing grain boundary. It is already known that the dislocations in lath martensite are character-
ized by the uniform distribution, and there is no inhomogeneous dislocation substructure generating a local lattice rotation within grain. This should be the reason why the recrystallization takes place through the bulging of pre-existing grain boundaries in lath martensite.

3.2. Necessary Conditions for Grain Boundary to Cause Bulging

In spite of the high density of grain boundaries in lath martensite, only the limited prior austenite grain boundaries cause bulging and form recrystallized grains as mentioned above. This fact indicates that there is some condition to be satisfied for the grain boundary migration. From the Figs. 2(c) and 2(d), two important features are noticed:

1. The bulging grain boundary has a misorientation angle between 15° and 45°, and the block boundaries do not migrate.

2. The bulging grain boundary moves from the lowly-dislocated grain (green-colored region) toward the highly-dislocated one containing some block boundaries as well (yellow-colored region). These two features suggest that both mobility of grain boundary and driving force for grain boundary migration must be considered for the conditions in grain boundary bulging.

As for the mobility of grain boundary, there are several previous studies discussing the effect of grain boundary character, and they concluded that high angle boundaries have a larger mobility than low angle boundaries but that the mobilities and activation energies for migration of high angle boundaries are dependent on the misorientation angle. For example, Titrov showed experimental data on texture formation which demonstrates that the boundaries with misorientation angle in the range of 15–45° migrated faster than other boundaries in various FCC and BCC metals including Fe–3%Si alloy. In the case of the example in Fig. 2(c), the bulging boundary was a high angle boundary with misorientation angle of approximately 26° which corresponds to the high mobility boundary. Assuming that such high mobility boundaries with misorientation angles between 15° and 45° can migrate preferentially during tempering, the distribution of them will be related with the distribution of nucleation sites of recrystallized grains. Figure 3 represents the fraction of grain boundary as a function of misorientation angle (>) (a) and grain boundary map showing the distribution of high mobility boundary with misorientation angle between 15° and 45° by the red lines (b) in the as-quenched martensite. Misorientation peaks are seen in (a) at 7°, 33°, 55° and 62°. The boundaries with misorientation angle of 33° are high mobility boundaries. On the basis of Kurdjumov–Sachs orientation relationship, the 7° boundaries could be regarded as sub-block boundaries, and the 55° and 62° boundaries correspond to block boundaries, although these peaks somewhat deviate from the exact K–S relationship. There are large amounts of high angle boundaries in the lath martensitic structure; however, the fraction of high mobility boundaries with misorientation angle of 15 to 45° is only 14% of all high angle boundaries. It also will be understood from (b) that the high mobility boundaries are almost limited to a portion of prior austenite grain boundaries. Although some high mobility boundaries exist also within prior austenite grains, they tend to be shortly-segmented and their area fraction is very small.

On the other hand, driving force for grain boundary mi-
Migration is known to arise from a difference in dislocation density on opposite sides of the grain boundary.\textsuperscript{9,12} From this point of view, lath martensite in as-quenched state unlikely to have a sufficient driving force for recrystallization because of its uniform dislocation distribution. For onset of the recrystallization during tempering, it is required that the recovery of martensite proceeds nonuniformly to generate a difference in dislocation density between neighboring two grains. The image quality maps of Fig. 4 show that the dislocation density distribution exhibits a uniform-to-nonuniform transition during tempering. The IQ map of as-quenched lath martensite (a) is colored uniformly with orange. As the recovery proceeds, however, the color (dislocation density) is changed differently grain by grain, and then finally, the nonuniformly recovered structure (c) is appeared. The nonuniform recovery behavior can be more quantitatively expressed by plotting the number fraction of IQ values as shown in (d). The IQ values of as-quenched martensite are within a narrow range from 20 to 60. As the recovery proceeds, the IQ value tends to be increased owing to the decrease in dislocation density, and besides, the peak width is gradually broadened. The broadening means the annihilation of dislocations has occurred nonuniformly in lath martensite and resulted in the wide scattering of dislocation density. Here, the grain boundaries pointed by the arrows in (c) should be noted. They lie between a highly-dislocated grain (orange or yellow) and a lowly-dislocated one (green). At such grain boundaries, the driving force for grain boundary migration should be increasing, and thus, the second required condition for the recrystallization by bulge nucleation and growth mechanism will be satisfied. The grain boundary satisfying both of the conditions could become a preferential nucleation site like the example shown in Fig. 2; however, the limitation of high mobility boundary and the necessity of nonuniform recovery should lead to the low frequency of recrystallized grain nucleation and the coarse-grained recrystallization structure formation in lath martensite.

5. Conclusions

Ultra-low carbon lath martensite undergoes recrystallization during tempering by the bulge nucleation and growth mechanism. However, the frequency of nucleation of recrystallized grains is significantly low, and thus, a coarse-grained recrystallization structure is formed after the completion of recrystallization. The low frequency of nucleation is related to the two necessary conditions for grain boundary to cause bulging, that is, sufficiently large mobility of grain boundary and driving force for grain boundary migration. The high mobility boundaries are almost limited to a portion of prior austenite grain boundaries, while nonuniform recovery during tempering is required to obtain the driving force for grain boundary migration. The limitation of such boundary satisfying these conditions leads to the low frequency of nucleation of recrystallized grains in lath martensite.

REFERENCES