EFFECT OF NB ON THE RECRYSTALLIZATION OF AUSTENITE CONTAINING HIGH DISLOCATION DENSITY

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Abstract

An 18%Ni martensitic steel can revert to austenite by the mode of martensitic shear lattice displacement by rapid heating above the reversion finishing temperature. The reversed austenite is characterized by a high density of dislocations and this highly dislocated austenite causes recrystallization during holding above the reversion temperature. The addition of Nb in solid solution affects the recrystallization of reversed austenite, but it is not so significant when the Nb content in steels is small. It was found that the recrystallization of reversed austenite is retarded in proportion to the amount Nb in solid solution. This is probably attributed to the grain boundary dragging effect by Nb segregated on the grain boundary. On the other hand, the existence of Nb(C,N) precipitates gives a marked retardation effect to the recrystallization of reversed austenite. As a result, it is suggested that the control of Nb(C,N) precipitation is most important to suppress the recrystallization of highly dislocated austenite when the Nb content in steels is low.

Introduction

Austenite grain size is one of important factors in terms of microstructure control because it gives a great influence to the resultant phase transformation on cooling. On the grain refinement of austenite, Nb is recognized as an excellent alloying element due to the grain boundary dragging effect by itself and secondly, the effective grain boundary and dislocation pinning by finely dispersed precipitates such as Nb(C,N). It is obvious that the Nb addition is very useful for the refinement of the microstructure in commercial plain carbon steels [1], but it is difficult to evaluate the contribution of Nb on the austenite grain refinement in plain carbon steels. The reason is that the austenitic structure has disappeared at high temperatures due to its transformation upon cooling.

On the other hand, author et al. has reported for an 18%Ni martensitic steel that it reverts to austenite by the mode of martensitic shear lattice displacement by rapid heating above the reversion finishing temperature [2]. The reversed austenite is characterized by a high density of dislocations and the highly dislocated austenite causes recrystallization after the reversion during holding above reversion temperature [3]. Such a behavior is very convenient for investigating the recrystallization of highly dislocated austenite because a high density of dislocations can be introduced into austenite without deformation and the distribution of dislocations is homogeneous throughout the material. In this paper, the effect of Nb addition on the recrystallization of highly dislocated austenite was investigated in terms of the effect of Nb itself in solid solution and precipitates of Nb(C,N) using the 18%Ni martensitic steel.

Experimental Procedure

Materials and heat treatment

Electrical iron, electrical nickel, and the other alloying elements were melted using a vacuum induction furnace and then cast in a metallic mold of 28 mm x 50 mm x 100 mm. The ingots of 1.5 kg each were homogenized at 1473°K for 36 ks and then hot-rolled to 10 mm thick plates. Table I lists the chemical composition of the steels obtained. The steel A is base 18%Ni steel. The steel B was prepared for investigating the effect of N in solid solution. The steel C, D and E are for investigating the effect of Nb itself in solid solution and the steel F is for the investigation of NbN precipitates. Martensitic transformation starting temperature (Ms) and reversion starting temperature (Rs) is controlled to be around 550°K and 830°K respectively for all of the steels because they affect the dislocation density of reversed austenite. After the solution treatment of 1523°K - 3.6 ks followed by water quenching, specimens cut from the steel plates were subjected to reversion treatment for 1.8 ks at the temperature between 873°K and 1273°K followed by water quenching. The specimens with reversion treatment were provided to the microstructure observation by optical microscopy (OM) and transmission electron microscopy (TEM).

Table I. Chemical composition (mass %) of the steels used. Ms and Mf temperature of the base steel are 553°K and 453°K. respectively.

Steel	Ni	Nb	Ν	Cr	Mn	
Base steel (A)	18.08	-	0.0015	0.22	0.36	
0.03N-steel (B)	17.03	-	0.0307	0.22	1.14	
0.1Nb-steel (C)	17.92	0.096	0.0036	0.22	0.36	
0.2Nb-steel (D)	17.01	0.19	0.0029	0.22	1.11	
0.3Nb-steel (E)	17.08	0.29	0.0024	0.22	1.12	
0.1Nb-0.01N-steel (F)	17.00	0.1	0.01	0.22	1.00	

* Si<0.01, P<0.01, S<0.01, C:0.0012-0.0016

Phase transformation kinetics of Fe-18%Ni alloy

Figure 1 shows the phase diagram of Fe-Ni alloy system. Ni is one of the strong austenite stabilizing elements so that To line lowers with increasing Ni content in Fe-Ni alloy. Besides, it is well known that the diffusivity of substitutional atoms such as Ni is very low in comparison with that of interstitial atoms such as C and N. Hence, high Ni alloy cannot undergo diffusive phase transformation from fcc to bcc on cooling and this results in martensitic transformation in high Ni steels. Ms temperature of Fe-18%Ni alloy (base steel) was 553°K. On the other hand, the base steel with martensitic structure causes martensitic shear reversion from bcc to fcc on heating because the To temperature of this steel (about 740°K) is not high enough for diffusive phase transformation. Martensitic reversion starting temperature (Rs) was 833°K for the base steel. As a result, the base steel undergoes twice of martensitic transformation on heating and cooling. Optical microstructures of the base steel are in Figure 2 for the same area before (a) and after the

heat treatment of rapid heating to 1073°K and the following water quenching (b). It should be noted that the same austenite grain boundary is kept after the heat treatment. This reveals the fact that no diffusive phase transformation occurs both on heating to 1073°K and on cooling from the temperature.



Figure 1. Phase diagram of Fe-Ni alloy system. Rs temperature of Fe-18%Ni alloy is 833K.



Figure 2. Optical microstructure for the same area of Fe-18%Ni alloy (base steel). Before (a) and after the heat treatment of rapid heating to 1073K and the following water quenching (b).

Recrystallization behavior of martensitically reversed austenite

Direct observation of the reversed austenite is difficult due to the martensitic transformation on cooling but the existence of highly dislocated reversed austenite can be proved by recrystallization of the reversed austenite. Figure 3 shows the microstructural change of lath martensite as a function of holding time at 1073° K. Unrecrystallized austenite and recrystallized austenite are distinguished from the morphology of lath martensite: Fine block structure is formed from unrecrystallized austenite (see Figure 2 (b)), while wide block structure is formed from recrystallized austenite as shown in Figure 3 (a). After the completion of recrystallization, the prior austenite grain size is refined to 100 µm or less and the whole surface is covered by the wide block structure as shown in the Figure 3 (b). The fraction of recrystallized austenite was measured by several OM observations.



Figure 3. Optical microstructures of the base steel. Water quenched after holding at 1073K for 0.6ks (a) and 36ks (b).

Experimental Results

Effect of nitrogen in solid solution on the recrystallization of highly dislocated austenite

Recrystallization behaviors of reversed austenite are shown for the base steel and the 0.03N-steel in Figure 4 as a function of holding temperature. In the case of 1.8 ks holding, recrystallization of reversed austenite easily occurs above 1000°K in both steels. No significant influence by nitrogen addition can be found on the recrystallization behavior of reversed austenite. Figure 5 is the calculated phase diagram for Fe-17%Ni-1%Mn-0.2%Cr-N alloy system. It is found that austenite single-phase region can be obtained above 1000°K independent of nitrogen content. The precipitation of CrN may affect the recrystallization behavior of reversed austenite below 1000 K, but we do not have to think about the effect in this case because the reversion finishing temperature of the 0.003N-steel is just below 1000°K.



Figure 4. Change in the fraction of recrystallized austenite as a function of holding temperature. Specimens were water quenched after holding at given temperature for 1.8ks.



Figure 5. Phase diagram of Fe-17%Ni-1%Mn-0.2%Cr-N alloy system.

Effect of niobium in solid solution on the recrystallization of highly dislocated austenite

Figure 6 shows the change in the fraction of recrystallized austenite as a function of holding temperature. Recrystallization of reversed austenite is surely retarded with increasing Nb content. This suggests the grain boundary dragging effect by Nb on the recrystallization of highly dislocated austenite. However, the steels used contain a small amount of carbon and nitrogen which produce Nb(C,N) so that we have to think about the effect of such a precipitate on the recrystallization behavior. The volume fraction of Nb(C,N) can be calculated from the content of C and N under the condition that the whole C and N combine with Nb. The Nb content subtracted the value for Nb(C,N) from the total Nb content gives the Nb content in solid solution.



Figure 6. Change in the fraction of recrystallized austenite as a function of holding temperature. Specimens were water quenched after holding at given temperature for 1.8ks.

Figure 7 shows the results of estimated volume fraction of Nb(C,N) and Nb content in solid solution. The volume fraction of Nb(C,N) was estimated at about 0.04% independent of the Nb content in steels and it was confirmed that the Nb content in solid solution surely increases with an increase of Nb content in steels. This result indicates that the retardation of recrystallization shown in Figure 6 is attributed to the grain boundary dragging effect by Nb in solid solution.



Figure 7. Volume fraction of Nb(C,N) and Nb content in solid solution in the steels with different Nb content.

Figure 8 shows the relation between the Nb content in solid solution and recrystallization temperature. The volume fraction of Nb(C,N) is as small as 0.04% but this figure suggests that the effect of such a small amount of precipitates can not be ignored in terms of the recrystallization of highly dislocated austenite.



Figure 8. Relation between the Nb content in solid solution and recrystallization temperature in the case of 1.8ks holding.

In order to make the effect of Nb(C,N) clearer, the 0.1Nb-0.01N-steel containing about 0.1vol% of Nb(C,N) was examined. Figure 9 shows the effect of Nb(C,N) precipitates on the recrystallization of reversed austenite. Small increment (about 0.06%) in the volume fraction of Nb(C,N) results in a marked retardation of highly dislocated austenite. Figure 10 displays the relation between the volume fraction of Nb(C,N) and the recrystallization temperature. The retardation of recrystallization by Nb(C,N) precipitates is evaluated at 2500°K/% in the case of 1.8 ks holding. This means that Nb(C,N) precipitates works effectively for the retardation of the recrystallization of highly dislocated austenite through the effect of grain boundary pinning and dislocation pinning. Figure 11 is the phase diagram of Fe-17%Ni-1%Mn-0.2%Cr-0.01N-Nb alloy system. For the 0.1Nb-0.01N-steel, it is expected that Nb(C,N) precipitates start decomposition from 1200°K and they completely disappear above 1500°K. The decomposition starting temperature for Nb(C,N) precipitates is just correspondent to the recrystallization temperature of reversed austenite. As mentioned above, the volume fraction of Nb(C,N) gives a large effect on the recrystallization of highly dislocated austenite. The recrystallization behavior is very sensitive to a small change in the volume fraction of Nb(C,N) precipitates. This is probably the reason why the recrystallization temperature of the 0.1Nb-0.01N-steel is just corresponding to the decomposition starting temperature for Nb(C,N) precipitate.



Figure 9. Change in the fraction of recrystallized austenite as a function of holding temperature. Specimens were water quenched after holding at given temperature for 1.8ks.



Figure 10. Relation between the Nb content in solid solution and recrystallization temperature in the case of 1.8ks holding.



Figure 11. Phase diagram of Fe-17%Ni-1%Mn-0.2%Cr-0.01N-Nb alloy system.

Conclusion

The effect of Nb additions on the recrystallization behavior of highly dislocated austenite was investigated using an 18%Ni martensitic steel which can revert to austenite by the mode of martensitic shear lattice displacement on heating and exhibits an austenitic structure containing a high density of dislocations. The results obtained are as follows:

1. The addition of Nb in solid solution affects the recrystallization of reversed austenite, but it has little effect when the Nb content in steels is small. It was found that the recrystallization of reversed austenite is retarded in proportion to the amount Nb in solid solution. This is probably attributed to the grain boundary dragging effect by Nb segregated on grain boundary. On the other hand, the existence of Nb(C,N) precipitates gives a marked retardation effect to the recrystallization of reversed austenite. As a result, it was suggested that the control of Nb(C,N)

precipitation is most important to suppress the recrystallization of highly dislocated austenite when Nb content in steels is small.

References

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