Effect of Initial Microstructure on Superplasticity in Ultrafine Grained 18Cr-9Ni Stainless Steel

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Ultrafine grained austenitic structure was obtained in 18Cr-9Ni stainless steel by thermomechanical treatment using reversion from deformation-induced martensite. The superplastic deformation behavior was investigated at 923 K for the steels containing various amounts of retained martensite particles in the initial structure before tensile testing. The retained martensite was effective for suppressing grain growth of austenite and necessary for the superplasticity although it was thermodynamically unstable phase and gradually decreased its volume fraction with superplastic deformation. Therefore, the superplastic elongation was strongly dependent on the initial volume fraction of the retained martensite. The total superplastic elongation was enlarged with increasing the initial amount of martensite, and the maximum elongation of about 270% was obtained when the volume fraction was controlled to be around 10 vol%. However, the increase in elongation was leveled off in the range above 15 vol% martensite. The effect of the retained martensite on the superplasticity was discussed in connection with the changes in volume fraction of the martensite, austenite grain size and deformation mechanism.

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1. Introduction

Metastable austenitic steels, such as 16Cr-10Ni or 14Cr-12Ni¹⁾ steels, form ultrafine grained structure through reversion from deformation-induced martensite generated by heavy cold working.^{2,3)} The ultrafine grained austenitic steels have not only high yield strength around 700 MPa at ambient temperature⁴⁾ but also excellent deformability at elevated temperature due to superplastic deformation.^{5,6)} In addition, the superplasticity of these steels occurs at much lower temperature compared with other stainless steels. It is well known that austenite-ferrite dual phase stainless steels exhibit superplasticity, but the temperature has to be elevated above 1050 K for obtaining elongation of more than 200%.⁷⁾ On the other hand, the superplasticity with total elongation of 430% was obtained at 923 K in the ultrafine grained austenitic stainless steel with a common chemical composition of Fe-18Cr-9Ni.⁶⁾ Besides, Katoh and Torisaka obtained more than 500% elongation at 973 K in a similar steel which was produced by using reversion from deformation-induced martensite formed through multi-direction upsetting.⁵⁾

According to our previous study,⁶⁾ the microstructure at deformation temperature should not be austenitic single structure to obtain large superplastic elongation in the ultrafine grained steel, because the austenite grains abruptly grow during the superplastic deformation, and this prevents further superplastic deformation.⁶⁾ Therefore, we used a small amount of retained martensite⁷⁾ which is left in the ultrafine grained austenitic structure even at elevated temperature. The martensite particles were very effective for suppressing grain growth of austenite by grain boundary pinning effect. However, the volume fraction of retained martensite gradually reduces with passing time and the microstructure finally approaches to full austenitic structure because the martensite

is thermodynamically unstable phase at the elevated temperature. From this reason, the superplastic deformation behavior would be more significantly dependent on the holding time at the deformation temperature in this ultrafine grained austenitic steel than in conventional superplastic materials containing thermally stable second phases. Therefore, it should be very important to evaluate the initial microstructure at first and then relate it with the following microstructural change during deformation.

In this study, Fe-18Cr-9Ni alloys with various initial microstructures were tensile tested at 923 K, and then the effect of the initial amount of retained martensite on superplastic elongation was investigated in connection with microstructural development during the tensile deformation.

2. Material and Experimental Procedure

The following conditions must be satisfied for obtaining the metastable austenitic steel with ultrafine grained structure:^{2,3)}

- (1) Deformation-induced martensitic single structure is formed by cold rolling at room temperature.
- (2) Austenitic single structure is formed after reversion of the deformation-induced martensite at around 900 K.
- (3) The reversed austenite does not undergo martensitic transformation on cooling.

One of the authors clarified the each condition in Fe-Cr-Ni ternary alloys and reported the structure diagram at 873 K as shown in Fig. 1.^{2,3)} The area enclosed by the three lines gives the chemical composition satisfying the three conditions. However, the 18Cr-9Ni steel being out of the area (shown by 'x') was selected as a specimen because the purpose of this study is to use a small amount of untransformed martensite which is retained after reversion for suppressing grain growth of the ultrafine grained austenite during superplastic deformation. The chemical composition of the steel used in this study was shown in Table 1.

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Fig. 1 Diagram showing microstructure obtained by reversion treatment of deformation-induced martensite in Fe-Cr-Ni ternary system metastable stainless steels. The γ_R , γ_C , α'_T and α'_F denote austenite reversed from martensite, cold-worked austenite, tempered martensite (retained martensite) and fresh martensite formed from the reversed austenite, respectively.^{2,3)}

Fig. 3 Change in volume fraction of deformation-induced martensite with increasing reduction by cold rolling after solution treatment.

Table 1 Chemical composition of 18Cr-9Ni steel used in this study. (mass%)

Cr	Ni	Si	Mn	P	S	C	N	Fe
17.85	8.81	0.06	0.26	0.008	0.010	0.005	0.019	bal.

Fig. 2 Thermomechanical treatment for obtaining ultrafine grained austenitic stainless steel.

An ingot produced by casting in an argon gas atmosphere was hot-rolled to break the dendritic structure, homogenized at 1523 K for 18 ks, and then subjected to the thermomechanical treatment shown in Fig. 2. The specimen was solution-treated at 1373 K for 1.8 ks, followed by air cooling, and then cold-rolled up to 90% maximum in reduction of area. The cold-rolled materials with deformation-induced martensitic structure were annealed at 923 K for various times (reversion treatment), followed by water cooling. Tensile tests were carried out at 923 K and an initial strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ for platelet test pieces with 0.5 mm thickness, 5 mm width and 10 mm gauge length. Microstructure was observed with a 200 kV transmission electron microscope (TEM). Volume fraction of martensite was estimated from the ratio of saturation magnetizations of the specimen and of full ferritic (martensitic) material.⁹⁾

3. Results and Discussion

3.1 Deformation-induced martensitic transformation and reversion in 18Cr-9Ni steel

Figure 3 shows the change in volume fraction of deformation-induced martensite as a function of the reduction by cold rolling after solution treatment. Although almost

full martensitic structure is formed by 50% reduction, the 90% cold-rolled material was used in this study because the further straining after completing martensitic transformation is spent on destruction of lath structure and formation of fine dislocation cell structure, which contributes to the ultra grain refinement of reversed austenite.

There are two types of mechanism for reversion from deformation-induced martensite to austenite in metastable austenitic steel.¹⁰⁾ One is 'diffusional reversion', in which the austenite grains nucleate and grow with diffusion of Cr and Ni atoms as well as Fe atoms. The other is 'martensitic shear reversion', in which the atomic arrangement is altered by homogeneously deforming the original bcc pattern into the new fcc structure without the diffusion of atoms. The reversion of 18Cr-9Ni steel was reported to proceed with the former mechanism.¹⁰⁾ In general, it takes longer to complete the austenitization in the diffusional reversion than in the martensitic shear reversion because of the necessity of diffusion. Figure 4 shows the change in volume fraction of martensite as a function of time of reversion treatment at 923 K in the 90% cold-rolled material. The amount of martensite gradually decreases with the treatment time through the reversion of martensite to austenite. The slow reversion rate demonstrates that it proceeds with the diffusional mechanism. It should be noted here that more than 5 vol% of martensite is retained even after 10 ks although the amount of martensite finally becomes very small.

3.2 Effect of initial amount of retained martensite on tensile deformation behavior at elevated temperature

In general, a great volume of second phase being stable even at high temperature is often used for suppressing grain growth in superplastic metallic materials such as dual phase stainless steel^{8,11)} and Ti-Al-V system alloy¹²⁾ because it is important to keep grain size fine during the deformation at the elevated temperature for obtaining large superplastic elongation. In that point, the thermally unstable martensite may be inadequate to maintain the ultrafine grained structure for a long time. However, in the case of a short time forming at a

Fig. 4 Change in volume fraction of martensite as a function of time of reversion treatment at 923 K after cold rolling by 90%.

high strain rate, the martensite would be usable for keeping grain size fine because of the slow reversion rate shown in the result of Fig. 4. To demonstrate the effectiveness of the martensite, the volume fraction of retained martensite was varied by reversion treatment time, and then the effect of the initial volume fraction on the deformation behavior was investigated by means of tensile testing at 923 K. Figure 5 represents TEM images showing microstructure of 18Cr-9Ni steels which were reversion-treated at 923 K for 0.6 ks (a)(b) or for 5.4 ks (c)(d). The dark field images (b) and (d) show the dispersion of retained martensite particles. Both specimens exhibit fine grained austenitic structure containing retained

Fig. 5 TEM images of initial microstructure observed before tensile testing. The reversion treatment after cold rolling was performed at 923 K for 0.6 ks (a)(b) or 5.4 ks (c)(d). The dark field images of (b) and (d) show the dispersion of retained martensite particles.

Fig. 6 Stress-strain curves of the 0.6 ks and 5.4 ks reversion-treated materials. The tensile tests were carried out at 923 K and an initial strain of $1.0 \times 10^{-3} \text{ s}^{-1}$.

martensite. The austenite grain sizes are small enough to cause grain boundary sliding which leads to superplasticity. The volume fraction of martensite is 12 vol% in the 0.6 ks material and 6 vol% in the 5.4 ks material. The particles of martensite mainly exist at triple junctions of austenite grain boundaries as pointed by the arrows in the picture. There is no significant difference in the size and morphology of the martensite particles between these specimens. Figure 6 shows the stress-strain curves obtained by the tensile tests at 923 K and an initial strain rate of 1.0×10^{-3} in the two kinds of specimens with the different initial microstructures which were. The stress-strain curves are characterized by the gradual softening after yielding and the large elongation more than 100% in both specimens, suggesting that they deformed superplastically. However, the values of elongation and maximum stress are obviously different between these materials. The elongation is larger by 85% and the maximum stress is lower by 26 MPa in the 0.6 ks material (12 vol% initial martensite) than in the 5.4 ks material (6 vol% initial martensite). This result indicates that the retained martensite plays an important role in the superplasticity, and the elongation depends on the initial amount of the martensite. Tensile tests were carried out also for other specimens with various initial amounts of martensite, and then the relation between the initial amount of martensite and the total elongation was obtained as shown in Fig. 7. The total elongation is increased with increasing the initial volume fraction of martensite, and the maximum elongation of 270% was obtained when the volume fraction is controlled to be

Fig. 7 Relation between initial volume fraction of retained martensite and total elongation obtained by tensile tests.

about 10 vol%. However, the increase in elongation is leveled off in the range above 15 vol% martensite, although it is generally accepted that the maximum elongation in super-plastic deformation is obtained when the volume fraction of the second phase is around 50%.^{11,12} The ineffectiveness of the increment of second phase is due to the thermal instability of the second phase martensite. As expected from the result of Fig. 4, the initial amount of martensite would rapidly decrease down to about 10 vol% in the early stage of tensile testing. Thus, the amount of the actually effective martensite should be less than 10 vol% even when the initial amount is much larger. Figure 8 shows change in the volume fraction of martensite as a function of the strain during tensile testing (solid lines) in the 0.6 ks material (12 vol% initial martensite) and the 5.4 ks material (6 vol% initial martensite). The broken lines also present the results from the unstrained region of tensile test pieces (shoulder section). It is confirmed that the reversion is promoted by straining and the decrease in volume fraction of martensite is more rapid in the strained gauge section than in the unstrained shoulder section. However, the amount of retained martensite is kept higher during testing by increasing the initial amount of martensite, and more than 5 vol% martensite is still retained even after 50% deformation in the 0.6 ks material. On the other hand, the amount of martensite is reduced to less than 3 vol% after the 50% deformation in the 5.4 ks material.

The change in the amount of retained martensite should be

closely related with the behavior of grain growth of austenite during tensile testing. Figure 9 represents the microstructure of the 0.6 ks material and the 5.4 ks material deformed by 20%, 40% and 60%. The measured volume fraction of martensite is also shown in each picture. It is clearly found

Fig. 8 Change in volume fraction of martensite during tensile testing at 923 K as a function of strain in the 0.6 ks and 5.4 ks reversion-treated materials. The broken lines show the results obtained from unstrained parts of the test pieces.

Fig. 9 TEM images showing microstructural change during tensile testing at 923 K in the 0.6 ks and 5.4 ks reversion-treated materials.

Fig. 10 Schematic illustrations showing the effect of initial microstructure on the microstructural development during deformation.

that the grain growth is retarded in the 0.6 ks reversion-treated material. For example, the grain size in the 60% deformed 0.6 ks material is kept below 1 μm . The specimens having such ultrafine grained structure (grain size < 1 μm) contains few dislocations within grains. This suggests that the specimens have deformed mainly with the grain boundary sliding mechanism. On the other hand, the 60% deformed 5.4 ks material, whose grain size has been enlarged above 1 μm , exhibits dislocation cell structure, meaning that the specimen has deformed not with grain boundary sliding but with dislocation slips within grains. Figure 10 is schematic illustrations showing the microstructural development as a function of deformation time (or strain) in tensile testing. When the material is sufficiently reversion-treated for obtaining thermodynamically stable microstructure (a), the amount of retained martensite becomes small and the austenite grains are easily coarsened in the early stage of deformation owing to the lack of pinning effect. This causes the dislocation slips within grains, leading to a fracture in a short time without further superplastic elongation. On the other hand, when the material is shortly reversion-treated (b), the greater amount of martensite is available for the pinning although the microstructure is far from the equilibrium state. As a result, the ultrafine grained structure is kept for a longer time, which gives larger superplastic elongation before the fracture.

4. Conclusions

The superplastic deformation behavior was investigated by means of tensile testing at 923 K in the ultrafine grained metastable 18Cr-9Ni austenitic stainless steel which was produced through reversion from deformation-induced martensite. The results obtained are summarized as follows.

- (1) The martensite retained in the ultrafine grained structure after reversion treatment is effective for suppressing grain growth of austenite and increasing superplastic elongation obtained by tensile testing.
- (2) Since the retained martensite in the initial structure is thermodynamically unstable, the volume fraction of the

martensite is gradually decreased with superplastic deformation.

- (3) The total elongation is increased with increasing the initial volume fraction of retained martensite up to about 10 vol% because the ultrafine grained structure is kept for a longer time by larger amount of retained martensite during the deformation. However, the increase in elongation is leveled off even if the initial amount of martensite is increased over 15 vol%.

- (4) Once the amount of retained martensite becomes very small (less than 5 vol%), the marked grain growth is occurred owing to the lack of pinning effect. This causes the dislocation slips within grains, leading to a fracture in a short time without further superplastic elongation.

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