High-Strain Rate Superplasticity and Role of Dynamic Recrystallization in a Superplastic Duplex Stainless Steel*

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Superplasticity of a 25Cr-7Ni-3Mo ferrite/austenite duplex stainless steel has been studied with particular emphasis on the microstructural change during deformation, in order to clarify roles of dynamic recrystallization in the superplastic deformation. A microduplex structure consisting of fine ferritic subgrains and austenite particles (grain size: 1 μm) is obtained by annealing at 1273 K after 90% cold rolling of a fully ferrite structure. The steel with this fine-grained structure exhibits high-strain rate superplasticity at 1273 K in the ferrite/austenite two-phase region. A maximum elongation over 1700% is obtained at a strain rate of 1.7×10^{-2} /s. Even at a higher strain rate of 1.7×10^{-1} /s the elongation is near 1000%. Ferrite/ferrite boundaries are low-angle boundaries before the superplastic deformation. These low-angle boundaries are changed to high-angle ones by dynamic recrystallization of the ferrite matrix in the early stage of deformation. During the deformation after the recrystallization of the ferrite matrix, dislocations are scarcely observed in the whole of specimens. It is concluded that grain-boundary sliding is the dominant mode of the superplastic deformation, and that the role of the dynamic recrystallization is to make the fine structure suitable for the grain-boundary sliding in the early stage of deformation. A mechanism of the dynamic recrystallization is briefly discussed.

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I. Introduction

The fine-structure superplasticity, in which materials deform extensively at elevated temperatures under small stresses by grain-boundary sliding, has been reported in a number of metals and alloys with fine grain sizes below 10 μm . It has been well known⁽¹⁾⁽²⁾ that ferrite/austenite duplex stainless steels are typical ferrous alloys showing the superplasticity, when the steels have a microduplex structure consisting of fine ferrite and austenite grains. Ameyama *et al.*⁽³⁾ studied the formation process of the microduplex structure of duplex stainless steels during annealing in the ferrite/austenite two-phase region after 90% cold rolling of a fully ferritic structure. They reported that ferrite matrix of the microduplex structure is in a recovery condition, namely, ferrite grains are subgrains. This result brings us to the question why the duplex stainless steels exhibit the superplasticity, in spite of the fact that ferrite/ferrite boundaries of the microduplex structure are low-angle ones which do not slide readily.

Recently, Maehara⁽⁴⁾ has studied the microstructural

change during superplastic deformation in duplex stainless steels. He has concluded that in the ferrite/austenite duplex phase structure, the dynamic recrystallization of the ferrite matrix occurs locally and intermittently, and the superplasticity can be obtained by a subtle balance between the local strain hardening and the subsequent recrystallization. This conclusion emphasizes that the mechanism of superplasticity of duplex stainless steels is not the grain-boundary sliding which has been considered as the dominant deformation mode in a number of superplastic materials. However, the roles of the dynamic recrystallization have not been clarified.

From a technological viewpoint, much attention has been paid to high-strain rate superplasticity. Wadsworth *et al.*⁽⁵⁾⁽⁶⁾ have reported tensile elongations over 500% at unusually high strain rates between 10^{-1} /s and 10^2 /s in aluminum alloys with very fine grain sizes below 1 μm . In the duplex stainless steels, however, such high-strain rate superplasticity has not been reported. In the literature⁽⁷⁾⁽⁸⁾, the maximum strain rate for superplastic elongations over 500% is in the range between 1×10^{-3} /s and 1×10^{-2} /s.

The primary purpose of the present study is thus to clarify the mechanism of appearance of the superplasticity in the duplex stainless steel with the microduplex structure. Especially, the roles of dynamic recrystallization in the superplastic deformation are examined. At the same time, it is intended to investigate the appearance of high-strain rate superplasticity in the duplex stainless steel by obtaining a finer microduplex structure than the conventional one with a grain size of several microns.

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II. Experimental Procedure

The material used is a 25Cr–7Ni–3Mo ferrite/austenite duplex stainless steel (SUS 329J1). Chemical composition of the material is 0.029C, 0.32Si, 0.84Mn, 0.022P, 0.002S, 25.10Cr, 7.15Ni, 3.10Mo, 0.14N, the balance Fe (mass%). The heat treatment and tensile test conditions are shown in Fig. 1. Two kinds of microduplex structures with different austenite particle sizes were achieved by two different thermomechanical treatments shown in Fig. 1(a) and (b). In the first heat treatment (Fig. 1(a)), plates of 15 mm thickness were solution-treated in vacuum for 3.6 ks at 1573 K in the ferrite single phase region and water quenched. The plates with a fully ferritic structure were cold-rolled by 90% in reduction. The plates were subsequently aged in vacuum for 1.8 ks at 1273 K in the ferrite/austenite two-phase region followed by water quenching. Tensile specimens with 10 mm gage length and 6 mm gage section width were then cut along the cold-rolling direction from the sheets of 1.5 mm thickness. These specimens showed a microduplex structure in which austenite particles of 2.5–3 μm diameter precipitate in the ferrite matrix. These specimens are hereafter referred to as “aged specimens”. The other heat treatment (Fig. 1(b)) was performed to obtain finer austenite particles. The tensile specimens were cut from the 90% cold-rolled sheets of 1.5 mm thickness. These specimens are hereafter referred to as “rolled specimens”. Tensile tests of the rolled specimens were conducted after 60 s holding at 1273 K. As it will be shown later (Figs. 2 and 3), in the rolled specimens, austenite

Fig. 1 Schematic diagrams of the heat treatments and tensile tests for (a) aged specimens and (b) rolled specimens.

Fig. 2 Optical micrographs showing microduplex structures of (a) the rolled and (b) the aged specimen before tensile deformation; (a) the specimen was annealed at 1273 K for 60 s after cold rolling, (b) the specimen was annealed at 1273 K for 1.8 ks after cold rolling.

particles of 1 μm diameter uniformly precipitated in the ferrite matrix just before the tensile deformation.

The temperature for tensile deformations was 1273 K in the ferrite/austenite two-phase region. The specimens were heated with an infrared ray furnace. The heating rate up to 1273 K was 1 K/s. The tensile deformations were conducted in a 90%N₂+10%H₂ mixture gas atmosphere on an Instron type machine with initial strain rates from $1.7 \times 10^{-4}/\text{s}$ to $1.7 \times 10^0/\text{s}$. In order to investigate the microstructural change during deformation, the specimens were cooled with He gas immediately (within 0.3 s) after various amounts of strain. Specimens for optical metallography were electrolytically etched with a 10% aqueous solution of oxalic acid. Foils for transmission electron microscopy were thinned in a solution of 10% perchloric acid + 90% acetic acid using a jet polisher. The foils were examined with a JEOL200CX and a JEOL100CX. Grain sizes (D) were obtained from an equation of $D = 1.128L$ (L : mean linear intercept)⁽⁹⁾ by a linear intercept method.

III. Results

1. Microstructure before superplastic deformation

Optical microstructures of the rolled and the aged specimens before the tensile deformation at 1273 K are

Fig. 3 TEM micrograph of the rolled specimen just before tensile deformation. The specimen was annealed at 1273 K for 60 s. Selected-area diffraction patterns were taken from the ferrite subgrains marked a, b and c in the micrograph.

shown in Fig. 2. The rolled specimen (Fig. 2(a)) was held for 60 s at 1273 K after 90% cold-rolling. Both specimens show the so-called microduplex structure where fine austenite particles precipitate in the ferrite matrix. The austenite particles are smaller in the rolled specimen than in the aged specimen, because of the shorter aging time at 1273 K in the rolled specimen. It is worth to note that in both of the specimens the volume fraction of austenite was 0.4 and did not change in subsequent static-holding up to 38 ks at 1273 K. It is thus clear that the precipitation of austenite is completed before the tensile deformations in both specimens.

Figure 3 shows a typical transmission electron micrograph of the rolled specimen just before the tensile deformation. The grain sizes of ferrite and austenite are ap-

proximately $1\ \mu\text{m}$. Dislocations are scarcely observed in both phases of ferrite and austenite. Selected-area electron diffraction patterns in Fig. 3 were taken from the ferrite grains marked a, b and c. The three diffraction patterns are almost identical to one another. This clearly indicates that the ferrite grains of the microduplex structure are subgrains and the ferrite matrix is in a recovery condition, as Ameyama *et al.*⁽³⁾ have pointed out. The TEM microstructure of the aged specimen was essentially the same as that of the rolled specimen, except for the grain size. Ferrite and austenite grains of the aged specimen were $2.5\text{--}3.0\ \mu\text{m}$ in diameter. In the optical microstructures (Fig. 2) of both specimens, ferrite/ferrite boundaries are not seen. This is because the ferrite/ferrite boundaries are low-angle boundaries and

hardly etched.

2. Superplastic deformation behavior

Figure 4 shows the variation in elongation with initial strain rate in the rolled and the aged specimens deformed at 1273 K. In the aged specimens, large elongations over 500% are obtained at relatively low strain rates below $1 \times 10^{-2}/s$, whereas the elongations do not exceed 200% at the higher strain rates of $5 \times 10^{-2}/s$ and $1.7 \times 10^{-1}/s$. This variation in elongation with strain rate in the aged specimens is similar to that reported in duplex stainless steels with a grain size of several microns⁽²⁾⁽⁷⁾⁽⁸⁾. On the other hand, the rolled specimens exhibit higher ductilities especially at high strain rates compared with the aged specimens. A maximum elongation over 1700% is obtained at a strain rate of $1.7 \times 10^{-2}/s$. Even at a higher strain rate of $1.7 \times 10^{-1}/s$ the elongation is near 1000%. This result emphasizes that the high-strain rate superplasticity can be also achieved in the duplex stainless steel like aluminum alloys⁽⁵⁾⁽⁶⁾ by obtaining the fine-grained microduplex structure with a grain size of about $1 \mu m$ before the deformation.

Figure 5 shows the effect of initial strain rate on the true stress-true strain curves of the rolled and the aged specimens. At high strain rates of $1.7 \times 10^{-1}/s$ and

Fig. 4 Variation in elongation with initial strain rate in the rolled and the aged specimens deformed at 1273 K.

Fig. 5 Effect of initial strain rate on the true stress-true strain curves of the rolled and the aged specimens deformed at 1273 K.

Fig. 6 Variation in peak stress and m -value with initial strain rate of the rolled and the aged specimens deformed at 1273 K.

$1.7 \times 10^{-2}/s$, both specimens show the peak stress. The flow stress gradually decreases with increasing strain after the peak stress. At a lower strain rate of $1.7 \times 10^{-3}/s$ the peak stress is not clearly seen. At a constant initial strain rate, the flow stress is lower in the rolled specimens (fine-grained structure) than in the aged specimens (coarse-grained structure). Figure 6 shows the variation in peak stress and m -value with initial strain rates of the rolled and the aged specimens. Both specimens show high m -values over 0.5 in the vicinity of a strain rate of $1 \times 10^{-2}/s$.

3. Microstructural change during superplastic deformation

Figures 7 and 8 show the optical microstructures at various stages of the deformation at 1273 K in the rolled and the aged specimens, respectively. The rolled specimens were deformed at an initial strain rate of $1.7 \times 10^{-2}/s$, while the aged specimens were deformed at $1.7 \times 10^{-3}/s$. Significant grain growth with increasing strain is observed in both specimens. The shape of grains does not change during the deformation. The grains are equiaxed in shape even after a large strain, i.e., 1300% elongation in the rolled specimen (Fig. 7(c)). It must be noted that ferrite/ferrite boundaries are clearly seen in all the optical microstructures shown in Figs. 7 and 8. This suggests that the ferrite/ferrite boundaries have become high-angle boundaries by recrystallization of the ferrite matrix during the deformation by 170% elongation in the rolled specimen and by 130% elongation in the aged specimen. Precipitation of the sigma phase was not observed in any specimens examined.

Figures 9 to 14 show the TEM micrographs at various stages of the deformation at 1273 K and at a strain rate of $1.7 \times 10^{-2}/s$ in the rolled specimens. The TEM micrograph shown in Fig. 9 was taken from the rolled specimen deformed up to 20% elongation (just after the peak strain). Each of the selected-area diffraction patterns taken from the ferrite grains marked a, b and c shows somewhat different contrast. This indicates that the misorientations between adjacent ferrite grains increase slightly from those before the deformation.

Fig. 7 Optical microstructures at various stages of deformation at 1273 K and at an initial strain rate of 1.7×10^{-2} /s in the rolled specimens; (a) 170% elongation, (b) 720% elongation, and (c) 1300% elongation.

Fig. 8 Optical microstructures at various stages of deformation at 1273 K and at an initial strain rate of 1.7×10^{-3} /s in the aged specimens; (a) 130% elongation, (b) 420% elongation, and (c) 700% elongation.

However, the misorientations are small (within several degrees) and the ferrite grains are still subgrains at 20% elongation. The dislocation density is relatively high in both phases of ferrite and austenite, suggesting the difficulty of grain-boundary sliding of subboundaries. Nevertheless, as described before (Figs. 5 and 6), the m -value obtained from the strain rate dependence of peak stress was high in the vicinity of a strain rate of 1.7×10^{-2} /s and the rolled specimens with the finer-grained structure showed the lower peak stress than the aged specimens. These results have been well established as the characteristic phenomena in the superplastic deformation involving the grain-boundary sliding as the dominant mechanism⁽¹⁰⁾⁽¹¹⁾. It is thus considered that the grain-

boundary sliding of ferrite/austenite boundaries occurs to some extent. However, since ferrite/ferrite boundaries were low-angle boundaries, slip deformation might be required in addition to the grain-boundary sliding of ferrite/austenite boundaries, resulting in a relatively high dislocation density at 20% elongation.

Three kinds of TEM microstructures were observed in the identical rolled specimen deformed up to 50% elongation. The first one was essentially the same as that shown in Fig. 9 (20% elongation). The misorientations between adjacent ferrite grains were small and the dislocation density was relatively high. Figure 10 shows a typical TEM micrograph of the second one which was most frequently observed among the three microstructures. The misorien-

Fig. 9 TEM micrograph of the rolled specimen deformed at 1273 K and at an initial strain rate of 1.7×10^{-2} /s up to 20% elongation (just after the peak strain). Selected-area diffraction patterns were taken from the ferrite subgrains marked a, b and c in the micrograph

tation between the ferrite grains marked a and b is large (46 degrees). The dislocation density is, however, still high in the ferrite grains. The third microstructure is shown in Fig. 11. Dislocations are scarcely observed in the ferrite grains marked a and b. The misorientation between those ferrite grains is 44 degrees. Consequently, at 50% elongation the misorientations between adjacent ferrite grains is increasing and some ferrite grains without dislocations are formed.

Further deformation up to 85% elongation changed the microstructure dramatically, as shown in Fig. 12. It must be emphasized that dislocations are scarcely observed in both phases of ferrite and austenite. It is clear from the selected-area diffraction patterns taken from the ferrite grains marked a, b, c and d that the misorientations between adjacent ferrite grains are large. The average ferrite grain size is $1.6 \mu\text{m}$, whereas the average austenite grain size is $1.5 \mu\text{m}$. It can be thus concluded that in the early stage of deformation (by 85% elongation) the recrystallization of the ferrite matrix is completed. This recrystallization changes the microstructure to the fine and equiaxed duplex structure with high-

Fig. 10 TEM micrograph of the rolled specimen deformed at 1273 K and at an initial strain rate of 1.7×10^{-2} /s up to 50% elongation. Selected-area diffraction patterns were taken from the ferrite grains marked a and b in the micrograph

Fig. 11 TEM micrograph of the rolled specimen deformed at 1273 K and at an initial strain rate of 1.7×10^{-2} /s up to 50% elongation. Selected-area diffraction patterns were taken from the ferrite grains marked a and b in the micrograph.

Fig. 12 TEM micrograph of the rolled specimen deformed at 1273 K and at an initial strain rate of 1.7×10^{-2} /s up to 85% elongation. Selected-area diffraction patterns were taken from the ferrite grains marked a, b, c and d in the micrograph.

angle ferrite/ferrite boundaries suitable for the grain-boundary sliding.

Figures 13 and 14 show TEM micrographs of the rolled specimens deformed up to 170 and 1300% elongations, respectively. Dislocations are scarcely observed in both microstructures. During the deformation from 85 to 1300% elongation, the dislocation density is very low in the whole of specimens and no work hardening structure is observed. These results strongly suggest that the deformation takes place mainly by the grain-boundary sliding. Maehara⁽⁴⁾ proposed that the dynamic recrystallization of ferrite matrix, which occurs repeatedly during the deformation, is the mechanism of superplasticity of the duplex stainless steels. In the present study, however, the work hardening structure necessary for the recrystallization is not observed during the deformation from 85 to 1300%

elongations.

Figure 15 shows the change in average grain size of austenite with holding time at 1273 K in the rolled specimens deformed at a strain rate of 1.7×10^{-2} /s. The holding time is the sum of deformation time and the static holding time (60 s) before the deformation. In the figure, the change in austenite grain size with static holding time at 1273 K in the rolled specimens is also shown for comparison. The arrow in the figure indicates the time for 85% elongation at which the recrystallization of the ferrite matrix is completed. After the recrystallization of the ferrite matrix, the grain growth rate in the deformed materials is far greater than that in the undeformed materials. Strain-enhanced grain growth has been observed and pointed out as one of the typical features of the microstructural change during the

Fig. 13 TEM micrograph of the rolled specimen deformed at 1273 K and at an initial strain rate of $1.7 \times 10^{-2}/s$ up to 170% elongation.

superplastic deformation whose dominant mechanism is the grain-boundary sliding⁽¹²⁾⁽¹³⁾. Therefore, the extensive grain growth in the deformed materials suggests that the deformation proceeds mainly by the grain-boundary sliding after the recrystallization of the ferrite matrix. This is consistent with the result that the dislocation density is very low in the whole of specimens during the superplastic deformation after the recrystallization of the ferrite matrix.

The rolled specimens exhibited a large elongation near

Fig. 15 Variation of austenite grain size in the rolled specimens during superplastic deformation at 1273 K and at an initial strain rate at $1.7 \times 10^{-2}/s$, and during static holding at 1273 K.

1000% at a high strain rate of $1.7 \times 10^{-1}/s$ (Fig. 4). The microstructural change during the deformation at $1.7 \times 10^{-1}/s$ was essentially the same as that at $1.7 \times 10^{-2}/s$. A typical TEM microstructure of the rolled specimen deformed at $1.7 \times 10^{-1}/s$ is shown in Fig. 16. The specimen was deformed up to 180% elongation. As is seen in selected-area diffraction patterns obtained from the ferrite grains marked a and b, the ferrite matrix is recrystallized and the ferrite/ferrite boundaries are converted to high-angle ones suitable for the grain-boundary sliding. However, the dislocation density is somewhat higher than that of the specimens deformed at a lower strain rate of $1.7 \times 10^{-2}/s$ (i.e., Fig. 13). This may be associated with a larger contribution of slip deformation at a higher strain rate.

Fig. 14 TEM micrograph of the rolled specimen deformed at 1273 K and at an initial strain rate of $1.7 \times 10^{-2}/s$ up to 1300% elongation.

Fig. 16 TEM micrograph of the rolled specimen deformed at 1273 K and at an initial strain rate of 1.7×10^{-1} /s up to 180% elongation. Selected-area diffraction patterns were taken from the ferrite grains marked a and b in the micrograph.

IV. Discussion

1. Role of the dynamic recrystallization in the superplastic deformation

Before the deformation at 1273 K, the ferrite/ferrite boundaries of the microduplex structure are low-angle boundaries. The microstructural observation showed that in the early stage of the deformation, the ferrite matrix was recrystallized and the ferrite/ferrite boundaries were converted to high-angle ones suitable for the grain-boundary sliding. The completion of recrystallization was confirmed at 85% elongation in the rolled specimens. Once the fine and equiaxed duplex structure with high-angle ferrite/ferrite boundaries was formed, the dislocation density was very low and the extensive grain growth occurred during the subsequent deformation. These results strongly suggest that the grain-boundary sliding is the dominant mode of the deformation

after the recrystallization of the ferrite matrix. The dynamic recrystallization of the ferrite matrix occurred only in one cycle in the early stage of the deformation. The conventional dynamic recrystallization, where the strain hardening and the subsequent recrystallization occur repeatedly during deformation, was not observed. From these results, it is concluded that the role of the dynamic recrystallization in the superplastic deformation is to make the fine structure suitable for the grain-boundary sliding in the early stage of the deformation.

2. Mechanism of the dynamic recrystallization

The ferrite matrix of the microduplex structure consists of ferrite subgrains and is in a recovery condition. Therefore, the ferrite matrix is able to recrystallize by static holding. However, a very long time is needed for completion of the recrystallization, when the specimens are held at 1273 K. In fact, when the rolled specimens were statically held for 38 ks at 1273 K, the recrystallization of the ferrite matrix was observed only in limited

regions where the austenite grains coarsened over $10\text{ }\mu\text{m}$ in diameter. On the other hand, the recrystallization was completed at 85% elongation (deformation time of 50 s), when the rolled specimen was tensile-deformed at 1273 K and at a strain rate of $1.7 \times 10^{-2}/\text{s}$ (Fig. 12). These results indicate that the subgrain structure of the ferrite matrix is very stable against the static holding at 1273 K but easily recrystallize by applying the tensile deformation at 1273 K. This result differs from that reported in ferritic steels. It has been well recognized in ferritic steels⁽¹⁴⁾ that the dynamic recrystallization is very hard to occur. Maehara⁽⁴⁾ considered that the occurrence of the dynamic recrystallization in the ferrite matrix of the microduplex structure is attributed to localization of deformation into the ferrite matrix due to the existence of relatively hard austenite particles.

In order to discuss the mechanism of the dynamic recrystallization during the superplastic deformation, it should be taken into account that in the process of conventional recrystallization by the nucleation and growth mechanism, subgrains are formed by recovery and particular subgrains among a number of subgrains can grow as recrystallized grains⁽¹⁵⁾. In the rolled specimen deformed up to 85% elongation at $1.7 \times 10^{-2}/\text{s}$, the average grain size of recrystallized ferrite was $1.6\text{ }\mu\text{m}$. Therefore, the ferrite subgrain structure with a fine grain size below $1.6\text{ }\mu\text{m}$ must be newly formed by 85% elongation, if the recrystallization occurred by the nucleation and growth mechanism. However, dislocations were rather uniformly distributed in ferrite grains and the finer subgrain structure was not observed in the rolled specimens deformed up to 20 and 50% elongations (Figs. 9 and 10). These results suggest that the dynamic recrystallization during the superplastic deformation occurs in a different manner from the nucleation and growth mechanism.

Similar microstructural changes during superplastic deformation have been reported in a number of aluminum alloys such as an Al-6%Cu-0.4%Zr alloy (Supral 100) and Al-Zn-Mg-Zr alloys⁽¹⁶⁾⁻⁽²²⁾. These aluminum alloys are in recovery conditions before the tensile deformation. The subgrain structure before the deformation changes to a fine recrystallized grain structure consisting of high-angle boundaries in the early stage of the deformation. Furthermore, misorientations between adjacent grains continuously increase with an increase in strain⁽¹⁸⁾⁽¹⁹⁾⁽²⁰⁾. This recrystallization has been called "continuous recrystallization"⁽¹⁸⁾⁽¹⁹⁾⁽²¹⁾ to distinguish from the conventional recrystallization ("discontinuous recrystallization") that occurs by the nucleation and growth mechanism. In the present Fe-25%Cr-7%Ni-3%Mo steel, the misorientation angle between adjacent ferrite grains increased during the deformation from 20 to 85% elongations. Ameyama *et al.*⁽²³⁾ have recently reported a similar continuous increase in the misorientation angle during superplastic deformation in an Fe-24%Cr-7%Ni duplex stainless steel with the microduplex structure. Therefore, it can be considered that the dynamic recrystallization in the duplex stainless steel occurs in the same manner as the dynamic con-

tinuous recrystallization in the superplastic aluminum alloys does.

Although the mechanism of the dynamic continuous recrystallization has not yet been clarified, Hales and McNelly⁽²¹⁾ have proposed a mechanism based on the microstructure observation in a superplastic Al-Mg alloy. They have observed that dislocations introduced to grains by slip deformation are absorbed into boundaries and rearranged during superplastic flow, and concluded that such absorption of dislocations into boundaries has the effect of increasing misorientations between adjacent grains. However, in single-phase alloys, the marked increase in misorientation angle between subgrains has not been reported in the dynamic recovery condition. This may be associated with operation of an identical slip system among subgrains in a grain in the single-phase alloys. In such a case, annihilation of dislocations by intersecting of dislocations with opposite forms each other may frequently occur at subboundaries. Unlike the single phase alloys, different slip systems may operate among ferrite subgrains in the microduplex structure, because of restriction of the deformation by the relatively harder austenite particles. It is thus expected that frequency of annihilation of dislocations in the process of dislocation rearrangement at subboundaries is much lower compared with the single phase alloys, and that the absorption of dislocations into the ferrite subboundaries has the effect of increasing misorientations between adjacent subgrains as proposed by Hales and McNelly⁽²¹⁾.

In such a process of the dynamic continuous recrystallization, the introduction of dislocations by slip deformation in the ferrite subgrains and subsequent rearrangement of the dislocations at the ferrite subboundaries should occur continuously until the grain-boundary sliding becomes the dominant deformation mode. This is consistent with the microstructural observation that in the early stage of the deformation a considerable amount of dislocations are seen in spite of the relatively high misorientations between adjacent ferrite grains (Fig. 10). The recrystallization by dislocation absorption into boundaries does not require either subboundary movement or coarsening of second particles. Therefore, the recrystallized structure should have the same grain size as that of subgrain structure before deformation, if the coarsening of second particles could be neglected. This is in good agreement with the results reported in aluminum alloys⁽¹⁹⁾⁽²⁰⁾; the finer grain size is obtained by the dynamic continuous recrystallization than by the static discontinuous recrystallization. Therefore, the dynamic continuous recrystallization is also important as one of the thermo-mechanical treatments for the grain refinement.

3. Appearance of high-strain rate superplasticity

It has been well established⁽¹⁰⁾⁽¹¹⁾ that there is a range of strain rates over which superplastic deformation can be expected, at a given temperature for a given superplastic material. Above a certain specific strain rate, superplastic flow is no longer the dominant process and another mode

of deformation becomes important⁽¹⁰⁾⁽¹¹⁾. It has been found⁽¹⁰⁾⁽¹¹⁾ that the maximum strain rate for superplastic flow becomes higher with decreasing grain size and is inversely proportional to the cube of grain size when grain-boundary diffusion is the rate controlling process. High-strain rate superplasticity can be thus expected by refining the grain size. Based on this point, Bieler *et al.*⁽⁶⁾ studied superplastic behavior in mechanically alloyed aluminum alloys with an extremely fine (submicron) grained structure. They reported tensile elongations near 500% at unusually high strain rates between $1 \times 10^0/\text{s}$ and $1 \times 10^2/\text{s}$.

When the structure contains fine second phase particles, the grain growth of the matrix phase is strongly influenced by the restraining force of the second particles. According to Zener's model⁽²⁴⁾, the grain size (D) of the matrix phase in two-phase structure can be written as

$$D = \beta(d/f) \quad (1)$$

where d and f are the grain size and the volume fraction of the second phase, respectively, and β is constant. Abe *et al.*⁽²⁵⁾ reported that the coefficient β is nearly $4/9$ for the static annealing in duplex stainless steels. Figure 17 shows the relation between the ferrite grain size (D) and the austenite grain size (d) during the superplastic deformation at 1273 K in the rolled and the aged specimens. The volume fraction of austenite (f) is 0.4 at 1273 K. It is seen that the experimental data obey Zener's relation (Equation (1)) and the coefficient β is approximately $4/9$, irrespective of the strain rate and the heat treatment before the tensile deformation. This indicates that the matrix grain size is controlled by the grain size of second phase particles even during the superplastic deformation. The recrystallized grain size of the ferrite matrix is thus determined by the grain size of the austenite particles. In the rolled specimen, the austenite particle size before the deformation was $1 \mu\text{m}$ and smaller than that in the aged specimen, so that the finer equiaxed duplex structure was obtained in the early stage of the deformation. This

finer structure made the maximum strain rate for the superplastic flow higher and the rolled specimens exhibited the high strain rate superplasticity. Consequently, the refinement of austenite particle size before the deformation is important for the appearance of high-strain rate superplasticity in the duplex stainless steels. The nature of the ferrite/ferrite boundaries before the deformation is not important, since the ferrite subboundaries can be readily converted to high-angle boundaries by the dynamic recrystallization in the early stage of the deformation. The present results suggest that the superplastic flow at high strain rates over $1.7 \times 10^{-1}/\text{s}$ is possible in the duplex stainless steels, if the austenite particle size of submicron is obtained before the deformation.

V. Conclusion

Superplasticity of a 25Cr-7Ni-3Mo ferrite/austenite duplex stainless steel has been studied with particular emphasis on the microstructural change during deformation in order to clarify the role of dynamic recrystallization in the superplastic flow. Main results obtained are as follows.

(1) A microduplex structure consisting of fine ferrite subgrains and austenite particles (grain size: $1 \mu\text{m}$) is obtained by annealing at 1273 K after 90% cold rolling of a fully ferritic structure. The steel with this fine-grained structure exhibits high-strain rate superplasticity at 1273 K in the ferrite/austenite two phase region. A maximum elongation over 1700% is obtained at a strain rate of $1.7 \times 10^{-2}/\text{s}$. Even at a higher strain rate of $1.7 \times 10^{-1}/\text{s}$ the elongation is near 1000%.

(2) The ferrite matrix of the microduplex structure before the superplastic deformation is in a recovery condition and ferrite/ferrite boundaries are low-angle boundaries. In the early stage of the deformation, the ferrite/ferrite boundaries are changed to high-angle boundaries by dynamic recrystallization, and the fine and equiaxed duplex structure suitable for grain-boundary sliding is formed. Dislocations are scarcely observed in the whole of specimens during the deformation after the recrystallization of the ferrite matrix.

(3) The grain growth during the deformation after the recrystallization of the ferrite matrix is much faster than that during the static holding without deformation. The experimental data on grain sizes of ferrite and austenite obey Zener's relation (Equation (1)) and the coefficient β is $4/9$.

(4) The grain-boundary sliding is concluded to be the dominant mode of the superplastic deformation from the results of the very low dislocation density and the extensive grain growth during the deformation after the dynamic recrystallization of the ferrite matrix. The dynamic recrystallization occurs only in one cycle in the early stage of the deformation. The conventional dynamic recrystallization, where the strain hardening and the subsequent recrystallization occur repeatedly during deformation, is not observed. The role of the

Fig. 17 Relation between ferrite grain size (D) and austenite grain size (d) during superplastic deformation at 1273 K. f is the volume fraction of austenite and is 0.4 at 1273 K.

dynamic recrystallization in the superplastic flow is to make the fine structure suitable for the grain-boundary sliding in the early stage of the deformation.

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