Superplastic Deformation Mechanism of δ/γ Duplex Stainless Steels*

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Synopsis

Superplasticity of δ/γ duplex stainless steels has been studied in relation to the microstructural change during deformation in order to elucidate the role of the second phase particles. In the case of deformation in the δ/γ duplex phase region, local strain concentration within the δ -ferrite matrix due to dispersion of relatively hard γ particles and the subsequent recrystallization occur repeatedly. When the alloys containing high Cr and Mo are deformed at temperatures around 900°C, σ phase particles precipitate via eutectoid decomposition of δ -ferrite into new γ and σ phases, and the γ/σ duplex phase structure forms. The dynamic recrystallization of γ phase matrix occurs locally and intermittently, and extremely large superplasticity is also obtained. Prior cold work largely accelerates these processes, and leads to equiaxed δ/γ or γ/σ duplex structure in the early stage of deformation. The superplasticity in both cases can be obtained by subtle balance between the local strain hardening and the subsequent recrystallization, and thus the flow stress exhibits large strain rate dependence. This model can also explain granular appearance of the fracture surface as a coalescence of microvoids induced by the individual hard phase particles. In order to obtain large superplasticity, the microstructure should consist of hard particles embedded within a soft matrix and the amount of hard particles should be at least greater than 10 % in the case of γ/σ duplex structure. The structure with hard phase matrix within which the soft second phase particles disperse do not exhibits superplasticity.

Key words: superplasticity; duplex stainless steel; hot deformation; dynamic precipitation; dynamic recrystallization; hot tensile testing.

I. Introduction

Superplasticity during isothermal deformation has been the subject of numerous investigations and a significantly wide variety of alloy systems are known to exhibit this property.¹⁻¹²⁾ Normally superplastic materials are characterized by large strain rate dependence of flow stress and extremely fine microstructure.³⁻¹²⁾ It is also interesting to note that most of superplastic materials consist of duplex phases,^{3,8,10)} but the role of the second phase has scarcely been examined.

Hayden *et al.*¹³⁾ reported superplasticity of δ/γ duplex stainless steels containing 26%Cr-6.5%Ni-0.5%Ti. Recently the present authors examined the superplasticity of δ/γ duplex stainless steels with particular attention to the microstructural change during

deformation,^{14,15)} and obtained the following results;

(1) Superplasticity is enhanced largely by the prior cold work after solution treatment of the materials.

(2) When the δ/γ duplex structure of high Cr and Mo steels decomposes into γ/σ duplex structure¹⁶⁻¹⁸⁾ in the early stage of deformation at temperatures around 900°C, large superplasticity is also obtained.

(3) Local strain concentration due to fine dispersion of hard second phase particles (γ in δ or σ in γ) and the subsequent recrystallization (dynamic recrystallization) of the soft matrix phase occur during the superplastic deformation and result in equiaxed δ/γ or γ/σ duplex structure.

In the above described dynamic recrystallization processes, the matrix should be a soft phase and the hard second phase particles have to disperse within it. Thus in the present study, the effects of γ/δ and σ/γ fractions on superplasticity in the cases of both harder and softer phase matrix are examined in order to elucidate the mechanism of superplasticity in δ/γ duplex stainless steels.

II. Experimental Procedure

The steels of which chemical compositions are given in Table 1 were prepared by a laboratory induction furnace. The ingots of 50 kg in weight were hot-forged and rolled into 12-mm thick plates. A part of these plates were solution-treated for 30 min at 1 250°C which is still in δ/γ duplex phase region, and quenched into water. Some of them were further cold-rolled into 6-mm thick plates (50 % in reduction). From these hot-rolled, solution-treated and cold-rolled plates, tensile specimens were machined in the direction parallel to that of rolling. The gage length, gage diameter and fillet radius were 8, 4 and 2 mm, respectively.

The specimens were set to a hot tensile machine, and were heated at a rate of 1°C/s to the deformation temperatures ranging from 800 to 1 100°C in a furnace attached to the tensile machine. After homo-

Table 1. Chemical composition of the materials used. (wt%)

Steel	C	Si	Mn	P	S	Ni	Cr	Mo	Cu	W	N
S 1	0.015	1.69	3.22	0.005	0.002	4.06	18.52	—	1.18	—	0.0094
S 2	0.015	1.73	3.42	0.005	0.002	3.96	18.77	—	1.20	—	0.0928
D 1	0.017	0.48	0.85	0.025	0.001	6.82	24.66	2.79	0.46	0.28	0.143
D 2	0.005	3.22	0.79	0.003	0.001	10.52	22.88	—	—	—	0.0912

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genizing for 5 min, they were pulled isothermally at a constant cross head speed from 0.5 to 100 mm/min (the initial strain rates ($\dot{\epsilon}$) being from 4.16×10^{-4} to $2.08 \times 10^{-1} \text{ s}^{-1}$) until final fracture occurred. The experiments were conducted in a flowing Ar gas atmosphere in order to suppress surface oxidation of fractured surfaces. In some cases, the deformations were interrupted at given elongations and cooled at the fastest rate available, *i.e.*, 5°C/s in the temperature between 1 000 and 500°C , in order to examine the microstructural change during deformation. On these specimens metallographic and fractographic examinations were conducted by means of optical, scanning electron (SEM) and transmission electron microscopy (TEM).

In order to understand the precipitation behavior of σ phase, the specimens prepared from the above described plates were aged for given times in a salt bath kept at 900°C . Sigma phase fraction was determined by means of X-ray diffraction method reported elsewhere.¹⁶⁻¹⁸⁾

III. Experimental Results

1. Tensile Properties

Examples of fractured tensile specimens are given in Fig. 1. Steels S1 and D1 exhibited large superplastic elongation especially in the priorly cold-rolled conditions in a wide range of deformation temperature and initial strain rate. The maximum elongations of each steel were greater than 700 and 2 500 %, respectively. While the elongations of Steels S2 and D2 were very small, *i.e.*, 100~300 % even in the cold-rolled specimens. Figure 2 shows the examples of flow stress–elongation curves of various steels. After passing the peak stress, the flow stress decreased gradually until final fracture occurred in Steels S1 and D1, but a sudden decrease corresponding to local necking was observed in Steels S2 and D2.

Figure 3 shows the variation of the tensile properties at 950°C with initial strain rate in cold-rolled specimens. In Steels S1 and D1 a large peak of superplastic elongation was observed at an initial strain

- (a) Undeformed
- (b) Steel S1 deformed at 950°C at $\dot{\epsilon} = 2.08 \times 10^{-3} \text{ s}^{-1}$ (725 % elongation)
- (c) Steel S2 at 900°C at $\dot{\epsilon} = 2.08 \times 10^{-3} \text{ s}^{-1}$ (193 %)
- (d) Steel D1 at 950°C at $\dot{\epsilon} = 2.08 \times 10^{-3} \text{ s}^{-1}$ (1 178 %)
- (e) Steel D2 at 900°C at $\dot{\epsilon} = 1.04 \times 10^{-2} \text{ s}^{-1}$ (284 %)

Fig. 1. Examples of fractured superplastic tensile specimens deformed after prior cold rolling.

rates around $2 \times 10^{-3} \text{ s}^{-1}$. While the elongations of Steels S2 and D2 were very small, and the strain rate showing peak elongation shifted slightly to lower $\dot{\epsilon}$ side. It can also be seen that the strain rate dependence of peak stress in Steels S1 and D1 was largest at the strain rate of peak elongation, but those in Steels S2 and D2 were very small. High flow stresses of Steels D1 and D2 compared with those of S1 and S2 are due to σ phase precipitation during deformation.¹⁹⁾

- (a) Steels S1 and S2 deformed at 950°C at $\dot{\epsilon} = 1.04 \times 10^{-2} \text{ s}^{-1}$
- (b) Steels D1 and D2 deformed at 900°C at $\dot{\epsilon} = 4.16 \times 10^{-3} \text{ s}^{-1}$.

Fig. 2. Stress–elongation curves of the cold-rolled specimens.

Fig. 3. Variation of tensile properties with initial strain rate in various steels; initially cold-rolled specimens deformed at 950°C .

Figure 4 shows the variation of the tensile properties in Steels D1 and D2 at $\dot{\epsilon}=2.08 \times 10^{-3} \text{ s}^{-1}$ with deformation temperature and the prior treatment. Although elongation of the solution-treated Steel D1 was rather small, it was largely increased by the prior hot or cold deformation, *i.e.*, the maximum elongation of hot-rolled and cold-rolled specimens were about 700 and 2500 %, respectively. It should be noted that two peaks of elongation were recognized in the deformation of hot-rolled and cold-rolled specimens at temperatures around 950 and 1050°C. In Steel D2, in addition to small elongation in all the conditions examined, superplasticity was hardly affected by the prior cold deformation. The peak stress of Steel D1 was decreased by prior cold deformation. Strengthening due to lowering the temperature below 950°C is mainly caused by the dynamic precipitation of σ phase.¹⁹⁾ Superplasticity of Steel S1 was largely increased by the prior cold work, but such effect was not observed in Steel S2 as shown in Fig. 5.

2. Precipitation Behavior of σ Phase

Figures 6 and 7 show the microstructures of the cold-rolled specimens heated for 5 min at 1050 and 950°C, respectively. In Steels S1 and D1 heated at 1050°C, fine γ particles precipitated in the reheating process and dispersed within δ -ferrite matrix in addition to coarse γ islands formed in the solution treatment process. In Steels S2 and D2 δ -ferrite islands were embedded within γ phase matrix conversely. Dispersion of γ particles formed in Steel S1 during reheating to 950°C in Steel S1 was much finer than that in the case of 1050°C as in Fig. 7(a). Although fine γ particles dispersed within δ islands in Steel S2 reheated to 950°C, the microstructure is completely different from that of Steel S1 in view of the matrix phase. In Steels D1 and D2, large amounts of σ particles formed within γ phase²⁰⁾ as well as in δ -ferrite as in Figs. 7(c) and 7(d).

Figure 8 shows the precipitation-time curves of σ phase in Steels D1 and D2 during isothermal aging at 900°C with and without prior cold work. Although the incubation time for the σ phase precipitation in Steel D2 was shorter than that in solution-treated D1, the precipitation was much faster in Steel D1 than D2 in the initially cold-rolled condition. It should also be noted that the saturated amount of σ phase was much larger in Steel D1 than in Steel D2: *i.e.*, 30 and 10 %, respectively.

3. Fractography and Metallography

Figures 9(a) and 9(c) illustrate the fractured surfaces of Steels S1 and S2 specimens deformed at 950°C at $\dot{\epsilon}=2.08 \times 10^{-3} \text{ s}^{-1}$ after the prior cold rolling, respectively. Granular undulations can be seen in the case of the Steel S1 specimen fractured after a large superplastic elongation of 700 %. It should be noted that the undulations correspond in dimension to the dispersion of hard γ particles within δ -ferrite matrix, as shown in Fig. 9(b). While in the Steel S2 specimen fractured at a small elongation of 150 %, the fractured surface was similar to that observed in

Fig. 4. Variation of tensile properties with deformation temperature and its effect of prior treatment; Steels D1 and D2 specimens deformed at $\dot{\epsilon}=2.08 \times 10^{-3} \text{ s}^{-1}$.

Fig. 5. Variation of tensile properties with deformation temperature and its effect of prior treatment; Steels S1 and S2 specimens deformed at $\dot{\epsilon}=2.08 \times 10^{-3} \text{ s}^{-1}$.

transgranular ductile fracture as in Fig. 9(c), and the traces of shear deformation were recognized as indicated by an arrow 1, though granular undulations were also observed in the bottom of dimples (arrow 2). The average distance between these dimples is almost equal to the separation between δ -ferrite islands elongated to the rolling direction (Fig. 9(d)). The fractured surfaces of Steel D1 exhibiting a large elongation at temperatures around 900°C was essentially the same as that in Steel S1, *i.e.*, granular undulations corresponding to the fine dispersion of σ

Fig. 6. Microstructures on the longitudinal cross section; Steels (a) S1, (b) S2, (c) D1 and (d) D2 specimens heated at 1 050°C for 5 min after prior cold rolling.

Fig. 7. Microstructures on the longitudinal cross section; Steels (a) S1, (b) S2, (c) D1 and (d) D2 specimens heated at 950°C for 5 min after prior cold rolling.

Fig. 8. Precipitation curves of σ phase at 900°C in Steels D1 and D2; the specimens isothermally aged after solution treatment or the subsequent cold rolling.

particles within the new γ matrix (Figs. 10(a) and 10(b)). The fracture appearance of Steel D2 with poor superplasticity, however, was a typical of microvoid coalescence mode of fracture as in Fig. 10(c).

Figures 11 to 13 show the microstructures after deformation of prior cold-rolled specimens examined by means of TEM. In Steel S1 deformed up to 350 % at 950°C at $\dot{\epsilon}=2.08\times10^{-3}\text{ s}^{-1}$, locally recrystallized and strain concentrated regions can be seen in the δ -ferrite matrix adjacent to the hard γ phase grains as in Figs. 11(a) and 11(b), respectively. In the Steel D2 specimen deformed up to 200 % at $\dot{\epsilon}=4.16\times10^{-3}\text{ s}^{-1}$ at 900°C where the dynamic precipitation of σ phase particles was almost completed, locally deformed and recrystallized regions in the γ phase matrix in contract with hard σ phase particles were

Fig. 9.
Fracture surfaces and corresponding microstructures on the longitudinal cross section; (a), (b) Steel S1 and (c), (d) Steel S2, deformed at 950°C at $\dot{\epsilon}=2.08 \times 10^{-3} \text{ s}^{-1}$ after prior cold rolling. The fracture elongations were 700 and 150 %, respectively.

Fig. 10.
Fracture surfaces and corresponding microstructures on the longitudinal cross section; (a), (b) Steel D1 and (c), (d) Steel D2, deformed at 900°C at $\dot{\epsilon}=2.08 \times 10^{-2} \text{ s}^{-1}$. The fracture elongations were 800 and 180 %, respectively.

observed as in Figs. 12(a) and 12(b), respectively. In Steel D2, however, such dynamic recrystallization of γ phase did not occur because the amount of σ phase particles was so small, and the structure was much coarser than that in Steel D1 as in Fig. 13.

VI. Discussion

1. Microstructural Change during Deformation and the Mechanism of Superplasticity

1. In the Case of δ/γ Duplex Structure

Large superplastic elongation was observed in Steels S1 and D1 in which fine γ particles dispersed within the δ -ferrite matrix at temperatures from 800 to 1100°C at the initial strain rates from 10^{-3} to 10^{-2} s^{-1} . During superplastic deformation microstructure changed considerably. In Steel S1 or D1 deformed

at temperatures above 1000°C, coarse γ islands were broken into fine particles and the Widmanstätten γ particles formed during reheating to the deformation temperatures grew into spherical particles, resulting in homogeneous dispersion of γ particles in the δ -ferrite matrix as in Fig. 9(b). The δ -ferrite matrix was deformed locally by relatively hard γ particles (Fig. 11(a)) and the subsequent recrystallization occurs (Fig. 11(b)) repeatedly. These processes lead to the finally stable equiaxed δ/γ duplex structure, and are closely related to the superplastic deformation mechanism. That is, the local strain concentration will induce the work hardening which is larger than the decrease of strength due to the local necking. Therefore the deformation will propagate in the region where local necking has not occurred and will reduce the cross section of such regions accompanied by the

Fig. 11. TEM micrographs showing mixture of (a) local strained and (b) recrystallized structures of δ -ferrite matrix surrounding γ particles in the same thin foil specimen; Steel S1 deformed to 350 % in elongation at 950°C at $\dot{\epsilon}=2.08 \times 10^{-3} \text{ s}^{-1}$ after prior cold rolling.

Fig. 12. TEM micrographs showing mixture of (a) local strained and (b) recrystallized structures of γ matrix surrounding σ particles in the same thin foil specimen; Steel D1 deformed to 200 % in elongation at 900°C at $\dot{\epsilon}=4.16 \times 10^{-3} \text{ s}^{-1}$ after cold rolling.

Fig. 13. TEM micrographs of Steel D2; the specimen deformed to 100 % in elongation at 900°C at $\dot{\epsilon}=4.16 \times 10^{-3} \text{ s}^{-1}$ after prior cold rolling.

subsequent strain hardening. Eventually the initially deformed regions will recrystallize and will be deformable. Successive occurrence of these processes will lead to the homogeneous superplastic deformation. This suggests that the superplasticity arises from a very subtle balance of strain rate and deformation temperature. That is, if the strain rate is too

small, strain accumulation enough for the recrystallization can not be obtained because of dynamic recovery as well as of γ phase coarsening. On the contrary, an extremely large strain rate will yield only overall work hardening of soft δ matrix without homogeneous dispersion of γ particles, resulting in poor superplasticity. Equiaxed structure formation during superplastic deformation can also be explained by this dynamic recrystallization model. This model is consistent with the fact that the structure is either coarsened or refined by the decrease or the increase of strain rate, respectively.^{14,15)}

In the superplastic deformation, the flow stress σ depends largely on the strain rate $\dot{\epsilon}$ as

$$\sigma = K\dot{\epsilon}^m$$

where K and m are constants and m is significantly large. This empirical relationship is also in keeping with the present dynamic recrystallization model, but the equation should be expressed theoretically in a different way. That is, in the region of low strain rate, since the dislocations introduced by deformation are subsequently rearranged or eliminated through recovery, the peak stress will not be largely affected by the strain rate, the m -value being significantly small. In the high strain rate region, the recovery and recrystallization can hardly occur and large work hardening will be introduced, but the strain rate dependence will also be small. Whereas, in the intermediate strain rate region, work hardening and recrystallization occur simultaneously, and a small increase of the strain rate will suppress the recrystallization considerably and will result in a large m -value.

2. In the Case of γ/σ Duplex Structure

The deformation of Steel D1 at temperatures around 900°C also provides a significantly large superplastic elongation especially in the initially cold-rolled specimens. In this case, dynamic precipitation of σ phase occurs mainly by the eutectoid decomposition of δ into new γ and σ phases, resulting in γ/σ duplex structure with 30 vol% of σ phase. In the cold-rolled specimens fine homogeneous dispersion of σ particles is easily attained in the early stage of deformation (Fig. 10(b)), because the prior plastic deformation accelerates the precipitation rate (Fig. 5) and induces the nucleation of σ particles within γ islands as well as in δ -ferrite (Fig. 7(c)). The effect of such dynamic phase change on superplasticity cannot be neglected in this case. Most of the superplastic deformation, however, proceeds in γ/σ duplex phase condition without any phase change. Precipitation rate of σ phase can be predicted from the aging curve of the specimens subjected to the same pretreatment, since it is scarcely influenced by superplastic deformation. For instance, in the case of 2 000 % elongation at 900°C at $2 \times 10^{-3} \text{ s}^{-1}$, the precipitation is completed before 300 % deformation, showing that the rest of 1 700 % elongation can be achieved in γ/σ duplex phase condition. Within new matrix phase of γ , local strain and the subsequent recrystallization occur repeatedly (Fig. 12), leading to equiaxed γ/σ duplex structure (Fig. 10(b)). Thus the dynamic recrystallization mechanism similar to that in δ/γ duplex phase structure will hold in this case. Two peaks of elongation against deformation temperature in Steel D1 (Fig. 4) are expected to arise from the above two states of δ/γ and γ/σ duplex structures, and the microstructural change in the dynamic recrystallization model can be depicted schematically in Fig. 14(a). Superplasticity is markedly enhanced by the prior plastic deformation through the acceleration of microstructural change into stable equiaxed structures.

2. Role of Second Phase Particles on Superplasticity

The superplasticity of Steel D2 deformed at temperatures around 900°C is very poor in spite of the similarly fast precipitation rate of σ phase as in D1. This can be ascribed to the difference in saturated fraction of σ phase, *i.e.*, 30 and 10 % in Steels D1 and D2, respectively. When the amount of hard second phase is too small, even if the dispersion is homogeneous, dynamic recrystallization of the soft matrix phase can not occur throughout the whole specimen, as depicted in Fig. 14(b).

Dispersion of soft phase particles, *e.g.*, δ in γ as in Steel S2 or in higher temperature region of D2 can not enhance dynamic recrystallization of the hard matrix phase, resulting in much poor superplasticity, as in Fig. 14(c). In this case, enhancement of superplasticity by prior deformation is negligibly small (Figs. 4 and 5).

Thus it can be concluded that the second phase particles in superplastic duplex phase alloys have important meaning not only for retaining the fine grain structure but also for the dynamic recrystallization of

Fig. 14. Schematic illustrations of duplex structures during superplastic deformation showing (a) dynamic recrystallization due to homogeneous dispersion of hard second phase particles, (b) insufficient dynamic recrystallization because of small amount of hard particles and (c) none dynamic recrystallization due to dispersion of soft phase particles.

soft phase matrix. Consequently a soft phase should be the matrix, and dispersion of a certain amount of hard second phase is necessary for the superplasticity.

3. Fractography

Fracture surface of the superplastic specimens consists of granular undulations corresponding to the dispersion of hard second phase particles (Figs. 9(a) and 10(a)). This can be explained as follows: disintegration of the subtle balance between local straining and recrystallization of soft phase matrix surrounding hard particles leads to a number of microvoids, and final fracture occurs by their sudden coalescence. This phenomenon does not conflict with the dynamic recrystallization model.

Equiaxed grain structure observed during superplastic deformation has been explained in terms of grain boundary sliding so far,²¹⁻²³⁾ though the homogeneous deformation can not be explained by this model. The feature of grain boundary sliding is probably due to microscopic observations of the deformation with dynamic recrystallization of matrix phase surrounding hard particles, and may be phenomenologically true, although it is not essential for superplasticity.

V. Conclusions

(1) Large superplastic elongations were observed in the tensile deformation of δ/γ duplex stainless steels with dispersion of γ particles in δ -ferrite matrix at initial strain rates from 10^{-3} to 10^{-2} s^{-1} at temperatures from 850 to 1 100°C.

(2) During the deformation large microstructural change was observed. In the case of δ/γ duplex phase condition, γ particles dispersed homogeneously, and local strain concentration of δ -ferrite matrix due to hard γ particles and subsequent recrystallization (dynamic recrystallization) occurred repeatedly, resulting in equiaxed δ/γ duplex structure.

(3) During the deformation of high Cr and Mo alloys at temperatures around 900°C, σ phase precipitation occurred mainly by eutectoid decomposition from δ into new γ and σ phases. After the reaction was completed in the early stage of the deformation, recrystallization of relatively soft new matrix phase of γ occurred dynamically due to dispersion of hard σ particles, resulting in stable equiaxed duplex structure of γ/σ .

(4) The superplasticity was largely enhanced especially by prior cold work *via* marked accelerating effect on the above described processes.

(5) Superplasticity of duplex phase alloys can be closely related to the microstructural changes, and can be obtained by the subtle balance between local work hardening due to hard second phase particles and the subsequent softening due to recrystallization within soft matrix. Large strain rate dependence of flow stress, *i.e.*, large m -value, in superplastic conditions can be explained by large change of average dislocation density within the matrix phase with small change of strain rate.

(6) Fracture surface of superplastic specimens consisted of granular undulations corresponding to the dispersion of hard second phase particles, as a result that a number of microvoids introduced by the unbalance between local straining and recrystallization within soft phase surrounding hard particles coalesced suddenly into final fracture.

(7) Superplasticity of the materials with soft phase dispersion within hard phase matrix was quite poor, and can not be enhanced by the prior deformation, consistent with the dynamic recrystallization model.

(8) Thus the hard second phase particles in superplastic duplex phase alloys has important meaning for the dynamic recrystallization of soft matrix phase as well as for retaining the fine grain structures, and a certain amount of the hard particles, *e.g.*, more than 10 % σ particles in γ/σ duplex structure, was found to be necessary.

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