In-situ Microstructural Observations

of Transfomation Superplasticity in

and

Pure

Micro-grid

Iron*

Analyses

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Synopsis
In the present paper, the microstructural changes associated with phase transformation and the straining behavior in polycrystalline structures due-
ing transformation superplastic deformation are investigated. In-situ ob-servations of microstructural changes during Ac3 transformation on rapid heating in pure iron have been carried out with a high temperature optical microscope and a dark field reflection high temperature microscope. Both of hot-stage microscopes were specially designed for this study. The dis-tribution of superplastic strain has been examined by microscopic strain analyses by using a micro-grid pattern with 12.7 pm intervals. The main results are as follows:
(1) Ac3 transformation process at a heating rate under 50 K/s is predominantly nucleation of austenite grains at the prior ferrite grain boundaries and triple points. A subsequent growth of grains into the prior ferrite matrices is observed. The growth of austenite grains is not always isotropic under a tensile stress.
(2) In the initial stage of transformation, superplastic strain is in-duced by the sliding at rya transformation interface along the prior ferrite grain boundaries. The observed superplastic strain is also associated with the grain rotation, corresponding to the growth of austenite grains which surround ferrite grains.
(3) In the intermediate stage of transformation, the sliding deforma-tion is generated at the migrating transformation interface associated with the growth of austenite grains. Accumulated strain by the sliding is left within the previously transformed region.
(4) These observations suggest that the sliding mechanism at the mi-grating transformation interface is a principal mechanism of transformation superplasticity.

Key words: transformation superplasticity; iron; in-situ observation; grid analysis; high temperature microscope.

I. Introduction

In previous papers, macroscopic deformation due to transformation superplasticity was analyzed in pure iron') and low carbon steels' under rapid heating and cooling cycles. With a micro-grid pattern spaced at 12.7 tm intervals, semi-macroscopic analyses have been performed on the superplastically deformed pure iron.' It was found that (i) the deformation was

principally incompressible and homogeneous and (ii) inhomogeneous distribution of strain along with a rotation component was observed.

II. Experimental Procedures

1. High Temperature Microscope

A high temperature microscope was developed spe-cially for the observation of superplastic deformation and microstructural changes during transformation

processes. A schematic diagram of the newly devel-

oped apparatus is shown in Fig. 1; the main functions are application of rapid heating and cooling cycles and loading of static tension to the specimen. The additional facilities are a chamber for atmospheric control, a supply of etchant for specimen surface, an optical microscope, a motor driven camera, and a

pen-recorder.
The chemical analysis and the specimen dimension
are shown in Table 1 and Fig. 2, respectively. The specimens were heated to a predetermined tempera-

Fig. 1. Schematic dia gram of test apparatus.

Fig.

Table 1.

2. Specimen dimension.

Chemical composition. (wt%)

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ture at a rate up to 200 K/s with resistance heating by using a 6 kVA transformer which was regulated with a current phase controller, and were cooled in the chamber at a selected rate by controlled jet of argon gas from a cooling nozzle. The specimen tem-perature was regulated by means of feedback control from the thermo-electromotive force of PR thermo-

couple spot-welded to the center of a specimen. The force. was simultaneously recorded on a pen-recorder. Hence, selection of thermal cycles between a tempera-ture interval can be achieved and an isothermal soak-

ing after rapid heating can be also performed.
The specimen was contained in an argon or vacu-um atmosphere inside the chamber, and subjected to a constant tensile load through the electrodes. The apparatus is equipped with an instantaneous etching system operated by triggering a magnetic valve of etching gases to reveal clearly the outline of grain boundaries or interfaces undergoing transformation and a dark field reflection high temperature micro-scope that exposes dynamically the surface relief due to transformation (see Fig. 3).
With this novel system, the microstructural changes during transformation superplastic deformation and the straining behavior were observed directly and con-currently on the same microscopic field of view and were recorded on 16-mm cine-films or 35-mm films by using a motor driven camera.

2. Strain Analysis with Micro-grid Technique
A grid analysis was applied to clarify the charac-teristic deformation behavior associated with trans-

formation superplasticity in a sub-macroscopic scale. The strain distribution in polycrystalline structures can be analyzed by measuring the displacement of each grid point which is inprinted on the surface of specimens by photo-etching technique. The spacing of the fiducial grid points corresponds to a gauge length in strain measurements. In the present study, the interval was selected to be 12.7 µm (2 000 lines/ inch in density) in order to detect the local deforma-

tion behavior at and near the transformation inter-

face. The process of the photo-etching is shown in Fig. 4.
The deformation behavior of specimen was ob-served on the same field of microscopic observation and simultaneously recorded on 35-mm films. In the strain analysis, the displacement of each grid was measured by magnification of the images on films with a laboratory automation system (see Fig. 5). Strain distributions were calculated and displayed on an X-Y plotter.

III. Results

1. In-situ Observation of Microstructural Changes during Ac3 Transformation during Rapid Heating
1. Structural Changes under No Load
In-situ observation of Ac3 transformation during rapid heating was carried out by the apparatus (the dark field reflection high temperature microscope with 35-mm motor driven camera). Figure 6 represents a sequence of films for four temperatures. The results indicate that the initiation of Ac3 transformation oc-

curs by formation of austenite grains at the prior fer-rite grain boundary or triple points at a heating rate of under 20 K/s followed by complete austenitizing. Without loading, the austenite grains are found to grow isotropically. Figure 7 shows the geometrical relationship between the prior ferrite grain boundaries and the contour of resultant austenite grain bound-

Fig. 3. Dark fie id reflection high temperature microscope.

Fig. 4. Technique of photo-etch
specimen surface.

ing of grid pattern on

Fig. 5. Laboratory automation system for g rid analysis.

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aries.
2. Structural Changes under Tensile Loading Transformation superplastic deformation is gener-ated under an applied load. Direct observation of the movement of transformation interfaces was carried out. The results are shown in Fig. Although the transformation proceeds similarly as without load-ing, austenite grains are found to grow anisotropically into ferrite matrix under loading of a tensile stress of

Fig. 6. In-situ observation of Ac3 transformation process at a heating rate of 20 K/ s in pure iron without load-
ing by dark field reflection high temperature micro-
scope.

Fig. 7. Relation between
Ac3 transformation
aries.

austenite grain boundaries after
and prior ferrite grain bound-

Fig. 8.
In-situ observation of Ac3 transformation
process in pure iron at a heating rate of 5 K/s under a tensile stress of 5 MPa.

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5 MPa.

3. Observations of Surface Morphology after Rapid Cooling from Transformation Temperature
When a specimen is rapidly heated to a tempera-ture just above the transformation start (Ac3s) point and then rapidly cooled, both of a-r and r-a trans-formation proceed simultaneously in a limited region of polycrystalline structures. The structure before heating, as shown in Fig. 9(a), exhibits the ferrite grain boundaries after light etching. Figure 9(b) shows the microstructure observed at room tempera-ture after the thermal history. In comparison with Fig. 9(a), variations are found locally in the proximity to ferrite grain boundaries. These variations were shown to be slight surface undulations formed during the cycle, as shown in a scanning micrograph of Fig. 9(c). The morphological changes of surface will be associated with partial austenitizing. Figure 9(d) shows the feature of a-r transformation fronts at an

instant at the temperature Tmax. Hence, the process of austenite grain growth during rapid heating can be determined. To clarify the influence of applied stress on the undulation, further experiments were carried out. Without loading, a slight undulation is found on the specimen surface, as shown in Fig. 10(a). The undulation will correspond to dilatation which is gen-erally useful to analyze the process of phase transfor-mation. Under a tensile stress, transformation super-plastic deformation is generated and simultaneously a remarkable undulation occurs. When the stress is

above the critical stress of superplasticity, i =20 MPa,3~ the induced deformation will be similar to creep deformation"3~ and reliefs caused by slip is no-ticed within grain interior (Figs. 10(d) and 10(e)). On the contrary, if the stress is below the critical stress, slip lines in grain interior can not be found in Fig. 10(c), although the offset at transformation front and slight rotations of each austenite grain are recognized. The fronts of austenite grains migrate anisotropically but smoothly into prior ferrite matrix, and the inter-faces of austenite and ferrite interface appear straight.

2. Strain Analysis of Superplastic Deformation
The relation between dilatation at Ac3 transforma-

tion under rapid heating and superplastic deforma-

Fig. 9.
Morphological changes on specimen surface (a) be-
fore and (b) after heating to a temperature just above Ac3s point under a tensile stress of 20 MPa, (c) same as (b), (d) a-r-a transformed region iden-tified with (a) and (b).

Fig. 10. Morphological changes on the specimen surface after heating to a temperature above Ac3s point
under various tensile stress (a) unloading, ((b),
(c)) 20 MPa, and ((d), (e)) 30 MPa.

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tion behavior under a torsional stress, was investi-gated by using a previously reported apparatus.') Both phenomena are associated with the microstruc-tural changes, as shown in Fig. 11. The process will be classified into the following three stages.
(I) Initial stage : nucleation of austenite grains at prior ferrite grain boundary or
triple point and the subsequent
grain growth
(II) Intermediate stage: the growth of austenite grains
(III) Final stage : coalescence of austenite grains to form stable austenite grain bound-
aries

The strain analyses were carried out at each stage to study how the superplastic straining propagates in polycrystalline structures. The specimen surface was buff-polished and then slightly etched to reveal ferrite grain boundaries. An orthogonal fiducial grid pat-tern with a spacing of 12.7 tm was photo-etched on the specimen surfaces. When the specimen was heated rapidly to l max in the Ac, transformation tem-perature range, the transformation process (I), (1+ II), or (1+11+111) proceeded by accompanying superplastic deformation. On cooling from Tmax, superplastic deformation was induced along with the progress of a--' transformation. After completion of the temperature cycle, the specimen exhibits the traces of transformation fronts produced by the strain generated under partial a-r and r-a transformation at Tmax

1. Straining Behavior at the Initial Stage of Transforma-tion

A specimen was rapidly heated at a rate of 50 K/s up to the temperature just above Ac3s under a tensile

stress, and then rapidly cooled. The thermal and strain histories are shown in Fig. 12(a). Figure 13(a) shows the appearance of the specimen surface; the traces of a-r-a transformation in the vicinity of prior ferrite grain boundaries are represented in dotted zones. With reference to straining behavior, displace-ments are noticed at the fiducial grid points which are located on the prior ferrite grain boundaries. This indicates that grain boundary slide is a predomi-nant process along the prior ferrite grain boundaries. Reference lines are drawn by interpolation the grid points as shown in Fig. 13(b), to make clear the slid-ing behavior. The rotation of each ferrite grain is detected by measuring the angle between a grid line of grain interior and the tensile direction. The rota-tion is found to be a result of the accumulation of

slides along the periphery of ferrite grain. These characteristics in the vicinity of grain boundaries were investigated by a scanning electron microscope. In the microphotographs of Fig. 14, the traces of local-ized transformation along with slip deformation are recognized in the vicinity of prior ferrite grain bound-aries. Especially in Fig. 14(c), the undulated zone along the prior ferrite grain boundary suggests that an extensive slip deformation has taken place in as-sociation with the progress of a-r-a transformation in the area.

2. Straining Behavior at the Intermediate Stage of Trans-formation

Strain analysis was carried out to see the migration behavior of transformation interfaces accompanied with growth of austenite grains. A specimen was rapidly heated at about 20 K above Ac3s point (Fig.

Fig. 11. Relation between the dilatometric change and su-perplastic deformation during Ac, transformation
in pure iron: heating rate=50 K/s; shear stress=
4 MPa.

Fig. 12. Rapidly heated to various temperatures Tmax and rapidly cooled cycle. Induced strain curves cor-
respond with each temperature cycle.

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12(b)) and then rapidly cooled. The surface mor-phology, as shown in Fig. 15 ((a), (b)), exhibits the trace of partial a-r-a transformation and the front of

Fig. 13. Microstructure after rapid heating to a tempera-
ture just above Ac3s and rapid cooling under a
tensile stress of 30 MPa in pure iron : grid points
are spaced at 12.7 µm intervals.

Fig. 15.
Grid analysis
deformation in

of transformation
pure iron.

superplastic

Fig. 14. Microstructure after rapid heating to a tempera-ture just above Ac,s and rapid cooling under a ten-
sile stress of 30 MPa. Growth of austenite grains
and sliding deformation are observed along prior
ferritic grain boundaries.

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transformation at Tmax. The grid displacement was analyzed on Fig. 15 and the results are illustrated in Fig. 16.4 Contour lines of an equivalent strain on the basis of Mises criterion are drawn in Fig. 16(a), and the distribution of principal strain vectors (s1, E2) is represented in Fig. 16(b). The front of transformed region at Tmax, obtained from Fig. 15(d), is shown in the same figure.
The dotted area subjected to a---a transformation is found to be distributed very similarly to the area where the values of equivalent strain are high. This evidence indicates that the expansion of a strained area is accompanied with the growth of austenite grains in polycrystalline structures due to the progress of a-r transformation.

With reference to the distribution of principal strains as shown by the dotted area in Fig. 16(b), the axis of principal strain s is inclined and determined to be about 45° to the direction of transformation front, as shown in Fig. 17((a), (b)). This suggests that the grain boundary sliding mechanism is dominant on the transformation front during instantaneous phase transformation.

Iv. Discussion

Transformation superplasticity is characterized by the m-value of about 1 in the equation c=kEm. When m is 1, the deformation exhibits a Newtonian viscous flow. In a previous paper3) on the micro-grid analy-ses of transformation superplasticity, it was found that: (i) the transformation superplastic deformation was incompressible and homogeneous on the basis of analysis of the trace of grid points during the propa-gation of deformation, and
(ii) the deformation mode was different from the modes off both plastic deformation at room temper-ature and creep deformation at a high temperature. Further analyses of macroscopic deformation were made under tensile stresses and torsional stresses.5>

Fig. 16. Grid analysis of transformation superplastic defor-mation. Distribution of (a) equivalent strain 5eq,
(b) principal strain si and E2.

Fig. 17.
Sweep of a/r interface and the
distribution of induced strain
during Ac, transformation.

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As a result, the three times-law of viscosity, A = 3~ (A: normal viscosity, : shear viscosity), in other equa-tions, Q =3z- or =t/3 (a: effective stress, z : shear stress, : effective strain rate, r: shear strain rate) were found. These findings indicate that transformation superplastic deformation are quite similar in character to viscous flow rather than plastic deformation.
On the other hand, the initial stress is reduced to zero as the transformation proceeds during stress re-laxation tests at a constant torsional strain.6~ This

suggests that liquid-visco-elasticity occurs during the phase transformation, but not firmo-visco-elasticity. The dynamic visco-elastic behavior under transforma-tion superplasticity was confirmed by an experiment of ultrasonic attenuation7~ and the internal friction by means of a torsionally forced vibration method. These facts indicate that a viscous-liquid structure might ap-pear transitorily at an afr transformation interface during transformation.
In the present study, the movement of an a/r trans-formation interface and the straining behavior during transformation were investigated in terms of the char-acteristic deformation in polycrystalline structures. First of all, the transformation process was classified into (I) initial stage, (II) intermediate stage, and (III) final stage, from the dilatometry and superplastic straining behavior.
In the stage (I), austenite grains nucleate at ferrite grain boundaries and triple points. The grains grow along the prior ferrite grain boundaries. In conse-quence, chains of austenite grains are formed to sur-round the prior ferrite grains. If the elastic viscous-liquid structure appears at the a/r interfaces during transformation, the binding force among ferrite grains might be relaxed, resulting in promotion of slides on the interface along prior ferrite grain boundaries and grain rotations and consequently in progress of super-plastic deformation (see Figs. 13 and 14).
In the stage (II), the movement of transformation interface during propagation of austenite grain growth was speculated on the basis of the results in Chap. III. 1 and was represented in Fig. 17(b) along with the distribution of the principal strains. In the figure, the principal strain is found to make 45° to the tan-gent at the instantaneous of interface. This rela-tionship indicates that the superplastic deformation process might be caused by the slip mechanism acting on the transformation interface. As the transforma-

tion proceeds, the transformation interface migrates forward, and while the superplastically strained area might simultaneously expand, resulting in the accu-mulated strain left in the previously transformed re-gion. These deformation mechanism was confirmed in the previous experiments in which the continuous microscopic observation and strain analyses were car-ried out on the superplastic deformation during Ac3 diffusional transformation in a 0.1 % C steel.8~ The

superplastic strain is generated on the sweeping inter-face, but is not generated in the non-transformed fer-rite region as shown in Figs. 10 and 15, and in the previously transformed austenite region.8~ Hence, these facts suggest that the characteristics of macro-

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scopic deformation similar to a Newtonian viscous flow will be due to the characteristics of the sweeping interface. On the other hand, an (a+r) duplex in equilibrium during isothermal soaking induced no superplastic strain.2~ However, above the critical stress of superplastic deformation of 20 MPa,4~ a sig-nificant strain was found to be induced by slip in a grain as shown in Fig. 10. Consequently, the macro-scopic deformation became to be similar to creep de-formation and the m-value became to be lower than 1.

As reported in the previous studies3~ regarding the stage (III), transformation interfaces were brought in contact on migration. Austenite grain boundary was newly formed, and subsequently to develop a stable grain boundary network after grain boundary migra-tion. At the same time, superplastic deformation is caused by the sliding along the austenite grain bound-ary.

With regard to the characteristics of deformation in a polycrystalline structures, inhomogeneous deforma-tion was found from the strain analyses, as shown in Figs. 13 and 16. Generally, to continue deformation without any cracks or voids in a polycrystalline struc-tures, the principal mechanisms such as grain bound-ary sliding or grain rotation should be accompanied with some accomodation mechanisms that can release a local strain concentration and maintain the mor-

phological continuity of aggregates. In ultra-fine grain superplasticity, some accomodation processes were proposed by Ashby and Verral,9~ Gifkins10~ and other investigators.
In the present study, transformation interfaces ex-hibited some distinctive behavior. Under a tensile

load, austenite grain growth was found to progress anisotropically and the morphology of the interfaces tended to be straight in trace, as shown in Figs. 8 and 10. In Fig. 18(a), an austenite grain grows smoothly in one direction. On the contrary, in Fig. 18(b), the surface relief indicates the successive nu-cleation of austenite grains during forward sweeping of the interface during Ac3 transformation. These observations indicate that:

(i) the transformation interface might have a great mobility due to its viscous-liquid structure, and
(ii) in the transformation superplasticity, the trans-formation interface may play a principal role in the deformation mechanism as well as the accomodation mechanism.

V. Conclusion

Microstructural changes during a transformation superplastic deformation process were observed under the specially designed high temperature microscope and strain analyses by a micro grid technique were carried out in pure iron. The results are as follows :
(1) Processes of Ac3 transformation during heating at a rate under 50 K /s include the nucleation of aus-tenite grains at the prior ferrite grain boundaries and triple points, and the subsequent grain growth into the prior ferrite matrix. The growth of austenite grains is not always isotropic under a tensile stress, and an a/r transformation interface tends to be

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straightly aligned.

(2) In the initial stage of transformation, super-plastic deformation is induced by the sliding on al r transformation interface along prior ferrite grain boundaries and the grain rotation, associated with the growth of austenite grains surrounding ferrite grains.
(3) In the intermediate stage of transformation, the sliding deformation is caused on the sweeping transformation interface associated with the progress of austenite grain growth, and the accumulated strain by the sliding is concentrated in the previously trans-formed regions.

(4) These observations indicate that the sliding mechanism on the migrating transformation interface is a principal mechanism governing the transforma-tion superplasticity.

Fig. 18.
Changes of specimen surface relief after heating to a temperature above Ac,s point under a tensile stress of 20 MPa in pure iron.

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2)

3)

4)
5)

6)

7)

8)

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10)

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