Research and Development of Superplastic Materials

— Recent Progresses and Future Prospects —*

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Key words: superplasticity; grain refinement; superplastic forming; internal cavity.

I. Introduction

Superplasticity, as a generally accepted metallurgical concept, is the phenomenon of enhanced ductility under a low resistance to deformation, associated with the microstructural behavior such as slip, twinning, grain boundary migration, phase transformation, precipitation, and recrystallization.

There are now a large number of investigations on the mechanisms and contributing factors of unusually high tensile elongation. Industrial applications of superplasticity have recently been developed for plastic forming. Other applications as the functional material are also in progress, because some of superplastic materials have the ability of diffusion bonding and the high damping capacity.

With the progress of fabrication technology, the extended applications of superplasticity are considered in terms of both intrinsic material factors and external factors such as stress state. Both factors are combined to optimize the ductility and strength for the object of fabrication with respect to materials and processes. Superplasticity can be developed as a novel and important means for microstructural control by thermo-mechanical processing as a combination of mechanical working and heat treatment.

The absence of a satisfactory mechanistic explanation for superplasticity and the related phenomena invalidates the definition of superplasticity. However, grain boundary sliding appears to be the dominant deformation mechanism during superplastic flow on the basis of microstructural observations on abnormal elongation. In the present review on the recent developments and applications of superplastic materials, emphasis will be focused on the fine grain superplasticity that is associated with equiaxed fine grain structure having high angle boundaries.

This review can not be extended to dynamic superplasticity accompanied by phase transformation and transformation-induced plasticity as well as creep behavior proceeding at the state of solid solubility limit or progressing concurrently with recrystallization and aging³.

II. Development of Superplastic Materials

In the early stage of development, superplastic materials were limited to the classical group of materials

that are based on eutectic or eutectoid compositions. These materials are characterized by the microduplex equiaxed grain structure with dispersion of a second phase sufficiently to inhibit grain coarsening during deformation. Findings of these microstructural characteristics lead to the developments of a variety of nonferrous alloys with the base of Ag, Al, Bi, Cd, Co, Cr, Cu, Mg, Pb, Sn, and Zn.

Progresses in grain refinement techniques have provided the various methods of synthesizing materials for attaining superplastic properties. Rapidly solidified powders and films have been found to prepare superplastic alloys with fine but non-equiaxed grain structures. A variety of alloys have been shown to be rendered superplastic: These materials include the alloys of normal grain size with a dispersion of fine second phase particles, those of controlled grain boundary structure and morphology, those exhibiting "temporary superplasticity" under deformation at a relatively high strain-rate to synchronize with the grain growth rate, various types of duplex alloys, age-hardening alloys, dispersion hardening alloys, alloys containing intermetallic compounds as well as single-phase alloys and pure metals. Thus a wide applications of commercial materials are expected as superplastic materials.

Superplasticity has been the subject of recent international conferences: "Superplastic Forming of Structural Alloys" at San Diego, USA, in 1982; "Superplasticity in Aerospace-Aluminium" at Cranfield, U.K., in July 1985; "Superplasticity" at Grenoble, France, in September 1985. Bilateral symposia on superplasticity between China and Japan have been held in Beijing in 1985¹⁾ and in Yokohama in 1986.²⁾

The trends of research and development of superplasticity, as seen in these international conferences, are the rapid progress toward industrial superplastic forming; applications and practical use of Al-Li alloys, Ti-6Al-4V alloys and superalloys as framework components and engine parts for air- and spacecraft. To ensure the reliability for structural use, extensive studies have been made on the cavitation, strength, toughness, and fatigue of superplastically formed materials. The fundamental physical metallurgy of superplasticity has been making progress for the identification of deformation mechanisms. Furthermore, the superplastic forming is now extended to the area of ceramics.

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In Japan, "the Research Group of Superplasticity in Japan" contributes to the effective interchange of information relevant to the research and development of superplasticity. "The Research and Development Institute of Metals and Composites for Future Industry" promotes the research activities of superplasticity, in particular, the development of Ni-base superalloys as heat-resistant materials and Ti-alloys as high toughness materials with high strength to density ratio. The activities have reported and compiled annually in the proceedings.³⁻⁶⁾

III. Grain Refinement for Superplasticity

The present concerns on commercial application of superplastic alloys are focused on the Al, Cu, Fe, Ni, and Ti based systems rather than the classical Zn-Al eutectoid alloy. The current trends of alloy and process design are mainly to attain the reduction of deformation resistance, the strengthening of grain boundary together with grain refinement and the moderate ductility in place of the traditional elongation as large as several thousand percent. The recent trends of de-

sign of superplastic materials and processing are directed toward superplastic forming of the materials of poor workability including intermetallic compounds and ceramics.

One of the authors has reviewed⁷⁾ on the various techniques of grain refinement for rendering superplasticity. These methods include (1) formation of duplex structures by eutectoid reaction, (2) eutectic reaction, (3) static recrystallization and precipitation after heavy deformation, (4) thermomechanical treatments,⁸⁾ (5) optimum thermal cycling, (6) spray atomization, compaction and sintering through cold isostatic pressing (CIP), hot isostatic pressing (HIP), or combined CIP and HIP, (7) spray atomization and collection process (Osprey process), (8) liquid dynamic compaction (LDC) process,⁹⁾ (9) hot extrusion of splat cooled ribbons, and (10) development of modulated structure by spinodal decomposition.

In the following, grain refinement techniques will be described for the typical alloy systems and the superplastic phenomena of the products. Table 1 is a summary of the methods for grain refinement of re-

Table 1. Grain refinement methods for recently developed superplastic alloys.

Alloy	Composition (wt%)	Grain refinement method
High strength aluminum alloy	Al-2.3%Mg-5.58%Zn-1.57%Cu-0.04%Si-0.04%Fe-0.2%Cr-0.05Ti (A7475, 7075)	Hot rolling (400°C)—overaging (480°C, 2 h)—FC—Cold rolling (>70%)—rapid heating (480°C, 5 min)—WQ Grain size: 15–20 μm
Supral 100	Al-6%Cu-0.4%Zr-0.25%Mg-0.1%Fe-0.05%Zn-0.1%Mn Modifications: Supral 210 and 220 with Mg, Si and Ge alloying	Solution treat. (530°C)—overaging (below 400°C)—heavy warm roll. (300°C)—annealing (500°C) Subgrain size: 1~2 μm; equiaxed fine grain structure after superplastic deformation at 500°C
Al-Li alloy	Al-2.5%Li-1.2%Cu-0.7%Mg-0.12%Zr (A8090) Modifications: A8091 and 2090	Solution treat. (560°C, 1 h)—WQ—warm working (300°C)—annealing (530°C, 1 h)—WQ Grain size: 2 μm
NEOPRAL 6/4 brass	Al-5%Mg-0.6%Cu-0.7%Mn-0.15%Cr Cu-40%Zn	Hot rolling—heavy cold rolling—annealing Heating (820°C, 40~60 min)—WQ—cold rolling (90%)—annealing (350~380°C, 1 h) Grain size: α; 3.5~5.1 μm; β; 4.2~4.8 μm
Cu-Al-Ni alloy Ti alloy	Cu-10%Al-6%Ni-4%Fe-1%Mn (C6301) Ti-6%Al-4%V	Hot rolling—heavy cold rolling—annealing Hot rolling (950°C)—cold rolling—annealing Grain size: 1 μm
GT-33 (powder)	Ti-6.5%Al-1.4%V-1.4%Sn-1%Zr-2.9%Mo-2.1%Cr-1.7%Fe-0.11%O	Test stage: Arc melting—hot rolling (850°C) Mass production: Arc melting—He gas atomizing—HIP (800~850°C, 1 h)
SP-35	Ti-3%Al-9.5%V-2.5%Mo	Hot forging (900°C)—hot rolling (880°C)—AC—annealing (780°C, 1 h)—FC—cold rolling (650°C, 2 h)—FC Equiaxed 1~3-μm α grains in β matrix
Mod. IN-100 (powder)	Ni-0.063%C-12.43%Cr-3.4%Mo-18.36%Co-4.27%Ti-4.84%Al-0.053%Zr-0.023%B-0.03%W-0.088%Fe-0.65%V	Ar gas atomizing—HIP (about 1 100°, 900 kgf/cm ² , 1 h)—hot rolling (850~950°C, 70%)—annealing (1 070°C, 1 h)
TMP-7 (powder)	Ni-0.07%C-8.1%Cr-8.3%Co-0.8%Ti-5.0%Al-0.05%Zr-0.01%B-12.4%W-4.5%Ta-0.9%Hf	He gas atomizing—HIP (about 1 100°C, 3 h)—extrusion—(1 150°C, extrusion ratio: 5) Grain size: 2 μm
High strength low alloy steel (HSLA)	Fe-0.15%C-0.19%Si-1.16%Mn-0.014%Al-0.014%Nb-0.019%P-0.006%S	Solution treat. (1 250°C, 1 h)—IBQ—cold rolling (80%)—annealing (865°C, 30 s)—IBQ α+γ microduplex (4 μm) structure
High speed tool steel	Fe-1.25%C-4%Cr-5%Mo-6%W-3%V-8%Co-<0.35%Si-<0.28%Mn-<0.07%Cu-<0.02%P-<0.01%S	Hot rolling (950°C, 78%)—solution treat. (1 240°C, 30 min)—hot rolling (950°C, 78%)—annealing (1 050°C, 5 min) Grain size: 3~5 μm
δ/γ duplex stainless steel	Fe-0.017%C-0.48%Si-0.85%Mn-6.82%Ni-24.66%Cr-2.79%Mo-0.46%Cu-0.28%W-0.025%P-0.001%S-0.143%N	Hot rolling (1 100~1 370°C)—WQ—cold rolling (50%) Elongation depends on the fraction of σ phase (in the case of fine dispersion of γ in δ ferrite (20 μm) matrix).

cently developed alloys. Extensive reviews¹⁰⁻¹³⁾ are also available for Al, Cu, and Ti based alloys and Ni powder alloys.

1. High Strength Aluminum Alloys

Commercially available alloys are Supral 100, Al-Li alloys, and NEOPRAL. Five methods, as shown in Fig. 1,¹⁴⁻¹⁷⁾ have been proposed for the grain refinement of these alloys. These methods utilize the pinning effects of dislocations by fine particles, with a size of about 0.75 μm , which are precipitated from solute atoms such as Cu, Mg, and Zn that are brought into solution in the process of solution treatment and overaging. The dislocations generated in deformation are inhibited to migrate and rearrange. As a consequence, the formation of cell structure and the nucleation of recrystallization grains on the interface of precipitates are promoted to provide the sites of recrystallization nuclei for grain refinement. How-

ever, the resultant grain size is difficult to refine below 12 μm . Therefore, the products are not satisfactory in reliability for the strength, since cavities are formed at a strain 232 % during superplastic deformation. Flow stress of A7475 alloy is known to decrease with decreasing grain size,¹⁸⁾ attempts have been made to find the effective means for refinement such as the use of spray atomized powders. The superplasticity of these alloys is thought to proceed by the mechanism of grain boundary sliding, as suggested in observations on the changes in size and morphology of grains and in preferred orientation. Significant contributions arise from dynamic recovery in the early stage and dynamic recrystallization in the latter stage.¹⁹⁾

The methods for grain refinement of Supral 100 and Al-Li²⁰⁾ alloy are not disclosed in detail. Both alloys may be refined by recrystallization after heavy deformation through the processing of solution treatment and warm working. In the warm working, fine dispersion of metastable Al_3Zr and stable CuAl_2 in Supral 100 and Al_3CuLi_3 (Al_2CuLi for higher contents of Cu) in Al-Li alloys will interact with dislocations to promote nucleation of recrystallization grains for grain refinement. These alloys can be refined further by dynamic recrystallization during superplastic deformation.²⁰⁾

A single phase alloy with the trade name of NEOPRAL has a uniform dispersion of fine particles and is refined by an optimum combination of hot working, heavy cold reduction, and recrystallization to render the superplasticity with an elongation above 700 % in the temperature range between 480 and 530°C.²¹⁾

Fig. 1. Techniques of grain refinement processing for superplasticity in high strength Al alloys.

2. Copper Alloys

Copper-40%zinc alloy, α - β brass, can be rendered a microduplex fine grain structure by the following processing; water quenching from β region to produce Widmanstätten structure, heavy cold reduction, annealing for static recrystallization. The product has a mixed structure of α grains of $3.5\sim 5.1\ \mu\text{m}$ and β grains of $4.2\sim 4.8\ \mu\text{m}$ and the maximum elongation of 460 % in $\alpha+\beta$ two phase region at 600°C .²²⁾ The JIS C6301 type alloy, with a composition of Cu-10%Al-6%Ni-4%Fe-1%Mn, can be rendered superplastic by static recrystallization through the processing of hot working, heavy cold working, and annealing: the flow stress between 2.5 and 20 MPa and m -value between 0.4 and 0.68 for a strain rate in the range of 1×10^{-3} to $1\times 10^{-1}\ \text{s}^{-1}$ in the α and κ two phase region, and the flow stress of 3 MPa and the maximum elongation of 5 500 % for a strain rate of $6.3\times 10^{-3}\ \text{s}^{-1}$ at 800°C when the α - and κ -phases present in nearly equal proportions.²³⁾

3. Titanium Alloys

Research and application of titanium alloys is mainly focused on Ti-6%Al-4%V. The alloy can be refined in the process of hot and cold working and annealing and has a flow stress below 10 MPa in the α and β two phase region for a wide range of temperature and strain rate.²⁴⁾ A high strength modification of Ti-6%Al-4%V, GT-33, has been designed by the National Research Institute for Metals in Japan to stabilize the β phase by addition of V, Mo, Cr, and Fe and to be equal in the proportion of α and β phases at 800°C . The hot rolled sheet of the alloy, produced by the consumable electrode arc melting, has a flow stress of 13 MPa and an elongation of 698 % at a strain rate of $6.7\times 10^{-4}\ \text{s}^{-1}$ at 850°C . The further treatment of heating at 850°C for 1 h and water quenching followed by aging for 4 h in the range between 550 and 600°C has been proved to achieve an elongation above 10 % and a strength to density ratio of $28\ \text{kgf/mm}^2/\text{g/cm}^3$ at a strain rate of $1\times 10^{-4}\ \text{s}^{-1}$ at 300°C , which are the target of commercial application. The mass production can be made in the following process: atomization in He gas, HIP treatment, and superplastic forging at 850°C .²⁵⁾ A newly developed alloy,²⁶⁾ SP-35 with the composition as shown in Table 1, is advantageous over Ti-6Al-4V to be rendered superplastic at lower temperatures: the alloy exhibits superplasticity at $7.24\times 10^{-4}\ \text{s}^{-1}$ in the temperature range between 700 and 750°C .

4. Nickel Alloys

The representative alloy is IN-100 with the composition of Ni-10%Cr-15%Co-5.5%Al-4.7%Ti-3%Mo-1%V-0.18%C-0.06%Zr. The alloy can be formed by the isothermal superplastic forming known as Gatorizing.²⁷⁾ The process developed by Pratt and Whitney uses the gas atomized powders as the starting materials. The powders are canned in a mild steel case and heavily worked by extrusion at $1\ 100^\circ\text{C}$ to produce a fine microduplex structure. The con-

solidated alloys, after further isothermal forging below the recrystallization temperature, have the m -value of 0.5 ± 0.05 in the range of 927 to $1\ 093^\circ\text{C}$.²⁸⁾ As shown in Fig. 2, a modified IN-100 is reported²⁹⁾ to have a maximum elongation of 560 % with the maximum m -value at $10^{-2}\ \text{s}^{-1}$, as a result of grain refinement to an average size of $1.5\ \mu\text{m}$ by the processing of HIP materials through extrusion of 72 % at $1\ 100^\circ\text{C}$ and annealing at $1\ 070^\circ\text{C}$ for 1 h. Moreover the alloy, packed within a case of S35C steel and heated at $1\ 100^\circ\text{C}$, has been found³⁰⁾ to be formed superplastically at a strain rate of $1.8\times 10^{-2}\ \text{s}^{-1}$ with an Inconel 713 die kept at 600°C . The possibility of low temperature superplastic forging saves the use of Ti-Zr-Mo alloy (TZM) die for economy.

The National Research Institute for Metals in Japan has developed an alloy TMP-7³¹⁾ by application of the computer aided alloy design technique. The alloy is processed to refine the microstructure by HIP of the atomized powders and can be superplastically forged at a strain rate of $1.05\times 10^{-3}\ \text{s}^{-1}$ at $1\ 050^\circ\text{C}$. Further solution treatment improves the property to attain nearly the target: a strength of 1 600 MPa and an elongation above 20 % at 760°C .

5. Carbon Steels

Ultra high carbon steels containing 1.6 % C that are processed to have a fine martensitic structure by heating rapidly just above the A_1 temperature with a rate of 100 to 200°C/s and rapid cooling to room temperature are reported³²⁾ to have an m -value of 0.5. Eutectoid steels such as SK-5 and hypoeutectoid steels have been shown^{33,34)} to be processable to fine grain sizes for superplasticity: ferrite grains below $2\ \mu\text{m}$ with a significant volume fraction of cementite below $0.4\ \mu\text{m}$.

Number in (): the total elongation (%)
Fig. 2. Variations of total elongation with deformation temperature and strain-rate in Modified IN-100 sheets.

Fig. 3. Grain refinement processing for superplasticity in ball bearing steel.

6. Alloy Steels

A ball bearing steel, SUJ-2, can be provided with a microduplex structure of ferrite about 0.8 μm and spherical cementite about 3 μm through the thermomechanical and thermal processing shown in Fig. 3 and shows superplasticity of an elongation of 846 % and m -value of 0.33³⁵⁾ at 730°C for a strain rate of $2.8 \times 10^{-3} \text{ s}^{-1}$. A high strength low alloy steel is reported³⁶⁾ to have an elongation of 738 % at 790°C for a strain rate of $8.3 \times 10^{-5} \text{ s}^{-1}$ by preparing a microduplex structure of α and γ both of about 3 μm in grain size through the processing shown in Table 1. Application of the thermomechanical process, illustrated in Fig. 4, to a high speed tool steel produces a microduplex structure for superplasticity as shown in Fig. 5³⁷⁾ and improves the workability. A 25%Cr-7%Ni-3%Mo steel cold rolled 50 % after quenching from 1 250°C shows superplasticity with an elongation of 2 000 % and m -value of 0.5^{38,39)} at 950°C for a tensile strain rate of $2 \times 10^{-3} \text{ s}^{-1}$, because of the concurrent grain refinement associated with precipitation of σ phase during superplastic deformation.

7. Intermetallic Compounds

Polycrystalline intermetallic compounds are generally brittle because of the lack of sufficient slip systems in ordered structure to provide compatibility of deformation at grain boundaries and the segregation of impurities along grain boundaries. Microalloying often improves the ductility of intermetallic compounds at room temperature. Polycrystals of Ni_3Al , which has a crystal structure of L1_2 type and is a microconstituent, γ' , of superalloys, fracture intergranularly in tensile testing without plastic deformation. However, addition of B less than 0.1 % improves the ductility, as shown in Fig. 6.⁴⁰⁾ This may be a result of increased cohesion at grain boundaries.

An optimum combination of deformation temperature and strain rate improves plastic formability of Sendust that has the composition of $\text{Fe}_3(\text{Al}_{0.35}\text{Si}_{0.65})$ and DO_3 type crystal structure with the slip systems equal to body centered cubic crystals. As shown in Fig. 7,⁴¹⁾ the flow stress has strong dependence on strain rate at high temperatures; steady state deformation arises in deformation at 850°C at a strain rate as slow as $1.1 \times 10^{-4} \text{ s}^{-1}$ and as large as 100 %. Addition of silver above the solubility limit in TiAl compounds of L1_0 type crystal structure is studied⁴²⁾ to improve the formability by precipitation of silver along grain boundary.

Fig. 4. Grain refinement processing for superplasticity in high speed tool steel.

Number in (): the total elongation (%)
Number in []: flow stress (MPa)
Fig. 5. Variations of total elongation with deformation temperature and strain-rate in high speed tool steel.

8. Ceramics

Partially stabilized zirconia (PSZ) that contains 3 mol% of Y_2O_3 in solid solution and that has fine grains (about 0.3 μm) of tetragonal crystal structure with a small amount of cubic crystals shows an elon-

Fig. 6. Enhancement of elongation by addition of B in polycrystalline Ni_3Al intermetallic compound.

Fig. 7. Stress-strain curves of polycrystalline Sendust.

gation of above 140 % at 1450°C, as shown in Fig. 8.⁴³⁾ The m -value is $1/1.9=0.5$ in tensile deformation and $1/2.1=0.5$ in compression. During deformation equiaxed grains of the tetragonal crystal are formed. Cavitation occurs in tensile deformation with an excessive strain rate, but it is suppressed in compression. Such a high ductility is considered to permit the forming of rings and pipes from sheet blanks by the use of SiC dies. The PSZ powders are reported⁴⁴⁾ to be consolidated up to the theoretical density by HIP treatment at 1450°C under a pressure of 150 MPa (1500 atm) after preliminary sintering in the range 1200 to 1400°C.

Hot pressed alumina becomes formable in compression by doping of MgO or MgO and Y_2O_3 . Moreover, doping of Cr in alumina permits forming as shown in Fig. 9.⁴⁵⁾ Also alumina base ceramics mixed with $\text{Ti}(\text{C}_{1-x}\text{N}_x)$ has been successfully de-

Fig. 8. Tensile tests of tetragonal zirconia partially stabilized by solid solution of 3 mol% Y_2O_3 . True stress-true strain curves are estimated on the assumption that the deformation progresses uniformly without local necking.

formed at high temperatures.⁴⁶⁾

IV. Mechanisms of Superplasticity

A model of grain boundary slide serves as a first approximation, because the contribution of grain boundary slide is estimated to amount 60 to 80 % of the total superplastic deformation. Figure 10 shows that the contribution of grain boundary slide depends on the strain rate in a single phase Al-Mg alloy with a grain size of about 8.3 μm .⁴⁷⁾ Although the strain rate dependence may vary with the material, composition, grain size, and deformation temperature, the dominant role of grain boundary slide can not be denied. Phenomenologically, superplastic deformation proceeds with grain-switching that the grains are interchanging and rotating, and so not elongating appreciably. However, the size and shape of grains can remain unchanged as a result of thermal activation processes during superplastic deformation such as the generation and movement of dislocations, recovery, precipitation and decompositions of second phases, dynamic recrystallization, and grain growth.

Gifkins⁴⁸⁾ has proposed an approach known as the core-mantle model to take into account the accommodation of grain boundary slide in terms of the motion of grain boundary dislocations. The mantle is the layer of geometrically necessary dislocations and takes part in the grain boundary slide. With a small grain size the mantle dominates overall deformation, and as conditions of stress and temperature favor the operation of grain boundary slide, the width of the mantle decreases. Dislocations moving along grain boundaries pile up at the triple edges of boundaries and dissociate into crystal dislocations in the mantles of sliding grains. The dislocations glide and climb of dislocations in the mantle and induce grain rotation along the neighboring grain boundaries, and eventually they annihilate or recombine to form new grain boundary dislocations. This mechanism confines the deformation to a narrow mantle, therefore the grain switching should be possible.

The model can be extended to the deformation of

Fig. 9. Formability of chromium doped alumina.

ceramics. Viscous slide along grain boundaries is also important in the intermetallic compound of TiAl containing Ag above the solubility limit, as mentioned above, and in the multiphase ceramics containing glassy phase. Sliding along interphase boundaries may proceed in fine ceramics with a composite of multiple phases. If the plastic deformation behavior has the dependence on strain rate, it is considered to be a type of superplasticity.

V. Superplastic Forming

1. Sheet Metal Forming

High ductility of superplastic materials is favorable for stretch forming. For alloys such as titanium alloys that can be joined in the solid state, it is possible to diffusion bond concurrently with superplastic forming, a process termed SPF/DB.⁴⁹⁾ Although superplastic materials have not satisfactory deep-drawability, a heavy drawing can be achieved with application

Fig. 10. Contributions of grain boundary slide, ϵ_{gbs} , slip in grain interior, ϵ_{slip} , and diffusional flow, ϵ_{diff} to the total elongation, ϵ_t , in superplastic deformation of Al-Mg alloy.

Fig. 11. Superplastic deep-drawing with local heating of deforming flange.

of a hydrostatic pressure on the tool side, or with heating of the deforming parts locally to a temperature of superplastic deformation⁵¹⁾ as shown in Fig. 11.⁵⁰⁾

Fig. 12.
Dimension of a disc formed successfully by superplastic forging; the superplastic deformation behavior is simulated numerically.

2. Forging and Extrusion

A block of superalloy of 85-mm diameter and 72-mm height has been successfully forged at 1 050°C into a disc of 150-mm diameter with bosh in an isothermal forging machine of 400-tf capacity.⁵²⁾ The die for forging is made of TZM for practical use. A numerical simulation has been made to analyze the superplastic deformation behavior of superalloys and titanium alloys, when a cylindrical block is forged into the predetermined shape as shown in Fig. 12. The simulation reproduces the distribution of strain, strain rate, and temperature at each element of the deforming material during forging to the finished shape. By selection of the sufficient number of mesh elements, the method is capable to simulate the flow even at the corners where the shape changes in a complicated way. Also the experimental simulation of metal flow can be made by the use of paraffine wax.⁵³⁾ Recently plasticine has been developed to have a various levels of *m*-value.⁵⁴⁾ These simulation techniques are believed to facilitate the effective and efficient design of die and forming operation.

VI. Reliability of Superplastically Formed Materials

In a number of superplastic materials, internal cavities form at the interfaces of second phase particles, at triple grain joints and grain ledges particularly under a state of tensile stress. This cavitation imposes the security of infallible performance in terms of the fatigue, creep, stress relaxation, corrosion, and wear in the structural use of superplastic materials.

Recently, extensive studies have been focused on the frequency of void formation in superplastic forming of high strength aluminum alloys which are difficult to refine the grain size below 10 μm. Figure 13 shows a series of microstructures in A7475 aluminum specimens deformed to failure under various conditions.¹⁹⁾ Microstructural changes in tensile deformation are shown in Fig. 14.¹⁹⁾ Figure 15⁵⁵⁾ shows the dependence of void volume on grain size in superplastic deformation, suggesting that a small number of cavities are formed when the grain size is refined.

Fig. 13. Microstructures in the vicinity of fractured region in superplastic A7075 Al alloy deformed to failure under various conditions.

Fig. 14. Microstructural changes during superplastic tensile deformation of A7475 Al alloy; deformation stage: (a) 10 %, (b) 150 % and (f) 380 % strain.

Figure 16⁵⁵⁾ illustrates the strain rate and temperature effects on cavity volume fraction. Cavitation may be suppressed by superposition of hydrostatic pressure by maintaining a back pressure as shown in Fig. 17.⁵⁶⁾ Imposed back pressure is also effective for enhancement of total and uniform elongations at room temperature.⁵⁵⁾ Figure 18 shows the critical temperature

Fig. 15. Effects of grain size on cavitation tendencies in superplastic A7475 Al alloy.

Fig. 16. Effects of strain-rate and temperature on cavity formation in superplastic A7475 Al alloy.

Fig. 17. Suppression of cavity formation by hydrostatic pressure in superplastic stretch-forming of A7475 Al alloy.

Fig. 18. Mapping of temperature and pressure for restoration of the initial density in superplastically formed products.

perature and high hydrostatic pressure.⁵⁷⁾

VII. Studies for Further Developments

1. Materials and Processing

In preparation of the fine grain structure for superplasticity, uniformity of grain size is often difficult to achieve in bulky materials such as large blocks and plates. In particular, the central layers of thermo-mechanically processed materials are usually hard to refine because of the inhomogeneity of plastic deformation and cooling in the process; a smaller plastic strain and cooling rate in the central layer of plates and strips. Bulky materials may be obtained by powder metallurgy of atomized powders, however, the problems associated with trapping of Ar-gas on the particle surface, retention of the prior particle boundary, and inclusion of the nonmetallic particles should be solved.

2. Workability

In the plastic forming of superplastic materials, the primary importance is the capability of forming into a net shape or near net shape without internal cavitation rather than the extremely high m -value and abnormal tensile elongation. The present knowledge of the mechanism of cavitation and the relationship between grain size and void formation is not sufficient. A guide principle for reduced cavitation is grain refinement in the present. Therefore the methods are expected to be developed for grain refinement below $10 \mu\text{m}$ in high strength aluminum alloys as mentioned above.

Formation of voids is considered to be suppressed or delayed under a state of compressive stress. Quantitative evaluation of the hydrostatic pressure effects is necessary on void formation during superplastic tensile deformation of various materials. Furthermore, the relation between void fraction and plastic strain must be clarified in the deformation under multiple stress states.

Especially these problems should be studied extensively for high strength aluminum alloys in which void

and pressure to restore the initial density after superplastic forming. Formed cavities can be effectively removed by the post treatment of HIP at high tem-

formation depends strongly on the dispersion of spherical precipitates and the imposed hydrostatic pressure.

3. Reliability of Superplastically Formed Products

The presence of fine voids may be inevitable after superplastic forming, even in an extremely grain refined materials. Since additional heat treatments for satisfying the material specification of formed articles may induce grain coarsening and change the microstructure and morphology of microconstituents, these microstructural and morphological changes should be studied for the various types of materials and forming procedures. Furthermore, it is necessary to assess the influence of voids on corrosion and fatigue at high and ambient temperature.

4. Diffusion Bondability

Titanium alloys are sometimes possible to diffusion bond during, before or after superplastic forming, a process as termed SPF/DB. Further exploitation of the SPF/DB method to aluminum and other alloys is expected as a result of the analysis of diffusion bondability which will depend on the grain size.

5. Standardization of Superplastic Testing

The parameter that is commonly selected as a measure of superplasticity is the strain-rate sensitivity of the flow stress, m , in the formulation of plastic strain rate $\dot{\epsilon}$ and flow stress σ , $\sigma = k\dot{\epsilon}^m$. Measurement of the m -value has been performed in several ways. Standardization of the measuring procedure is necessary to characterize the superplastic behavior of various materials on a common basis and to facilitate the discussion and comparison of superplasticity.

6. Development of High Capacity Superplastic Forging Machine

A press machine of 400-tf capacity is necessary to form a test sample of superalloy turbine disc of 150-mm diameter by isothermal forging. Equipment of a high capacity machine is required to form discs of 400-mm diameter with blades.

7. Development of Ceramic Dies

The TZM dies are too expensive for commercial use. Development of long life dies, if necessary of a separate type, made of ceramics by HIP is required for forging of the discs as large as 400-mm diameter. Moreover, optimum lubricants are required for the ceramic dies.

8. Temperature and Strain-rate Controlled Superplastic Forming

Superplastic forming process must be designed to optimize the temperature and strain-rate for the forming materials by reducing the flow stress at lower strains and by suppressing the cavitation at higher strains.

VIII. Summary

Superplasticity, which is expected to take an important part in the field of shaping and fabrication

of metals in the later 1980s, has made a rapid and remarkable progresses in the recent years. In particular, the Research and Development Institute of Metals and Composites for Future Industry in Japan started a project for high performance crystal-controlled alloys in 1981 especially to develop the superplastic materials based on superalloys and titanium alloys. The project is a joint work of industry, university, and government by setting a goal in 1989. Intermetallic compounds and ceramics are included as the objects of superplastic materials.

In the process of research and development, a prefix of "super" is often used. In near future, superplastic materials will be widely applied in commercial production as the materials for plastic forming without emphasis on the characteristics of "super".

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