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REVIEW

Seventy-five years of superplasticity: historic developments and new opportunities

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Abstract On this seventy-fifth anniversary of the first scientific report of true superplastic flow, it is appropriate both to look back and examine the major developments that established the present understanding of superplasticity and to look to the future to the new opportunities that are made possible by new processing techniques, based on the application of severe plastic deformation, that permit the production of fully dense bulk materials with submicrometer or nanometer grain sizes. This review proposes a minor modification to the present definition of superplasticity, it provides an overview of the current understanding of the flow of superplastic metals and ceramics and then it examines, and gives examples of, the new possibilities that are now available for achieving exceptional superplastic behavior.

Introduction

When a polycrystalline material is pulled in tension, it invariably breaks after pulling out to a relatively small strain. However, under some limited experimental conditions the material may pull out to exhibit a very large neck-free elongation prior to failure. These high tensile elongations, often exceeding 1000%, are examples of the occurrence of *superplasticity*. This topic has been the subject of numerous review articles but it is sufficient to note that detailed reports and references to these earlier commentaries may be found in two reports on superplastic flow [1, 2].

Superplasticity is an important field of scientific research both because it presents significant challenges in the areas of flow and fracture and because it forms the underlying basis for the commercial superplastic forming (SPF) industry in which complex shapes and curved parts are formed from superplastic sheet metals. This industry is now more than 40 years old and currently processes thousands of tons of metallic parts for a wide range of industries with special emphasis on the aerospace and automotive sectors but with numerous applications in architecture and consumer products [3].

It is now well established that two basic requirements must be fulfilled in order to achieve superplastic flow [4]. First, superplasticity requires a very small grain size, typically smaller than ~ 10 µm. Second, superplasticity is a diffusion-controlled process operating within the regime of high temperature deformation and therefore it requires a relatively high testing temperature typically at or above $\sim 0.5\ T_m$, where T_m is the absolute melting temperature of the material. In practice, these two requirements tend to be incompatible because grain growth occurs at elevated temperatures in pure metals and solid solution alloys. This means in practice that superplastic metals are generally either two-phase or they contain a fine dispersion of a second phase to inhibit grain growth.

As will be shown in a later section, the current year of 2009 represents the 75th anniversary since the first report of true superplastic flow in the scientific literature. At the same time, very recent developments in the field of materials science are opening up the topic of superplasticity in new and exciting ways and they are providing opportunities for

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achieving exceptional superplastic properties that cannot be attained in more conventional superplastic alloys. Accordingly, it is now an appropriate time to take stock of the present situation and to examine both past developments and future opportunities.

This paper is designed with several specific objectives. First, a brief but general background to high temperature flow and superplasticity is given in the following section. Next, the formal definition of superplasticity, first introduced in 1991, is re-examined in the following section and some additions are proposed that will help to avoid the various mis-interpretations of superplastic flow that have become apparent in the more recent literature. The next section presents a brief summary of the history of superplasticity and summarizes the major scientific advances occurring over the last four decades. Finally, the last section provides a forward look to new developments that suggest there is now an opportunity to achieve unusual and important results within the overall framework of superplastic flow.

General principles of high temperature flow including superplasticity

The rate of superplastic flow is controlled by the rate of diffusion and accordingly the flow process occurs within the general regime of high temperature creep. For flow processes at elevated temperatures, it is now well established that the steady-state strain rate, $\dot{\varepsilon}$, may be expressed by a relationship of the form [5, 6]

$$\dot{\varepsilon} = \frac{ADG\mathbf{b}}{kT}\left(\frac{\mathbf{b}}{d}\right)^{p}\left(\frac{\sigma}{G}\right)^{n} \tag{1}$$

where D is the appropriate diffusion coefficient [$=D_o \exp(-Q/RT)$, where D_o is a frequency factor, Q is the activation energy, and R is the gas constant], G is the shear modulus, $\mathbf{b}$ is the Burgers vector, k is Boltzmann's constant, T is the absolute temperature, d is the grain size, σ is the applied stress, p and n are the exponents of the inverse grain size and the stress, respectively, and A is a dimensionless constant. In practice, therefore, the various potential creep mechanisms are dictated by the appropriate values for Q, p, n, and A.

In the creep literature, it is a standard procedure to plot, on double-logarithmic scales, the measured steady-state strain rate against the applied stress so that the slope of the plot is then equal to n ($=\partial \ln \dot{\varepsilon}/\partial \ln \sigma$). In the superplastic literature, the samples are often tested at constant strain rate and the steady-state flow stress is plotted against the imposed strain rate, again on a double-logarithmic scale, so that the slope of the plot gives the strain rate sensitivity, m ($=\partial \ln \sigma/\partial \ln \dot{\varepsilon}$). In practice, both approaches are equivalent and equally acceptable because the value of m is equal to $1/n$.

Earlier reviews discussed the potential creep mechanisms that may become dominant under different experimental conditions [7, 8]. At very high stresses and strain rates, the linear relationship between strain rate and stress breaks down in the region of power-law breakdown. However, there is generally a very wide range of intermediate stresses where there is a linear relationship between $\dot{\varepsilon}$ and σ with a stress exponent either close to ~ 5 if dislocation climb is the rate-controlling process or equal to 3 when dislocation glide is dominant. The climb and glide of dislocations are sequential processes that occur intragranularly so that $p = 0$ in Eq. 1. Other mechanisms become important at even lower stresses including, for example, Nabarro-Herring [9, 10] and Coble [11] diffusion creep, Harper-Dorn creep [12, 13], and grain boundary sliding [14]. These mechanisms all have low stress exponents and, depending on the mechanism, they may occur intergranularly so that $p > 0$. As will be demonstrated, the regime of superplasticity lies in this region at low stresses. The important requirement in investigations of superplastic flow is therefore to determine the appropriate values for the various parameters in Eq. 1.

The definition of superplasticity

A formal definition of superplasticity was first proposed at the International Conference on Superplasticity in Advanced Materials held in Osaka, Japan, in 1991 (ICSAM-91) [15]:

> Superplasticity is the ability of a polycrystalline material to exhibit, in a generally isotropic manner, very high elongations prior to failure.

This definition is fundamentally correct and it has been cited widely in the superplasticity literature [2]. Nevertheless, the definition does not incorporate any specific minimum tensile elongation that is a necessary prerequisite in order to achieve superplastic flow in metals and in this respect the definition breaks down because some recent reports have claimed the occurrence of superplastic flow under conditions where closer inspection suggests this claim is not justified. In order to understand this difficulty, it is necessary to re-examine the factors influencing the total elongations achieved in tensile testing.

An early analysis of experimental data, published in 1969, showed that the measured elongations to failure increased with increasing values of the strain rate sensitivity, m [16]. The relevant plot is shown in Fig. 1 where this is taken from an earlier report [4] and includes additional experimental data for the exceptionally superplastic Zn–22%Al eutectoid alloy [17] and the Pb–62%Sn eutectic

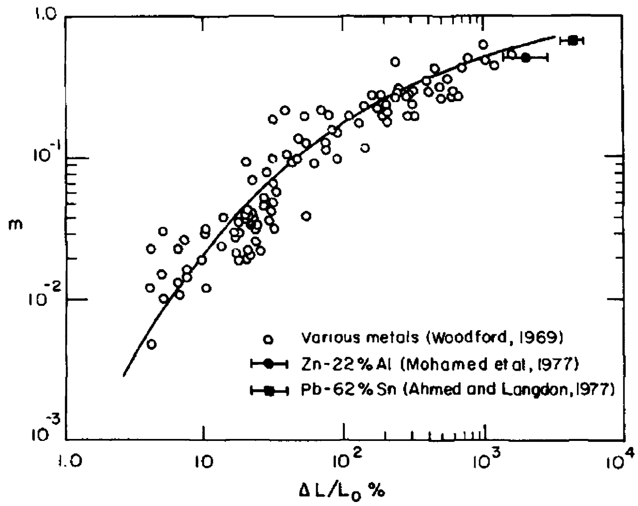

Fig. 1 The variation of the strain rate sensitivity, m, with the elongation to failure, $\Delta L/L_0\%$, for various metals [16]: the illustration is taken from an earlier report [4] and includes data for the highly superplastic Zn–22%Al [17] and Pb–62%Sn alloys [18]

alloy [18]: the plot shows m as a function of $\Delta L/L_0\%$, where ΔL is the total increase in length at the point of fracture and L_0 is the initial gauge length. It is readily apparent from Fig. 1 that a high value of m, and consequently a low value of n, is a necessary criterion in order to achieve superplastic ductilities.

As noted in the preceding section, if the flow process is controlled by dislocation glide then the stress exponent is given by $n = 3$ and the strain rate sensitivity is $m \approx 0.33$. Creep controlled by dislocation glide occurs in solid solution alloys, such as the Al–Mg system, where solute atoms segregate preferentially at dislocations and these solute atmospheres are then dragged by the moving dislocations: the same mechanism has also been designated solute-drag creep in some recent reports. The theoretical model for this process predicts $n = 3$ [19] and this is in excellent agreement with experimental data. Furthermore, theoretical models and experimental data show there may be a transition to dislocation climb with $n \approx 5$ at even lower stresses [20] and the dislocations may break away from their solute atmospheres to give an increase in the stress exponent at high stresses [21]. Nevertheless, and depending upon the solute content, there is usually a reasonably wide range of stress where dislocation glide is the dominant process and $n = 3$.

Reference to Fig. 1 shows that when $n = 3$ and $m \approx 0.33$ the metals will exhibit tensile elongations to failure of the order of ~200–300%. This is consistent with an early report of tensile elongations in an Al–Mg alloy [22] and with several more recent descriptions of flow in solid solution alloys. For example, in an examination of the tensile ductility of Al–Mg alloys it was stated that [23]:

> As a consequence of the high strain rate sensitivity of $m = 0.33$ associated with solute-drag creep, tensile elongations of up to 325% have been achieved. Such elongations are close to those found in superplastic deformation.

Interest in the dislocation glide or solute-drag process has been heightened in recent years by the development, in the General Motors R&D Center, of the Quick Plastic Forming (QPF) technology which was introduced specifically as a hot blow-forming process for the production of high volumes of aluminum panels for use in automotive applications [24, 25]. This technology is based on the utilization of a special grade of the AA5083 Al–Mg alloy having a fine grain size and it is dependent upon operating primarily, although not exclusively, within the regime of dislocation glide. Numerous reports of the flow behavior and fracture properties of this alloy are now available [23, 26–31] but it is important to note that many of these experimental data are not within the regime of true superplasticity. Instead, it is more reasonable to follow earlier studies of the creep behavior of Al–Mg solid solution alloys and to label this type of behavior as examples of either "extended ductility" [32] or "enhanced ductility" [33].

In order to avoid interpreting creep controlled by viscous glide as true superplastic behavior, it is necessary to adapt the earlier definition of superplasticity and to introduce additional experimental requirements that will unambiguously identify superplastic flow. The two most appropriate additional parameters are the measured elongations to failure and the values of the strain rate sensitivity. Thus, experiments on coarse-grained Al–Mg alloys showed that the elongations to failure may be up to and slightly exceeding 300% in flow controlled by viscous glide [34] whereas much higher elongations are attained in a true superplastic condition. This suggests it may be reasonable to adopt a tensile elongation of ~400% as evidence for superplastic flow in metals. Alternatively, the flow mechanism for viscous glide requires that $m \approx 0.33$ whereas in superplastic flow the strain rate sensitivity is $m \approx 0.5$. Making use of these two requirements, it is now proposed that superplastic flow is most readily defined in the following form:

> Superplasticity is the ability of a polycrystalline material to exhibit, in a generally isotropic manner, very high elongations prior to failure. The measured elongations in superplasticity are generally at least 400% and the measured strain rate sensitivities are close to ~0.5.

An alternative possibility is to note that true superplasticity requires a failure of the material by quasi-stable flow in which the tensile specimens pull out to a point so that $A_f/A_i \rightarrow 0$, where A_f and A_i are the cross-sectional areas at the point of failure and within the initial gauge length, respectively [35]. However, this criterion is not a unique distinguishing feature for superplastic flow because many superplastic alloys fail through the development of internal cavitation where the values of A_f/A_i may be relatively high.

The historic developments in superplasticity

Historical considerations suggest the ability to achieve superplastic-like flow in metals was probably understood by artisans in ancient times: examples include the use of arsenic bronzes in Turkey in the early Bronze Age, the development of Damascus steels from 300 BC to the late nineteenth century [2, 36] and the introduction of Wootz steels in ancient India [37]. Nevertheless, it was only in the twentieth century that the first scientific reports began to appear documenting the potential for achieving relatively high tensile elongations in metals.

The first such report is generally attributed to Bengough [38] in 1912 where an elongation of 163% was achieved in a brass and later to experiments by Jenkins [39] in 1928 where elongations of ~300% were reported in Cd–Zn and Pb–Sn alloys. Nevertheless, these elongations fail to meet the definition of superplastic behavior proposed in the preceding section and in this respect the first unambiguous report of superplasticity may be traced to the work of Pearson [40] which was published exactly 75 years ago in 1934. In this latter work, Pearson achieved a remarkable tensile elongation of 1950% in the Pb–Sn eutectic alloy and then coiled the broken specimen for easy photography as shown in Fig. 2.

Pearson's experiments were conducted in the UK but nevertheless they attracted little or no attention in the west. However, the research was continued by Bochvar and Sviderskaya [41] in the Soviet Union and they used the Russian word *sverkhplastichnost′* (meaning "ultrahigh plasticity") in the title of a paper describing their results with Zn–Al alloys. Indeed, it was the subsequent translation of this Russian title in Chemical Abstracts in 1947 that introduced the word *superplasticity* into the English language [42]. Additional developments in the Soviet Union included the publication of the first book on superplasticity by Presnyakov [43], first published in the Russian language in 1969 but later translated into English for publication in 1976, and the founding in 1985 of the Institute of Metals Superplasticity Problems of the Russian Academy of Sciences in the city of Ufa in the western Urals region of Russia. This institute was, and remains, the only institute in the world devoted exclusively to research in the field of superplasticity and scientists from this institute have made many important scientific contributions in the superplastic literature [44].

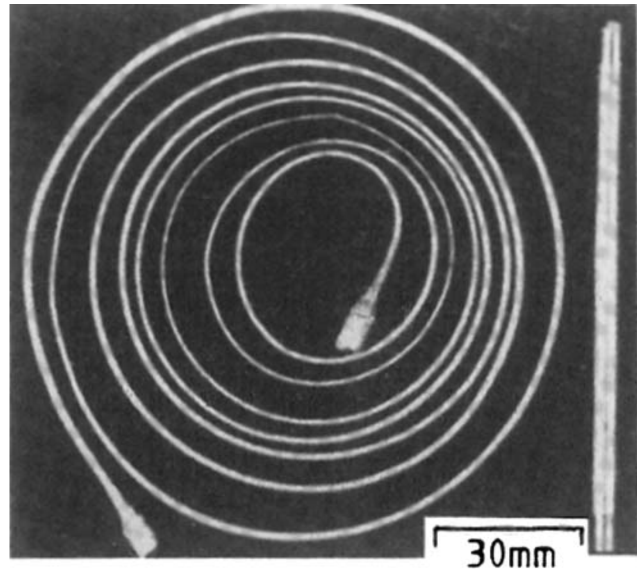

Fig. 2 The first scientific demonstration of true superplasticity showing an elongation of 1950% in the Pb–Sn eutectic alloy: the broken specimen is coiled for easy photography [40]

The re-introduction of superplastic research in western countries was prompted by a review in 1962 of the Russian research by Underwood [45]. This led to the introduction of a research program on superplastic flow and forming at M.I.T. [46] and the gradual dissemination of interest and research in superplasticity around the world. However, there was only a very limited understanding of superplastic flow at that time and in the subsequent years critical experiments were undertaken to provide a comprehensive and consistent understanding of the principles of superplasticity. The following sections review some of these more important developments which have led to an overall understanding of the superplastic phenomenon.

The variation of flow stress and tensile ductility with strain rate

In high temperature creep, double-logarithmic plots of strain rate against stress generally display separate flow regions having lower values of the stress exponent, n, with decreasing stress [7, 8]. However, in early experiments on superplastic materials there appeared to be a division in the experimental evidence between two different trends. On the one hand some experiments suggested that, as in conventional creep data, there was a transition to n close to 1 at the lowest stresses [47, 48] whereas on the other hand some experiments suggested a transition to a higher value of n at the lowest stresses [49–51].

This apparent dichotomy may be checked by conducting separate tensile tests on a series of specimens over a wide range of strain rates and then plotting the results in the form of the measured elongations to failure and the flow stress against the imposed strain rate. This result is shown in Fig. 3 for a superplastic Zn–22%Al eutectoid alloy where the upper plot shows the elongation to failure, $\Delta L/L_0\%$, and the lower plot shows the flow stress, σ, as a function of the initial imposed strain rate, $\dot{\varepsilon}$ [52]. These results demonstrate the occurrence of three distinct regions of flow with superplastic elongations up to >2000% occurring in region II over a limited range of intermediate strain rates spanning about two orders of magnitude. Although these maximum elongations are very high, even larger elongations to failure, up to a maximum of 7550%, were subsequently reported for the Pb–62%Sn eutectic alloy [53]. It is apparent from Fig. 3 that within region II the strain rate sensitivity is $m \approx 0.5$ and the elongations to failure are high, whereas at low strain rates in region I and at high strain rates in region III the strain rate sensitivities decrease to $m \approx 0.2$ and there are corresponding reductions in the tensile elongations in these two regions. Thus, the variation of ductility with the value of m matches the behavior anticipated from Fig. 1.

The results in Fig. 3 were subsequently augmented by obtaining sets of similar data on the Zn–22%Al eutectoid alloy at different testing temperatures [17]. Thus, these and other results provide clear evidence for the division of flow

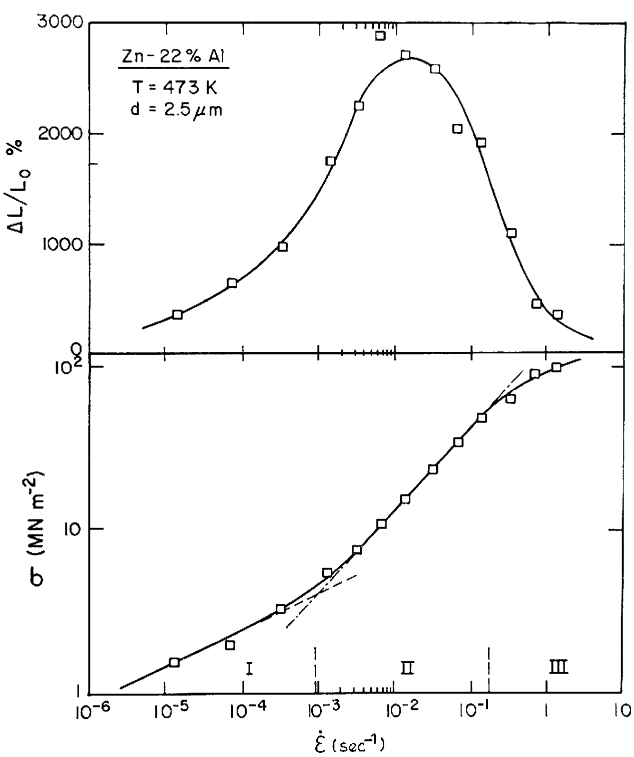

Fig. 3 Elongation to failure (*upper*) and flow stress (*lower*) plotted against the initial strain rate for a Zn–22%Al eutectoid alloy [52]

into three discrete and well-defined regimes labeled I, II and III. In addition, the erroneous reports of a high value of m at the lowest strain rates was attributed to a failure to allow for the occurrence of a primary stage of creep in creep testing experiments [54].

The significance of the three regions of flow in superplastic materials

As described in an earlier report [55], any understanding of superplasticity must account for the three distinct flow regions visible in Fig. 3. Very early experiments in superplasticity showed that the grains move over each other in the superplastic region II and grain boundary sliding is therefore an important, and possibly dominant, flow process in this region [56]. At higher stresses, the transition to region III where $m \approx 0.2$ and $n \approx 5$ corresponds to the anticipated transition to an intragranular dislocation climb process [5, 7, 8]. It is less easy to explain the transition to region I at low stresses with $m \approx 0.2$ because generally it is anticipated that the stress exponent decreases, and therefore the strain rate sensitivity increases, at lower levels of the applied stress.

An early proposal suggested that region I was due to a threshold stress which arose from fluctuations in the grain boundary areas [57] but this model is based on diffusion paths which are physically unrealistic and therefore it must be rejected [58]. An alternative and more realistic interpretation was later developed where region I was attributed to the segregation of impurity atoms at the grain boundaries and the consequent interaction between these impurities and the moving boundary dislocations that contributed to grain boundary sliding [59, 60]. The pinning of boundary dislocations by impurity atoms leads to a temperature-dependent threshold stress and the predictions are generally consistent with the experimental evidence.

Additional data are now available supporting this interpretation of region I. Figure 4 shows the experimental results obtained on two different grades of the Zn–22%Al alloy where the data are plotted as the shear strain rate, $\dot{\gamma}$, against the shear stress, τ, for tests conducted using double-shear samples; the solid points are for an alloy containing 180 ppm of impurities and the open points are for a very high purity alloy containing only 6 ppm of impurities [61]. It is apparent that both alloys give identical results in region II and in the transition to region III but at low stresses there is a transition to region I in the lower purity material but there is no evidence for the presence of region I in the very high purity alloy. Similar results were also reported in other investigations [62, 63] and extensive investigations were subsequently conducted to evaluate the influence of impurities on grain boundary sliding during superplastic flow [64–67].

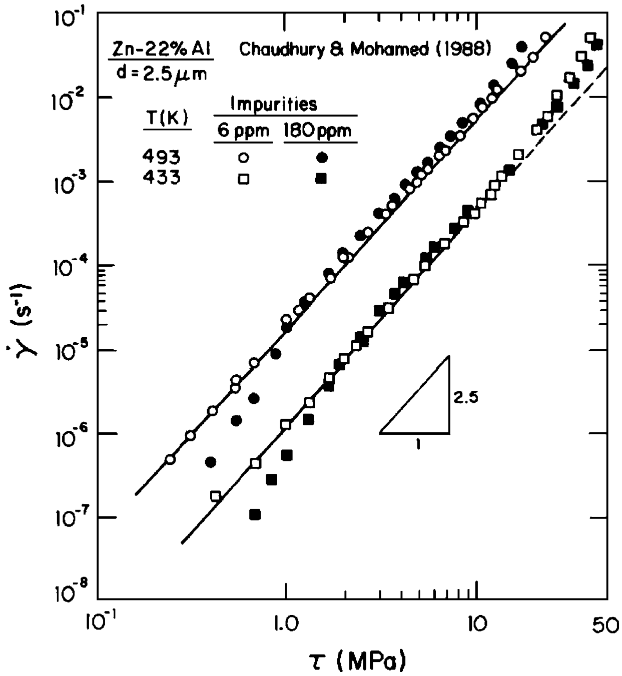

Fig. 4 Shear strain rate versus shear stress for Zn–22%Al alloys tested with two different impurity levels: the behavior of the two alloys deviates in region I at low stresses [61]

The significance of cavitation in superplasticity

Although early experimental evidence suggested that grain boundary sliding plays a major role in superplastic flow, there was a general consensus that the occurrence of exceptionally high elongations to failure was indicative of an absence of the development of any internal cavities. Thus, an early review of superplasticity noted explicitly that void formation was not observed in superplastic metals [68]. Nevertheless, careful experiments on a commercial Zn–22%Al eutectoid alloy, containing ~190 ppm of impurities, showed this view was incorrect and instead very extensive internal cavitation was visible even in a specimen pulling out in region II to a total elongation of ~2600% [69]. Later experiments showed there was also significant levels of cavitation in a high purity Zn–22%Al eutectoid alloy containing only ~15 ppm of impurities [70]. The results from these two sets of experiments confirmed the importance of cavitation in materials undergoing superplastic flow even under conditions where the tensile samples exhibit exceptionally high elongations.

In these early experiments on the Zn–22%Al alloy, the internal cavities were visible in the form of cavity stringers aligned essentially parallel to the tensile axis. A later investigation was conducted using a superplastic quasi-single phase copper alloy containing a dispersion of Co-rich particles and in this material it was demonstrated that the cavity stringers were consistently aligned along the prior rolling direction (RD) [71]. This effect is visible in Fig. 5 where the samples in (a) and (b) have the rolling directions lying either horizontal along the tensile axis or perpendicular to the tensile axis, respectively. In Fig. 5a there is considerable cavity interlinkage near the fracture tip but away from the tip the cavity stringers are clearly aligned along the tensile axis. By contrast, in Fig. 5b the stringers lie parallel to the rolling direction and essentially perpendicular to the tensile axis. Additional information is also available on the formation of cavity stringers in two-phase alloys [72, 73].

It is well established that the growth of cavities under conditions of high temperature creep occurs either through absorbing vacancies in diffusion growth [74] or by plastic flow in the surrounding matrix in plasticity-controlled growth [75]. However, the situation changes for superplastic materials because the grain size is exceptionally

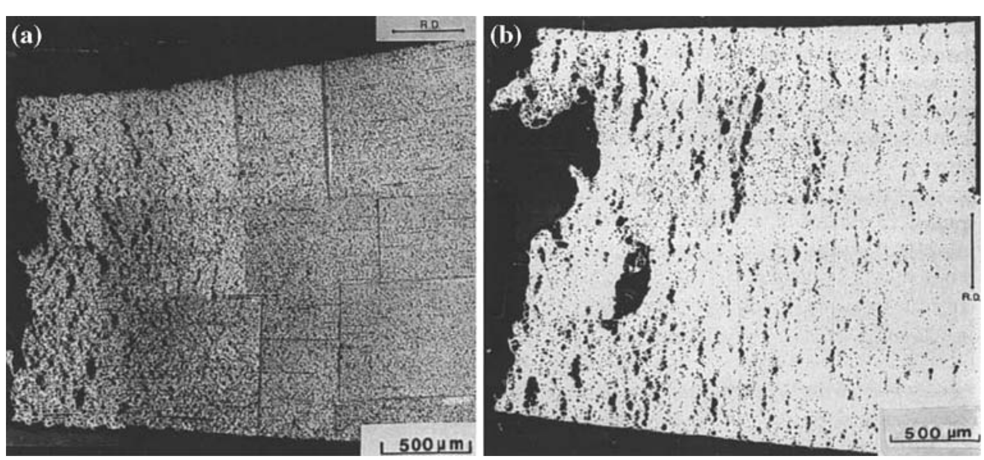

Fig. 5 Examples of internal cavitation in a superplastic quasi-single phase copper alloy containing a dispersion of Co-rich particles: the cavity stringers consistently align parallel to the prior rolling direction which lies along the tensile axis in (**a**) and perpendicular to the tensile axis in (**b**) [71]

small and this means the cavity size may exceed the grain size so that several boundaries impinge upon a single cavity. This then leads to enhanced vacancy diffusion along these multiple boundary paths, thereby giving the process of superplastic diffusion growth [76]. Calculations show this growth process leads to a condition in which the rate of change of the cavity radius with strain is independent of the instantaneous cavity radius and inversely proportional to the square of the grain size. In practice, this growth mechanism becomes important at lower strain rates and when the specimen grain size is smaller than ~ 5 μm.

The development of internal cavitation in superplastic metals presents a potential limitation on the use of these materials for superplastic forming operations. However, it is quantitatively possible to measure the extent of any internal cavitation using a nondestructive procedure based on photo-acoustics [77, 78].

A comprehensive review is available describing many of the features associated with cavity development in superplastic materials [79].

The nature of the flow mechanism in the superplastic region II

Very early experiments on the Zn–22%Al alloy established the importance of grain boundary sliding in superplastic flow [56]. A theoretical model was developed in which groups of grains in reasonable alignment slide together as units to form stress concentrations at obstacles such as triple points. These stress concentrations are accommodated by dislocation slip within the blocking grain and the consequent pile up of intragranular dislocations at the opposite grain boundary. The rate-controlling process in the model is then the removal by climb into the grain boundary of dislocations at the head of the pile-up. This model predicts a strain rate of the form given in Eq. 1 with $A \approx 12$ [80], $n = 2$, $p = 2$, and $D = D_{gb}$, where D_{gb} is the coefficient for grain boundary diffusion. Almost identical relationships were also derived in later modifications of this basic model with the same values for n, p, and D but with values of A of ~ 2 [81] and ~ 64 [82].

Since this type of model predicts accommodation of the sliding process through the intragranular movement of dislocations by slip, it is important to confirm the occurrence of slip during the superplastic process. This confirmation was achieved in two different ways. First, transmission electron microscopy was used with a superplastic copper alloy to show that during deformation in region II there is an accumulation of matrix dislocations in coherent twin boundaries within the grains [83]. Second, the intragranular strain was measured directly in a superplastic Pb–62%Sn eutectic alloy using scanning electron microscopy at elongations up to a total of 800% [84]. These measurements revealed the occurrence of a non-uniform and oscillatory intragranular strain within the Sn and Pb phases but with this intragranular strain making no net contribution to the total elongation of the specimen.

Using this approach, it is possible to develop a unified model for grain boundary sliding both under creep conditions when the grain sizes are large and in superplastic conditions when the grain sizes are very small. Subgrains are formed within the grains during dislocation climb in the non-superplastic region III and these subgrains have a well-defined average size, λ, which varies inversely with the applied stress for both metals [85] and ceramics [86]. Calculations show that the transition from region III to the superplastic region II occurs approximately at the condition where $d \approx \lambda$ so that subgrains are not formed in superplastic flow [87]. Thus, this difference in microstructure permits the development of a unified model for creep and superplasticity, as depicted schematically in Fig. 6 [6].

Under creep conditions with large grain sizes and $d > \lambda$, as given in Fig. 6a, a stress concentration at the triple point A is accommodated by intragranular slip which impinges on the subgrain boundary at B. By contrast, superplastic conditions with small grain sizes and $d < \lambda$, as given in Fig. 6b, leads to a stress concentration at the triple point C and accommodation by intragranular slip which impinges on the opposing grain boundary at D. For superplastic conditions in region II with $d < \lambda$ this model leads to Eq. 1 with $A \approx 10$, $n = 2$, $p = 2$, and $D = D_{gb}$ whereas for conventional creep conditions with $d > \lambda$ the model leads to Eq. 1 with $A \approx 10^3$, $n = 3$, $p = 1$, and $D = D_\ell$ where

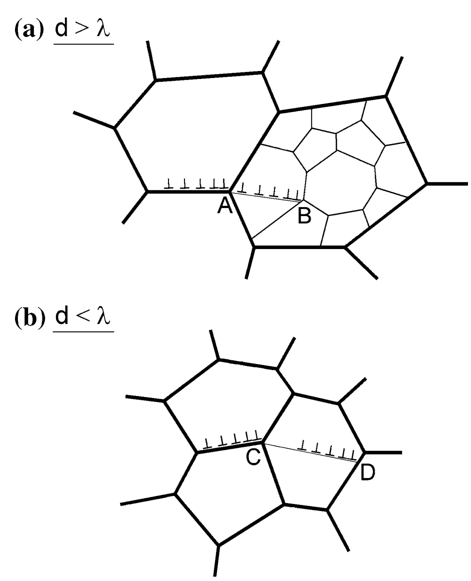

Fig. 6 Schematic illustration of a unified model for grain boundary sliding in **a** conventional creep when $d > \lambda$ and **b** superplasticity when $d < \lambda$ [6]: the principles of the model are described in an earlier report [14]

D_ℓ is the coefficient for lattice self-diffusion [14]. The result for superplasticity is therefore consistent with the earlier calculations [56] but the result now also incorporates grain boundary sliding under conventional creep conditions.

The contribution of grain boundary sliding to the total strain

Numerous procedures have been developed for measuring the contribution of grain boundary sliding under creep conditions [6, 88, 89] and the same techniques may be used for materials deformed under superplastic conditions. These procedures lead to a contribution of grain boundary sliding to the total strain, ξ, which is given by

$$\xi = \frac{\varepsilon_{gbs}}{\varepsilon_t} \tag{2}$$

where ε_{gbs} and ε_t are the strain due to sliding and the total strain, respectively.A comprehensive summary of the values reported for ξ in superplastic alloys showed that the measured values were generally $\sim$50–70% in the superplastic region II but with significantly lower values of ξ in regions I and III [90]. These and other similar measurements [91] suggest there is a "missing strain" of the order of $\sim$30–50% in region II which is not directly due to grain boundary sliding. However, this interpretation is incorrect because very careful analysis reveals limitations in the experimental procedures used to measure the sliding contributions. Specifically, it can be shown that the measured sliding contribution will be $\sim$45–90% even when all of the deformation occurs by sliding and the associated accommodation mechanism [92]. Furthermore, since the accommodation of sliding is less severe at the specimen surface, estimates of the sliding contributions from surface marker lines will tend to lie nearer the lower end of this predicted range. Accordingly, the analytical evidence demonstrates that grain boundary sliding accounts for essentially all of the strain occurring under the optimum superplastic conditions in region II despite measured values of ξ which are consistently in the range of $\sim$50–70% [92].

The occurrence of superplasticity in ceramics

It is well known that ceramic materials are inherently brittle and therefore it is reasonable to anticipate they will not exhibit superplastic elongations. Nevertheless, tensile elongations of >100% were reported for a 3 mol% yttria-stabilized polycrystalline tetragonal zirconia [93, 94], termed 3Y-TZP, and this initiated widespread research into the flow and fracture of potentially superplastic ceramic materials and to the publication of an early review on superplasticity in ceramics [95]. Although these early

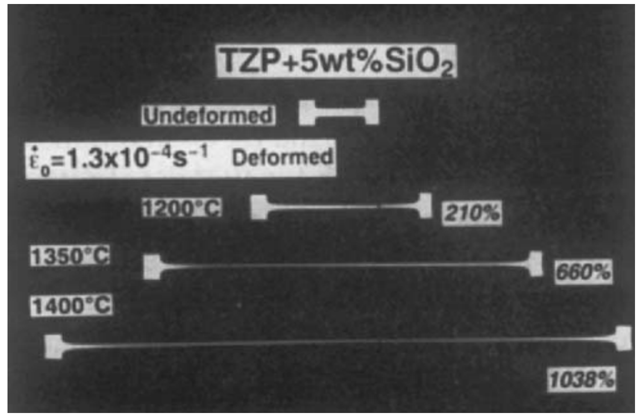

Fig. 7 Examples of exceptional superplasticity in a polycrystalline tetragonal zirconia doped with 5% silica [97]

reports documented only relatively modest elongations by comparison with superplastic metals, later investigations demonstrated the ability to achieve much larger elongations to failure. Examples of these high tensile elongations include $\sim$1050% without failure at a strain rate of 0.4 s^{-1} in a ZrO_2–Al_2O_3-spinel composite [96] and 1038% in a polycrystalline tetragonal zirconia doped with 5 wt% silica when testing at 1673 K at a strain rate of 1.3×10^{-4} s^{-1} [97]: the latter sample is shown in Fig. 7 together with other samples tested under different conditions. Later experiments on the ZrO_2–Al_2O_3-spinel composite gave a remarkable tensile elongation of 2510% when testing at 1923 K at a strain rate of 8.5×10^{-2} s^{-1} [98].

Many of the characteristics of superplastic flow in ceramic materials are similar to those already documented for metals. For example, there is often the development of damage by cavitation in superplastic ceramics [99–101]. There are also examples where the flow properties in a ceramic are interpreted, as in metals, in terms of a model of grain boundary sliding accommodated by intragranular dislocation slip: an example includes results obtained on an alumina-zirconia-mullite composite [102].

Nevertheless, there are some potential difficulties in understanding the flow mechanisms in superplastic ceramics because, although superplasticity is interpreted in terms of the occurrence of grain boundary sliding, it is now well established that sliding cannot occur in a polycrystalline material without the development of an accommodation process in the form of dislocation slip within the adjacent grains. This process of accommodated sliding forms the basis for the fundamental superplastic mechanism as shown in Fig. 6b and it means in practice that any interpretation of superplasticity in terms of grain boundary sliding, as proposed for the 3Y-TZP ceramic when testing at 1673 K [103], must also allow for the occurrence of intragranular slip. However, there are questions concerning whether intragranular dislocation movement is a viable mechanism in

3Y-TZP at the low stresses used to achieve superplasticity at a temperature of 1673 K [104]. Calculations suggest there is little or no dislocation slip in fine-grained 3Y-TZP at the low stresses associated with superplastic flow [105, 106] and instead the flow behavior under superplastic conditions may be interpreted in terms of Coble diffusion creep controlled by movement of the Zr^{4+} ions with interface-controlled diffusion creep leading to an increase in the stress exponent, and a consequent decrease in m, at the lowest stresses in region I [107]. The proposal of interface-controlled diffusion creep is based on an earlier concept developed for metals [108] and it provides an excellent prediction of the creep and superplastic behavior reported experimentally for 3Y-TZP over a range of temperatures and grain sizes [109].

Future opportunities in superplasticity

As noted earlier, superplastic flow is achieved in metallic alloys having grain sizes smaller than $\sim$10 μm [4]. This means that, in traditional practice, superplastic materials are prepared by thermo-mechanical processing and the alloys typically have grain sizes of $\sim$3 to 5 μm. Very recent developments have demonstrated the potential for producing bulk fully-dense metals with grain sizes that are much smaller than those used in conventional superplastic testing. These new developments are based on the processing of metals through the application of severe plastic deformation (SPD) in which the materials are very heavily strained through the introduction of a high density of dislocations but without incurring any significant changes in the overall dimensions of the samples [110]. The subsequent re-arrangement of these dislocations into low-angle boundaries and their further evolution into high-angle boundaries forms the basis for the production of materials with exceptionally small grain sizes.

Several different SPD processing procedures are now available but the two procedures receiving most interest are equal-channel angular pressing (ECAP) [111] and high-pressure torsion (HPT) [112]. In ECAP the sample, in the form of a short rod or bar, is pressed through a die constrained within a channel that is bent within the die through a sharp angle often, but not always, equal to 90°. In HPT the sample is generally in the form of a thin disk, similar to a coin, and it is placed between heavy anvils and subjected to an applied pressure and concurrent torsional straining. Very recent investigations have explored the potential for applying HPT to larger bulk samples in the form of short cylinders [113, 114]. The procedures of ECAP and HPT are both capable of producing exceptional grain refinement but typically the materials processed by ECAP have ultrafine grain sizes within the submicrometer range whereas HPT may produce submicrometer materials or materials having a true nanometer grain size where the latter is defined as a grain size of <100 nm [115].

A very early investigation showed the potential for achieving superplastic-like properties in materials processed using SPD procedures. In an experiment on an Al–4% Cu–0.5% Zr alloy, an elongation of 250% was achieved in a sample processed by HPT with an initial measured grain size of $\sim$0.3 μm [116]. In the following two sections, examples are presented for superplastic flow in materials processed by ECAP and HPT, respectively.

Superplasticity achieved through processing by ECAP

As noted in an earlier section, the strain rate in the superplastic region II is consistent with Eq. 1 with an exponent for the inverse grain size of $p = 2$ [14]. This led to the early recognition that a reduction in grain size by an order of magnitude, typically from $\sim$ 3 μm to $\sim$ 300 nm, should increase the range of strain rates associated with superplasticity by two orders of magnitude [117]. This means it may be possible to use SPD processing in order to achieve high strain rate superplasticity where this is defined as superplastic flow occurring at strain rates at and above 10^{-2} s^{-1} [118].

The first direct example of this effect was in 1997 when high strain rate superplasticity was achieved in two commercial aluminum alloys with elongations up to >1000% at a strain rate of 1.0×10^{-2} s^{-1} [119]. Subsequent more detailed experiments on an Al–3% Mg–0.2% Sc alloy gave exceptional superplastic elongations at very rapid strain rates as shown in Fig. 8 where the open points were obtained after processing by ECAP at room temperature and testing in tension at different temperatures and the solid points are for samples processed by cold rolling and tested in tension at 673 K [120]. These results confirm the potential for achieving high strain rate superplasticity and they demonstrate also that the same results cannot be achieved through cold rolling. Additional experiments showed that this Al–Mg-Sc alloy exhibits almost identical superplastic properties when samples are cut in three orthogonal directions within the as-pressed billets [121].

The superplastic forming industry uses sheet metals and therefore it is important to determine whether the superplastic properties attained by ECAP are also achieved if the billets are cold-rolled (CR) into sheets. The experimental results are shown in Fig. 9 using the same Al–Mg-Sc alloy as in Fig. 8 and they confirm that the superplastic properties are retained when the as-pressed billets are rolled into sheets [122]. This result demonstrates the potential for using ECAP in the production of materials for use in the superplastic forming industry. Furthermore, experiments showed that simple blow-forming may be used for the

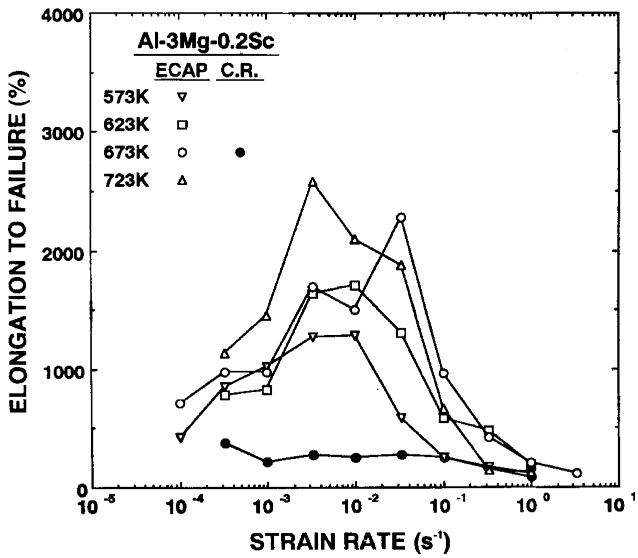

Fig. 8 Elongation to failure versus strain rate for samples of the Al–3% Mg–0.2% Sc alloy processed by ECAP at room temperature and then pulled to failure at different temperatures: the solid points are for specimens of the same alloy prepared by cold rolling and tested at 673 K [120]

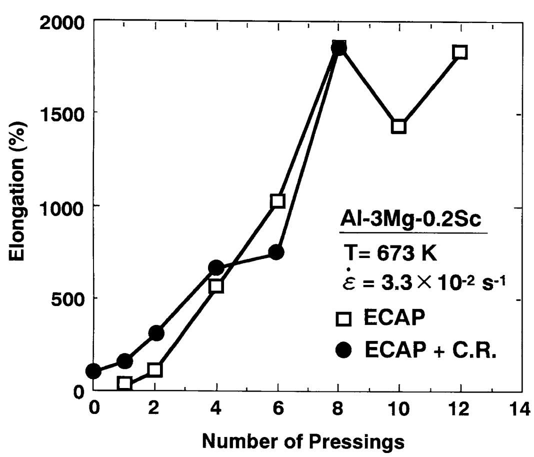

Fig. 9 Elongation to failure versus number of passes for an Al–3% Mg–0.2% Sc alloy processed either by ECAP or by ECAP and cold rolling into a sheet [122]

rapid production of domes using disks cut from the as-pressed billets. These tests were conducted using the same Al–Mg–Sc alloy and disks were cut from the billets, inserted into a biaxial gas-pressure forming facility and then formed rapidly into domes at a temperature of 673 K using a gas pressure of 1 MPa [123]. The result is shown in Fig. 10 where the disk on the left was cut from the billet without blow-forming and the other two disks were subjected to a gas pressure for 30 and 60 s, respectively. Measurements of the local thicknesses around the domes confirmed that the thinning was reasonably uniform which

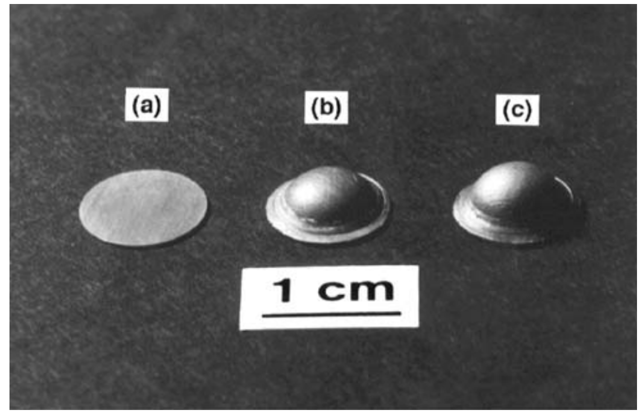

Fig. 10 Domes produced by blow-forming in disks of the Al–3% Mg–0.2% Sc alloy cut from billets processed by ECAP: the disk at (*a*) shows the initial condition without blow-forming and the disks at (*b*) and (*c*) were held at 673 K under a gas pressure of 1 MPa for 30 and 60 s, respectively [123]

is consistent with the high strain rate sensitivity in the superplastic regime.

A recent comprehensive review provided a tabulation of all of the results available to date documenting superplastic elongations in metals processed by ECAP [124]: these metals include a range of Al, Cu and Mg alloys plus the Zn-22% Al eutectoid alloy.

Although processing by ECAP may be used for the production of superplastic magnesium alloys, these materials generally require the introduction of a preliminary grain refinement through extrusion [125, 126]. This two-step process, termed EX-ECAP, is capable of producing excellent results, especially when combined with other modifications in the processing procedures. For example, a combination of extrusion and ECAP, together with the use of an ECAP die having an internal angle of 135°, produced superplastic elongations of up to ~1780% in a Mg–8%Li alloy [127]. An example of exceptional superplasticity in a commercial magnesium alloy is shown in Fig. 11 for a ZK60 Mg–5.5% Zn–0.5% Zr alloy processed by extrusion and ECAP with a die angle of 90° and then pulled to failure at 473 K to give an elongation to failure of 3050% [128]. This result is the highest tensile elongation achieved to date for a magnesium-based alloy processed and tested under any conditions including ECAP and it provides a very clear demonstration of the remarkable results that may be

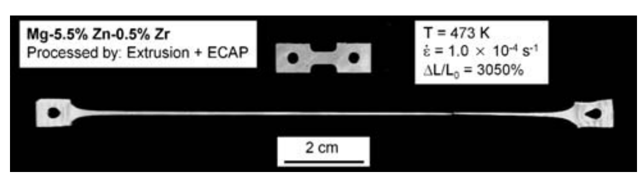

Fig. 11 An exceptional elongation of 3050% recorded in a commercial extruded ZK60 magnesium alloy after processing by ECAP [128]

attained through SPD processing. High elongations were also achieved in this alloy over a range of testing conditions [129] and strategies were developed for achieving high strain rate superplasticity in magnesium-based alloys [130].

Superplasticity achieved through processing by HPT

Several reports are now available documenting the occurrence of true superplastic elongations in disk samples processed by HPT: examples include ~620% in a magnesium AZ61 alloy [131], ~750% in an Al-1420 alloy [132], and 810% in an Mg–9% Al alloy [133]. There is also a report of a tensile elongation of 1510% in a specimen of an Al–3% Mg–0.2% Sc alloy cut from an HPT ring sample [134]. Nevertheless, and despite the relatively smaller grain sizes produced in processing by HPT, these elongations are lower than those generally reported when processing using ECAP.

It is probable that the explanation for these lower elongations lies in the very small thicknesses of the gauge sections within the miniature tensile specimens that are cut from the HPT disks or rings. This trend would be consistent with the very early recognition that the measured elongations in failure are dependent upon the precise shape and configuration of the test specimens [135]. To check this possibility, recent experiments were conducted on bulk HPT samples of the Al–3% Mg–0.2% Sc alloy using small cylinders with heights and diameters of 8.57 and 10.0 mm, respectively [136]. Figure 12 shows an example of the exceptional superplasticity achieved in this alloy using a very small tensile specimen, with a gauge length of 1 mm, cut from a central horizontal section through the cylinder. For this condition, the measured grain size was ~130 nm and the elongation to failure was 1600% when testing at 573 K using a strain rate of 3.3×10^{-3} s^{-1}. This elongation to failure is the highest reported to date in any sample processed by HPT.

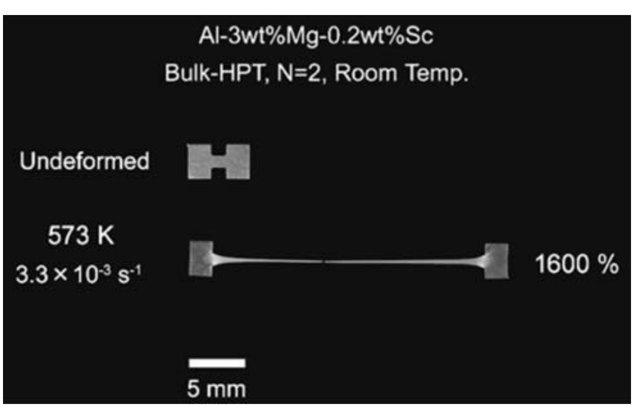

Fig. 12 An example of superplasticity in an Al–3% Mg–0.2% Sc alloy processed by HPT and pulled to failure at 573 K [136]

Summary and conclusions

1. The first scientific report of true superplastic behavior in metallic alloys occurred 75 years ago with a reported elongation to failure of 1950% in the Pb–Sn eutectic alloy. Since that time, numerous experimental investigations have led to a detailed understanding of the flow and fracture properties of superplastic materials. In addition, the field of superplasticity has expanded to also include ceramic materials.
2. The formal definition of superplasticity was first proposed in 1991. Minor changes in the definition are now required to avoid mis-interpretations of superplasticity under conditions where the rate-controlling mechanism in solid solution alloys is dislocation glide and the dragging of solute atmospheres. An alternative definition is now proposed.
3. The fundamental framework of superplastic flow is well understood in conventional metallic alloys where the grain sizes are typically ~3–5 μm. However, there are new opportunities for achieving exceptional superplastic results in materials processed by severe plastic deformation, as in equal-channel angular pressing or high-pressure torsion, where the grain sizes are typically smaller than in conventional alloys by at least an order of magnitude.
4. The small grain sizes produced by these new techniques introduce the possibility of achieving superplastic forming capabilities at very high strain rates. Examples of this effect are now available but more investigations are needed to fully explore this possibility.

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