New developments in the processing of metallic alloys for achieving exceptional superplastic properties

Chuan Ting Wang[1,a], Zheng Li[2,b], Jing Tao Wang[2,c], Terence G. Langdon[3,d,*]

[1]School of Mechanical Engineering, Nanjing University of Science and Technology, Nanjing 210094, China

[2]School of Materials Science and Engineering, Nanjing University of Science and Technology, Nanjing 210014, China

[3]Materials Research Group, Department of Mechanical Engineering, University of Southampton, Southampton SO17 1BJ, U.K.

[a] ctwang@njust.edu.cn, [b] lizheng@njust.edu.cn, [c] jtwang@njust.edu.cn, [d] langdon@soton.ac.uk

Keywords: Equal-Channel Angular Pressing, High-Pressure Torsion, Severe Plastic Deformation, Tube High-Pressure Shearing, Ultrafine-Grained Materials

Abstract. The process of superplasticity has a long history dating back to the early experiments of Pearson conducted in the U.K. in 1934. Since that time, superplasticity has become of increasing importance because of the recognition that superplastic forming provides a simple procedure for the processing of complex and curved parts for use in a wide range of industrial applications. The fundamental requirement for superplastic flow is a small grain size typically smaller than ~10 µm. These fine grains were achieved traditionally through the use of appropriate thermo-mechanical processing which provided a procedure for developing microstructures having grain sizes of the order of a few micrometers. Over the last two decades the processing procedures have been further developed through the use of techniques based on the application of severe plastic deformation (SPD) where it is possible to achieve ultrafine-grained materials with grains sizes in the submicrometer or even the nanometer range. Early SPD experiments were conducted using the processes of equal-channel angular pressing or high-pressure torsion but more recently a new and improved technique was developed which is known as tube high-pressure shearing (t-HPS). Experiments show that t-HPS provides a capability of producing exceptional superplastic elongations with, for example, an elongation of ~2320% in a Bi-Sn alloy when tested at a strain rate of 10^{-4} s^{-1} at room temperature. This report examines these recent developments with an emphasis on the potential for improving the superplastic capabilities of metallic alloys.

Introduction

When metals are pulled in tension, they generally break at relatively low elongations of the order of ~50-100% or less. However, some interesting and unusual results started appearing at the beginning of the last century with a report in 1912 of an elongation of 163% in brass [1] and a later report in 1928 of elongations of ~300% in Cd-Zn and Pb-Sn alloys [2]. In order to firmly place these results in perspective, it is necessary to have a unique and unambiguous definition of the meaning of superplasticity. This definition was achieved by critically examining an early plot of the strain rate sensitivity, m, against the elongation to failure for a very large number of metals, where m is defined as $\partial \ln \sigma / \partial \ln \dot{\varepsilon}$ where σ is the applied stress in creep experiments or the measured flow stress in conventional tensile testing and $\dot{\varepsilon}$ is the measured steady-state strain rate in creep or the strain rate imposed on the sample in tension [3]. This plot was published in 1969 and it showed that the elongation increased with increasing values of m so that a high value of m is a critical requirement for high elongations in tension.

To definitively establish a value of m for superplastiuc flow, it is first necessary to examine the meaning of the strain rate sensitivity and this requires an examination of the different flow mechanisms that may occur during the plastic deformation of metals.

Flow mechanisms in metals during plastic deformation

When flow occurs in metals tested in either creep conditions of constant stress or in conventional tensile testing, the strain rate may be expressed by a relationship of the form [4,5]

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

where D is the diffusion coefficient, G is the shear modulus, **b** is the Burgers vector, k is Boltzmann's constant, T is the absolute temperature, d is the grain size, A is a constant having a value of ~10, p is the exponent of the inverse grain aize and n is the stress exponet which is equal to $1/m$. Inspection shows that, under any selected testing conditions, the only variables in Eq. 1 are A, D, p and n. Therefore, in order to use Eq. 1 in superplasticity, it is necessary to conduct experiments to determine the values of these various terms.

As discussed in detail in other reports [6,7], the earliest descriptions of superplastic properties provided inconclusive results because tests were conducted in tension at a fixed strain rate at an elevated temperature in order to measure the flow stress and then the same sample was used to measure additional flow stresses at other strain rates without removing the sample from the testing facility. This procedure is expeditious but it fails to take account of the grain growth which will occur during the testing procedure. Accordingly, a set of careful experiments was conducted on the Zn-22% Al eutectoid alloy where each flow stress was measured on a different sample at the same tempetrature of 473 K but different strain rates and all samples had the same initial grain size of 2.5 µm [8]. Later experiments were conducted at the additional temperatures of 423 and 503 K and the full results are shown in Fig. 1 where each datum point was obtained from a different sample, ΔL is the total increase in length and L_o is the initial length of each sample [9].

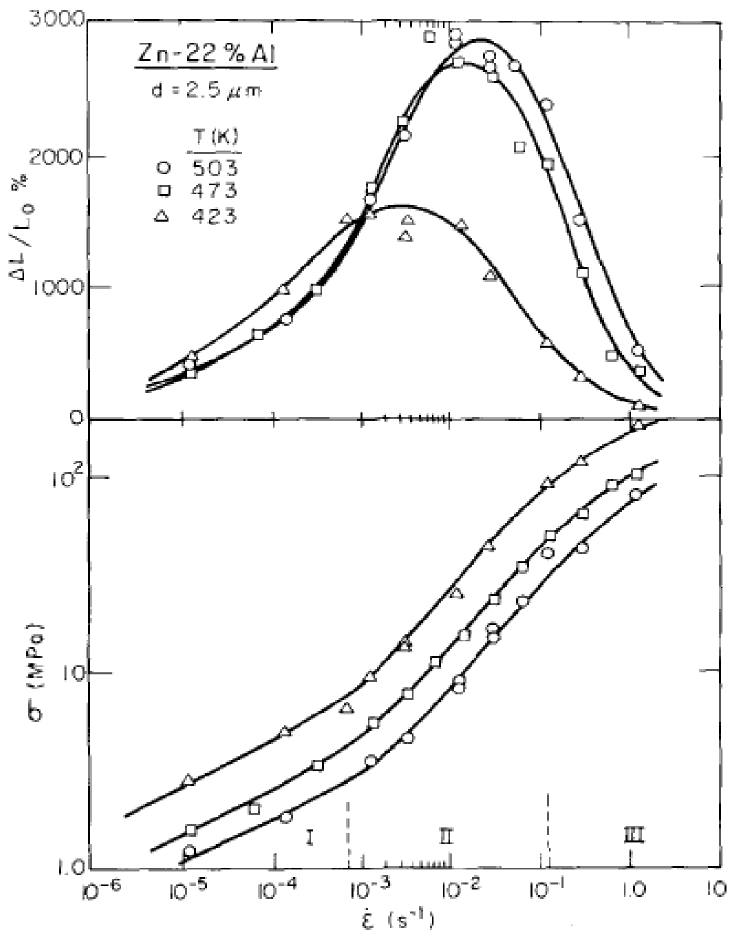


Fig. 1 Elongation to failure (upper) and measured flow stress (lower) plotted against the imposed strain rate for the Zn-22% Al eutectoid alloy [9].

It is readily apparent from inspection of Fig. 1 that all experimental points are mutually consistent. High superplastic elongations occur at intermediate

Materials Research Proceedings **32** (2023) 3-14 https://doi.org/10.21741/9781644902615-1

strain rates in the area labelled region II and there are lower elongations at both slower strain rates in region I and faster strain rates in region III. Typically, the high elongations occur over about two orders of magnitude of strain rate and the slopes of the lines in Fig. 1 indicate strain rate sensitivities of about ~0.2 in regions I and III and ~0.5 in region II.

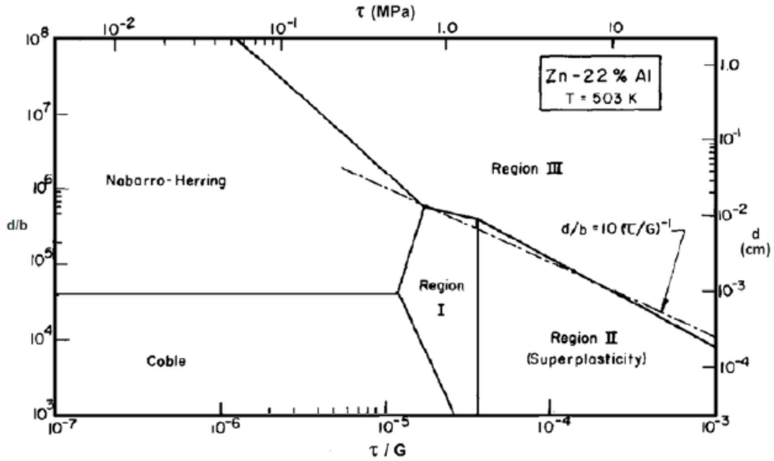


Fig. 2 Deformation mechanism map for Zn-22% Al alloy at 503 K showing the region of superplastic flow in grain size-stress space [10].

The optimum procedure for obtaining a simple visual presentation of these results is to use the data to plot a deformation mechanism map of the normalized grain size against the normalized stress. A map was constructed using the results obtained at a temperature of 503 K and it is shown in Fig. 2 where region II is the area of superplastic flow in grain size-stress space, regions I and III are the areas of lower elongations and τ is the shear stress [10]. The map in Fig. 2 also includes the theoretical predictions for diffusional creep where vacancies flow through the lattice in Nabarro-Herring creep [11,12] and along the grain boundaries in Coble creep [13]. It is apparent that at higher stresses there is no superplasticity when the grain size is large but as the grain size is reduced there is a sharp transition from the non-superplastic region III to the superplastic region II.

To obtain a better understanding of this transition, it is important to note that a very wide range of creep date, covering many different metals, shows that subgrains are formed within the grains under normal creep conditions and the average size of these subgrains, λ, varies inversely with the applied stress through a general relationship of the form [14]

$$\frac{\lambda}{\mathbf{b}} = B\left(\frac{\tau}{G}\right)^{-1} \qquad (2)$$

where B is a constant having a value of ~10. Similar relationships were also reported for ceramic [15] and geological [16] materials. Setting the grain size equal to the subgrain size so that $d = \lambda$, Eq. 2 is plotted as a broken line in Fig. 2 where this line is in remarkably good agreement with the boundary separating the fields for regions II and III. This plot shows unambiguously that superplastic flow requires a refined grain size that is sufficiently small that no subgrain boundaries are formed within the grains.

When a material pulls out to a very large superplastic elongation, the grains remain essentially equiaxed but they move with respect to each other so that the major flow process in superplasticity is grain boundary sliding [17]. Nevertheless, sliding cannot occur in isolation and it must be accommodated by some limited intragranular slip to prevent the opening of voids within the material. This suggests a mechanism for sliding as depicted schematically in Fig. 3 [18]. Thus, sliding under conventional creep conditions is shown in Fig. 3(a) where $d > \lambda$ so that sliding produces a stress concentration at the triple point A, dislocations move into the next grain and then pile up at the first subgrain boundary at B and climb into the boundary. By contrast, when $d < \lambda$ in superplasticity, the stress concentration at the triple point C generates dislocations in the next grain which cross the grain and then pile up and climb into the opposite grain boundary at D. By modelling these two processes it was shown that the mechanism of superplasticity leads to Eq. 1 with $A \approx 10$, $D = D_{gb}$ for grain boundary diffusion and p and n both equal to 2. Excellent experimental evidence is now available demonstrating the occurrence of an accommodating intragranular slip during superplastic flow and this provides direct support for this model [19-22].

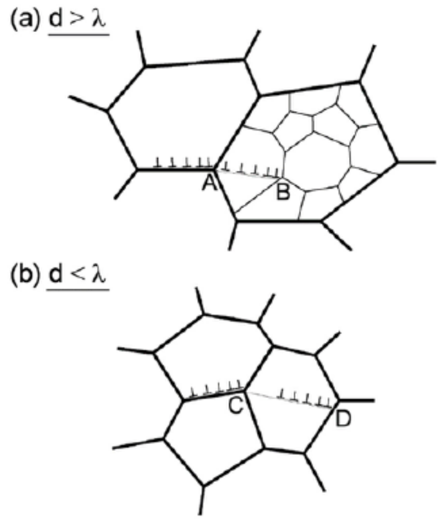


Fig. 3 Grain boundary sliding in (a) creep with $d > \lambda$ and (b) superplasticity with $d < \lambda$ [18].

In conventional creep, flow generally occurs by a dislocation climb process where the diffusion coefficient is the value for lattice self-diffusion, $n \approx 4.5$ and $p = 0$. However, in solid solution alloys the rate of glide may become slower than the rate of climb because of the segregation of solute atoms around the moving dislocations and in these conditions the diffusion coefficient is the value for interdiffusion of the solute with $n = 3$ and $p = 0$. This means that the strain rate sensitivity in creep controlled by dislocation glide is $m \approx 0.3$ and this is quite high so that, since the elongation increases with increasing values of m, dislocation glide will lead to some reasonably high elongations prior to failure. It is important, therefore, to establish a criterion for superplasticity that avoids any confusion with metals deforming by the viscous glide process. Accordingly, a careful analysis of published data was conducted for the elongations achieved in metals deforming by dislocation glide and it was found that these elongations may be as high as, and even exceed, a value of 300% [23]. For example, there is a report of an elongation of 325% in an Al-Mg alloy deforming by dislocation glide with a measured strain rate sensitivity of $m = 0.33$ [24]. Based on extensive analysis of published data, superplasticity was formally defined as a measured elongation of at least 400% and a strain rate sensitivity close to $m \approx 0.5$ [23].

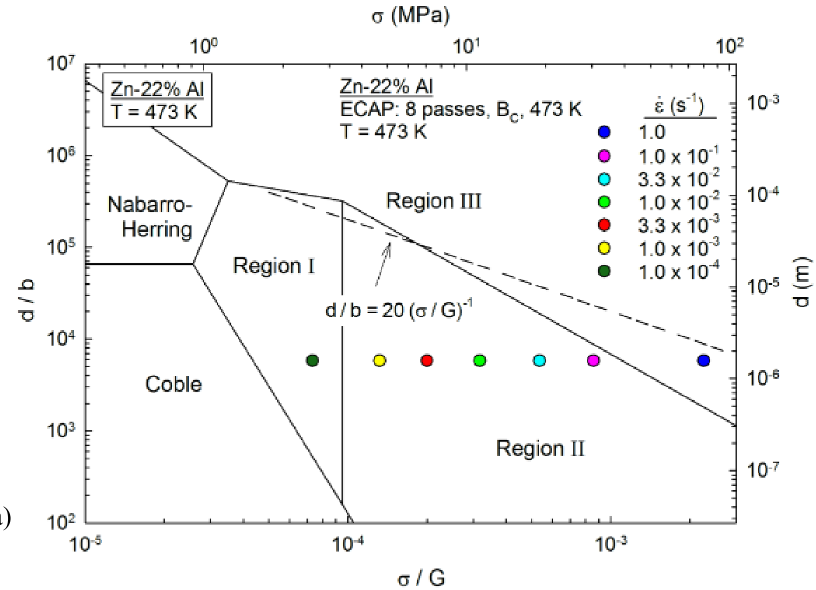
Recent developments in the superplasticity of metals

Superplasticity has become an important flow process over the last two or three decades, mainly because of the development of industrial superplastic forming processes which provide the opportunity for fabricating relatively complex curved shapes from sheet metal. The potential uses of superplastic forming are very wide ranging from automotive applications to consumer products and various uses in architectural design. This processing is now employed annually to physically form many thousands of tons of sheet metals [25] and some reviews are available describing these developments as superplasticity has progressed from a laboratory curiosity to a viable and valuable industrial tool [26,27]. There are several recent developments within the field of superplasticity but four of these developments have important implications and they will be reviewed briefly in this report.

First, superplasticity requires a small grain size that is typically below ~10 μm and, because it is a diffusion-controlled process, the testing should be performed at an elevated temperature that is generally above ~$0.5T_m$ where T_m is the absolute melting temperature. These characteristics were discussed in detail in an early review [6]. Materials suitable for superplastic testing were traditionally fabricated using thermo-mechanical processing but this has a limitation because it is difficult or even impossible to achieve grain sizes smaller than ~2-5 μm. A classic publication in 1988 showed that it was possible to use a new processing technique, based on the application of severe plastic deformation (SPD), to achieve a submicrometer grain size in an Al-Cu-Zr alloy [28]. This led to widespread use of SPD in the processing of ultrafine-grained (UFG) metals and the various processing procedures for achieving significant grain refinement in bulk solids were described in a detailed review [29]. Basically, there are two major SPD procedures and these are equal-channel angular pressing (ECAP) where a rod or bar is pressed through a die constrained within a channel that is bent through a sharp angle within the die [30] and high-pressure torsion (HPT) where the sample, generally in the form of a thin disk, is subjected to an applied pressure and concomitant torsional straining [31]. In practice, processing by HPT has an advantage over ECAP because it produces materials having smaller grain sizes [32] and a higher fraction of high-angle grain boundaries which contributes to easier grain boundary sliding during plastic flow [33]. Finally, an advantage of producing UFG microstructures is that it leads to the occurrence of superplastic flow at high strain rates [34] and this is advantageous for use in superplastic forming operations [35].

Second, reports of superplasticity have been extended to new materials such as high-entropy alloys (HEAs). The first report was a superplastic elongation of 1240% in an AlCoCrCuFeNi alloy processed by multiaxial forging [36] and there were later reports of superplasticity in a CoCrFeNiMn HEA [37] and an elongation of up to 2000% in an $Al_9(CoCrFeNiMn)_{91}$ (at.%) alloy [38]. A comprehensive review is now available summarizing the properties of the traditional CoCrFeNiMn HEA [39]. More recently, results have become available showing the occurrence of superplastic elongations in multi-principal element alloys (MPEAs) [40,41].

Third, an important requirement in attaining superplastic results for any selected alloy at a fixed temperature is to summarize the data in a simple format so that predictions are then possible concerning the limitations of the superplastic regime in terms of stress and grain size. This is most easily accomplished by using the experimental data to construct a deformation mechanism map of the type shown earlier in Fig. 2. Within these maps, it is advantageous to include the predicted regimes for the Nabarro-Herring and Coble creep mechanisms since these processes are well-established with good theoretical relationships. The development of deformation mechanism maps, and the various types of maps that are available, were discussed in an earlier report [42]. Examples of this approach are given in Fig. 4 where results are plotted for a Zn-22% Al eutectoid alloy tested at a temperature of 473 K after processing by (a) ECAP for 8 passes at 473 K and (b) HPT for 1 to 5 turns at room temperature (RT) under an applied pressure of 6.0 GPa [42]. These maps show the lines for the subgrain size based on Eq. 2 and all experimental points lie within the correct fields.



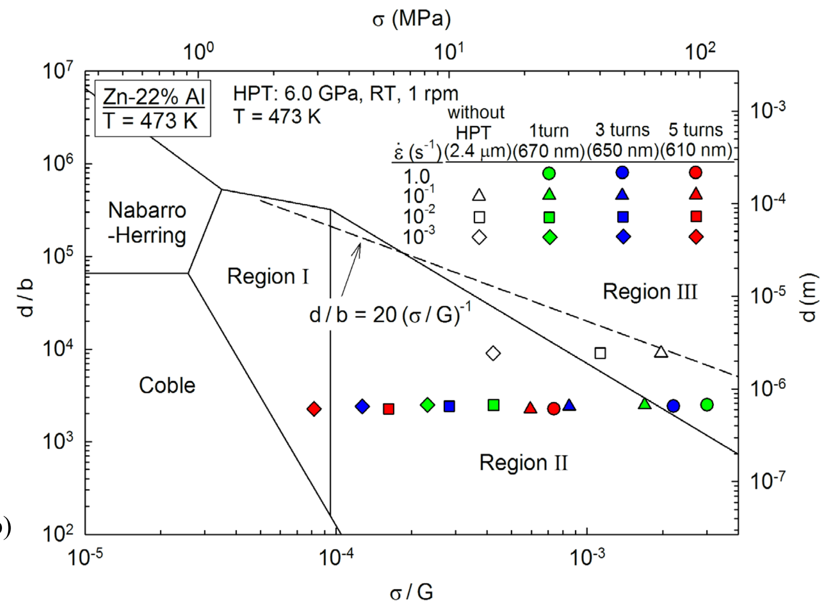


Fig. 4 Deformation mechanism maps for Zn-22% Al tested at 473 K after processing by (a) ECAP and (b) HPT [42].

Fourth, calculations show that the relationship developed earlier for superplastic flow [18], based on grain boundary sliding with intragranular accommodation, is in excellent agreement with experimental data available for both Al and Mg alloys tested after processing by either ECAP or HPT [43]. Specifically, in plots of the temperature and grain size compensated strain rate against the normalized stress, the experimental datum points lie close to, and scatter about, the predicted values for the strain rate in superplasticity. However, this is a direct test of the validity of this approach at elevated temperatures where the relationship incorporates the occurrence of rapid diffusion and a negligible supersaturation of vacancies. In order to extend this same analysis to flow at low temperatures, it is necessary to modify the relationship to permit a supersaturation of vacancies. This approach was used effectively in a recent model which led to good agreement with experimental data for several different metals [44] and, more recently, a high-entropy alloy [45]. A very recent comprehensive analysis demonstrates that this approach is in good agreement with more than 2300 individual datum points taken from the literature covering multiple materials over a very wide range of grain sizes, strain rates and temperatures [46].

The introduction of a new procedure for achieving grain refinement and superplasticity

The preceding analysis emphasized the importance of the SPD procedures of ECAP and HPT which are now widely used to achieve very significant grain refinement. Nevertheless, an alternative procedure was developed recently and preliminary experiments show this procedure has a capability of producing exceptionally high superplastic elongations.

This new procedure is tube high-pressure shearing (t-HPS) where a sample, in the form of a tube, is subjected to shearing under the action of a hydrostatic pressure. A detailed description of this procedure was presented earlier [47-49] and a schematic illustration is shown in Fig. 5 where (a) is conventional HPT for a solid disk, (b) is a torsion/twisting process for a tube and (c) is the new process of t-HPS as applied to a tube: in Fig. 5 the radius is r, the height is h and the angle of rotation is θ. Inspection shows that HPT and torsion/twisting are similar processes because in both cases a vertical line scribed parallel to the axis on the outer surface will be sheared into a curve as indicated by the broken line whereas a radial line on the upper surface will remain straight. Conversely, in t-HPS the outer surface of the tube is rotated so that a vertical line will remain parallel to the tube axis during deformation but a radial line will shear into a curve as shown by the broken line in Fig. 5(c).

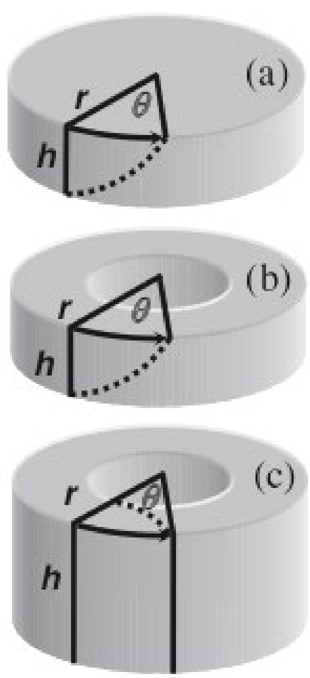


Fig. 5 Principles of (a) HPT, (b) torsion/twisting and (c) t-HPS [47].

Experiments were conducted where t-HPS was applied to alloys of Pb-40% Sn and Pb-62% Sn (wt. %) by constraining these alloys within an outer cylinder under a hydrostatic pressure of 1.0 GPa and with the outer cylinder rotated up to 40 or 50 turns for these two alloys, respectively; this gives equivalent true strains for these alloys of ~1600 and ~2000, respectively, thereby providing a good mechanical mixing between the Pb and Sn [50]. Full details of the grain sizes for the α and β phases and the measured α/β phase volume ratios were given for the alloys in the as-cast, rolled and t-HPS conditions in a tabulation presented earlier [50]. However, as an example, for as-cast, rolled and t-HPS the α phase sizes were ~2.3, ~2.0 and ~0.7 μm in the Pb-62% Sn alloy and ~1.0, ~1.8 and ~0.8 μm in the Pb-40% Sn alloy, respectively. Tensile specimens were

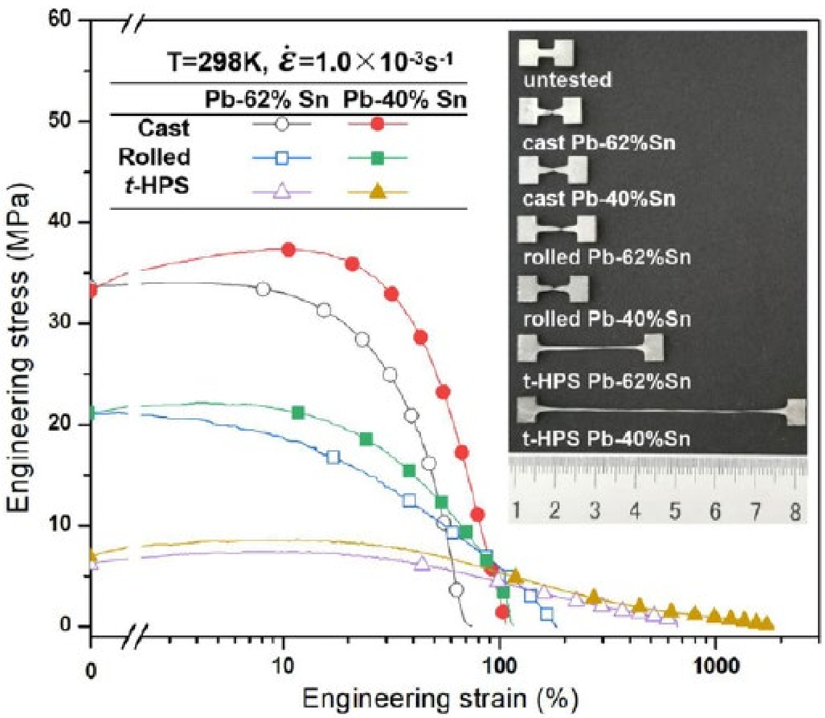


Fig. 6 Engineering stress-strain curves for two Pb-Sn alloys tested in tension at room temperature: the inset shows samples after pulling to failure [50].

machined for these three separate fabrication conditions and then the samples were pulled in tension to failure at room temperature (298 K) at a strain rate of 1.0×10^{-3} s^{-1}. The resultant engineering stress-strain curves are shown in Fig. 6 and the inset at upper right shows the appearance of the specimens after fracture. The results from these tests demonstrate that the as-cast condition gives elongations up to ~100% but this was increased to almost ~200% by rolling where this is not sufficient to demonstrate the occurrence of superplastic flow. These elongations are consistent with an earlier report for flow in a Pb-62% Sn alloy [51]. Nevertheless, processing by t-HPS gave good superplastic properties with an elongation of ~670% in the Pb-62% Sn alloy which represents the highest elongation achieved at this strain rate at room temperature when using an alloy prepared by casting and extrusion. Furthermore, there was an unprecedented elongation of ~1870% in the Pb-40% Sn alloy processed by t-HPS where this elongation exceeds all earlier reported values by a factor of at least three times. This unusual result is due to the presence of equiaxed grain after t-HPS, the grain size of ~0.7 μm and the mixing of the Pb and Sn domains in nearly equal proportions.

Very recently, tests were conducted by processing a cast Bi-43% Sn (wt. %) alloy by t-HPS using a procedure described earlier [52]. Samples were processed through 20 and 100 revolutions at room temperature and then tensile tests were undertaken at 298 K using different strain rates from 1.0×10^{-4} to 1.0×10^{-2} s^{-1}. The results are shown in Fig. 7 for the 20 turns condition where the maximum elongation was 1820% at a strain rate of 1.0×10^{-4} s^{-1}. Figure 8 gives results after 100 turns where the maximum elongation was 2320% at this strain rate. The occurrence of remarkable superplasticity at RT is evident from the absence of any localized necking within the gauge length [53].

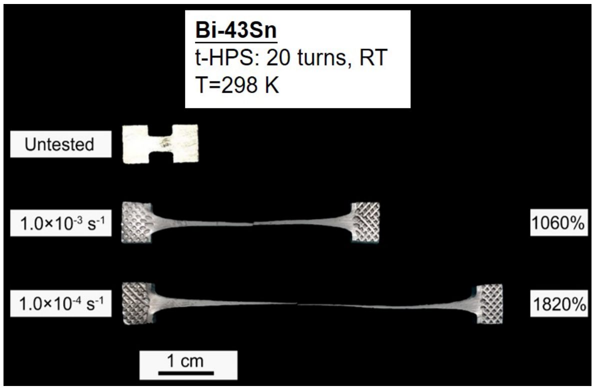


Fig. 7 Appearance of Bi-43% Sn samples after processing by t-HPS for 20 turns and pulling to failure at two different strain rates at 298 K.

Fig. 8 Appearance of Bi-43% Sn samples after processing by t-HPS for 100 turns and pulling to failure at 298 K.

Summary and conclusions

1. Superplasticity occurs in metals when the grain size is equal to or smaller than the subgrain size. A model based on grain boundary sliding is in excellent agreement with the available data.
2. Recent developments include the use of severe plastic deformation to achieve exceptional grain refinement, the extension of superplasticity to new materials such as high-entropy alloys and the visual display of the superplastic regime through the use of deformation mechanism maps.
3. Tube high-pressure shearing is a new processing technique providing the capability of fabricating metals that exhibit exceptionally high superplastic elongations.

Acknowledgements

This work was supported in part by the National Natural Science Foundation of China (Grant No. 52074160) and in part by the European Research Council (ERC Grant Agreement No. 267464-SPDMETALS).

References

[1] G.D. Bengough, A study of the properties of alloys at high temperatures. J. Inst. Metals 7 (1912) 123-178.

[2] C.H.M. Jenkins, Strength of Cd-Zn and Sn-Pb alloy solder. J. Inst. Metals 40 (1928) 21-32.

[3] D.A. Woodford, Strain rate sensitivity as a measure of ductility, Trans. ASM 62 (1969) 291-293.

[4] T.G. Langdon, An analysis of flow mechanisms in high temperature creep and superplasticity, Mater. Trans. 46 (2005) 1951-1956. https://doi.org/10.2320/matertrans.46.1951

[5] T.G. Langdon, Identifying creep mechanisms in plastic flow, Z. Metallk. 96 (2005) 522-531. https://doi.org/10.3139/146.101066

[6] T.G. Langdon, The mechanical properties of superplastic materials, Metall. Trans. A 13A (1982) 689-701. https://doi.org/10.1007/BF02642383

[7] T.G. Langdon, Overview: Using severe plastic deformation in the processing of superplastic materials, Mater. Trans. (2023) in press. https://doi.org/10.2320/matertrans.MT-MF2022021 https://doi.org/10.2320/matertrans.MT-MF2022021

[8] H. Ishikawa, F.A. Mohamed, T.G. Langdon, The influence of strain rate on ductility in the superplastic Zn-22% Al eutectoid, Phil. Mag. 32 (1975) 1269-1271. https://doi.org/10.1080/14786437508228105

[9] F.A. Mohamed, M.M.I. Ahmed, T.G. Langdon, Factors influencing ductility in the superplastic Zn-22 Pct Al eutectoid, Metall. Trans. A 8A (1977) 933-938. https://doi.org/10.1007/BF02661575

[10] F.A. Mohamed, T.G. Langdon, Deformation mechanism maps for superplastic materials, Scripta Metall. 10 (1976) 759-762. https://doi.org/10.1016/0036-9748(76)90358-6

[11] F.R.N. Nabarro, Deformation of crystals by the motion of single ions, in: Report of a Conference on Strength of Solids, The Physical Society, London, U.K. (1984), pp. 75-90.

[12] C. Herring, Diffusional viscosity of a polycrystalline solid, J. Appl. Phys. 21 (1950) 437-445. https://doi.org/10.1063/1.1699681

[13] R.L. Coble, A model for boundary diffusion controlled creep in polycrystalline materials, J. Appl. Phys. 34 (1963) 1679-1682. https://doi.org/10.1063/1.1702656

[14] J.E. Bird, A.K. Mukherjee, J.E. Dorn, Correlations between high-temperature creep behavior and structure, in: (D.G. Brandon and A. Rosen, Eds.), Quantitative Relation Between Properties and Microstructure Israel Universities Press, Jerusalem, Israel (1959) pp. 255-342.

[15] W.R. Cannon, T.G. Langdon, Review: Creep of ceramics, Part 2 An examination of flow mechanisms, J. Mater. Sci. 23 (1988) 1-20. https://doi.org/10.1007/BF01174028

[16] R.J. Twiss, Theory and applicability of a recrystallized grain size paleopiezometer, Pure Appl. Geophys. 115 (1977) 227-244. https://doi.org/10.1007/BF01637105

[17] T.G. Langdon, An evaluation of the strain contributed by grain boundary sliding in superplasticity, Mater. Sci. Eng. A 174 (1994) 225-230. https://doi.org/10.1016/0921-5093(94)91092-8

[18] T.G. Langdon, A unified approach to grain boundary sliding in creep and superplasticity, Acta Metall. Mater. 42 (1994) 2437-2443. https://doi.org/10.1016/0956-7151(94)90322-0

[19] L.K.L. Falk, P.R. Howell, G.L. Dunlop, T.G. Langdon, The role of matrix dislocations in the superplastic deformation of a copper alloy, Acta Metall. 34 (1986) 1203-1214. https://doi.org/10.1016/0001-6160(86)90007-6

[20] R.Z. Valiev, T.G. Langdon, An investigation of the role of intragranular dislocation strain in the superplastic Pb-62% Sn eutectic alloy, Acta Metall. Mater. 41 (1993) 949-954. https://doi.org/10.1016/0956-7151(93)90029-R

[21] Y. Xun, F.A. Mohamed, Slip-accommodated superplastic flow in Zn-22wt% Al, Phil. Mag.

83 (2003) 2247-2266.

[22] Y. Xun, F.A. Mohamed, Superplastic behavior of Zn-22% Al containing nano-scale dispersion

particles, Acta Mater. 52 (2004) 4401-4412. https://doi.org/10.1016/j.actamat.2004.03.039

[23] T.G. Langdon, Seventy-five years of superplasticity: historic developments and new opportunities, J. Mater. Sci. 44 (2009) 5998-6010. https://doi.org/10.1007/s10853-009-3780-5

[24] E.M. Taleff, G.A. Henshall, T.G. Nieh, D.R. Lesuer, J. Wadsworth, Warm-temperature tensile ductility in Al-Mg alloys, Metall. Mater. Trans. A 29A (1998) 1081-1091. https://doi.org/10.1007/s11661-998-0300-1

[25] A.J. Barnes, Superplastic forming 40 years and still growing, J. Mater. Eng. Perform. 16 (2007) 440-454. https://doi.org/10.1007/s11665-007-9076-5

[26] T.G. Langdon, The background to superplastic forming and opportunities arising from new developments, Solid State Phenom. 306 (2020) 1-8. https://doi.org/10.4028/www.scientific.net/SSP.306.1

[27] J. Wongsa-Ngam, T.G. Langdon, Advances in superplasticity from a laboratory curiosity to the development of a superplastic forming industry, Metals 12 (2022) 1921. https://doi.org/10.3390/met12111921

[28] R.Z. Valiev, O.A. Kaibyshev, R.I. Kuznetsov, R.Sh. Musalimov, N.K. Tsenev, Low-temperature superplasticity of metallic materials, Dokl. Akad. Nauk SSSR 301 (1988) 864-866.

[29] R.Z. Valiev, Y. Estrin, Z. Horita, T.G. Langdon, M.J. Zehetbauer, Y.T. Zhu, Producing bulk ultrafine-grained materials by severe plastic deformation, JOM 58(4) (2006) 33-39. https://doi.org/10.1007/s11837-006-0213-7

[30] R.Z. Valiev, T.G. Langdon, Principles of equal-channel angular pressing as a processing tool for grain refinement, Prog. Mater. Sci. 51 (2006) 881-981. https://doi.org/10.1016/j.pmatsci.2006.02.003

[31] A.P. Zhilyaev, T.G. Langdon, Using high-pressure torsion for metal processing: Fundamentals and applications, Prog. Mater. Sci. 53 (2008) 893-979. https://doi.org/10.1016/j.pmatsci.2008.03.002

[32] A.P. Zhilyaev, B.K. Kim, G.V. Nurislamova, M.D. Baró, J.A. Szpunar, T.G. Langdon, Orientation imaging microscopy of ultrafine-grained nickel, Scripta Mater. 46 (2002) 575-580. https://doi.org/10.1016/S1359-6462(02)00018-0

[33] J. Wongsa-Ngam, M. Kawasaki, T.G. Langdon, A comparison of microstructures and mechanical properties in a Cu-Zr alloy processed using different SPD techniques, J. Mater. Sci. 48 (2013) 4653-4660. https://doi.org/10.1007/s10853-012-7072-0

[34] R.Z. Valiev, D.A. Salimonenko, N.K. Tsenev, P.B. Berbon, T.G. Langdon, Observations of high strain rate superplasticity in commercial aluminum alloys with ultrafine grains sizes, Scripta Mater. 37 (1997) 1943-1950. https://doi.org/10.1016/S1359-6462(97)00387-4

[35] Z. Horita, M. Furukawa, M. Nemoto, A.J. Barnes, T.G. Langdon, Superplastic forming at high strain rates after severe plastic deformation, Acta Mater. 48 (2000) 3633-3640. https://doi.org/10.1016/S1359-6454(00)00182-8

[36] D.G. Shaysultanov, N.D. Stepanov, A.V. Kuznetsov, G.A. Salishchev, O.N. Senkov, Phase composition and superplastic behavior of a wrought AlCoCrCuFeNi high-entropy alloy, JOM 35 (12) (2013) 1815-1828.

[37] H. Shahmir, J. He, Z. Lu, M. Kawasaki, T.G. Langdon, Evidence for superplasticity in a CoCrFeNiMn high-entropy alloy processed by high-pressure torsion, Mater. Sci. Eng. A685 (2017) 342-348. https://doi.org/10.1016/j.msea.2017.01.016

[38] N.T.C. Nguyen, P. Asghari-Rad, P. Sathiyamoorthi, A. Zargaran, C.S. Lee, H.S. Kim, Ultrahigh high-strain-rate superplasticity in a nanostructured high-entropy alloy, Nature Commun. 11 (2020) 2736. https://doi.org/10.1038/s41467-020-16601-1

[39] H. Shahmir, M.S. Mehranpour, S.A.A. Shams, T.G. Langdon, Twenty years of the CoCrFeNiMn high-entropy alloy: achieving exceptional mechanical properties through

microstructure engineering, J. Mater. Res. Tech. 23 (2023) 3362-3423. https://doi.org/10.1016/j.jmrt.2023.01.181

[40] P. Asghari-Rad, N.T.C. Nguyen, A. Zargaran, P. Sathiyamoorthi, H.S. Kim, Relation of phase fraction to superplastic behavior of multi-principal element alloy with multi-phase structure, Scripta Mater. 207 (2022) 114239. https://doi.org/10.1016/j.scriptamat.2021.114239

[41] N.T.C. Nguyen, P. Asghari-Rad, H. Park, H.S. Kim, Differential superplasticity in a multi-phase multi-principal element alloy by initial annealing, J. Mater. Sci. 57 (2022) 18154-18167. https://doi.org/10.1007/s10853-022-07616-8

[42] M. Kawasaki, T.G. Langdon, The many facets of deformation mechanism mapping and the application to nanostructured materials, J. Mater. Res. 28 (2013) 1827-1834. https://doi.org/10.1557/jmr.2013.55

[43] M. Kawasaki, T.G. Langdon, Review: achieving superplastic properties in ultrafine-grained materials at high temperatures, J. Mater. Sci. 51 (2016) 19-32. https://doi.org/10.1007/s10853-015-9176-9

[44] R.B. Figueiredo, T.G. Langdon, Deformation mechanisms in ultrafine-grained metals with an emphasis on the Hall-Petch relationship, J. Mater. Res. Tech. 14 (2021) 137-159. https://doi.org/10.1016/j.jmrt.2021.06.016

[45] R.B. Figueiredo, W. Wolf, T.G. Langdon, Effect of grain size on strength and strain rate sensitivity in the CrMnFeCoNi high-entropy alloy, J. Mater. Res. Tech. 20 (2022) 2358-2368. https://doi.org/10.1016/j.jmrt.2022.07.181

[46] R.B. Figueiredo, M. Kawasaki, T.G. Langdon, Seventy years of Hall-Petch, ninety years of superplasticity and a generalized approach to the effect of grain size on flow stress, Prog. Mater. Sci. 137 (2023) 101131. https://doi.org/10.1016/j.pmatsci.2023.101131

[47] J.T. Wang, Z. Li, J. Wang, T.G. Langdon, Principles of severe plastic deformation using tube high-pressure shearing, Scripta Mater. 67 (2012) 810-813. https://doi.org/10.1016/j.scriptamat.2012.07.028

[48] Z. Li, P.F. Zhang, H. Yuan, K. Lin, Y. Liu, D.L. Yin, J.T. Wang, T.G. Langdon, Principle of one-step synthesis for multi-layered structures using tube high-pressure shearing, Mater. Sci. Eng. A658 (2016) 367-375. https://doi.org/10.1016/j.msea.2016.02.024

[49] Z. Li, Y. Liu, J.T. Wang, T.G. Langdon, Tube high-pressure shearing: a simple shear path to unusual microstructures, Mater. Trans. (2023) (doi.org/10.2320/matertrans.MT-MF2022051).

[50] K. Lin, Z. Li, Y. Liu, E. Ma, J.T. Wang, T.G. Langdon, Exploiting tube high-pressure shearing to prepare a microstructure in Pb-Sn alloys for unprecedented superplasticity, Scripta Mater. 209 (2022) 114390. https://doi.org/10.1016/j.scriptamat.2021.114390

[51] M.M.I. Ahmed, T.G. Langdon, Ductility of the Pb-Sn eutectic at room temperature, J. Mater. Sci. Lett. 2 (1983) 59-62. https://doi.org/10.1007/BF00725431

[52] C.T. Wang, Z. Li, Y. He, J.T. Wang, T.G. Langdon, Microstructural evolution and tensile testing of a Bi-Sn (57/43) alloy processed by tube high-pressure shearing, Crystals 11 (2021) 1229. https://doi.org/10.3390/cryst11101229

[53] T.G. Langdon, Fracture processes in superplastic flow, Metal Sci. 16 (1982) 175-183. https://doi.org/10.1179/030634582790427208