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SUPERPLASTIC FORMING AT HIGH STRAIN RATES AFTER SEVERE PLASTIC DEFORMATION

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Abstract—An Al-3% Mg-0.2% Sc alloy was fabricated by casting and subjected to severe plastic deformation through equal-channel angular pressing to a strain of ~ 8. The grain size after pressing was ~ 0.2 μm and increased to − 1.1 μm when holding at 673 K for 10 min. Very high tensile elongations were recorded at 673 K with a maximum elongation of ~ 2280% when testing with an initial strain rate of 3.3×10^{-2} s^{-1}. The strain rate sensitivity was measured as ~ 0.5 at strain rates in the vicinity of 10^{-2} s^{-1}. Small disks were cut from the rods after pressing and these disks were successfully formed into domes at 673 K using a biaxial gas-pressure forming facility and forming times up to a maximum of 60 s. Measurements of the local thicknesses at selected points around the domes revealed reasonably uniform thinning which is consistent with the high strain rate sensitivity of this alloy. *© 2000 Acta Metallurgica Inc. Published by Elsevier Science Ltd. All rights reserved.*

Keywords: Aluminum alloys; Forming; High temperature mechanical properties; Microstructure

1. INTRODUCTION

The superplastic forming industry is now well-established for the fabrication of complex parts from sheet metals [1]. However, the utilization of this technology is currently limited because of the relatively low strain rates associated with the forming process. For example, the production forming rates for the standard superplastic Al-2004 and Al-5083 alloys are within the range of ~ 10^{-3}–10^{-2} s^{-1} so that the forming times are generally ~ 20–30 min for each component [2]. These slow rates of forming necessarily restrict the commercial applications of superplastic forming to a limited range of low-volume high-value components, primarily for use in the aerospace industry and building design, and they prevent, on economic grounds, an expansion of superplastic forming into the fabrication of high-volume components associated with the automotive and consumer product industries. An important current objective is therefore to process materials which are capable of achieving superplastic ductilities at high strain rates, typically in the range of ~ 10^{-2}–1 s^{-1}, so that the forming time for each component may be reduced below ~ 60 s.

Experiments have shown that the rate of flow in superplasticity varies inversely with the grain size raised to a power of ~ 2 and there is evidence from standard superplastic alloys that the superplastic regime is displaced to faster strain rates when the grain size of a material is reduced [3]. Observations of this type led to the suggestion that it may be possible to increase the strain rate for optimum superplasticity by making a substantial reduction in the grain size to the submicrometer or even the nanometer level [4]. Although these very small grain sizes are difficult or even impossible to attain using the standard procedures associated with thermomechanical processing, nevertheless there is good evidence that the grain sizes of polycrystalline samples may be very significantly reduced by imposing an intense plastic strain through a process such as equal-channel angular (ECA) pressing [5, 6].

It was shown recently that, provided the ultrafine grain sizes introduced by ECA pressing are reasonably stable at the high temperatures required for superplastic flow, it is possible to achieve high tensile ductilities in ECA-pressed aluminum alloys when

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testing at very rapid strain rates [7–13] including the rates associated with high strain rate superplasticity (HSR SP) which are defined formally as $\geq 10^{-2}$ s^{-1} [14]. Unfortunately, the experimental data available to date are rather limited because of the small numbers of samples available for tensile testing after the ECA pressing procedure. Accordingly, the present investigation was conducted with two objectives: (i) to provide a more rigorous appraisal of the potential for achieving HSR SP in tensile testing after ECA pressing by conducting tests over a wide range of strain rates and (ii) to evaluate the possibility of using ECA-pressed materials for superplastic forming operations at high strain rates by making use of a simple gas-pressure forming facility.

2. EXPERIMENTAL MATERIAL AND PROCEDURES

2.1. *Preparation of the material*

The experiments were undertaken using an aluminum alloy with a composition, in wt %, of 3% Mg and 0.2% Sc. This alloy was selected for two reasons. First, earlier experiments demonstrated the possibility of obtaining reasonably homogeneous microstructures and very substantial grain refinement in this alloy, with measured average grain sizes of the order of ~ 0.2 μm after ECA pressing [8, 13]. Second, high elongations have been reported in this alloy after ECA pressing, with a maximum elongation of ~ 1560% at a testing temperature of 673 K and an imposed strain rate of 3.3×10^{-2} s^{-1} [13]. It is also well established that the presence of small amounts of Sc in aluminum-based alloys increases the recrystallization temperature [15, 16].

The alloy was prepared by arc melting, in an argon atmosphere, Al of 99.99% purity and Sc of 99.999% purity to form initially an Al-3% Sc alloy, and this alloy was then remelted with additional Al and 3% Mg of 99.9% purity to give an Al-3% Mg-0.2% Sc alloy. The alloy was cast into a steel mold to form an ingot with dimensions of $17 \times 55 \times 120$ mm^3, the ingot was homogenized in air at 753 K for one day and then each surface was ground to remove ~ 1 mm. The ingot was cut into bars with dimensions of $15 \times 15 \times 120$ mm^3 and these bars were swaged into rods having diameters of 10 mm and the rods were cut into cylindrical samples for ECA pressing with lengths of ~ 60 mm.

All samples were subjected to a solution treatment of 1 h at a temperature of 883 K prior to ECA pressing where this temperature was selected because it was found by using differential scanning calorimetry that the onset of incipient melting occurs in this alloy at a temperature slightly above 883 K. The measured initial grain size after this solution treatment was ~ 200 μm.

2.2. *Procedure for ECA pressing*

Full details of the principles of ECA pressing were given earlier [17, 18] including information on the procedure for estimating the strain imposed on a sample when it is pressed through an ECA die [19]. In the present investigation, the ECA pressing was conducted using a solid die fabricated from high strength tool steel and with a single channel, circular in cross-section, forming an L-shaped configuration. The diameter of this channel was 10 mm, the angle subtended by the two portions of the channel, Φ, was 90° and the angle defining the outer arc of curvature at the point of intersection of the two portions of the channel, ψ, was 45°. It can be shown from first principles that these values of Φ and ψ lead to an imposed strain of ~ 1 on each passage of the sample through the die [19] and, since the cross-sectional dimensions of the sample remain unchanged on each pass, repetitive pressings may be conducted to achieve high total strains.

All of the ECA pressing was performed at room temperature with the samples coated in molybdenum disulfide as a lubricant and using a hydraulic press operating at a pressing speed of ~ 19 mm s^{-1}. Each sample was pressed through the die for a total of 8 passes giving an imposed strain of ~ 8 and the samples were rotated by 90° in the same direction between each pressing using the procedure designated as route B_C [20]. Processing using route B_C was adopted because earlier experiments on pure aluminum demonstrated that this is the optimum procedure for most rapidly achieving a homogeneous array of equiaxed grains separated by grain boundaries having high angles of misorientation [21].

2.3. *Microstructural characteristics after ECA pressing*

Samples were prepared for microstructural examination by transmission electron microscopy (TEM), in both the ECA-pressed condition and after pressing and subsequent annealing, using the procedure described in detail earlier [22]. All samples were examined in an Hitachi H-8100 transmission electron microscope operating at 200 kV and selected area electron diffraction (SAED) patterns were taken from regions having diameters of 12.3 μm.

2.4. *Tensile testing after ECA pressing*

An important requirement of this work was to evaluate the tensile ductilities which may be achieved by ECA pressing and especially the potential for attaining superplasticity at high strain rates. Accordingly, tensile specimens were machined from the samples after ECA pressing with the gauge lengths lying parallel to the longitudinal axes and with gauge lengths and cross-sectional areas of 5 mm and 2×3 mm^2, respectively. These samples were pulled to failure in air using a testing machine operating at a constant rate of cross-head displacement and with initial strain rates in the range from 1×10^{-4} to 3.3 s^{-1}. All of the tensile tests were conducted at 673 K because there is evidence that this temperature may represent an optimum condition for achieving high elongations to failure [13] and the testing temperature was maintained constant during each test to within ± 1 K. Each sample was heated to the testing temperature over a period of ~ 30 min and then held at this temperature for ~ 10 min in order to reach thermal equilibrium.

2.5. *Superplastic forming into a dome*

The potential for achieving rapid superplastic forming was evaluated by cutting disks from the rods after ECA pressing and using a simple biaxial gas-pressure forming facility equipped with a high temperature furnace. These disks had thicknesses of ~ 0.3 mm, they were cut perpendicular to the longitudinal axes of the rods, and they were inserted individually into a forming facility where they were clamped around the periphery so that there was an unrestricted area, having a diameter of 7.0 mm, available for the forming operation. The chamber was then evacuated, heated to 673 K, and a constant pressure of argon gas was applied for a short but controlled period of time to form the unrestricted portion of the disk into a dome. After the forming operations, selected domes were carefully sectioned and measurements were taken to determine the thickness of the material at points around the dome from the base to the pole.

3. EXPERIMENTAL RESULTS

3.1. *Microstructures after ECA pressing and static annealing*

Careful inspection of large areas within samples following ECA pressing revealed a reasonably homogeneous microstructure with an area fraction of > 90% where the grains were equiaxed and separated by high angle grain boundaries and an area fraction of < 10% where the grains were separated by low angle sub-boundaries. Similar observations for this alloy were reported earlier [13]. The grain size after ECA pressing was measured as ~ 0.2 μm.

An important requirement for achieving HSR SP is the ability to retain an ultrafine grain size up to temperatures which are sufficiently high that deformation can take place by diffusion-controlled processes such as superplastic flow. Fig. 1 shows the microstructure in this alloy following ECA pressing, heating to a temperature of 673 K and then holding at this temperature for a period of 10 min. In this condition, there is a homogeneous microstructure of equiaxed grains separated by high angle boundaries and with an average grain size of ~ 1.1 μm. It is apparent, therefore, that the grain size remains very small even at this high temperature and this grain stability is attributed to the presence of fine and stable Al_3Sc precipitates [15, 23].

3.2. *Elongations to failure in tension*

Samples were pulled to failure in tension at a temperature of 673 K after holding at temperature for 10 min to attain thermal equilibrium. Thus, the microstructures at the onset of the tensile testing were similar to that shown in Fig. 1.

The true stress is plotted against the true strain for each of these samples in Fig. 2 and inspection shows there is a peak stress at low strains followed by strain softening. Fig. 3 gives a plot of the maximum flow stress as a function of the initial strain rate and it is apparent that this curve shows evidence of a sigmoidal shape, as in standard superplastic alloys, with a maximum strain rate sensitivity, m, of ~ 0.5 at a strain rate slightly above ~ 10^{-2} s^{-1}. The curve in Fig. 3 suggests that the maximum elongations to failure will occur in the vicinity of ~ 10^{-2}–10^{-1} s^{-1} and this is confirmed in Fig. 4 where the samples are shown after failure: the upper sample is untested and the other samples are placed in order of the testing strain rates from the fastest rate at the top (3.3 s^{-1} to an elongation of ~ 120%) to the slowest rate at the bottom (1.0×10^{-4} s^{-1} to an elongation of ~ 710%). Inspection shows the alloy exhibits exceptionally high tensile ductilities after ECA pressing, especially at strain rates close to ~ 10^{-2} s^{-1} where the maximum elongation to failure is ~ 2280% at a strain rate of 3.3×10^{-2} s^{-1}. The very uniform deformation visible within the gauge length of this and other samples demonstrates conclusively the occurrence of superplastic flow in this alloy after ECA pressing.

3.3. *Superplastic forming under biaxial conditions*

Small disks cut from the ECA-pressed rods were superplastically formed into domes at a temperature of 673 K and examples are shown in Fig. 5 where there is an untested disk on the left and two disks subjected to a gas pressure of 10 atmospheres, equivalent to 1 MPa, for periods of 30 s in the center and 1 min on the right. Despite the relatively low pressure available in the gas-forming facility, it is evident from the smooth nature of the two domes shown in the center and right in Fig. 5 that this alloy is capable of forming a complex shape at a very rapid strain rate.

The two domes shown in Fig. 5 were sectioned and measurements were taken to determine the local thickness of the sheet at angular increments of 0°, 30°, 60° and 85° with respect to the center of the undeformed disk: these angles are defined schematically in Fig. 6 where 0° is the pole of the dome and 85° is very close to the undeformed sheet at the base. The results from these two sets of measurements are given in Fig. 7 where the local thickness is plotted against the angular increments. These measurements show that the dome deforms uniformly about the pole and, as expected, the thicknesses are smaller after the longer forming time of 60 s. It is important to note there was no evidence for the development of any cracking or excessive deformation in the vicinity of the pole even after a forming time of 60 s.

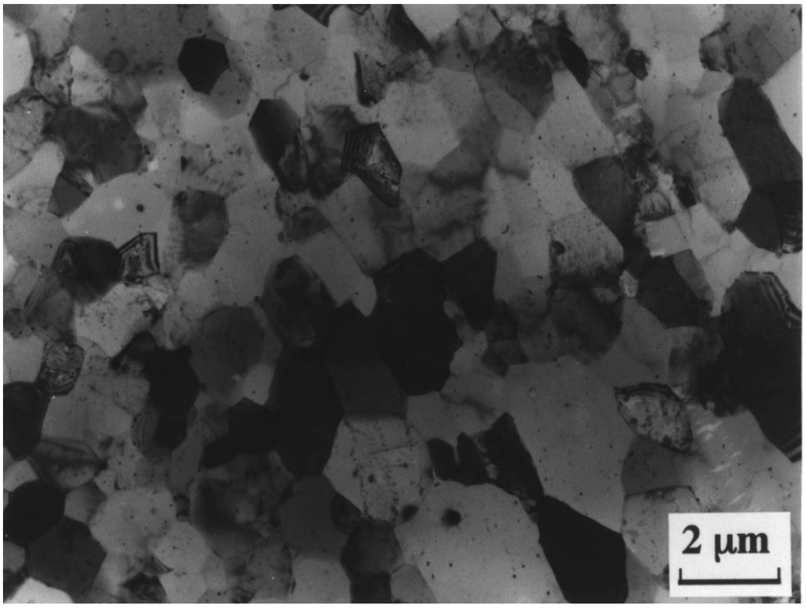

Fig. 1. Microstructure after ECA pressing, heating to 673 K and holding at temperature for 10 min.

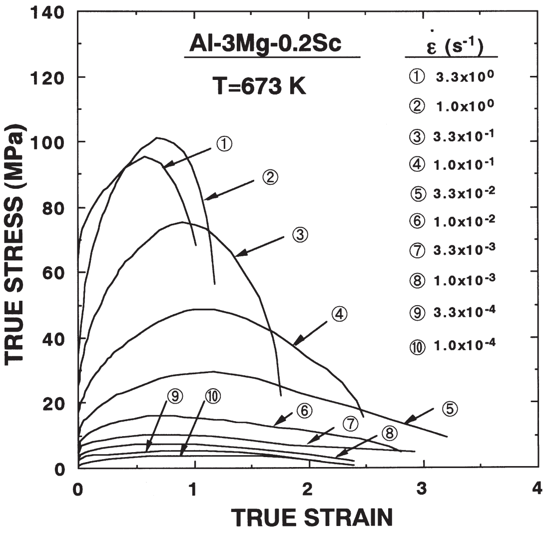

Fig. 2. True stress versus true strain for samples tested in tension at 673 K.

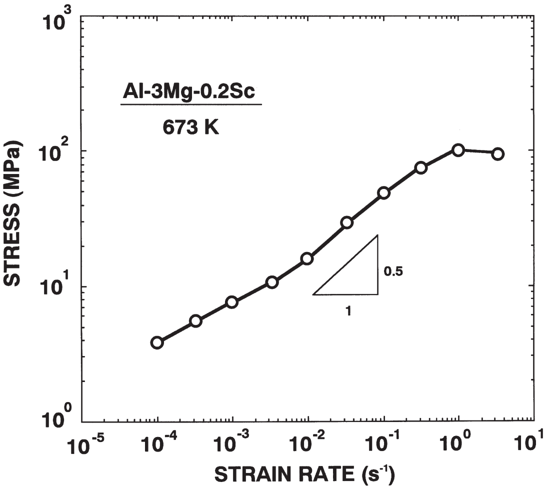

Fig. 3. Variation of maximum flow stress with initial strain rate for samples tested at 673 K.

4. DISCUSSION

4.1. Characteristics of the Al-Mg-Sc alloy after ECA pressing

The results from this investigation lead to three important conclusions concerning the Al-3% Mg-0.2% Sc alloy.

First, ECA pressing is a very effective processing tool for reducing the grain size of this alloy to the submicrometer level. The as-pressed grain size of ~ 0.2 μm is slightly smaller than the grain size of ~ 0.27 μm reported earlier for an Al-3% Mg alloy [24] and this difference is probably due to the presence of some Sc in solid solution since it has been established that the ultimate stable grain size in Al-Mg alloys decreases with the presence of additional Mg in solid solution [24]. In addition, these ultrafine grains are remarkably stable in this alloy at elevated temperatures so that it is possible to retain a grain size close to ~ 1 μm at a temperature as high as 673 K.

Second, the as-pressed alloy exhibits extremely high tensile ductilities at 673 K with elongations to failure exceeding 2000% under optimum conditions: these elongations are exceptionally large for a solid solution alloy and they are comparable to the elongations generally reported in highly superplastic two-phase alloys such as the Zn-22% Al eutectoid [3].

Third, these very high tensile elongations occur at strain rates extending up to and above 10^{-2} s^{-1} so that the alloy exhibits HSR SP after ECA pressing.

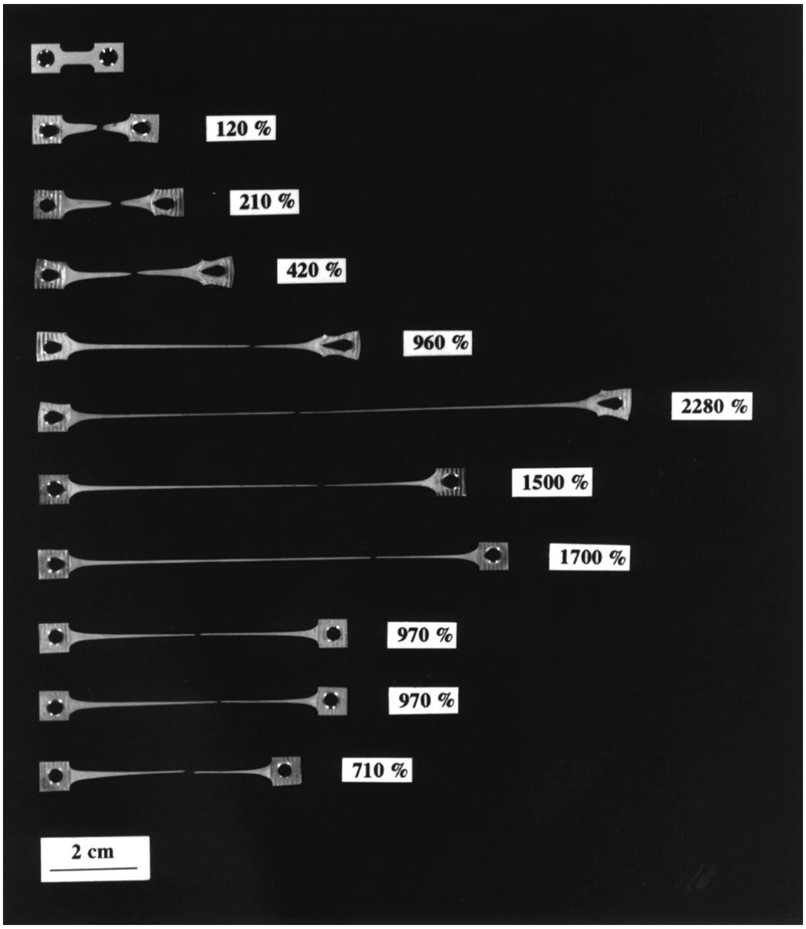

Fig. 4. Samples of the Al-Mg-Sc alloy after deformation by equal-channel angular pressing and pulling to failure at 673 K: the upper sample is untested and the other samples were pulled, in descending order, at initial strain rates of 3.3 s^{-1} (120%), 1.0 s^{-1} (210%), 3.3×10^{-1} s^{-1} (420%), 1.0×10^{-1} s^{-1} (960%), 3.3×10^{-2} s^{-1} (2280%), 1.0×10^{-2} s^{-1} (1500%), 3.3×10^{-3} s^{-1} (1700%), 1.0×10^{-3} s^{-1} (970%), 3.3×10^{-4} s^{-1} (970%) and 1.0×10^{-4} s^{-1} (710%).

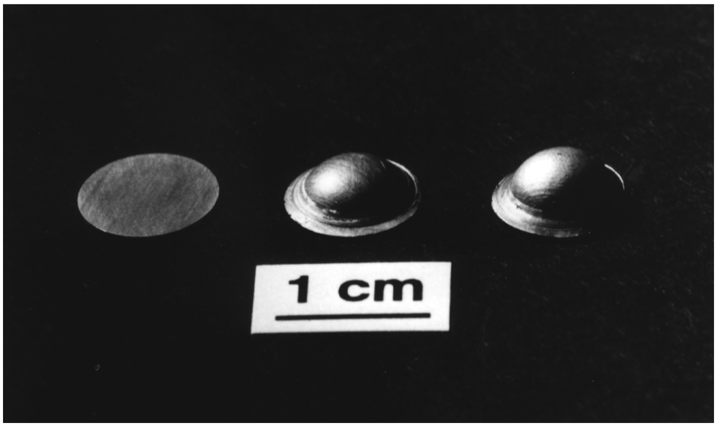

Fig. 5. Examples of superplastic forming at high strain rates in a gas-pressure forming facility: the disk on the left is untested and the other disks were held at 673 K and subjected to a gas pressure of 10 atmospheres for 30 s (center) and 1 min (right), respectively.

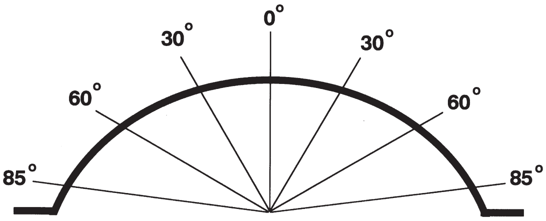

Fig. 6. Schematic illustration of the angular increments where the dome thicknesses were measured.

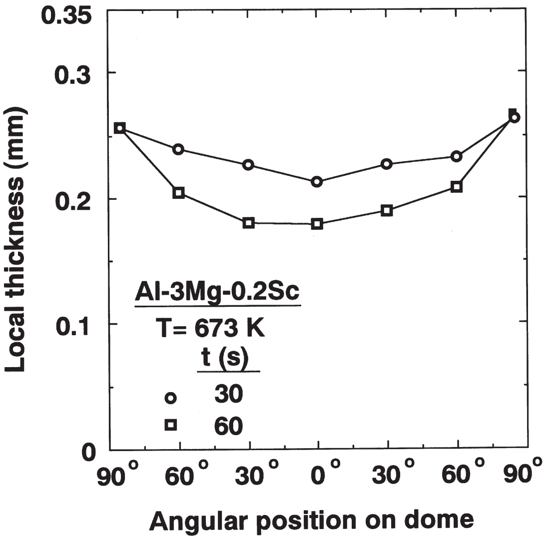

Fig. 7. Variation of the local thickness with the angular position around the dome for the two domes shown in Fig. 5.

Thus, ECA pressing may be used to attain a microstructure in which it is possible to achieve superplastic forming at very high strain rates.

It is important to note that superplastic forming at high strain rates has been reported also for an Al-6061/20% SiC composite where an Al-6061 matrix alloy was reinforced with SiC whiskers [25]. However, the rapid forming in the composite occurred by a different flow mechanism because it was achieved at a temperature of 873 K which is higher than the matrix solidus temperature of 855 K so that the material was in a semi-solid state. By contrast, the present results provide a very clear demonstration of high strain rate superplastic forming which occurs by conventional superplasticity in an unreinforced Al-based alloy at a temperature where no liquid phase is present.

4.2. Potential for using this approach in industrial superplastic forming

The measurements of the dome thicknesses after superplastic forming are important because an ideal superplastic sheet will have the capability of bulging into a hemisphere with little significant variation in the local thickness at any point on the surface. For the two domes illustrated in Fig. 5, the measurements in Fig. 7 show that the equivalent strains at the two poles of the domes are ~ 0.29 and ~ 0.40 after forming for 30 and 60 s corresponding to forming rates of ~ 1×10^{-2} and ~ 7×10^{-3} s^{-1}, respectively.

From the detailed analysis of sheet metal forming by Cornfield and Johnson [26], it is possible to define a thinning factor, ξ, as the ratio of the measured thickness at any point on the dome to the average thickness which would be attained assuming constant volume in the sheet metal and uniform deformation during the forming process. Thus, considering the dome on the right in Fig. 5 where the deformation is a maximum, the measured height of the dome is ~ 2.46 mm and the unrestricted diameter at the base is 7.0 mm so that the height to base ratio is ~ 0.35. Using the measured thickness at the pole and noting that the dome forms part of a hemisphere, the thinning factor is estimated for this condition as $\xi \simeq 0.89$. This value of ξ is consistent with the theoretical expectations for a highly superplastic material since it has been shown that, when the height to base ratio of a dome is ~ 0.35 and the strain rate sensitivity of the sheet metal is $m \simeq 0.5$ as demonstrated in Fig. 6, the theoretical value for ξ is ~ 0.85 [26]. These measurements of dome thickness demonstrate that the Al-Mg-Sc alloy used in this investigation exhibits reasonably uniform thinning during the forming operation because of the high strain rate sensitivity of the alloy.

Any successful industrial utilization of a superplastic aluminum alloy exhibiting HSR SP will require, in addition, that the high ductilities are achieved without significant increases in the corresponding flow stresses at these fast strain rates. In the present investigation, Fig. 3 shows that the measured flow stress at 10^{-2} s^{-1} is ~ 10 MPa for a temperature of 673 K with a corresponding value of $m \simeq 0.5$. For the Al-2004 alloy (Al-6% Cu-0.5% Zr), which is used extensively in commercial superplastic forming operations, it has been reported that $m \simeq 0.4$–0.5 and the flow stress is ~ 9 MPa at the optimum forming temperature of 753 K when using a strain rate of 3×10^{-3} s^{-1} [27]. The similarities in the flow stresses for these two alloys, despite the higher forming temperature for the Al-2004 alloy, confirms that the high ductilities in Fig. 4 are achieved at fast strain rates without the introduction of high flow stresses.

4.3. Comparison with other data showing rapid superplastic forming in Al alloys

To date, attempts to develop a superplastic forming capability in aluminum-based alloys at high strain rates have centered almost exclusively on conventional Al-Mg alloys where the strain rate sensitivity is $m \simeq 0.3$. However, this approach leads both to high flow stresses and, because of the relatively low value of m, to significant variations in the local thickness for any domes produced by superplastic forming.

A very recent example of this effect, including both high flow stresses and severe non-uniformity in the local thinning, is provided by the report of Uchida *et* *al.* [28] in which an Al-Mg alloy, designated Sumitomo SX01, was used to produce a hemisphere at a temperature of 743 K. From the published experimental data, the thinning factor in this material may be estimated as $\xi \simeq 0.4$ and this low value of ξ is again consistent with the theoretical expectations for a material having $m \simeq 0.3$ [26]. It is reasonable to conclude, therefore, that a material with $m \simeq 0.3$ will not provide the uniformity of thinning which is a desirable property for any successful utilization of high strain rate superplastic forming in industrial practice. By contrast, the present results show that the addition of Sc to an Al-3% Mg alloy increases the strain rate sensitivity to ~ 0.5 and thereby provides a relatively uniform thinning during the forming process.

4.4. Comparison with other results on Al-Mg-Sc alloys

There are some earlier reports of superplasticity in Al-Mg-Sc alloys where the materials were subjected to extensive thermomechanical processing. Sawtell and Jensen [29] reported elongations up to 1020% without failure in Al-4% Mg-0.5% Sc and Al-6% Mg-0.5% Sc alloys at temperatures in the range of 672–811 K and with strain rates up to ~ 10^{-2} s^{-1} and Nieh *et al.* [30] tested an Al-6% Mg-0.3% Sc alloy and obtained elongations up to a maximum of ~ 1130% at 798 K using a strain rate of 1.4×10^{-2} s^{-1}. Comparing these results with the present investigation, it is apparent the elongations are lower after thermomechanical treatment by more than a factor of two. This difference arises because the ECA pressing procedure is exceptionally effective in producing an array of extremely small grains separated by high angle grain boundaries.

Mukai *et al.* [31] reported subjecting an Al-4% Mg-0.5% Sc alloy to ECA pressing at temperatures of either 473 and 673 K but the subsequent elongations to failure in tensile testing were $<$ 400% at a temperature of 748 K with a strain rate of 1×10^{-2} s^{-1}. These very low elongations are probably a consequence of the nature of the ECA pressing procedure used in these experiments. For the sample pressed at 673 K, the grain size was reported as ~ 25 μm which is too large to achieve high tensile ductilities; for the sample pressed at 473 K, it was reported that the grain size was ~ 0.5 μm but this sample was pressed to a total strain of only ~ 5 which is insufficient to achieve a homogeneous array of grains separated by high angle grain boundaries in an Al-4% Mg alloy [24].

5. SUMMARY AND CONCLUSIONS

1. An Al-3% Mg-0.2% Sc alloy was fabricated by casting and then subjected to equal-channel angular (ECA) pressing at room temperature to a strain of ~ 8. The ECA pressing reduced the grain size from an initial value of ~ 200 μm to a final value of ~ 0.2 μm.
2. The ultrafine grain size introduced by ECA pressing was very stable at elevated temperatures: a grain size of ~ 1.1 μm was recorded after holding for 10 min at 673 K.
3. Very high tensile elongations were obtained in this alloy at 673 K: these elongations were > 1000% at strain rates in the vicinity of 10^{-2} s^{-1} with a maximum elongation of ~ 2280% when using an initial strain rate of 3.3×10^{-2} s^{-1}.
4. The strain rate sensitivity was $m \simeq 0.5$ at strain rates in the range of ~ 10^{-2}–10^{-1} s^{-1} and the measured flow stresses at these fast rates were comparable with a conventional commercial superplastic alloy (Al-2004).
5. Small disks, cut from the ECA-pressed rods, were successfully formed into domes using a biaxial gas-pressure forming facility operating at 673 K and with forming times up to a maximum of 60 s.
6. Measurements of the local thicknesses at selected points around the dome revealed a reasonably uniform thinning and results consistent with the theoretical expectations for the forming of a sheet metal with $m \simeq 0.5$.

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