Achieving superplasticity in a Bi-Sn alloy processed by equal-channel angular pressing

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Abstract. Equal-channel angular pressing (ECAP) was utilized to process a Bi–42Sn eutectic alloy at room temperature using processing route A for 1, 2, 4, and 8 passes. Tensile testing was performed at room temperature under initial strain rates in the range of 1.0×10^{-5} to 1.0×10^{-2} s^{-1}. The results demonstrate that processing by ECAP improves the ductility in this material and the elongations to failure increased with decreasing strain rate in all samples. The largest elongation to failure, ~1300%, was recorded in the sample processed by ECAP for 8 passes at an initial strain rate of 1.0×10^{-5} s^{-1}. This elongation is much improved by comparison with the as-cast Bi–42Sn alloy.

1. Introduction

The ability of a polycrystalline material to exhibit, in a generally isotropic manner, very high elongations prior to failure is known as superplasticity. The measured elongations in superplasticity are generally at least 400% and the measured strain rate sensitivities are close to ~0.5. Superplasticity is a diffusion-controlled process and therefore it requires a relatively high testing temperature, typically at or above $0.5T_m$ where T_m is the absolute melting temperature of the material [1]. In addition to high testing temperatures, the materials should have a small and stable grain size, typically smaller than ~10 μm, to exhibit superplastic flow [2]. Superplastic behaviour is usually observed either in two-phase eutectic/eutectoid materials or materials containing finely dispersed second phase particles to inhibit grain growth at high temperatures [1]. The reason is that it is hard for both of the two necessary conditions of superplasticity to be satisfied simultaneously in pure metals and solid solution alloys since grain growth occurs at high temperatures.

Materials processed by severe plastic deformation (SPD) methods are also capable of exhibiting high elongations to failure, and in some cases superplastic behaviour, when tested at high temperatures, typically more than $0.5T_m$. Large amounts of strain may be imposed to the working material using SPD techniques since the imposed strain makes no significant change in the overall dimensions of the material. Therefore, exceptional grain refinements may be achievable in metals through SPD methods. Equal-channel angular pressing (ECAP) is one of the well-known SPD methods which uses relatively large bulk samples and has other advantages such as simplicity in operation. For this reason it is generally considered superior to most other SPD methods [3,4]. Studying the superplastic behaviour in materials processed by SPD methods, including ECAP, has been the subject of much research and it has been established that ECAP-processed materials can exhibit excellent superplasticity. For example, an elongation of 3050% was achieved in a magnesium ZK60 alloy after processing for 2 passes of ECAP and then testing in tension at 473 K using a strain rate of 1.0×10^{-4} s^{-1} [5]. Similarly, a superplastic elongation of 3060% was achieved in a Pb-62% Sn eutectic alloy processed by ECAP for 16 passes under a strain rate of 1.0×10^{-3} s^{-1} at 413 K [6].

True superplastic behaviour, with an elongation to failure of more than 400%, was reported for the first time in an extruded Bi-Sn eutectic alloy after ageing for 7 days [7]. This material showed an elongation to failure of ~1950% when tested under a constant stress of ~1.7 MPa. However, there are very few investigations of the effect of SPD processing on the superplastic behaviour of this material. In the only study conducted to date, the Bi-Sn eutectic alloy was processed by high-pressure torsion (HPT) and the ductility of the material was significantly improved in tensile testing at room temperature. A superplastic elongation of ~1200% was achieved after processing by HPT through 10 turns and pulling to failure at an initial strain rate of 1.0×10^{-4} s^{-1} [8]. Nevertheless, the effect of processing by ECAP on the eutectic Bi-Sn alloy has not been studied. Therefore, the aim of this research was to investigate the superplastic behaviour of this material after ECAP processing.

2. Experimental material and procedures

The alloy was cast Bi-42% Sn from which rods with 60 mm lengths and cross-sectional dimensions of 10×10 mm^2 were machined for ECAP processing. This alloy has a low melting temperature of 411 K and experiences self-annealing when held at room temperature of 298 K. Therefore, no heat treatment was performed on the alloy before ECAP processing. The ECAP was conducted at room temperature using a die with an internal channel angle, φ, of 135°. The pressing was conducted using route A in which the sample is not rotated between consecutive passes [9]. The pressing rate was 1 $mm.s^{-1}$ and the ECAP was conducted for 1, 2, 4 and 8 passes. The processed samples were aged at room temperature for 40 days.

Two tensile specimens were cut from the longitudinal sections of the billets parallel to the pressing direction using electro-discharge machining with gauge dimensions of $4.0 \times 3.0 \times 2.0$ mm^3. Tensile tests were performed under constant cross-head speeds with initial strain rates from 1.0×10^{-5} to 1.0×10^{-2} s^{-1} at room temperature. The stress-strain curve was plotted for each specimen and the ultimate tensile strength was measured from the curve. The elongations to failure were estimated carefully by measuring the gauge lengths before and after tensile testing.

3. Results and discussion

Representative plots of engineering stress against elongation to failure are demonstrated in Fig. 1 for the samples processed by ECAP for 8 passes, aged at room temperature for 40 days and pulled to failure at room temperature under different initial strain rates. The values of flow stress and elongation to failure for the samples processed by ECAP through 1, 2, 4 and 8 passes followed by aging at room temperature for 40 days were derived from such plots and the results are presented in Table 1. The corresponding data for the as-cast condition is also included. It is seen in Table 1 that processing by ECAP leads to a decrease in the flow stress of the as-cast material and by decreasing the strain rate the flow stress decreases implying that this material has positive strain rate sensitivity, m. Furthermore, processing by ECAP leads to an increase in the elongation to failure and samples processed to higher numbers of ECAP passes show higher elongations to failure. The total elongation to failure increases with decreasing imposed stain rate.

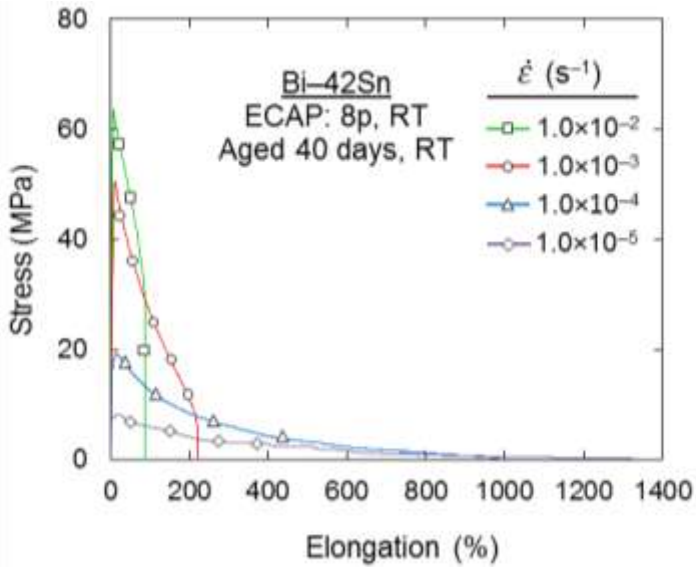

Fig. 1. Engineering stress-elongation curves of the Bi-42Sn alloy processed by ECAP for 8 passes at different initial strain rates in the range of 1.0 × 10^{-5} to 1.0 ×10^{-2} s^{-1}.

Table 1. Mechanical properties of the Bi-42Sn alloy in the as-cast condition and after ECAP processing for different numbers of passes

Condition	Strain rate (s^{-1})	Flow stress (MPa)	Elongation to failure (%)
As-cast	1.0 × 10^{-2}	81	32
	1.0 × 10^{-3}	71	53
	1.0 × 10^{-4}	54	92
	1.0 × 10^{-5}	38	112
1p	1.0 × 10^{-2}	83	45
	1.0 × 10^{-3}	65	112
	1.0 × 10^{-4}	40	150
	1.0 × 10^{-5}	24	175
2p	1.0 × 10^{-2}	79	51
	1.0 × 10^{-3}	61	143
	1.0 × 10^{-4}	36	172
	1.0 × 10^{-5}	16	245
4p	1.0 × 10^{-2}	78	80
	1.0 × 10^{-3}	55	172
	1.0 × 10^{-4}	24	389
	1.0 × 10^{-5}	15	596
8p	1.0 × 10^{-2}	72	88
	1.0 × 10^{-3}	57	225
	1.0 × 10^{-4}	25	988
	1.0 × 10^{-5}	14	1325

Figure 2 shows the variations of the elongation to failure versus the imposed initial strain rate for samples processed for different ECAP pass numbers. This plot more effectively demonstrates the effects of number of ECAP passes and initial strain rate on the elongation to failure of different samples. As mentioned earlier, the Bi-42Sn eutectic was the first alloy in which real superplasticity with an exceptional elongation to failure of ~1950% was recorded after extrusion and ageing at room temperature for 7 days [7]. With such a background, it is expected that by grain refinement through SPD techniques higher superplastic elongations may be achievable in this alloy. It is apparent from Fig. 2 that Bi-42Sn has a very low elongation to failure in the as-cast condition which is not strongly dependent on the initial strain rate. Under a strain rate of 10^{-2} s^{-1}, the as-cast Bi-42Sn is very brittle and has an elongation to failure of 32%. Under the same strain rate, the samples processed for 1, 2, 4 and 8 passes of ECAP possess elongations of 45, 51, 80, and 88%, respectively. A significant improvement in the elongation to failure is observed after ECAP for 4 passes, especially at lower initial strain rates. An elongation to failure of ~600% was recorded at the lowest initial strain rate of 1.0×10^{-5} s^{-1}. The elongation to failure in the sample processed for 8 passes is more strain rate sensitive and under the lowest strain rate of 1.0×10^{-5} s^{-1} a total elongation to failure of ~1300% was achieved, where this is a considerable improvement compared to the as-cast material. The as-cast Bi-42Sn eutectic alloy has a typical lamellar structure containing coarse lamellae of Bi and Sn which are broken down during ECAP processing leading to an improved elongation to failure in the processed samples especially after 4 passes of ECAP.

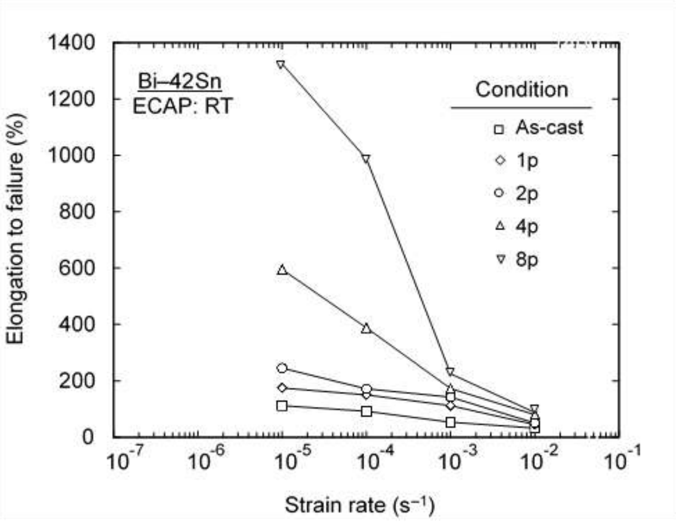

Fig.2. Variation of the elongation to failure versus the initial strain rate for the Bi-42Sn alloy processed by ECAP for different passes and pulled to failure at room temperature

The results presented in Fig. 2 indicate that the values of elongations of almost all samples are less than 400% and therefore, the flow behaviour of the material cannot be considered as superplastic under these conditions. The exceptions are the samples processed for 4 passes under an initial strain rate of 1.0×10^{-5} s^{-1} with total elongations of 600%, and those processed for 8 passes and tested under initial strain rates of 1.0×10^{-4} and 1.0×10^{-5} s^{-1} with total elongations of 990% and 1325%, respectively. These values confirm the occurrence of superplasticity in these conditions. The appearance of the specimens of the Bi-42Sn alloy processed for 8 passes after testing to failure at room temperature with different initial strain rates are demonstrated in Fig. 3. Close inspection shows that the superplastic samples pull out in quite a uniform manner with no evidence for the formation of any necking within the gauge length which is an important additional requirement of true superplastic flow [10].

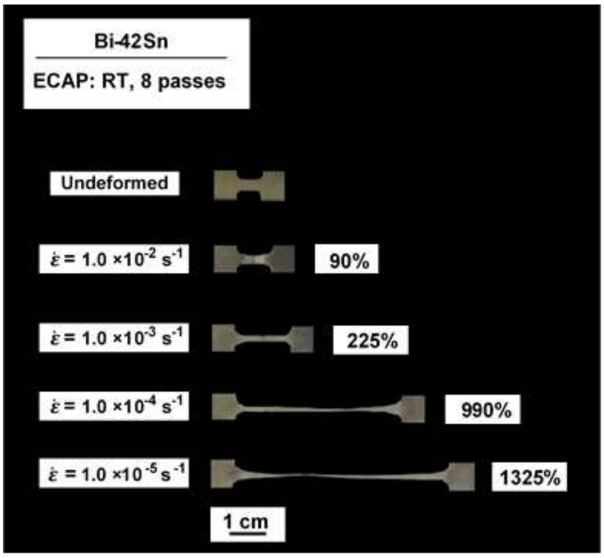

Fig. 3. Appearance of specimens of Bi-42Sn alloy, processed by ECAP for 8 passes, after testing to failure at room temperature with different initial strain rates.

For a better understanding of the dominant operating mechanisms during deformation of the Bi-42Sn alloy after the ECAP process, the flow stress was plotted logarithmically against strain rate using the data from Table 1, and the resulting diagram is presented in Fig. 4. In conventional superplastic alloys, three distinct regions of flow are observed in the flow stress-strain rate curve; over a range of intermediate strain rates high elongations to failure are recorded and the strain rate sensitivity is typically ~0.5. This is region II. At low strain rates in region I and at high strain rates in region III the strain rate sensitivity decreases [11]. However, the experimental range of strain rates in the present study was not sufficient to reveal the three regions of flow generally associated with conventional superplastic alloys where the measured elongations decrease at both high and low strain rates.

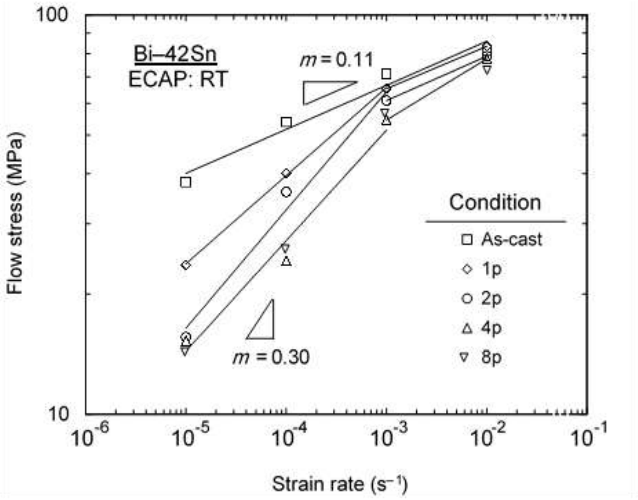

Fig. 4. Flow stress versus strain rate for the Bi-42Sn alloy processed by ECAP. Values of the strain rate sensitivity, *m*, are shown for the as-cast material and the alloy processed for 8 passes.

Values of the strain rate sensitivity, *m*, for the tested samples were derived from the data reported in Fig. 4. The measured *m*-value for the as-cast material was ~0.11 over three orders of magnitude of strain rate. As expected, no superplastic behaviour was observed in this condition which is in agreement with results of tensile tests. The same is true for the samples processed through 1 and 2 passes of ECAP. The variations of flow stress against strain rate in these samples tend to show regions II and III of a typical curve of a conventional superplastic alloy. However, the *m*-value in region II was smaller with values of ~0.22 and ~0.29 for the samples processed by ECAP for 1 and 2 passes, respectively. It is apparent that the values of strain rate sensitivity do not tend to increase significantly by increasing the numbers of passes and the samples processed for 4 and 8 passes have *m*-values of ~0.28 and ~0.3, respectively. These *m*-values are lower than the usual value of $m = 0.5$ associated with superplastic flow [12]. A value of $m = 0.3$ is observed where dislocation glide is the rate-controlling process [13]. However, dislocation glide does not give total elongations of more than 400%. Therefore, the maximum total elongation of ~1300% cannot be associated with dislocation glide. Microstructural observations in an HPT-processed Bi-42Sn alloy showing an elongation to failure of 1220% at an initial strain rate of 1.0×10^{-4} s^{-1} revealed that Bi-Sn and Sn-Sn boundaries have excellent grain boundary sliding behaviour whereas cavities are formed on Bi-Bi boundaries. Therefore, the enhanced superplastic flow behaviour of the fine-grained Bi-Sn alloy was attributed to the homogeneously distributed Bi-Sn and Sn-Sn boundaries after HPT processing [14]. In the present study, it is likely that grain boundary sliding is the dominant operating mechanism and the observed *m*-value of ~0.3 is probably due to the occurrence of grain growth during tensile testing which may lead to an apparent strain rate sensitivity of $m \approx 0.3$ in superplastic alloys [15].

4. Conclusions

ECAP was found to be a very effective way to achieve high elongations in the Bi-42Sn eutectic alloy. A sample processed for 8 passes showed an elongation to failure exceeding 1300% when testing at an initial strain rate of 1.0×10^{-5} s^{-1} and this is a considerable enhancement compared to the coarse-grained as-cast sample that only exhibited an elongation to failure of 112% under the same conditions. By producing more refined microstructure, for example by processing through larger numbers of passes, higher elongations to failure are expected in the Bi-42Sn alloy. The calculated value for strain rate sensitivity for the samples processed for 8 passes was ~0.3 over two orders of magnitude of strain rate. This implies that grain growth may operate during the deformation together with grain boundary sliding.

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