



Achieving superplasticity at room temperature in a Pb-Sn eutectic alloy through high-pressure torsion and self-annealing

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ABSTRACT

A Pb-62% Sn eutectic alloy was processed by high-pressure torsion (HPT) for one turn and stored at room temperature (RT) up to 8 months to investigate the influence of self-annealing on the mechanical behaviour. The hardness decreased significantly immediately after HPT but there was a gradual increase in hardness as the storage time increased. Electron backscatter diffraction (EBSD) revealed a significant grain refinement immediately after HPT and grain growth during storage at RT. A high fraction of high-angle grain boundaries was achieved after HPT and grain boundary sliding (GBS) was observed in the processed alloy near the hardness indents. The HPT processed samples were tested in tension at RT after different storage times using strain rates from 1.0×10^{-4} to $1.0 \times 10^{-1} \text{ s}^{-1}$. The results showed that, irrespective of the storage times, all processed samples exhibited excellent RT superplasticity at $1.0 \times 10^{-4} \text{ s}^{-1}$ and a highest elongation of 630% was recorded after storage for 4 days at RT. A detailed investigation showed that the mechanical behaviour of the alloy was highly dependent on the strain rate. Specifically, there was a transition point at a strain rate of $\sim 1.0 \times 10^{-2} \text{ s}^{-1}$ where strain softening was apparent with active GBS below the transition point and a higher strength due to grain boundary strengthening was observed at higher strain rates above the transition point.

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Introduction

Processing through the application of severe plastic deformation (SPD), using techniques such as equal-channel angular pressing (ECAP) [1] and high-pressure torsion (HPT) [2], has attracted much attention in the last three decades because it has the potential to introduce significant grain refinement to the sub-micrometer and nanometer ranges in polycrystalline materials [3–5]. Furthermore, the processing of metals by HPT is especially attractive because it introduces exceptionally small grain sizes [6, 7] and a high fraction of high-angle grain boundaries [8] which is an important requirement for achieving a superplastic forming capability.

The grain refinement achieved through SPD processing introduces ultrafine-grained metals which demonstrate excellent mechanical properties at both low and high temperatures [9, 10]. Thus, at low temperatures the strength of the material increases with a reduction in grain size through the conventional Hall–Petch relationship [11, 12] and at elevated temperatures, typically above $\sim 0.5T_m$ where T_m is the absolute melting temperature, diffusion becomes important so that there is a potential for achieving superplasticity [13].

Practically, the steady-state strain rate, $\dot{\epsilon}$, during plastic flow is described by the phenomenological creep relationship [14]

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

where A is a dimensionless constant, D is the appropriate diffusion constant [$= D_0 \exp(-Q/RT)$ where D_0 is a frequency factor, Q is the activation energy, R is the gas constant and T is the absolute temperature], G is the shear modulus, $\mathbf{b}$ is the Burgers vector, k is Boltzmann's constant, d is the grain size, σ is the flow stress and n and p are the stress and inverse grain size exponents, respectively. Thus, the stress exponent, n , is given by $\partial \ln \dot{\epsilon} / \partial \ln \sigma$ and the strain rate sensitivity, m , is the inverse of the stress exponent defined as $\partial \ln \sigma / \partial \ln \dot{\epsilon}$.

It is now well established that the measured elongations to failure in tensile testing increase with increasing values of the strain rate sensitivity, m [15, 16]. In practice, care must be taken in any attempts to identify

true superplasticity because in solid solution alloys the creep behavior may be controlled by dislocation glide where solute atoms segregate around the moving dislocations to form Cottrell atmospheres [17]. This leads to a situation where glide is slower than climb [18] and the strain rate is then characterized by a stress exponent of $n = 3$ so that the strain rate sensitivity is $m \approx 0.33$. This relatively high value of m gives tensile elongations of the order of ~ 200 – 300% or even slightly higher as, for example, in an Al–2.8 Mg alloy where the highest recorded elongation was 325% [19]. This behavior is not superplastic but instead it was designated “enhanced ductility” [20].

An analysis showed that the major flow mechanism in superplasticity is grain boundary sliding (GBS) [21] and this requires both a very small grain size, typically below $\sim 10 \mu\text{m}$, and a testing temperature above $\sim 0.5T_m$ [22]. A comprehensive model for GBS in superplasticity leads to a predicted strain rate given by Eq. (1) with $n = 2$, $p = 2$, $D = D_{gb}$ where D_{gb} is the coefficient for grain boundary diffusion and a value for the dimensionless constant of $A \approx 10$ [22]. In order to avoid the complications of enhanced ductility when glide is the rate-controlling mechanism, superplasticity is defined formally as a tensile elongation of at least 400% and it requires a measured strain rate sensitivity which is close to ~ 0.5 [23]. A recent review provides a detailed description of GBS under creep and superplastic conditions and it shows that the predicted strain rates from Eq. (1) are in excellent agreement with experimental data for a wide range of materials including Al and Mg alloys and high-entropy alloys [24].

Superplastic flow was initially considered a scientific curiosity which attracted little attention but in due course it was recognized that superplasticity may be used to form sheet metals and this provided an opportunity for achieving exceptional curved surfaces that were not readily achievable by other more conventional means. This led to the development of the superplastic forming industry [25–27] which now annually processes many thousands of tons of sheet metals and is used to produce a wide range of products ranging from the bodies of high-performance automobiles to applications in the aerospace and architectural industries and smaller products such as the cases for personal computers.

As already noted, superplasticity requires a temperature above $\sim 0.5T_m$ so that the basic step of superplastic forming is quite slow because it is necessary to heat the sheet metal to the required temperature. This limitation has led to much interest in attempting to achieve superplastic flow at room temperature with attention given primarily to two-phase alloys such as Zn-22% Al and various Al alloys. However, a close examination of these reports shows that a significant number failed to achieve true superplasticity because the maximum tensile elongations were below the required minimum value of 400% [28–33] and therefore the behavior is not easily separated from conventional dislocation glide. Nevertheless, there are several reports documenting true superplastic flow at room temperature and these cover a wide range of metals including Al-30% Zn [34], Mg-8% Li [35], various Zn-Al [36–40] and Zn-Cu [41] alloys and the Zn-22% Al eutectoid alloy [40, 42–44].

The present investigation was based on the Pb-62% Sn eutectic alloy where this is an ideal structure for achieving superplastic flow because the two-phase microstructure prevents significant grain growth during tensile deformation. Furthermore, the two-phase alloy has a low melting temperature of $T_m = 456$ K so that testing at room temperature (RT) corresponds to $>0.6 T_m$ and therefore it should be relatively easy to achieve superplasticity at this low temperature. Several reports are now available documenting the occurrence of true superplastic flow in the Pb-62% Sn alloy when testing in tension at RT using samples produced by cold rolling or extrusion [45–50], by producing materials by ECAP [51, 52] or using the new processing technique of tube high-pressure shearing (t-HPS) [53]. The highest elongation achieved in this way at RT was 2230% [47] which provides an excellent demonstration of superplasticity but falls short of the ductilities that may be achieved in this alloy when testing at higher temperatures as, for example, the maximum elongations of 4850% [54] and 7550% [55] which were attained when testing at 413 K. To date, there are no reports of superplasticity in the Pb-62% Sn alloy after processing by HPT. Accordingly, the present investigation was initiated to examine the microstructural evolution and the subsequent hardness values attained in the Pb–Sn alloy due to self-annealing at RT and the effect of this evolution on the potential for achieving superplastic flow. The investigation was

designed to build upon earlier reports that demonstrated significant grain refinement when processing the alloy by HPT [56–59].

Experimental material and procedures

The experiments were conducted using a Pb-62% Sn eutectic alloy supplied by Solder Chemistry (Landshut, Germany). The alloy was supplied as a casting billet with dimensions of $34 \times 20 \times 15$ mm³. The material was machined into rods of 10 mm diameter and then cut into individual billets with lengths of ~ 60 mm. No specific annealing treatment was performed because the very low melting point of 456 K leads to self-annealing at ambient temperature. Billets of the alloy were sliced into disks having thicknesses of ~ 1.2 – 1.5 mm and then each side of each disk was carefully polished using abrasive papers to give a series of HPT disc samples with thicknesses of $\sim 0.80 \pm 0.01$ mm. The typical microstructure of the alloy prior to processing by HPT contained reasonably homogeneously distributed Sn-rich and Pb-rich phases with an average grain size of $2.5 \mu\text{m}$ for the Pb–Sn alloy as reported earlier [56, 58].

The HPT processing was conducted under quasi-constrained conditions [60, 61] using an HPT facility consisting of massive upper and lower anvils each containing, at the centres of the inner surfaces, a circular depression with a diameter of 10 mm and a depth of 0.25 mm. For processing, a disk was placed in the depression on the lower anvil, the anvil was brought into a position such that a pressure was imposed and then concurrent torsional straining was introduced through rotation of the lower anvil. A compressive pressure was applied using a load of ~ 235 kN corresponding to an imposed pressure, P , of 3.0 GPa. Two sets of disks were processed by HPT for a total number of torsional revolutions, N , of 1 turn at a fixed rotational speed of 1 rpm. The processed disks were then stored at RT for different time periods up to a total of 8 months in order to investigate the self-annealing behaviour.

The microstructures were examined at the edges of the processed disks after different periods of storage using electron backscatter diffraction (EBSD). For

sample preparation, the processed discs were carefully ground and polished with abrasive papers and diamond paste. Each polished disk was etched for ~ 5 s in a solution of 25 ml H₂O, 5 ml HCl with a concentration of 37% and 5 g of NH₄HO₃. The EBSD patterns were collected with a step size of 0.15 µm and a clean-up procedure was then followed where the total number of modified points was less than 10%. The average grain sizes were measured using the line intercept method directly from both the SEM and OIM images where the individual Pb-rich phase was measured as one grain. This procedure was followed because the Sn-Sn and Pb-Sn boundaries are the preferred interfaces for GBS during plastic deformation in the Pb-Sn alloy [62].

Subsequently, all of the HPT-processed disks having different storage times were polished again to achieve mirror-like surfaces and these surfaces were used to record the values of the Vickers microhardness, Hv. The hardness measurements were taken with an FM-300 microhardness tester equipped with a Vickers indenter using a load of 10 gf and a dwell time of 15 s. Two different procedures were used to measure the distribution of Hv values on the surfaces of the disks after different storage times. First, the average values of Hv were recorded on the processed discs after storage for 0 (less than 4 h of storage after HPT processing), 4 and 9 days by taking measurements at selected positions along the disk diameters with the individual points separated by fixed incremental distances of 0.5 mm. These measurements were then plotted as Hv against the distance from the centre of each disk and the error bars were recorded at the 95% confidence level based on measurements from four separate adjacent indentations. Second, the microhardness values at over 5 points were taken at the edge of each disk and these locations were carefully monitored and re-measured for up to 20 days at RT.

A different set of HPT disks was tested in tension to evaluate the superplastic flow after storage at RT for 1, 4 and 12 days. Two tensile specimens were cut from each disk with gauge lengths of 1.5 mm and cross-sectional areas of 1.0 × 0.6 mm². As in earlier experiments [63], the gauge lengths were machined to exclude the central regions of the disks to avoid any inhomogeneities in these centre regions. All specimens were tested at RT using a Zwick testing machine operating at a constant cross-head speed with initial strain rates from 1.0 × 10⁻⁴ to 1.0 × 10⁻¹ s⁻¹. These specimens were used

to measure the elongations to failure after different storage periods.

Experimental results

Microstructural evolution during self-annealing

Microstructural observations were undertaken at the edges of the Pb-Sn disks processed by HPT through 1 turn after 0 day of self-annealing followed by different storage times of 2, 6 and 11 days at RT. Representative phase maps (a, b, c and d) and orientation imaging (OIM) maps for the Sn-rich (e, f, g and h) and the Pb-rich phases (i, j, k and l) are shown in Fig. 1 at the upper, middle and lower rows, respectively, where the left column shows the edge of the disk a few hours after HPT processing and the second, third and fourth columns show the microstructures after storage for 2, 6 and 11 days at RT, respectively. In the OIM maps, high-angle grain boundaries (HAGBs) with misorientations of > 15° are shown as thicker black lines and the thinner black lines represent low-angle grain boundaries (LAGBs) of 2–15°.

Inspection of the maps in the left column of Fig. 1 shows that both the Pb and the Sn phases are refined and homogeneously distributed through HPT. However, it is evident from the remaining three columns that self-annealing of the Pb-Sn alloy leads to a recovery of the refined microstructure by HPT so that the average grain size of the Sn phase and the average phase size of the Pb phase increases with increasing storage time. It is important to note that these averages were calculated as the average size of the Sn grains and the average size of the Pb phases where every Pb phase was treated as a single grain even though there were smaller grains within each Pb phase. This procedure was followed because, based on an earlier investigation [62], there was an insignificant contribution to GBS from the Pb-Pb interfaces by comparison with the Sn-Sn and Sn-Pb interfaces. It is also apparent that each phase tends to agglomerate so that the sizes of both the Pb and the Sn phases become progressively larger with the self-annealing time where this is especially clear for the phase maps in the upper row of Fig. 1. The OIM maps also suggest a higher fraction of HAGBs in the alloy immediately after HPT such that the microstructural recovery by self-annealing at RT

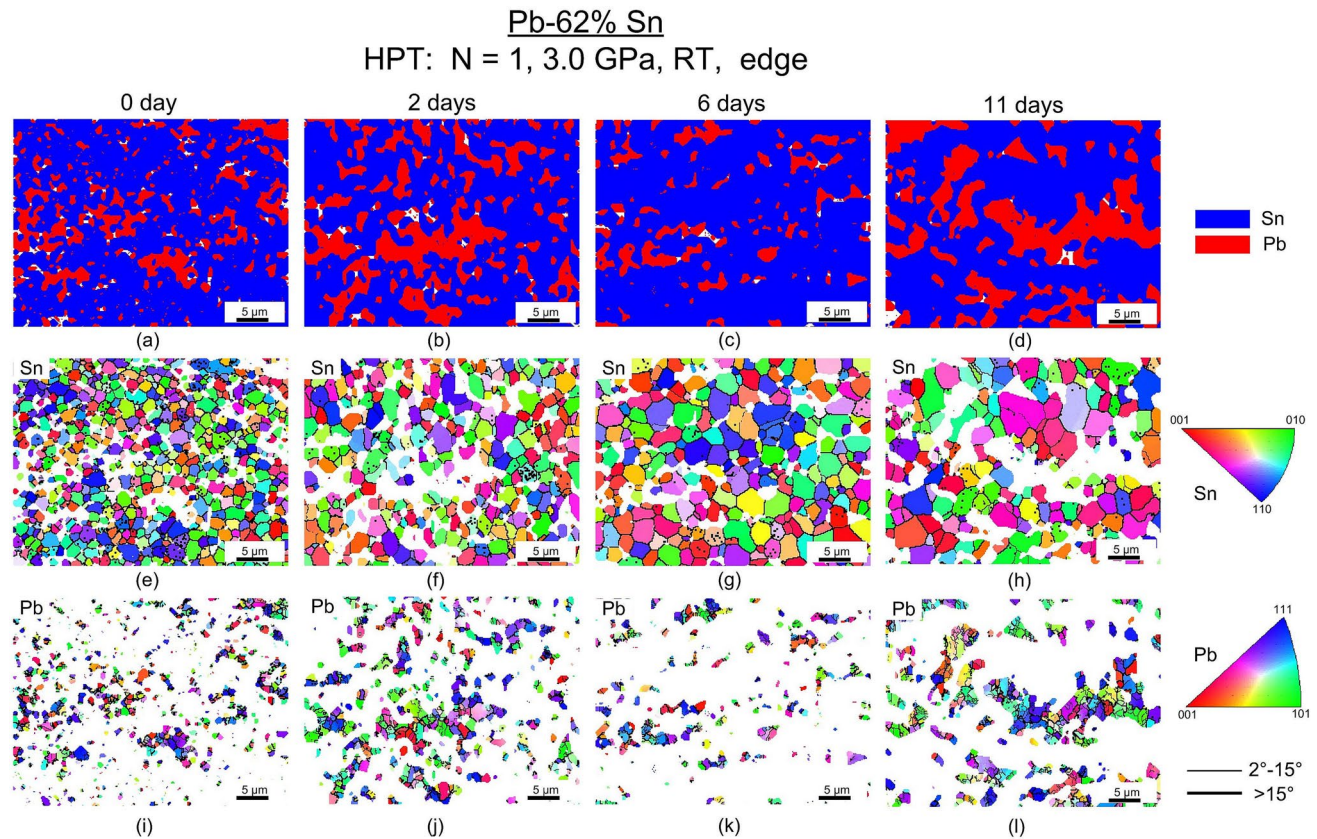


Figure 1 Representative phase maps (upper row) and OIM maps (central and lower rows) for Sn-rich and Pb-rich phases where **a**, **e** and **i** shows the edge of the disk after HPT processing (0 day), and **b**, **f** and **j**, **c**, **g** and **k** and **d**, **h** and **l** show the microstructures

after storage at RT for 2, 6 and 11 days, respectively: in the OIM maps, high-angle grain boundaries with misorientations of $>15^\circ$ are shown in thicker black lines and the thinner black lines represent low-angle grain boundaries of $2\text{--}15^\circ$.

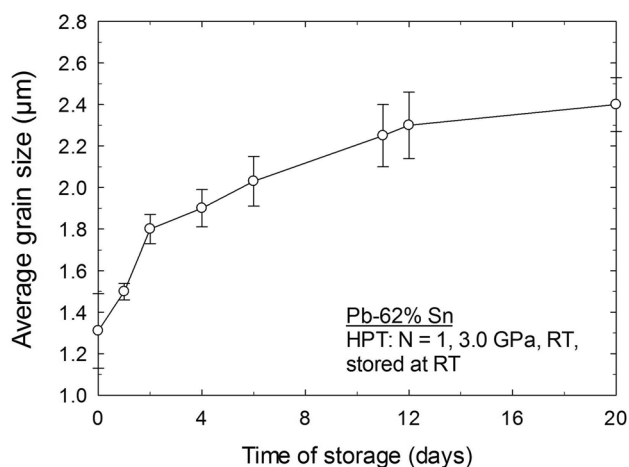


Figure 2 The average grain sizes against the storage time for the Pb–Sn alloy after HPT and subsequent RT storage up to 20 days: the average grain size considers the average of the Sn grains and the Pb phases in each sample due to the very small size of the Pb phases compared with the Sn grains.

has a small effect on the fractions in each phase of the alloy during storage at RT.

It was noted earlier that there was an initial grain size of $\sim 2.5\ \mu\text{m}$ in the Pb–Sn alloy prior to the HPT processing [56, 58]. Thereafter, Fig. 2 plots the average grain size against the storage time at RT up to a total of 20 days after processing by HPT through 1 turn. It is apparent that the grains grow after storage at RT and this growth is significant within the first 12 days of storage but less pronounced after 12 days.

Quantitative information on the grain boundary misorientations are summarized in Fig. 3 where the number fractions are plotted as a percentage against the misorientation angle for (a)–(b) the as-cast alloy without processing, (c)–(d) the alloy immediately after HPT and (e)–(f) the alloy after storage for 11 days, with the Sn and Pb phases in the left and right columns, respectively. Since the Sn phase has a tetragonal

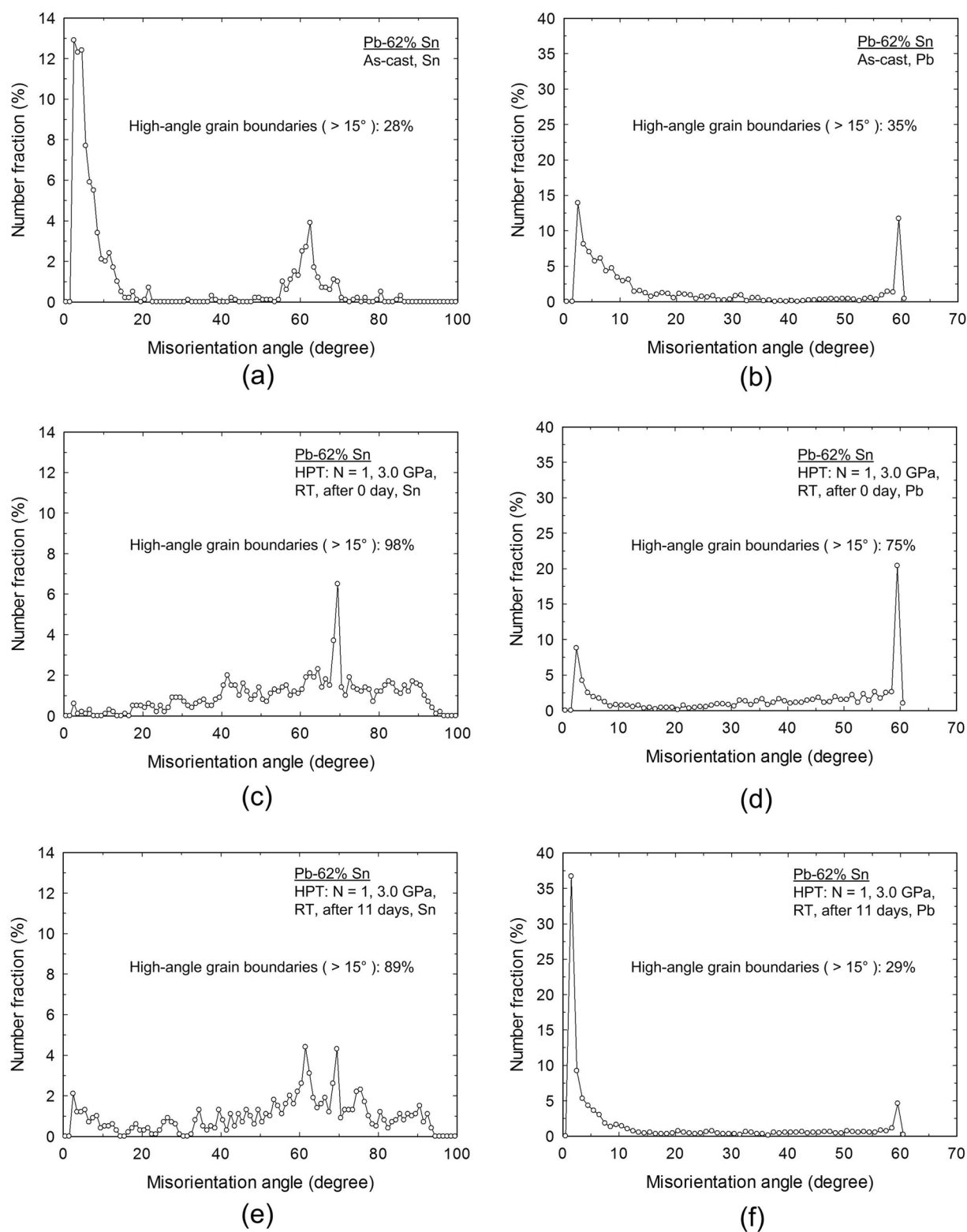


Figure 3 The plots of number fraction in percentage against the misorientation angle for **a-b** the as-cast alloy without processing, **c-d** the alloy immediately after HPT and **e-f** the alloy after stor-

age for 11 days: the grain boundaries were identified for either Sn phase (left column) or Pb phase (right column).

structure, the misorientation angles are measured in the range of 0° – 98° in Fig. 3(a), (c) and (e).

Since the cyclic twin nucleation mode is preferred during the solidification of the Sn-based alloy [64], it means that a large fraction of LAGBs is observed in the as-cast sample as shown in Fig. 3(a–b). However, immediately following the HPT processing there is a very high fraction of HAGBs in the alloy as shown in Fig. 3(c–d) and measurements for the Sn and Pb phases give fractions of 98% and 75% of HAGBs, respectively. Thereafter, although there is only a minor reduction in the HAGB fraction in the Sn phase after storage at RT for 11 days, the Pb phase recovers by self-annealing to give a reduced HAGB fraction of 29%.

Microhardness evolution during self-annealing

The results of the Vickers microhardness measurements in the Pb–Sn alloy after HPT with increasing storage time are shown in Fig. 4 where (a) gives the variation of hardness with distance from the centre of the disks after HPT for 1 turn and storage at RT for 0, 4 and 9 days and (b) shows the variation of hardness at the edges of the disks with increasing time of storage up to 20 days. The hardness value of ~ 10 Hv for the as-cast condition prior to HPT is marked with a dashed line in Fig. 4a and details of the hardness measurements were also reported earlier [57].

Several important trends are visible in Fig. 4(a). First, the hardness after HPT is consistently lower than in the as-cast condition. Second, there are high hardness values at the central regions but the hardness decreases towards the peripheries for all conditions and a minimum hardness of $H_v \approx 1.5$ was recorded at the edge of the disk immediately after HPT. Third, there is an increase in the hardness values with increasing storage time and the hardness increases significantly within ~ 1.5 mm of the centre of the disk. Fourth, there is essentially a plateau region with $H_v \approx 9$ in the central regions of the disks where the hardness remains reasonably constant after 4 and more days of storage. This contrasts with the edges of the disks where the hardness increases with increasing storage time.

This early rapid increase in hardness at the edges of the processed disks is shown in Fig. 4(b) where the hardness increases up to storage for about 6 days and thereafter there is a gradual increase in hardness towards $H_v \approx 7$ without complete saturation after 20 days of storage. These measurements demonstrate that the Pb–Sn alloy exhibits dramatic strain softening immediately after HPT together with significant grain refinement but this is followed by hardening during storage with, as shown in Fig. 2, significant grain growth.

An additional microstructural investigation was conducted to check for any changes associated with straining through the use of the microhardness

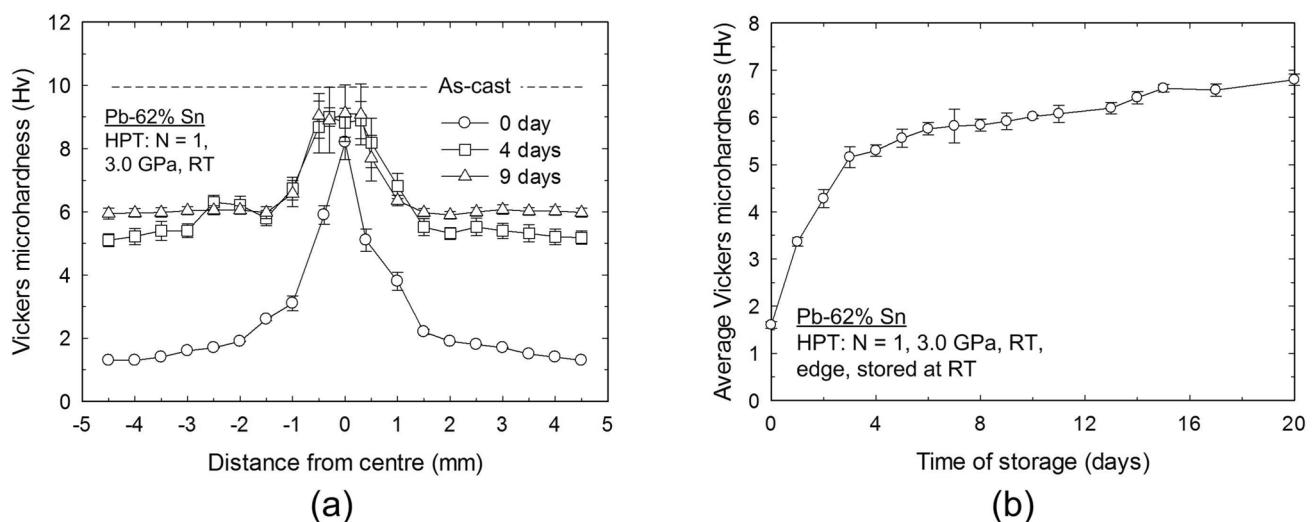
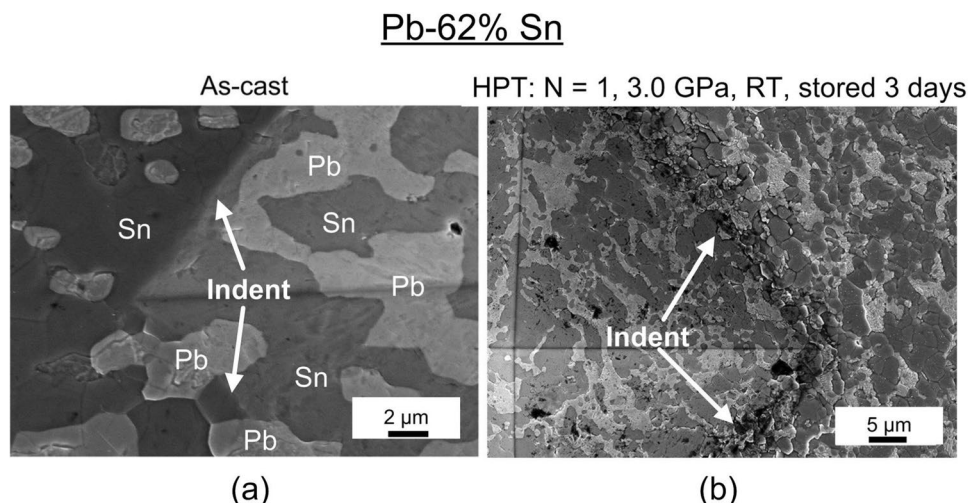


Figure 4 a Variation of hardness versus distance from center of the disks after HPT (0 day) and after storage at RT for 4 and 9 days and b the variation of hardness at the edges of the disks with increasing time of storage up to 20 days.

Figure 5 SEM images with the indent marks taken at the edges of the Pb–Sn disks **a** in the as-cast condition and **b** after processing by HPT for one turn and storing at RT for 3 days.



indenter. Figure 5 displays SEM images of Vickers indentations taken near the edges of the disks for (a) the as-cast alloy before HPT processing and (b) the alloy processed by HPT for 1 turn at 3.0 GPa and stored for 3 days at RT. The Sn-rich phase is dark grey and the Pb-rich phase is light grey in these SEM images. For the as-cast condition in Fig. 5(a) the edges of the indentation marks are smooth whereas for the sample after HPT and 3 days storage in Fig. 5(b) there is clear evidence for GBS such that the grains at the edges of the indentations are forced to slide against each other to accommodate the deformation. This type of behavior is generally accepted as evidence for the occurrence of GBS adjacent to the indentations [65–73]. Furthermore, it is easily recognized that most sliding occurs at the Sn–Sn and Pb–Sn interfaces but not at the Pb–Pb interfaces within the Pb phases in the processed sample.

Tensile testing at room temperature

The microstructure after straining by indentation demonstrates the occurrence of GBS at RT in the Pb–Sn alloy after HPT and it is reasonable to anticipate, therefore, that it will exhibit superplastic flow in tensile testing at RT. Accordingly, samples were processed by HPT and then tested in tension at RT at strain rates from 1.0×10^{-4} to $1.0 \times 10^{-1} \text{ s}^{-1}$ and additional samples were also tested after HPT and storage at RT for 1 and 12 days. The stress–strain curves are shown in Fig. 6 together with the curve for an as-cast sample having the same sample dimensions and tested at $1.0 \times 10^{-1} \text{ s}^{-1}$.

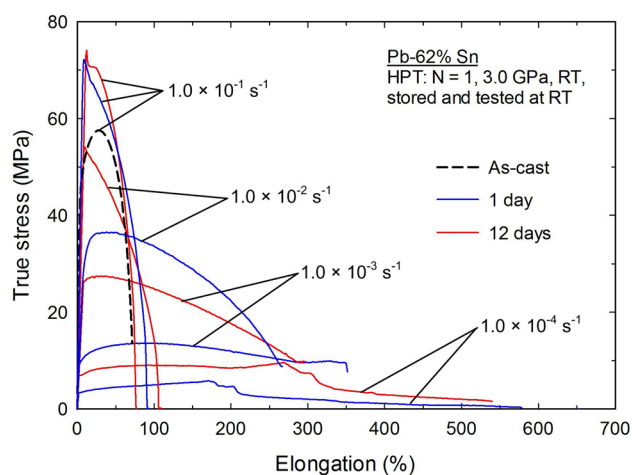


Figure 6 Stress–strain curves obtained from tensile testing at 1.0×10^{-4} to $1.0 \times 10^{-1} \text{ s}^{-1}$ at RT for the Pb–Sn alloy after HPT and subsequent storage for 1 and 12 days; for comparison, an as-cast sample having the same sample dimensions was tested at $1.0 \times 10^{-1} \text{ s}^{-1}$ and the result is shown as a dashed line.

The curves for different strain rates in Fig. 6 demonstrate several important trends in the plastic flow of the Pb–Sn alloy after HPT. First, the samples after HPT are strain rate sensitive at RT so that higher elongations to failure are achieved at the slower strain rates and there are higher yield strengths at faster strain rates. Second, the HPT samples after storage for 12 days show higher yield strengths than after 1 day of storage at all strain rates under the present testing conditions. This result is reasonable since, as shown in Fig. 4(b), the hardness increases with increasing storage time at the edges of the alloy disks after processing

by HPT. Third, both samples after HPT with storage for 1 and 12 days exhibit excellent superplastic elongations of $> 550\%$ when testing at RT at a strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$ whereas the samples have elongations of $< 400\%$ and they are not superplastic at faster strain rates. Fourth, the as-cast sample shows a lower yield strength compared with the HPT processed and self-annealed samples at the rapid strain rate of $1.0 \times 10^{-1} \text{ s}^{-1}$.

Figure 7 shows the specimens pulled to failure at four different strain rates after HPT for one turn and stored for 4 days at RT where the upper specimen is untested. Superplastic flow was achieved at strain rates at and below $1.0 \times 10^{-3} \text{ s}^{-1}$ with the highest elongation of 630% at $1.0 \times 10^{-4} \text{ s}^{-1}$ in a specimen showing uniform deformation with no evidence of necking within the gauge length as is typical of true superplastic behaviour [74]. By contrast, the abrupt failure at the fastest strain rate of $1.0 \times 10^{-1} \text{ s}^{-1}$ denotes quasi-brittle behaviour which is also a characteristic of superplastic materials at high strain rates above the

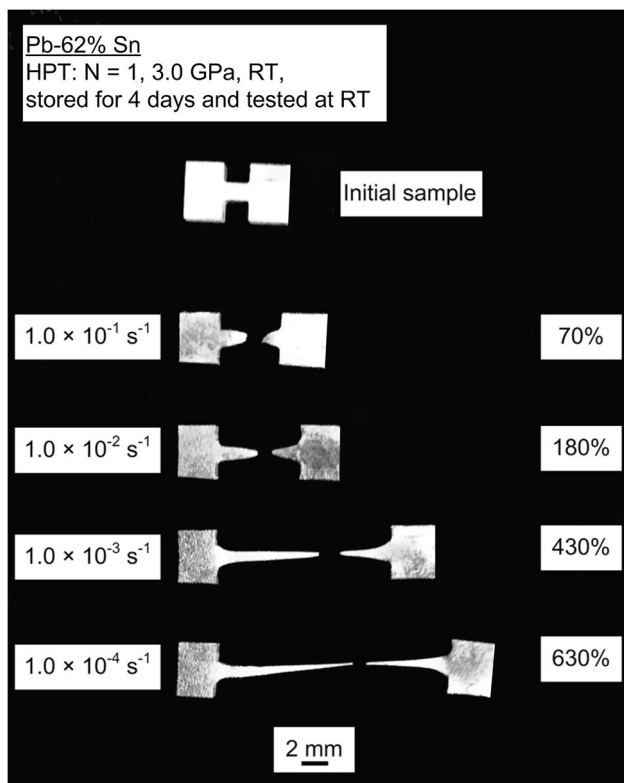


Figure 7 Samples processed by HPT for one turn and stored for 4 days and then pulled to failure at different initial strain rates where the upper specimen is untested.

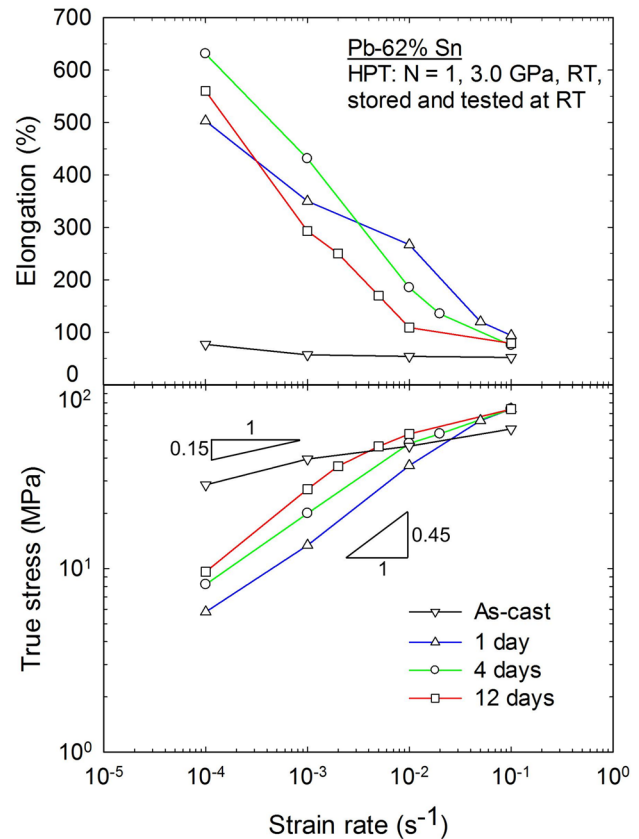


Figure 8 Elongation to failure (upper) and flow stress (lower) plotted against the initial strain rate for the Pb–Sn alloy in the as-cast condition and processed by HPT for one turn and stored for 1, 4 and 12 days.

limited strain rate range associated with conventional superplasticity [74].

The tensile testing results are summarized in Fig. 8 in plots of the measured elongations to failure (upper) and the flow stresses (lower) against the initial strain rate for the as-cast material and for samples after HPT and storage for 1, 4 and 12 days. Inspection of the upper plot in Fig. 8 shows that all HPT samples exhibit typical superplastic elongations of at least 400% at a strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$. It is noted also that, although the Pb–Sn alloy is two-phase and is ideal for achieving superplastic flow, the as-cast samples exhibit elongations to failure of $< 100\%$ at all strain rates and this is due to two limiting factors. First, by the very small gauge lengths of the tensile specimens which were machined from the disks fabricated for later processing by HPT [55, 75–77] and second, by the low fraction of HAGBs in the as-cast alloy as documented in Fig. 3(a)–(b). Nevertheless,

despite the inherent difficulty in achieving superplastic flow with these very small sample dimensions, the alloy exhibited excellent superplastic ductilities for all storage conditions at the lowest experimental strain rate. The lower plot of Fig. 8 shows that the strain rate sensitivity, m , is ~ 0.15 for the as-cast alloy at all strain rates whereas a higher value of $m \approx 0.45$ was attained for the HPT processed alloy at strain rates slower than $\sim 1.0 \times 10^{-2} \text{ s}^{-1}$ where this is consistent with the requirement of $m \approx 0.5$ for true superplastic flow [22, 23].

Discussion

The interplay between strain softening and anneal hardening

In this study the microstructure of the as-cast Pb-62% Sn eutectic alloy was successfully refined through HPT processing at RT and the alloy exhibited grain growth by self-annealing when samples were stored at RT as shown in Fig. 1. The samples with refined grains after HPT processing demonstrate a strain softening behaviour in which the hardness values for the HPT samples are much lower than for the as-cast sample in the absence of HPT processing. Thus, the as-cast hardness was $H_v \approx 10$ and the lowest recorded hardness was $H_v \approx 1.5$ at the disk edge immediately after HPT as shown in Fig. 4. Nevertheless, the hardness increased through self-annealing with increasing storage time and there was a saturated average hardness value of $H_v \approx 7$ after a reasonably long period of self-annealing at RT as shown in Fig. 4(b).

Strain softening after HPT processing is different from the general behaviour for most metals where there is strain hardening and ultimately a high saturated hardness with increasing imposed strain [78–81]. In order to understand this behavior, therefore, it is necessary to review the three different types of variation of hardness with strain for metals processed by HPT as summarized earlier [82], where this variation is dependent upon the nature of recovery in the material. Accordingly, most metals are of type 1 and exhibit strain hardening in the initial stages of processing by HPT where the hardness simply increases above the initial value and finally stabilizes at a saturation value: this behavior is designated “Without Recovery”. Alternatively, and as first reported for high-purity

aluminum [83], in type 2 materials there is a very high stacking fault energy so that the material exhibits easy cross-slip and rapid microstructural recovery and the hardness then exhibits a bell-shaped curve with an initial increase, a decrease and then a saturation value that is higher than the initial value but lower than the peak value: this behavior is designated “With Recovery”. There are several reports confirming this type of hardness behavior [84–89]. Third, in type 3 materials the measured hardness values all lie below the initial hardness prior to HPT processing despite the grain refinement that is achieved by HPT; this behavior is designated “With Weakening” and it leads ultimately to a low value of the saturation hardness where this weakening matches the behavior shown in Fig. 4(a) in the present investigation. Similar weakening was reported also for the Zn-22% Al eutectoid alloy [90–92] and the weakening in these two-phase alloys is due to the loss of precipitates as a consequence of the high pressures experienced in HPT processing. There are also examples of a different weakening effect in metals such as Pb, Sn and In where the hardness values are slightly lower in samples processed by HPT compared with the initial annealed condition due to dynamic softening in materials having low melting temperatures [89]. A review is available summarizing experimental reports of the hardness behavior during HPT for various metals and alloys [93].

The significance of grain boundary sliding on strength

The Pb-62% Sn alloy has a low melting temperature so that RT corresponds to a temperature above $\sim 0.6T_m$. This means that, as demonstrated in the present experiments, it is possible to achieve excellent superplastic elongations when testing at RT at a strain rate of $1.0 \times 10^{-4} \text{ s}^{-1}$. Several reports are now available demonstrating RT superplasticity in this Pb–Sn alloy as summarized in Table 1 where the grain sizes, tensile strain rates and the measured elongations to failure are listed, with the elongations defined as $\Delta L/L_0$ where L_0 is the initial gauge length and ΔL is the increase in gauge length to the point of fracture, respectively. The various samples were prepared by cold rolling [45–48], extrusion + annealing [49, 50], ECAP [51], ECAP + cold rolling [52] and in the present investigation by HPT and self-annealing.

Inspection shows that all of the materials listed in Table 1 have grain sizes below 10 μm and superplastic

Table 1 Elongations of the Pb-62% Sn alloy at RT

Processing method	d^* (μm)	Strain rate (s^{-1})	$\Delta L/L_0$	Reference
Cold rolling	6.1	6.6×10^{-2}	120%	Ahmed and Langdon [47]
		1.3×10^{-2}	130%	
		6.6×10^{-3}	140%	
		6.6×10^{-4}	210%	
		1.3×10^{-4}	330%	
		6.6×10^{-5}	650%	
		6.6×10^{-6}	2230%	
Cold rolling + 10% Prestrain	6.1	6.6×10^{-5}	725%	Ahmed and Langdon [46]
Cold rolling	6.1	6.6×10^{-3}	100%	Ahmed and Langdon [45]
		6.6×10^{-5}	775%	
Cold rolling	3.3	6.6×10^{-2}	75%	Ahmed and Langdon [48]
		6.6×10^{-3}	75%	
		6.6×10^{-4}	290%	
		6.6×10^{-5}	550%	
Extrusion + Annealing	2.5	1.0×10^{-2}	200%	Ha and Chang [50]
		1.0×10^{-3}	600%	
		1.0×10^{-4}	1200%	
Hot extrusion + Annealing	2.2	1.3×10^{-5}	830%	Soliman [49]
		1.3×10^{-4}	1610%	
		3.3×10^{-4}	500%	
		6.6×10^{-4}	300%	
		1.3×10^{-3}	190%	
		1.3×10^{-2}	40%	
		1.3×10^{-5}	770%	
	3.3	5.3×10^{-5}	1340%	
		1.3×10^{-4}	680%	
		1.3×10^{-4}	550%	
		3.0×10^{-4}	270%	
		1.3×10^{-3}	130%	
		1.3×10^{-5}	600%	
		4.0×10^{-5}	370%	
	6.0	1.3×10^{-4}	190%	
		3.3×10^{-4}	110%	
		1.3×10^{-3}	90%	
		1.3×10^{-5}	880%	
		4.0×10^{-5}	290%	
		1.3×10^{-4}	90%	
		3.3×10^{-4}	60%	
ECAP	6.0	1.3×10^{-3}	50%	El-Danaf et al. [51]
		1.0×10^{-2}	50%	
		5.0×10^{-3}	80%	
		1.0×10^{-3}	170%	
		5.0×10^{-4}	310%	
		1.0×10^{-4}	590%	
		5.0×10^{-5}	490%	
ECAP + Cold rolling	5.9	1.0×10^{-2}	20%	Lugon et al. [52]
		1.0×10^{-3}	60%	
		1.0×10^{-4}	190%	

Table 1 continued

Processing method	d^* (μm)	Strain rate (s^{-1})	$\Delta L/L_0$	Reference
t-HPS	1.1	1.3×10^{-2}	670%	Lin et al. [53]
HPT + Self-annealing	1.5	6.6×10^{-3}	90%	Current work
		6.6×10^{-4}	120%	
		1.0×10^{-2}	270%	
		1.0×10^{-3}	350%	
		1.0×10^{-4}	500%	
	1.9	1.0×10^{-1}	80%	
		2.0×10^{-1}	140%	
		1.0×10^{-2}	190%	
		1.0×10^{-3}	430%	
		1.0×10^{-4}	630%	
	2.3	1.0×10^{-1}	80%	
		1.0×10^{-2}	110%	
		5.0×10^{-3}	170%	
		2.0×10^{-3}	250%	
		1.0×10^{-3}	290%	
		1.0×10^{-4}	560%	

*The grain sizes for cold rolling and ECAP are the phase size without considering grains in the Sn-rich and Pb-rich phase and the grain size for HPT is for the Sn grains and the Pb phase

elongations of $> 400\%$ were observed typically at strain rates of the order of $\sim 1.0 \times 10^{-4} \text{ s}^{-1}$. The superplastic elongations were assisted by the high fractions of HAGBs in the materials after SPD processing as shown in Fig. 3 since early experiments on aluminum tricrystals demonstrated conclusively that high-angle grain boundaries are required in order to achieve GBS [94]. The low fractions of HAGBs in the as-cast material, as shown in Fig. 3(a)-(b), also account for an inability to achieve superplastic elongations in the as-cast material, as shown in Fig. 8, despite the very small grain size in the material prior to HPT processing. In addition, an earlier investigation on the Pb–Sn alloy demonstrated that the microstructure in the as-cast alloy was divided into large domains where each represented a nucleus during solidification and within each domain there were three major orientations with a twin relationship of $62.8^\circ < 100^\circ$ which represented the sixfold cyclic twin [59]. In practice, a large fraction of LAGBs is formed during solidification to accommodate the misfits of these sixfold cyclic twins and the consequent interlocked twin grains in each domain further hinder the ability for achieving GBS which is additional evidence supporting the poor elongations in the as-cast alloy.

Although the length of storage time and the degree of self-annealing provide no clear differences in the elongations to failure under the present testing

conditions as shown in Fig. 6, the Vickers microhardness and the tensile strength exhibit clear dependencies on the grain sizes. In practice, plots of the average grain size and the Vickers microhardness against the time of storage, as shown in Figs. 2 and 4(b), respectively, exhibit similar trends so that the grain growth through self-annealing provides increasing Hv with increasing storage time at RT. Furthermore, the plot of flow stress versus strain rate reveals a higher flow stress with increasing storage time at RT at all strain rates in the present testing conditions as shown in the lower plot in Fig. 8. It is concluded from Fig. 8 that the strength of the Pb–Sn alloy is strongly affected by the grain size and it is noted that there is a similar strain rate sensitivity over a strain rate range from 1.0×10^{-4} to $1.0 \times 10^{-3} \text{ s}^{-1}$. This means that the fine grain size after 1 day of storage provides more grain boundaries and thus promotes GBS which then weakens the strength whereas a coarser grain size after 12 days reduces the volume of grain boundaries and therefore hinders GBS and thereby enhances the strength. At the faster strain rates, this difference in terms of the flow stress for different storage conditions is reduced significantly. A similar tendency was noted in other work on the Pb–Sn alloy where at fast strain rates the difference in flow stress was small regardless of the difference in grain size [49]. This change occurs because there is a transition

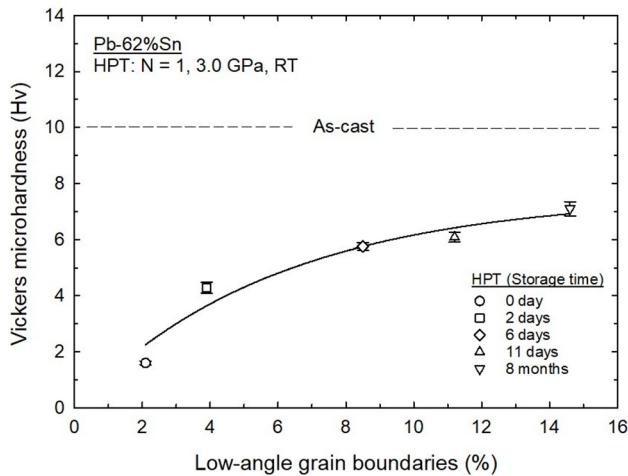


Figure 9 Vickers microhardness plotted against the fraction of LAGBs for the Pb–Sn alloy after HPT and RT storage for 0, 2, 6 and 11 days and for 8 months where the upper dashed line denotes the hardness of ~ 10 in the as-cast condition.

to deformation by dislocation creep rather than by GBS at the faster strain rates. In Fig. 8 it is noted also that in the fast strain rate region of 10^{-2} to 10^{-1} s^{-1} the measured flow stresses are higher than for the as-cast condition whereas in the microhardness measurements in Fig. 4(a) all values are lower than for the as-cast condition. This behavior is due to the slow strain rate of less than $\sim 10^{-1} \text{ s}^{-1}$ associated with conventional Vickers microhardness testing [92].

The grain boundary type should affect the strength of the alloy since GBS plays a major role in the deformation of superplastic materials. Accordingly, the Vickers microhardness was plotted against the fraction of LAGBs in Fig. 9 after HPT and storage at RT for 0, 2, 6 and 11 days and for 8 months, with the upper dashed line denoting the hardness of $H_v \sim 10$ in the as-cast condition. Thus, the microhardness values increase as the fraction of LAGBs increases and the fraction of HAGBs decreases, and this again demonstrates that the strength of the alloy relies on HAGBs and the presence of GBS.

Summary and conclusions

1. A Pb–62% Sn eutectic alloy was processed by HPT for 1 turn under 3.0 GPa at RT and the microstructure was successfully refined but with the occurrence of grain growth through self-annealing dur-

ing storage at RT. There was an immediate strain softening after HPT processing then the hardness increased during self-annealing.

2. Processing by HPT provided a significant increase in the fraction of HAGBs in the alloy. Inspection of the indentation marks on the alloy after HPT showed clear evidence for a high level of GBS at the preferred sliding interfaces of Sn–Sn and Pb–Sn.
3. A series of the processed samples having different storage times was tested in tension at RT. The results demonstrated that, despite the storage time, all processed alloy exhibited excellent RT superplasticity at $1.0 \times 10^{-4} \text{ s}^{-1}$ with a high strain rate sensitivity of $m \approx 0.45$ and a highest elongation of 630% after storage for 4 days at RT.

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Author contributions

Nian Xian Zhang: Methodology, Collecting data, Formal Analysis, Writing—Original Draft. Megumi Kawasaki: Writing—Reviewing and Editing. Yi Huang: Formal Analysis, Supervision, Writing—Reviewing and Editing. Terence G. Langdon: Conceptualization, Supervision, Writing—Original Draft.

Data availability

Data will be made available upon reasonable request.

Declarations

Conflict of Interest All authors declare no financial or non-financial competing interests.

Ethical approval Not Applicable.

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