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Tensile behavior of an eutectic Pb–Sn alloy processed by ECAP and rolling☆

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ABSTRACT

A eutectic lead–tin alloy was prepared by melting the commercially pure metals and cast in cylindrical molds with 10 mm diameter. The billets were processed by 4 passes of ECAP using a die with 90° between channels through route A and rolled to a final thickness of ∼1 mm. The microstructure was determined by optical microscopy. Tensile tests were carried out at room temperature in the strain rate range between 10^{-4} and 10^{-2} s^{-1}. The results show the elongation to failure increases at low strain-rates. The strain-rate sensitivity parameter was determined and a maximum value of ∼0.4 was observed at the lowest strain-rate.

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1. Introduction

Superplasticity refers to properties of high neck-free elongations in materials after tensile tests, at high temperature. The strain rate sensitivity m ($= \partial \ln \sigma / \partial \ln \dot{\varepsilon}$) is used as a parameter to evaluate superplasticity – σ is the flow stress and $\dot{\varepsilon}$ is the strain rate. Formally, superplasticity is defined as the ability of a material to exhibit elongation of at least 400% with strain rate sensitivity of approximately 0.5 [1]. Materials exhibiting high strain rate sensitivity ($m > 0.4$) are considered superplastic when compared to conventional materials ($m < 0.2$). Experiments show that high strain rate sensitivity is related to small grain sizes, usually <10 μm [2]. The use of severe plastic

deformation, as equal-channel angular pressing (ECAP), has been shown as an effective procedure for achieving grain refinement, since the grains can be reduced to the submicrometer range [3,4].

The eutectic Pb–62%Sn alloy may exhibit superplasticity even at room temperature. Experiments show that Pb–62%Sn specimens with fine grain size exhibited higher ductility than their coarse grained counterparts. Large elongations are observed at low strain rates (<10^{-3} s^{-1}) and shift to even lower strain rates with increasing the grain size [5]. It is known now that the eutectic lead–tin alloy exhibits superplasticity at room temperature after ECAP processing. The aim of the present paper is to evaluate the influence of rolling after ECAP on the tensile behavior of this alloy.

2. Experimental procedure

The Pb–62 wt% Sn alloy was prepared by melting commercially pure lead and tin and casting in a cylindrical mold of 10 mm diameter. The cast material was cut into billets with 60 mm length and processed by 4 passes of Equal-Channel Angular Pressing (ECAP), using a die with 90° between channels. Processing was carried out using route A in which the billets are not rotated between passes of ECAP [6]. Following ECAP, the billets were rolled to produce sheets with 1 mm thickness. Tensile specimens with 10 mm gauge length and $2 \times 1\,\mathrm{mm}^2$ cross section were machined out of the rolled sheets. The specimens were tested in an Instron machine operating at a constant cross-head displacement rate with initial strain rates in the range from 1.0×10^{-5} to $1.0 \times 10^{-2}\,\mathrm{s}^{-1}$. The specimens were pulled to failure except for the specimen tested at the lowest strain rate of $1.0 \times 10^{-5}\,\mathrm{s}^{-1}$ which was pulled only to a strain of 0.1. All tensile tests were carried out at room temperature. The load data was converted into engineering stress and the engineering strain was determined using a video extensometer that tracked the position of two marks in the gauge length of the specimens during testing. After being pulled to failure the pieces of the specimens were put together and the final gauge lengths were measured. The total elongation was determined as the elongation of the gauge length divided by the initial gauge length.

The microstructure of the material was evaluated after ECAP and rolling. A sample was cut from the rolled sheet, mounted using polymeric resin, ground with emery paper up to 4000 grit size, followed by polishing in Al_2O_3 suspension and then the final polishing step was carried out using OPS suspension. The final polishing step revealed the different phases of the alloy. After polishing, the sample was subjected to 0.1 tensile straining in order to reveal the grain boundaries. The microstructure was analyzed using optical microscopy and the grain size was determined by the mean linear intercept method.

3. Results

The microstructure of the Pb–62% Sn alloy consists of a distribution of two phases which are distinguished using optical microscopy. The distribution of the phases exhibits a slight alignment to the ECAP and rolling direction, which is attributed to the elongation of the phases in this direction. Fig. 1 shows a representative image of the microstructure of the eutectic alloy with the rolling direction aligned horizontally. An average grain size of 5.9 μm was determined by the linear intercept method.

Fig. 2 shows the tensile specimens after testing at different strain rates and an untested specimen for comparison. It is observed that the elongation increases with decreasing the strain rate. The maximum elongation, 190%, was obtained at the lowest strain rate, $10^{-4}\,\mathrm{s}^{-1}$. A well defined neck can be distinguished in the samples pulled to failure at $10^{-2}\,\mathrm{s}^{-1}$ and $10^{-3}\,\mathrm{s}^{-1}$ but it is not clearly resolved in the sample pulled at $10^{-4}\,\mathrm{s}^{-1}$.

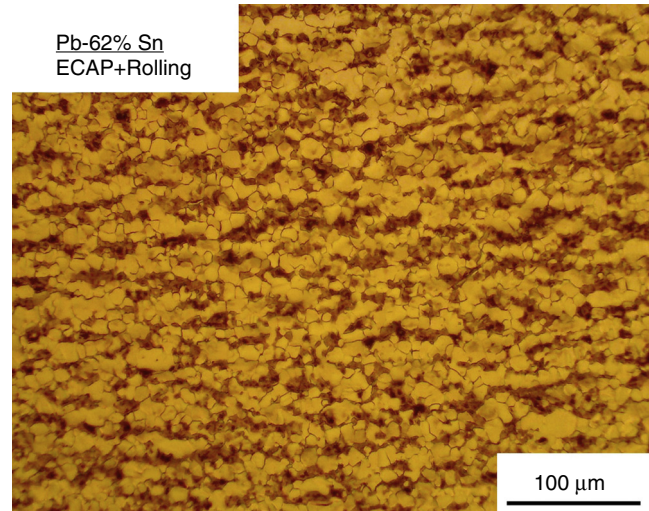
Pb-62% Sn
ECAP+Rolling

100 μm

Fig. 1 – Microstructure of the Pb–62% Sn alloy after processing by ECAP and rolling.

Fig. 3 shows the plot of the engineering stress as a function of engineering strain for tests at different strain rates, at room temperature. It is observed that the flow stress decreases significantly with decreasing strain rate. Also, a sharp decrease in the engineering stress is observed before failure in the samples pulled at 10^{-3}–$10^{-2}\,\mathrm{s}^{-1}$ range while the sample pulled at the lowest strain rate, $10^{-4}\,\mathrm{s}^{-1}$, exhibits a stress vs. strain curve with a long and slow reduction in stress. This observation suggests the occurrence of flow concentration in the samples tested at higher strain rates and an absence of flow concentration at the lowest strain rate. This is in agreement with the absence of necking in the latter.

The variation of flow stress with strain rate is shown in Fig. 4 using a double logarithmic scale. The flow stress was determined as the peak value in the engineering stress vs. strain curves and includes data for the test at $10^{-5}\,\mathrm{s}^{-1}$. The strain rate sensitivity parameter, $m = \partial \ln \sigma / \partial \ln \dot{\varepsilon}$, is determined by the slope of the segments in this graph. A slope of
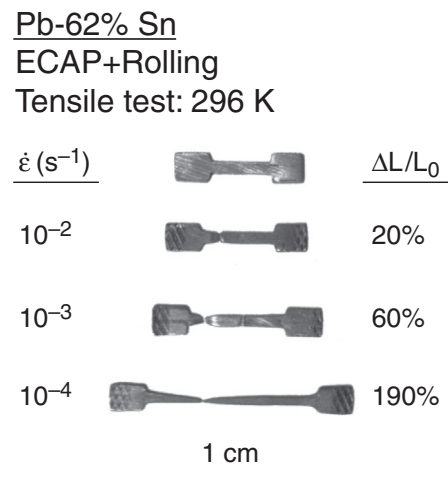

Pb-62% Sn
ECAP+Rolling
Tensile test: 296 K

$\dot{\varepsilon}\,(\mathrm{s}^{-1})$		$\Delta L/L_0$
10^{-2}		20%
10^{-3}		60%
10^{-4}		190%

1 cm

Fig. 2 – Appearance of the tensile specimens after being pulled to failure at room temperature and different strain rates.
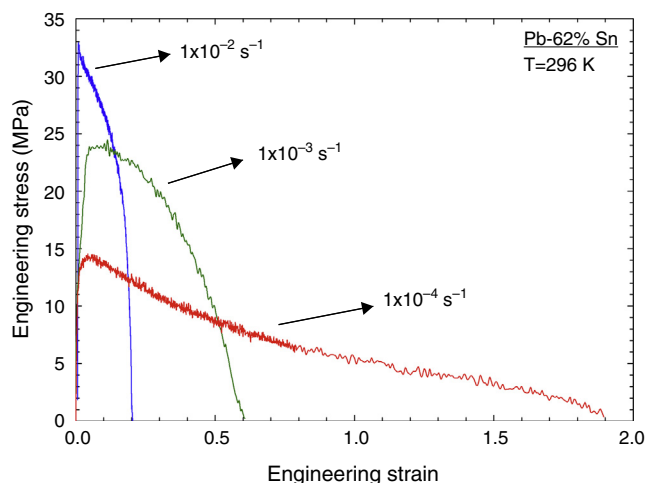
Fig. 3 – Engineering stress vs. engineering strain curves determined by tests at room temperature at different strain rates.

0.4 is shown for comparison. It is observed that the strain rate sensitivity is smaller at the higher strain rates and increases at the lower strain rates. The slope between 10^{-5} and $10^{-4}\,\text{s}^{-1}$ agrees with a strain rate sensitivity of $m = 0.4$.

4. Discussion

4.1. Elongation

The present results show that the eutectic alloy processed by casting followed by ECAP and rolling exhibits a very refined grain structure. The average grain size is within the range of a few micrometers, which is one of the requisites for superplastic behavior. The neck-free appearance of the specimen, the slow reduction of the flow stress in the engineering stress vs. strain curve and the high strain rate sensitivity observed at the lowest strain rates support the occurrence of superplastic deformation at room temperature in this material. However, the relatively low elongation to failure, 190%, observed at the
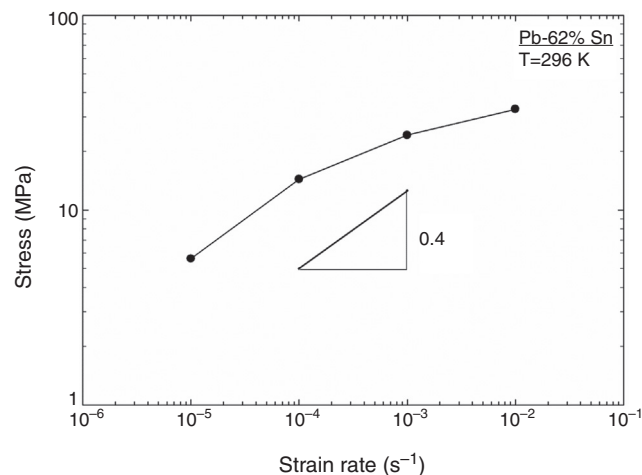

Fig. 4 – L logarithmic plot of flow stress as a function of strain rate.

lowest strain rate is out of the range of usual elongations for superplasticity. Thus, a brief discussion on reported elongations for similar alloys is given below.

El-Danaf et al. [4] reported an elongation of 600% at room temperature for the eutectic Pb–62%Sn alloy, with an average grain size of 6.0 μm, at the strain rate of $1.0 \times 10^{-4}\,\text{s}^{-1}$. Soliman [5] reported a maximum elongation of ~600% at room temperature, for the eutectic Pb–62%Sn alloy with grain size of 6.0 μm, at a strain rate of $1.5 \times 10^{-5}\,\text{s}^{-1}$. These values of elongation are significantly higher than the observed in the present experiments although the grain size and testing conditions are not very dissimilar. Also, Kawasaki et al. [7] reported a maximum elongation of 2665% at 150 °C in a similar alloy with a grain size of 6.0 μm, at a strain rate of $1.0 \times 10^{-3}\,\text{s}^{-1}$.

Despite the similar grain sizes and testing conditions, the specimen geometry reported in the literature are significantly different from the present experiments, specially the gauge aspect ratio. The ratios of the gauge cross-section by the gauge length are ~1.2 [4,5] and 1.5 [7], while it is 0.2 in the present experiments. The ratio of the cross section area by the gauge length plays a key role on final elongation of the specimens [8]. An early paper [9] suggested that the final elongation of a specimen pulled to failure in creep is a function of the stress exponent. The proposed relationship was:

$$\varepsilon_f = \frac{K}{n-1}$$

where ε_f is the final elongation, K is a constant between 2 and 3, and n is the stress exponent ($n = 1/m$). It follows that the final elongation predicted for a stress exponent $n = 2.5$ is in the range 133–200% depending on K. Therefore, the maximum elongation observed in the present experiments is within the predicted range of elongation in creep.

It is worth noting that the minimum strain rate used in the present experiments was $1.0 \times 10^{-4}\,\text{s}^{-1}$ and the elongation increases with decreasing the strain rate. Thus, it is expected that higher elongations are attained at lower strain rates. In fact, elongations of 550% and 650% were reported in a lead–tin alloy, with grain sizes of 3.3 μm and 6.1 μm respectively, tested at $6.6 \times 10^{-5}\,\text{s}^{-1}$ at room temperature [10]. Also, an elongation of >2000% was reported in a similar alloy tested at $6.6 \times 10^{-6}\,\text{s}^{-1}$ [11].

It has also been reported that pre-straining the lead–tin alloy at faster strain rates reduces the elongations observed at posterior deformation at low strain rates [12]. Therefore, pre-straining the material at a high strain rate by rolling could decrease the elongations observed during tensile tests in the present experiments since tensile testing is carried out at lower strain rates. However, the pre-straining effect on elongation was attributed to the formation of necking [12] and this is not observed in the present experiments.

4.2. Rate controlling mechanism

The strain rate in superplasticity, $\dot{\varepsilon}_{sp}$, is given by the relationship below [13]:

$$\dot{\varepsilon}_{sp} = \frac{AD_{GB}Gb}{kT}\left(\frac{\sigma}{G}\right)^2\left(\frac{b}{d}\right)^2$$
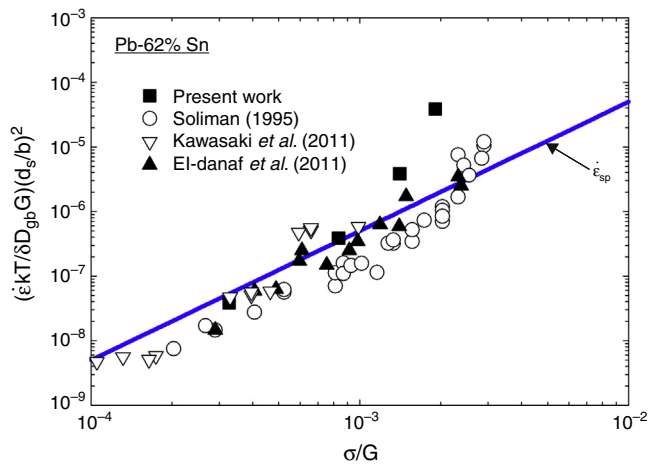
Fig. 5 – Strain rate normalized by the temperature and grain size effect and plotted as a function of the flow stress normalized by the shear modulus. Data from the literature [3,4,6] is also shown.

where A is a constant, D_{GB} is the diffusion coefficient for grain boundary diffusion, G is the shear modulus, b is the Burgers' vector modulus, k is the Boltzmann constant, T is the absolute temperature, σ is the flow stress and d is the spatial grain size. In order to clarify the rate controlling mechanism operating in the present experiments the experimental data is compared to the theoretical prediction for superplasticity in Fig. 5. Additional data from the literature [4,5,7] are also shown for comparison. The values of A, D_{GB}, G and b were collected from the literature [14]. It is observed that the present experimental data for the lower strain rates agree well with the data from the literature and with the theoretical prediction for superplastic flow in the eutectic Pb–62% Sn alloy. This suggests the material exhibits superplasticity at the lowest strain rates in the present experiments.

5.   Conclusions

1. The eutectic lead–tin alloy exhibits a fine grain structure ($d \approx 5.9\,\mu$m) after processing by ECAP and rolling.
2. Tensile testing at room temperature showed increased elongation to failure at low strain rates with a maximum elongation of 190% at $10^{-4}\,s^{-1}$.
3. The elongation to failure is lower than the observed in the literature but agrees with a model for elongation of samples subjected to tensile creep. The reduced elongation is attributed to the reduced gauge cross section by gauge length ratio.

4. The experimental data agrees with the model for grain boundary sliding creep.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgments

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