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The influence of prestrain on ductility in the superplastic Pb–Sn eutectic alloy

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Experiments were conducted to investigate the effect of prestraining on the ductility of the superplastic Pb–62 wt % Sn eutectic alloy at room temperature. It is shown that prestraining at a fast strain rate in region III leads to a decrease in the elongation to failure on subsequent testing at a slower rate in region II. This decrease is due to the development of strain inhomogeneities during the prestrain. There is a further decrease in elongation if a holding time is inserted between the prestrain in region III and the subsequent testing in region II. It is shown also that prestraining at a slow strain rate leads to a sharp increase in the elongation to failure on subsequent testing at a fast strain rate. The reasons for these trends are discussed with reference to the occurrence of grain growth and the uniformity of strain.

1. Introduction

It has been established in several creep investigations that prestraining at room temperature, usually up to a maximum strain of ~ 25%, leads to a significant decrease in the measured elongations to failure when the material is subsequently creep tested at elevated temperatures [1–3]. It has been shown also, using quantitative optical metallography, that this drop in ductility is due to an increase in the density of grain boundary cavities as a result of the prestraining treatment [4, 5].

It is surprising to note that there have been very few investigations of the effects of prestraining on superplastic materials. Furthermore, the limited data at present available relate to the subsequent flow characteristics rather than to the overall ductilities, and the experiments also have generally been performed on fairly complex alloy systems. For example, Immarigeon and Floyd [6] investigated the effect of prestraining at fast rates at a temperature of 1050° C on the subsequent flow properties at the same temperature of a nickel-based superalloy powder compact with a very small grain size.

The present investigation was initiated to provide the first detailed information on the influence of prestraining on the ductility of a typical simple superplastic alloy.

The experiments were conducted using the Pb–62 wt % Sn eutectic alloy tested at room temperature. This alloy was selected for three reasons. First, earlier experiments established that the Pb–Sn eutectic alloy exhibits relatively low elongations to failure ($\lesssim 500\%$) at initial strain rates above $\sim 10^{-4}\,\text{sec}^{-1}$ at room temperature, whereas there is a sharp increase in the measured ductilities (up to $> 1000\%$) at lower strain rates [7]. Second, the measured elongations to failure suggest a transition from non-superplastic behaviour in region III to superplastic behaviour in region II when the initial strain rate is decreased below $\sim 10^{-4}\,\text{sec}^{-1}$, and this transition is confirmed by detailed measurements showing that the strain is localized within a sharp neck in region III whereas the deformation in quasi-uniform at the lower strain rates in region II [8]. Third, although the Pb–Sn eutectic alloy generally exhibits very little cavitation, there is evidence for cavity formation in this alloy when testing to fracture in region III at room temperature [9].

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2. Experimental material and procedure

The experiments were performed using a Pb–62 wt% Sn alloy prepared from 99.999% purity lead and 99.995% purity tin. Details of the preparation of the material are given elsewhere [7], and the final alloy contained the following impurities in ppm: Ag 0.2, Al 0.5, Au 1, Bi 0.7, Ca 0.1, Cd 0.1, Cu 2, Fe 1, In 1, Mg, 0.2, Mn 0.1, Si 0.3 and Ti 0.5.

Tensile specimens were cut from sheets, parallel to the rolling direction, with an initial gauge length of 1.27 cm. The as-prepared material had an initial average spatial grain size, d, of 3.3 μm, where d is defined as $1.74 \times \bar{L}$ where $\bar{L}$ is the value of the mean linear intercept on a polished section. Each specimen was annealed in a silicone oil bath for 1 h at a temperature of 433 ± 1 K, and then cooled rapidly in air to room temperature to give a pre-test grain size of 6.1 μm.

As noted in earlier work [10], the stability of the initial grain size has a marked influence on the measured elongations to failure. Since the present experiments were designed to check the influence of prestraining on ductility, it was important to ensure that the various specimens were in an identical condition prior to testing. Accordingly, all specimens were stored in liquid nitrogen after the annealing treatment to prevent any additional grain growth.

All of the tests described in this report were conducted in air at room temperature (298 ± 2 K) using an Instron testing machine operating at a constant rate of cross-head displacement.

The influence of prestraining was investigated by performing three different types of experiment:

1. Initially, several specimens were pulled to different amounts of prestrain, up to a maximum of 60%, at an initial strain rate, $\dot{\epsilon}_1$, of 1.3×10^{-2} sec^{-1}. Earlier tests showed that this strain rate is within region III and the anticipated elongation to failure is ~ 110% [7], so that the specimens used in this work were prestrained up to a maximum of about one-half of the total possible ductility. Each test was terminated at a selected prestrain, and testing was then continued to failure, without interruption, at a cross-head speed which was equivalent to an initial strain rate at zero strain, $\dot{\epsilon}_2$, of 6.6×10^{-5} sec^{-1}. The latter strain rate is within region II and the anticipated elongation to failure, in the absence of a prestrain, is ~ 650% [7]. A base strain rate of 6.6×10^{-5} sec^{-1} was selected because earlier detailed measurements showed that the strain is quasi-uniform under these conditions, and necking is then diffuse rather than localized [8].

2. A second batch of specimens was prestrained at $\dot{\epsilon}_1 = 1.3 \times 10^{-2}$ sec^{-1} to a total prestrain of 10%, each specimen was held at room temperature for an amount of time, Δt, varying from 20 to 260 h, and testing was then continued to failure at the base strain rate of $\dot{\epsilon}_2 = 6.6 \times 10^{-5}$ sec^{-1}. These tests were designed to examine the influence on ductility of a prestrain plus a holding time.

3. Finally, experiments were conducted to determine the effect of prestraining at a slower, rather than a faster, initial strain rate. Several specimens were tested to various prestrain levels, up to a maximum of 200%, at an initial strain rate of $\dot{\epsilon}_1 = 6.6 \times 10^{-6}$ sec^{-1}, and then, without interruption, they were pulled to failure at $\dot{\epsilon}_2 = 1.3 \times 10^{-4}$ sec^{-1}. Earlier tests showed that the initial strain rate of $\dot{\epsilon}_1$ is in region II, and it gives a very high elongation to failure (of the order of ~ 2230%) [7] so that, for these tests, the maximum prestrain was only ~ 10% of the anticipated ductility. Earlier experiments also established that the deformation remains very uniform up to > 400% at a strain rate of 6.6×10^{-5} sec^{-1}, where the elongation to failure is ~ 775% [8]; in view of the much higher anticipated elongation at an initial strain rate of $\dot{\epsilon}_1 = 6.6 \times 10^{-6}$ sec^{-1}, it is reasonable to assume that the prestraining is also very uniform in these specimens, even at the maximum prestrain of 200%. The upper strain rate of 1.3×10^{-4} sec^{-1}, which was used for testing to failure, is in the transition between regions II and III and the anticipated ductility in the absence of prestraining is ~ 325% [7].

3. Experimental results

3.1. Effect of prestraining in region III and testing to failure in region II

Specimens were prestrained for different amounts, from 0% to a maximum of 60%, at $\dot{\epsilon}_1 = 1.3 \times 10^{-2}$ sec^{-1} and then pulled to failure without interruption (so that $t = 0$) at $\dot{\epsilon}_2 = 6.6 \times 10^{-5}$ sec^{-1}.

Fig. 1 shows a plot of the flow stress achieved at the lower strain rate of 6.6×10^{-5} sec^{-1} as a function of the amount of prestrain. This plot indicates that the flow stress is ~ 24 MPa in the absence of a prestrain,* but there is a tendency

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*The plots of true stress against true strain in the absence of a prestraining treatment were shown earlier [7].

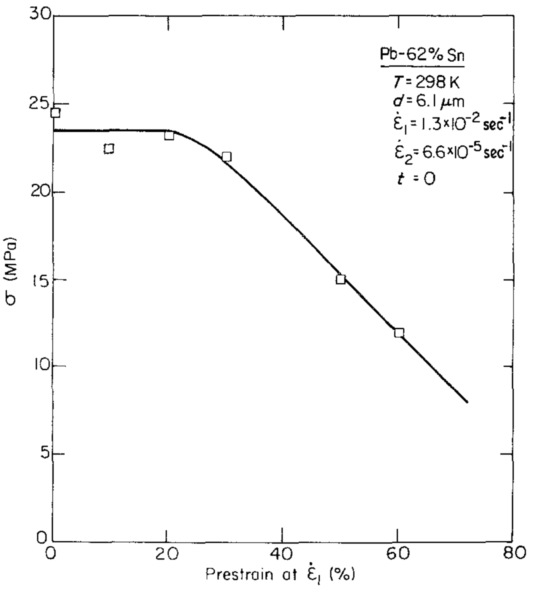

Figure 1 Flow stress at $\dot{\epsilon}_2 = 6.6 \times 10^{-5}$ sec^{-1} against amount of prestrain at $\dot{\epsilon}_1 = 1.3 \times 10^{-2}$ sec^{-1}.

for the flow stress to decrease sharply with increasing prestrain to a value of ~ 12 MPa when the prestrain at $\dot{\epsilon}_1$ is 60%.

The drop in the measured flow stress shown in Fig. 1 suggests that the prestraining at $\dot{\epsilon}_1$, especially to the larger prestrains, introduces necking within the specimen gauge length, and this suggestion is further supported by the decrease in the total elongation to failure, $\Delta L/L_0$ (%), given in Fig. 2 as a function of the prestrain at $\dot{\epsilon}_1$: in this plot, L_0 is the initial gauge length (1.27 cm) and ΔL is the total increase in length at the point of failure. Fig. 2 shows that the maximum elongation in the absence of prestrain is ~650%, there is a slight increase to ~ 725% for a prestrain of 10% and, thereafter, there is a sharp drop to a maximum ductility of ~300% at a prestrain of 30%. For prestrains above 30% at $\dot{\epsilon}_1$, the elongation to failure levels off at ~300%.

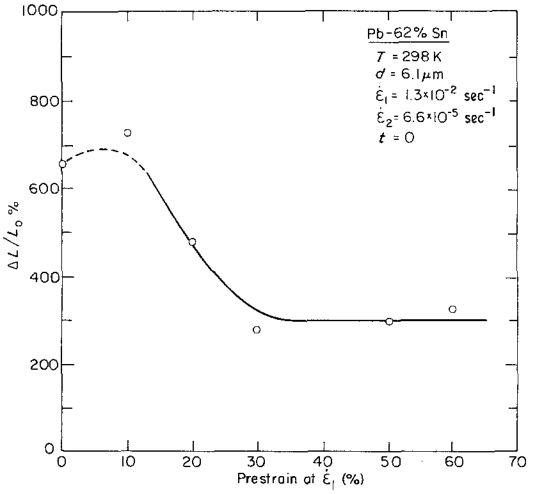

Figure 2 Elongation to failure at $\dot{\epsilon}_2 = 6.6 \times 10^{-5}$ sec^{-1} against amount of prestrain at $\dot{\epsilon}_1 = 1.3 \times 10^{-2}$ sec^{-1}.

In practice, it is important to note that this type of data, involving absolute measures of the elongations to failure, is subject to some experimental scatter between different specimens. For example, it was reported earlier that two similar tests on identical specimens with a grain size of 6.1 μm, conducted at room temperature with the same initial strain rates of 6.6 x 10^{-5} sec^{-1}, gave elongations to failure in the absence of a prestrain of ~ 650 and ~ 775%, respectively [8]. In view of this inherent scatter, it is reasonable to conclude that the apparent initial increase in fracture elongation, at a prestrain of 10% (as indicated by the broken line in Fig. 2), is probably not a true characteristic of the material. On the other hand, there is no doubt that prestraining at $\dot{\epsilon}_1$ to 20% or higher leads to a genuine decrease in the elongations to failure.

Fig. 3 shows the specimens after failure: specimen A is untested, and specimens B to F were pulled to failure at $\dot{\epsilon}_2 = 6.6 \times 10^{-5}$ sec^{-1} after prestrains at $\dot{\epsilon}_1 = 1.3 \times 10^{-2}$ sec^{-1} of 0% (B), 10% (C), 20% (D), 30% (E) and 50% (F), respectively. The higher ductility is clearly visible in specimen C, tested to 10% prestrain, but it should be noted that there is some evidence for necking within the gauge length other than at the point of failure. Specimens B and D pull out reasonably uniformly, but at the lower elongations, in specimens E and F, there is a small but observable increase in area at the point of failure. This increase is a characteristic of failure by necking [11], although the increase is small by comparison with the blunt fracture surfaces observed earlier in the Pb–Sn eutectic after testing to failure in region III without a prestrain at an initial strain rate of 6.6 x 10^{-3} sec^{-1} [8].

3.2. Effect of prestraining in region III with a holding time

To check whether the elongations to failure are affected by holding at room temperature after the

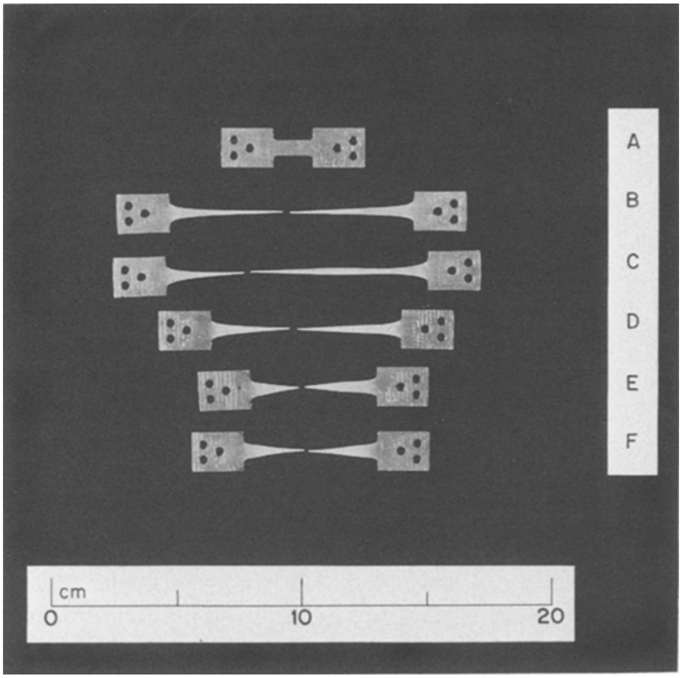

Figure 3 Appearance of specimens after prestraining at $\dot{\epsilon}_1 = 1.3 \times 10^{-2}$ sec^{-1} and testing to failure at $\dot{\epsilon}_2 = 6.6 \times 10^{-5}$ sec^{-1}: specimen A is untested, and specimens B to F were prestrained for 0, 10, 20, 30 and 50%, respectively.

prestrain and prior to testing, several specimens were pulled to a prestrain of 10% at $\dot{\epsilon}_1 = 1.3 \times 10^{-2}$ sec^{-1}. As indicated in Fig. 2, this small amount of prestrain gives no significant change in the elongation to failure in the absence of a hold at room temperature.

Fig. 4 shows $\Delta L/L_0$ (%) against the period of hold, Δt, for a series of specimens subsequently tested to failure at $\dot{\epsilon}_2 = 6.6 \times 10^{-5}$ sec^{-1}. The data show that the elongation to failure decreases with increasing hold time up to ~ 50 to 100 h, and thereafter the elongation stabilizes at ~ 350 ± 50%.

Typical specimens at failure are shown in Fig. 5: specimen A is again untested, and the other specimens were pulled to failure after a prestrain of 10% and hold times of 0 h (B), 24 h (C), 48 h (D), 72 h (E), 120 h (F) and 216 h (G), respectively*. As previously, these specimens show evidence of necking and a consequent small increase in the area of the fracture tip at the lower elongations.

3.3. Effect of prestraining in region II and testing to failure at a faster strain rate

Several specimens were prestrained at $\dot{\epsilon}_1 = 6.6 \times 10^{-6}$ sec^{-1} in region II and then pulled to failure, without an intermediate hold time, at $\dot{\epsilon}_2 = 1.3 \times 10^{-4}$ sec^{-1} at the transition between regions II and III. The results are shown in Fig. 6 plotted as $\Delta L/L_0$ (%) against the amount of prestrain at $\dot{\epsilon}_1$, and it is apparent that prestraining introduces significant increases in the elongations to failure above the value of ~ 325% which is obtained without a prestrain.

At first sight, it seems likely that the prestrain at $\dot{\epsilon}_1$ introduces a uniform elongation throughout the gauge length, so that the elongation to failure at $\dot{\epsilon}_2$ is simply the sum of the anticipated elongation at this strain rate (~ 325%) and the value of the prestrain. However, close inspection of Fig. 6 shows that, although this is correct at the smaller prestrains (for example, the prestrain of 100% gives $\Delta L/L_0 \simeq 430\%$), the trend tends to break down at the larger prestrains where the elongations are markedly higher than the sum of the two strains (for example, the prestrain of 200% leads to fracture at ~ 700% rather than, as anticipated, at ~ 525%).

The fractured specimens are shown in Fig. 7: specimen A is again untested, and the other specimens were prestrained at 6.6×10^{-6} sec^{-1} for 0% (B), 50% (C), 100% (D), 150% (E) and 200% (F), respectively. These specimens tend to show diffuse necking within the gauge length:

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*Specimen B in Fig. 5 is therefore identical to specimen C in Fig. 3.

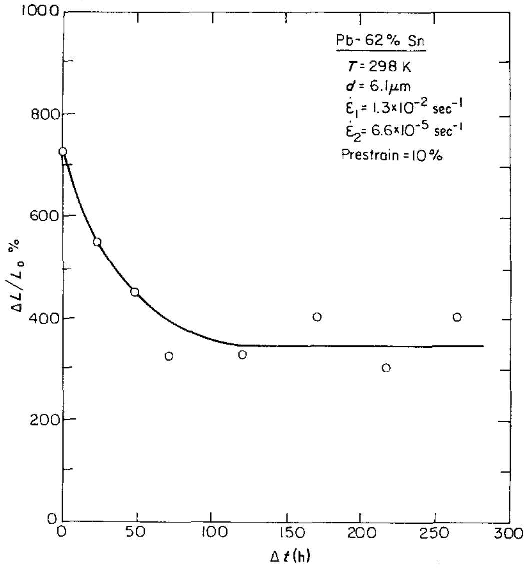

Figure 4 Elongation to failure at $\dot{\epsilon}_2 = 6.6 \times 10^{-5}$ sec^{-1} against holding time following a prestrain to 10% at $\dot{\epsilon}_1 = 1.3 \times 10^{-2}$ sec^{-1}.

examples of diffuse necks which have not led to failure are clearly visible in specimens C and F.

4. Discussion

The primary characteristic of a superplastic alloy is the very large elongation to failure which is attained over a limited range of strain rates. There have been numerous previous investigations of the various factors, such as strain rate, temperature and grain size, which affect the measured elongations to failure in superplastic materials [12–14], and the present results supplement this earlier work by providing information on the influence of a prestraining treatment.

There are three important conclusions from the present set of experiments: (i) prestraining at a faster strain rate in region III tends to decrease the elongation to failure when testing at a slower rate in region II (Fig. 2), (ii) there is a further decrease in the elongation to failure if a holding time is inserted between the prestrain in region III and the subsequent testing in region II (Fig. 4), and (iii) prestraining at an even slower rate in region II tends to increase the elongation to failure when subsequently testing at a faster rate, and this increase is larger than anticipated from a simple summation of the amount of prestrain and the anticipated fracture elongation at the faster rate (Fig. 6).

These results are examined in the following sections.

4.1. Effect of prestraining at a faster strain rate without a holding time

It was noted earlier that creep experiments consistently show that a prior prestraining treatment leads to a subsequent decrease in the measured fracture strain [1–3]. This trend is similar to the

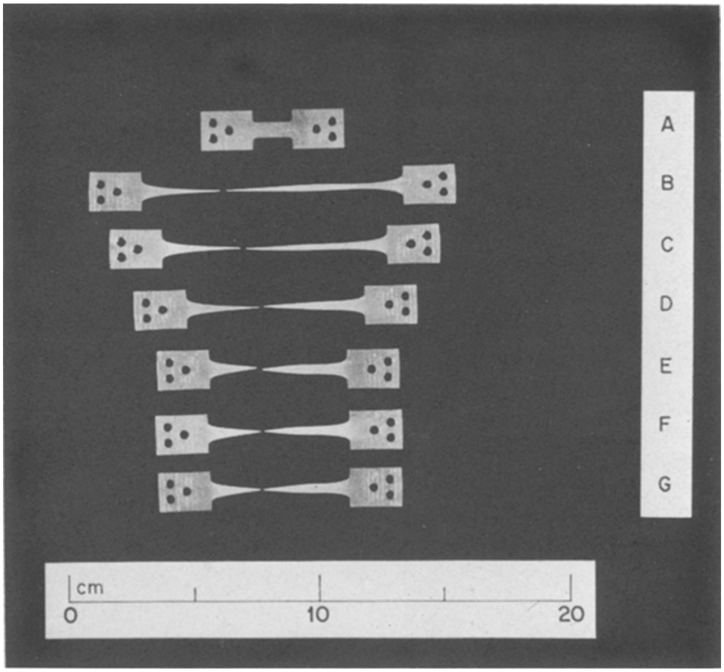

Figure 5 Appearance of specimens after prestraining to 10% at $\dot{\epsilon}_1 = 1.3 \times 10^{-2}$ sec^{-1}, holding at room temperature, and then testing to failure at $\dot{\epsilon}_2 = 6.6 \times 10^{-5}$ sec^{-1}: specimen A is untested, and specimens B to G were held for times of 0, 24, 48, 72, 120 and 216 h, respectively.

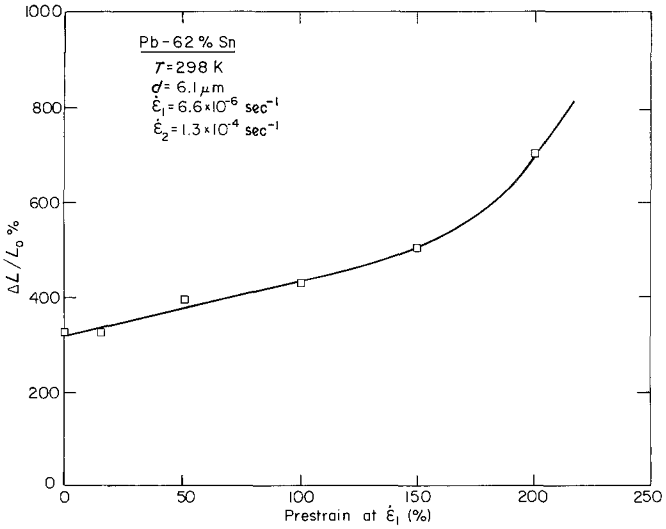

Figure 6 Elongation to failure at $\dot{\epsilon}_2 = 1.3 \times 10^{-4}$ sec^{-1} against amount of prestrain at $\dot{\epsilon}_1 = 6.6 \times 10^{-6}$ sec^{-1}.

results shown in Fig. 2. However, it is well established in creep testing that the decrease in elongation is due to the formation of grain boundary cavities during the prestrain [4, 5]. It is very unlikely that this explanation applies also to the superplastic Pb–Sn eutectic alloy, because cavitation is generally not important in this alloy and the experimental observations of cavitation relate only to specimens taken to failure at room temperature in region III [9].

It is probable that the explanation for the effect of the prestrain in superplasticity lies instead in the rapid development of strain inhomogeneities along the gauge length when testing in region III. The development of non-uniformities at low total strains was noted earlier in the Pb–Sn eutectic alloy [8], so that the specimens prestrained at $\dot{\epsilon}_1 = 1.3 \times 10^{-2}$ sec^{-1} contain the early stages of a neck. This reduced section continues to pull out when the specimen is taken to failure at $\dot{\epsilon}_2 = 6.6 \times 10^{-5}$ sec^{-1}. Thus, the final elongation at failure is intermediate between the value anticipated at $\dot{\epsilon}_1$ (~ 110%) and the value attained when testing only at $\dot{\epsilon}_2$ (~ 650%). The presence of non-uniformities of strain within the gauge length is consistent also with the decrease in flow stress at the higher prestrains, as shown in Fig. 1.

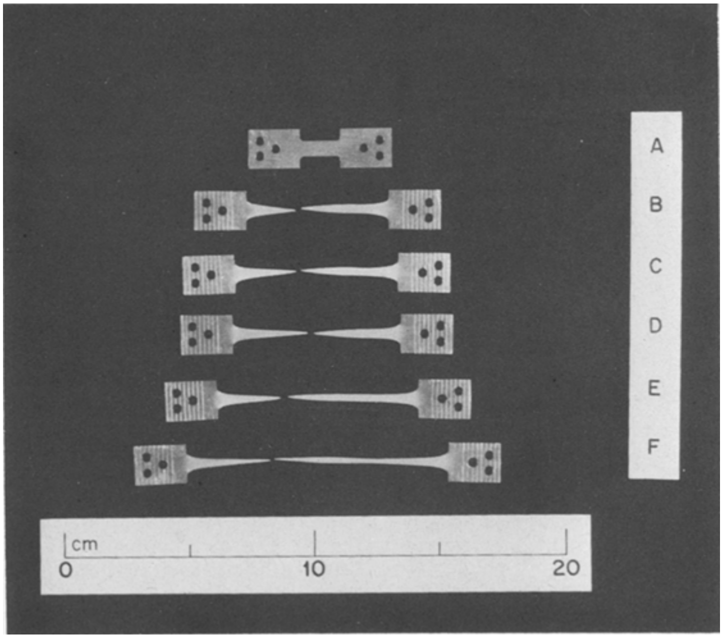

Figure 7 Appearance of specimens after prestraining at $\dot{\epsilon}_1 = 6.6 \times 10^{-6}$ sec^{-1} and testing to failure at $\dot{\epsilon}_2 = 1.3 \times 10^{-4}$ sec^{-1}: specimen A is untested, and specimens B to F were prestrained for 0, 50, 100, 150 and 200%, respectively.

4.2. Effect of prestraining at a faster strain rate with a holding time

The influence of a holding time between the initial prestrain and the subsequent testing, as shown in Fig. 4, is the opposite of the effect observed in normal tensile testing where there is often an increase in the total elongation if testing is performed with interruptions. The latter behaviour has been interpreted in terms of the development of thermal gradients along the gauge length and the influence of these gradients on the stability of plastic flow [15].

In the present experiments, the introduction of a holding time leads to a subsequent decrease in the elongations to failure, and it seems likely that this trend is associated with the occurrence of grain growth at room temperature. Although it is known that the average grain size changes by only a small amount when testing in region III at room temperature [10], it was established in many earlier experiments on superplastic alloys that grain growth is enhanced by deformation [13, 16] so that it occurs more rapidly in regions of non-uniform strain such as at necks within the gauge length. In this connection, it should be noted that holding the Pb–62 wt% Sn alloy at room temperature prior to testing is particularly undesirable because of the low absolute melting point of this alloy.*

4.3. Effect of prestraining at a slower strain rate without a holding time

The effect of prestraining at a lower strain rate ($\dot{\epsilon}_1 = 6.6 \times 10^{-6}\ \text{sec}^{-1}$) is to give a higher fracture elongation at the testing strain rate ($\dot{\epsilon}_2 = 1.3 \times 10^{-4}\ \text{sec}^{-1}$), as shown in Fig. 6.

This result is easily understood when it is noted that all of the tests were conducted using a testing machine operating at a constant rate of cross-head displacement. When the prestrain at $\dot{\epsilon}_1$ is small, up to ~ 50%, the specimens pull out uniformly at the low strain rate, and subsequent testing at $\dot{\epsilon}_2$ leads to a fracture elongation which is essentially the sum of the prestrain and the elongation anticipated at the faster strain rate. At the higher prestrains, however, the specimens again pull out uniformly [8], but this gives rise to an increase in the effective gauge lengths of the specimens so that, due to the use of a testing machine with a constant cross-head speed, the subsequent testing is conducted essentially at a lower initial strain rate.

For example, with a uniform prestrain of 200% and an initial gauge length of 1.27 cm, calculations show that the initial strain rate at $\dot{\epsilon}_2$ is effectively reduced by about one-third of an order of magnitude. It was shown earlier that the elongations to failure, in the absence of a prestrain, increase very rapidly between initial strain rates of $1.3 \times 10^{-4}\ \text{sec}^{-1}$ (~ 325%) and $6.6 \times 10^{-5}\ \text{sec}^{-1}$ (~ 650%) [7, 10]. Thus, this sharp increase in elongation is consistent with the data shown in Fig. 6.

5. Summary and conclusions

1. Experiments on the superplastic Pb–62 wt% Sn eutectic alloy at room temperature show that prestraining at a fast strain rate in region III leads to a decrease in the elongation to failure when testing at a slower rate in region II. This decrease is associated with the development of strain inhomogeneities, in the form of necking, during the prestrain.
2. There is a further decrease in elongation if a holding time is inserted between the prestrain in region III and the subsequent testing in region II. This decrease is attributed to the occurrence of grain growth at room temperature.
3. If specimens are prestrained at a slow strain rate and then tested to failure at a faster strain rate, there is a sharp increase in the elongation to failure. This increase arises because the specimens pull out uniformly at the slower rate and subsequent testing with a constant cross-head speed is then equivalent to using a lower initial strain rate.

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*The absolute melting point, T_m, is 456 K: room temperature corresponds to ~ 0.65 T_m.

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