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Evaluation of mechanical properties for fundamental studies in structural superplasticity

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It is shown that even when all the specimens used in a testing programme are taken from a single industrially prepared sheet, significant scatter in the mechanical properties of a superplastic alloy may be present but will not be revealed if the data are presented as log (stress)–log (strain rate) plots. A comparison of the linear stress–strain curves of different specimens for consistency is necessary before using them for microstructural and texture studies.

1. Introduction

Watts *et al.* [1] have stated that they prepared Al–Cu–Zr alloys of different compositions at two different laboratories and tested them for their superplastic properties without distinguishing between the sources of supply. Langdon [2] has concluded that in his experiments on a Zn–Al eutectoid alloy, the ductility data were "highly reproducible". These statements seem to imply that there are no major problems with regard to the reproducibility of results during superplastic deformation.

Yet, tests on ostensibly identical material deformed under similar conditions have often led to different conclusions with regard to the operating mechanisms and both Edington *et al.* [3] and Padmanabhan and Davies [4] have commented on the considerable scatter in the values of the elongation to fracture and the strain-rate sensitivity index, m, reported in the literature. Padmanabhan and Lücke [5] have also noted that a major source of conflict in assessing the role of crystallographic slip in superplastic flow is the subtle (and in most cases unnoticed) differences in the starting and testing conditions, which make direct comparisons difficult.

A paper has recently been presented [6], which assesses briefly the role of slip in a relatively coarse-grained Al–Cu–Zr alloy by evaluating the starting material, the changes due to thermal effects and those due to deformation using the orientation distribution function (ODF) analysis. During this investigation it was realized that significant scatter could exist in the mechanical properties and this should be carefully considered before specimens are accepted for texture measurements. This paper discusses these findings.

2. Experimental procedure

Two types of specimen were used. Tensile specimens were made from an alloy of composition equivalent to Supral 100 that was prepared in the laboratory (Alloy A). Here the required number of specimens were obtained from three melts made under "identical" settings of the furnace. Tensile specimens were also made from a large sheet each of commercial Supral 100 (Alloy B) and a superplastic Al–Ca–Zn alloy (Alloy C) supplied by Alcan, UK and Alcan, Canada, respectively. (In these two latter cases the specimens needed for an entire campaign of experiments were taken from a single sheet.)

In case of Alloy A three melts of 12 kg each were prepared in an electric resistance heating furnace, maintaining the operating conditions identical. The alloy was melted at 900 °C, covered with a flux, degassed with commercially available degassing tablets and poured into ingots of size 140 mm × 120 mm × 50 mm. (Commercial grade aluminium and copper were used individually but zirconium was added in the form of an Al–Zr master alloy.)

The ingots were homogenized at 370 °C for 20 h and forged at 300 °C (in one heating) from 50 mm to 28 mm thickness. Then they were rolled at 310 °C from a thickness of 28 mm to a thickness of 6 mm, imparting 15% reduction per pass and reheating for 10 min between passes. Such a treatment gave rise to an as-rolled grain size of 20.2 ± 2.5 μm, which was useful in checking if the mechanism of deformation changed drastically as one went from an ultra-fine-grained to a relatively coarse grained material [6].

From the rolled plates, tensile specimens of gauge dimensions 25 mm × 5.25 mm × 4 mm (ASTM sub-standard size, Fig. 1) were machined, keeping the axis

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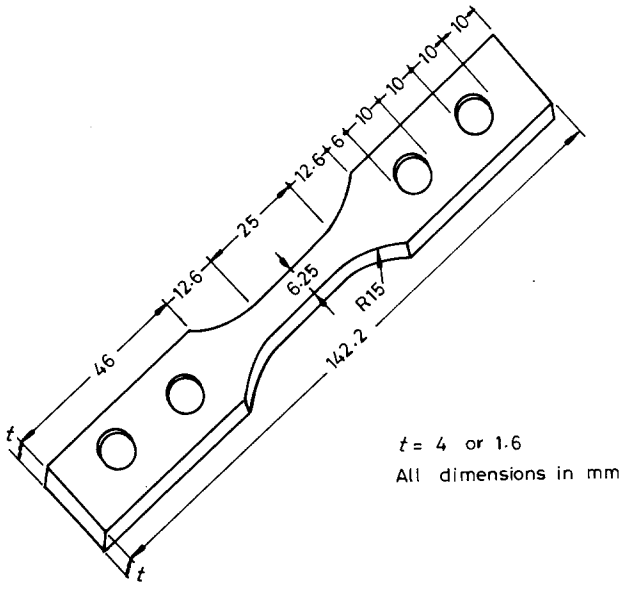


Figure 1 The tensile specimen used in the investigation.

of the specimens always parallel to the rolling direction. One batch of specimens was tested in this as-rolled condition. Another batch was soaked at 490 °C for 20 min and air-cooled before tensile testing (initial grain size 21.8 ± 3.3 μm). These two starting conditions will be referred to as unstabilized, U, and stabilized, S, microstructures, respectively, and denoted by the suffixes U and S in the figures for Alloy A.

The sheets of Alloys B and C had a thickness of 1.6 mm. Here also ASTM sub-standard size (Fig. 1, with thickness 1.6 mm) was chosen for the tensile specimens. In these two alloys, specimens with the axis parallel as well as perpendicular to the rolling direction were made. This is indicated in the relevant figures by the suffixes L (longitudinal) and T (transverse) respectively.

Alloy Supral 100 (B) was tested in the as-received condition. The average two-dimensional grain size of the starting material was 2.2 ± 0.3 μm. For good superplastic effects, the Al–Ca–Zn alloy specimens were heat treated at 540 °C (813 K) for 30 min and air cooled. In this condition this alloy had a two-dimensional matrix grain size in the range 2–3 μm and contained calcium- and zinc-rich precipitate particles of average size 1.0 ± 0.5 μm [7].

In case of the experimental Alloy A, three tensile specimens were selected at random for chemical analysis. In case of Alloys B and C the compositions were supplied by the manufacturers, which were confirmed to be accurate in our laboratory. The compositions (wt %) are listed in Table I.

First, the room-temperature (20 °C) true stress–true strain curves were determined for the three alloys. Then, the experimental alloy (A) and Supral 100 (B) were tested at 730 K (457 °C) and the Al–Ca–Zn alloy (C) at 825 K (552 °C), their respective optimal temperatures for superplastic deformation [8, 9]. Using a furnace with three independently controlled heating coils, a minimum accuracy of ± 1.5 °C was ensured over the whole length of the specimen in all the tests. On an electromechanical universal testing machine the specimens were tested to failure at ten different (constant) extension rates in the range 0.40–99.00 mm min^{-1}.

By comparing the directly measured specimen elongation with the output from an X–Y recorder in the case of a few chosen specimens which covered the whole load range involved, a calibration chart was prepared. Using this chart, the true elongation in the gauge portion of all the specimens was estimated by eliminating the contributions from flow in the grip regions and the elastic response of the testing system.

The microstructural/texture changes that accompanied the heating of the specimens to the temperatures of superplastic deformation and the additional changes brought about by the deformation are not considered in this paper.

3. Results and discussion

The room-temperature true stress (σ), true strain (ε) curves obtained at a constant crosshead speed of 2.5 mm min^{-1} (initial strain rate $\dot{\varepsilon}_0 = 1.57 \times 10^{-3}\ s^{-1}$) are given in Fig. 2. Supral 100 (B) and the Al–Ca–Zn alloy (C) were tested in the as-received condition. The flow stress, ultimate tensile strength (UTS) and elongation values of these two alloys are in broad agreement with the results quoted by Wadsworth *et al.* [10]. Those authors, however, did not compare the response of the two alloys in the longitudinal (rolling) and transverse directions of the sheeets.

When this is done in Fig. 2, it is seen that Supral 100 is more anisotropic in its room-temperature mechanical response than the Al–Ca–Zn alloy. (This anisotropic behaviour could have resulted from two

TABLE I The compositions (wt %) of the alloys used in the present study

(a) Experimental alloy (A)

Cu	Zr	Fe	Zn	Si	Ni	Bi	Mg	Mn	Ti	Al
5.80	0.39	0.24	0.16	0.13	0.062	0.036	0.01	<0.01	0.006	bal.

(b) Supral 100 (B)

Cu	Zr	Fe	Si	Ni	Mg	Mn	Zn	Ti	Ga	B	Al
6.5	0.40	0.15	0.074	0.072	0.036	0.028	0.019	0.005	55 p.p.m.	7 p.p.m.	bal.

(c) Al–Ca–Zn alloy (C)

Ca	Zn	Fe	Si	Mg	Al
4.87	4.64	0.13	0.09	0.01	bal.

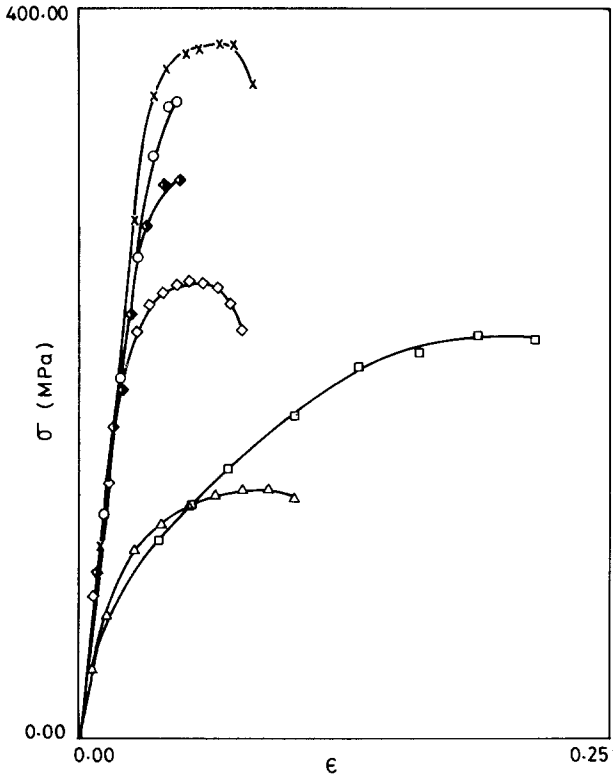


Figure 2 True stress, σ,–true strain, ε, plots (linear scale) at room temperature for the three alloys, determined at an initial strain rate of $1.7 \times 10^{-3}\ s^{-1}$. (△) A_U, (□) A_S, (×) B_L, (◇) B_T, (○) C_L, (◈) C_T.

sources: (a) grain shape, (b) texture.) Then, one would expect the superplastic behaviour of Supral 100 to be more anisotropic than that of the Al–Ca–Zn alloy. (In Fig. 2 and the following figures, no necking correction was made. As superplastic deformation involves only diffuse necks almost until the end, and the main aim is to compare the curves, this will not introduce any serious errors.)

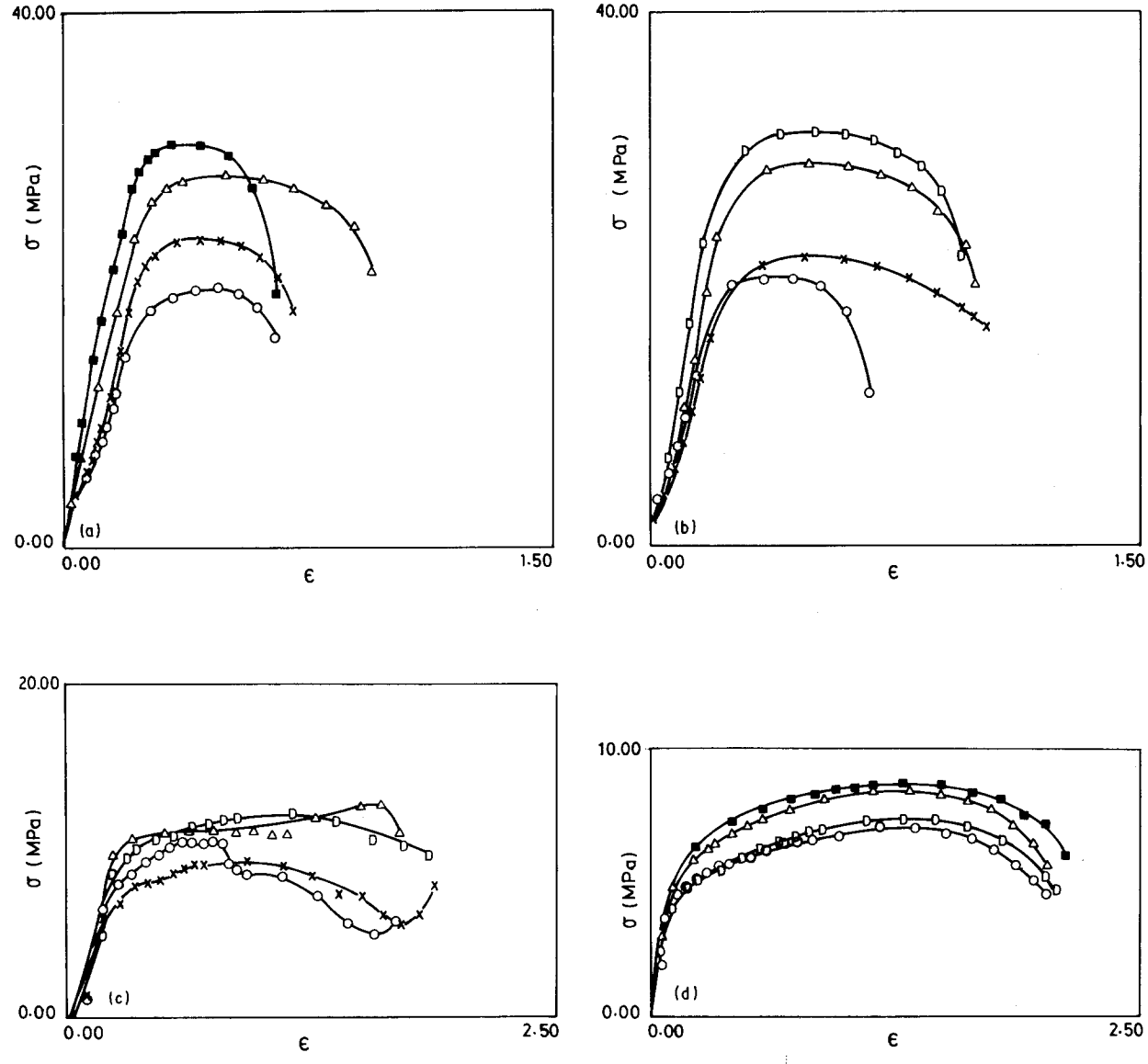
The observation that the flow stress of the laboratory made alloy (A) was considerably less than that of Supral 100 is in line with the Hall–Petch relationship (grain sizes 20.2 μm compared to 2.2 μm). It is also reasonable that in the experimental alloy a stabilized microstructure gave rise to greater ductility than an unstabilized variety.

In Fig. 3a–e the scatter that was present in the tensile tests at the temperatures of superplastic deformation is shown by taking some typical examples. The following observations are pertinent. (i) The scatter in the (linear) true stress–true strain curves can be quite significant even when all the specimens are taken from a single industrially prepared sheet. (ii) In the experimental alloy (A) in both the microstructural states at all strain rates there was more or less uniform scatter in the flow stress value for a given strain (see Fig. 3a, b). As most texture measurements concerning superplasticity have been made on experimental alloys prepared in the laboratory, it is essential that this scatter is reduced to a bare minimum by repeated testing. It is not clear from the published results if such care has been taken in the past. (iii) In the Supral 100 alloy, in both the rolling and transverse directions of the sheet, and in the Al–Ca–Zn alloy along the rolling direction of the sheet, the scatter in the true stress–true strain curves was much less than in the laboratory-made alloy (Fig. 3c, d), because in these cases the specimens for an entire campaign of experiments were obtained from a single sheet. (iv) This, however, was not the case with the Al–Ca–Zn alloy tested in the transverse direction of the sheet, even though here also the required number of specimens was obtained from a single sheet (Fig. 3e).

These are complicating results, and are often ignored. Preliminary studies have revealed that the differences in the texture and grain shape from place to place in the same sheet are not so high as to account for the entire scatter. Thus, it is conceivable that in some regions of the sheet a greater concentration of the fine, boundary pinning particles (which are mainly responsible for maintaining the fine grain size in these alloys at the temperatures of superplastic deformation) is present compared with other areas and this might have led to significant differences in the mechanical properties. If this is the case, the fracture behaviour should also be different and this aspect will be considered elsewhere. Only recently [11, 12] the importance of grain-shape anisotropy and aligned microstructures in understanding superplastic deformation has been emphasized. An extension of the rigorous nature of this analysis to include the uniformity of grain-boundary particle distribution, whenever specimens are selected for fundamental studies, is desirable.

Therefore, it is important to evaluate the variation in the true stress–true strain curves at closely spaced strain rate/extension rate intervals and check if the equation for superplastic flow, $\sigma_s = K\dot{\varepsilon}_s^m$ (where σ_s is the steady state flow stress, $\dot{\varepsilon}_s$ is the true strain rate in the steady state region of flow, m is the strain-rate sensitivity index and K is a temperature- and grain-size dependent material constant) applied for all the specimens chosen for further microscopic/texture examination. This was done here for all three alloys. Such selected curves were then used as master curves against which the flow curves of the specimens deformed to different extents at a given extension rate were compared. Only those specimens whose flow curves practically superimposed in the relevant strain range of comparison with the master curves were selected for an investigation of the texture changes that accompany superplastic deformation. The master curves generated for the three alloys are presented in Fig. 4a–f.

In Fig. 5, following the usual practice, the steady state flow stress at the temperatures of superplastic deformation is plotted against the corresponding steady state strain rate on a double logarithmic scale. In this figure even the data obtained from those specimens that exhibited anomalous behaviour are included (indicated by arrows). Notwithstanding this, mean straight lines could be drawn and an average value of m assigned in each case, practically over the entire range of strain rate covered in the present experiments. This clearly shows the drawback (a) in taking specimens for microstructural/texture examination to identify the roles of different mechanisms in



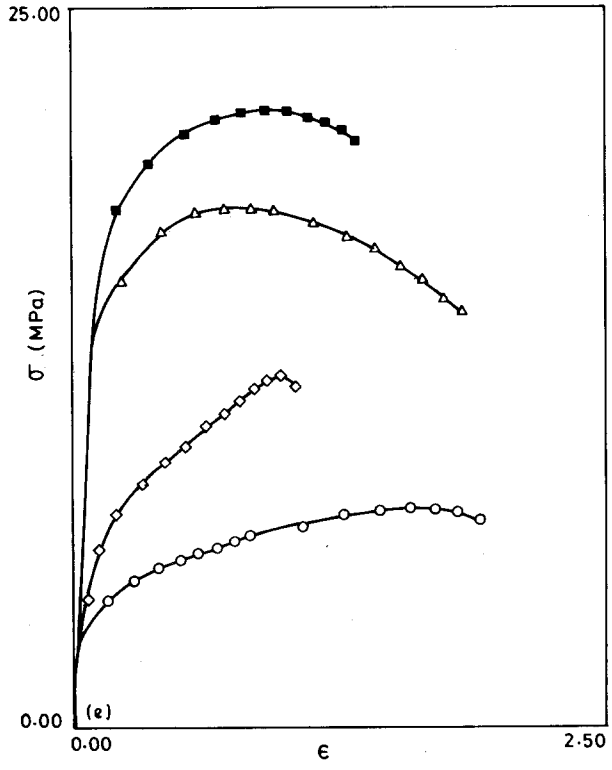


Figure 3 True stress–true strain plots (linear scale) at the temperatures of superplastic deformation (730 K for the experimental alloy (A) and Supral 100 (B), and 825 K for the Al–Ca–Zn alloy (C), which reveal the significant scatter present in the mechanical data: (a) experimental alloy, unstabilized microstructure (A_U), $\dot{\varepsilon}_0 = (\blacksquare, \triangle)$ $6.6 \times 10^{-3}\ s^{-1}$, $(\times, \bigcirc)$ $1.7 \times 10^{-3}\ s^{-1}$; (b) experimental alloy, stabilized microstructure (A_S), $\dot{\varepsilon}_0 = (\square, \triangle)$ $4.0 \times 10^{-3}\ s^{-1}$, $(\times, \bigcirc)$ $1.7 \times 10^{-3}\ s^{-1}$; (c) Supral 100 (B), $\dot{\varepsilon}_0 = (\square, \triangle)$ $4.0 \times 10^{-3}\ s^{-1}$, BT, $(\times, \bigcirc)$ $1.7 \times 10^{-3}\ s^{-1}$, BL; (d) the Al–Ca–Zn alloy tested in the sheet rolling direction (C_L) $\dot{\varepsilon}_0 = (\blacksquare, \triangle)$ $6.6 \times 10^{-3}\ s^{-1}$, $(\square, \bigcirc)$ $4.0 \times 10^{-3}\ s^{-1}$; (e) the Al–Ca–Zn alloy tested in the transverse direction of the sheet (C_T), $\dot{\varepsilon}_0 = (\blacksquare, \triangle)$ $6.6 \times 10^{-2}\ s^{-1}$, $(\diamond, \bigcirc)$ $2.7 \times 10^{-3}\ s^{-1}$.

the different regions of superplastic flow merely because the data points corresponding to these specimens fall on a smooth line (straight or curved) in a $\log \sigma_s$–$\log \dot{\varepsilon}_s$ plot, and (b) in comparing directly the results of different authors merely because their experiments correspond to a common/comparable average value of m.

This point is driven home forcefully in Fig. 6a–c, in all of which the elongation to fracture appears to go through a maximum with strain rate. (The lines have been drawn through points that corresponded to the

most consistent mechanical data.) But, it is important to note the significant scatter for all materials, except the Al–Ca–Zn alloy tested with the tensile axis parallel to the sheet rolling direction. The maximum in elongation at an intermediate strain rate cannot be understood if, as suggested by Fig. 5, m is a constant over the entire strain rate range of the tests.

A direct correlation could be achieved if the elongation to fracture was related to the local (and not the mean) value of m obtained using the strain rate change

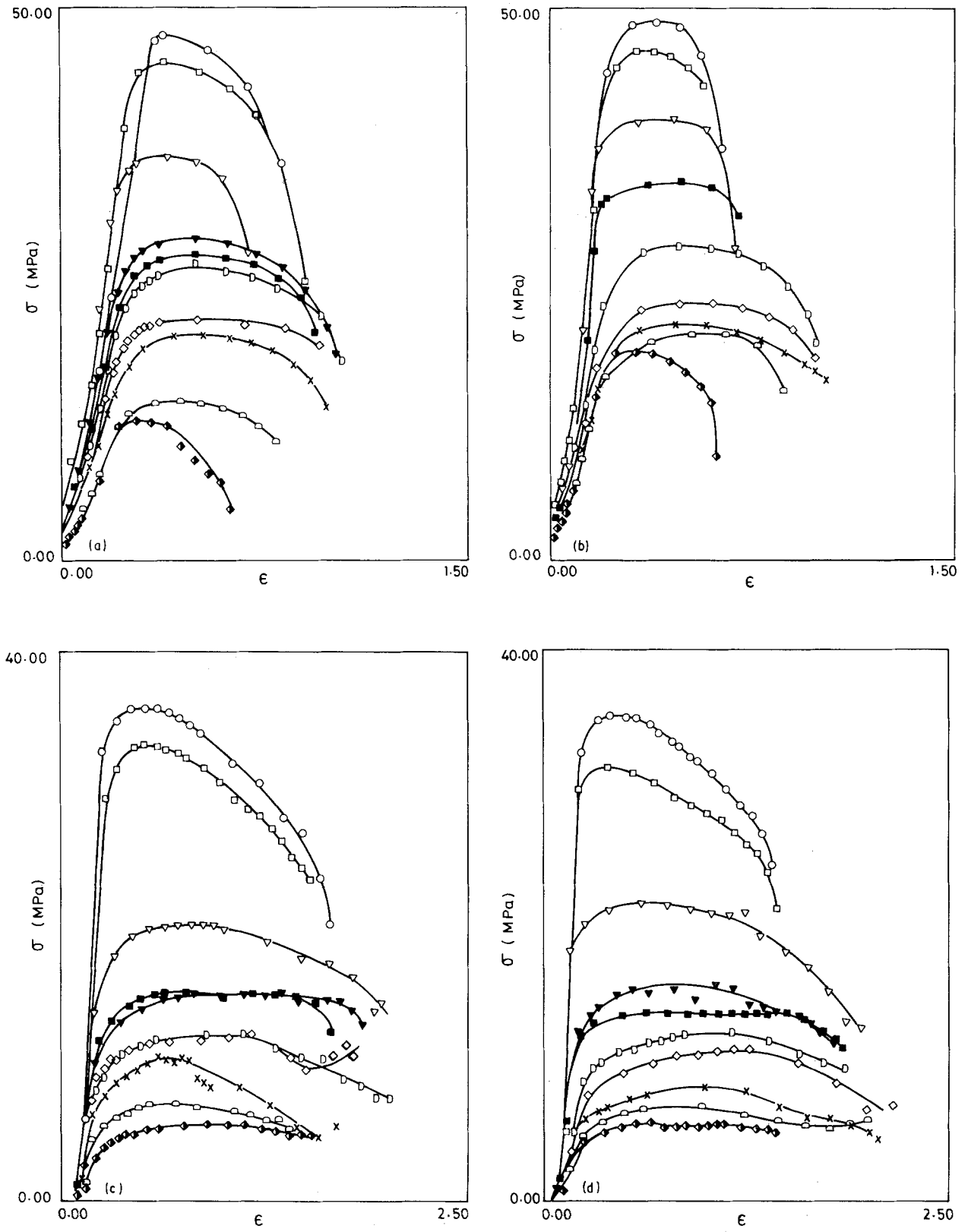


Figure 4 True stress–true strain plots (linear scale) at different constant initial strain rates (test temperature: 730 K for the laboratory alloy (A) and Supral 100 (B); 825 K for the Al–Ca–Zn alloy (C)) which served as master curves for selecting specimens for subsequent structural investigations (see text): (a) laboratory-made alloy, unstabilized microstructure (A_U); (b) laboratory-made alloy, stabilized microstructure (A_S); (c) Supral 100 tested in the rolling direction of the sheet (B_L); (d) Supral 100 tested in the transverse direction of the sheet (B_T); (e) the Al–Ca–Zn alloy tested in the rolling direction of the sheet (C_L); (f) the Al–Ca–Zn alloy tested in the transverse direction of the sheet (C_T). Initial strain rate (s^{-1}): (○) 6.6×10^{-2}, (□) 5.0×10^{-2}, (▽) 1.7×10^{-2}, (▼) 8.3×10^{-3}, (■) 6.6×10^{-3}, (D) 4.0×10^{-3}, (◇) 2.7×10^{-3}, (×) 1.7×10^{-3}, (⌓) 8.3×10^{-4}, (◆) 2.7×10^{-4}.

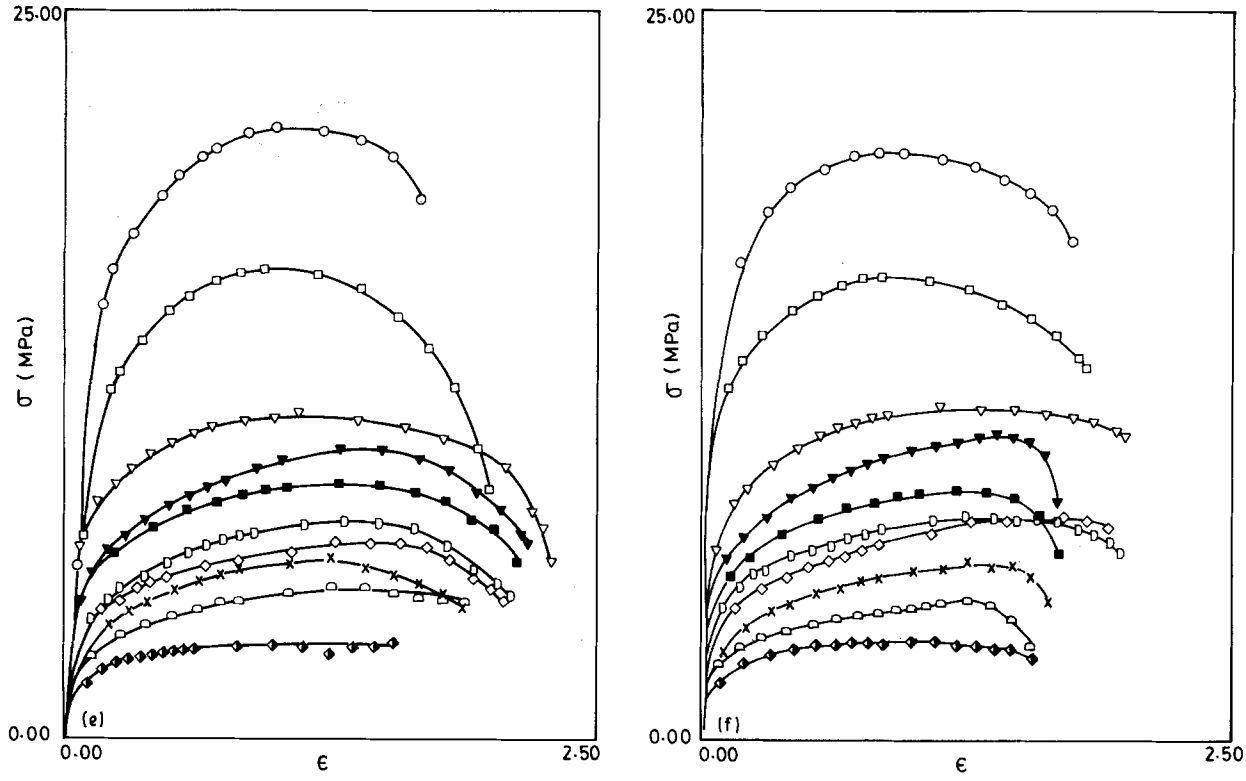


Figure 4 Continued.

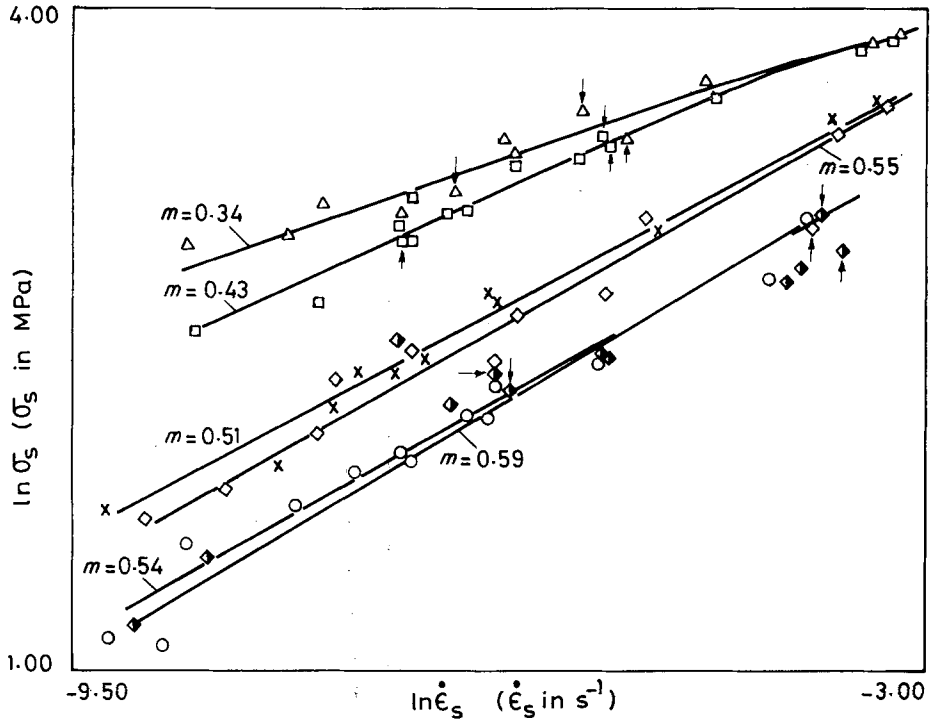
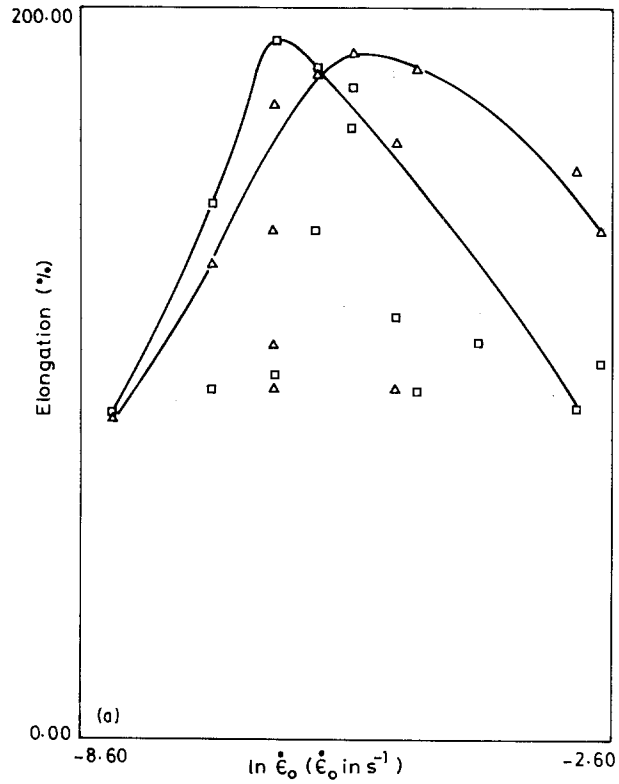


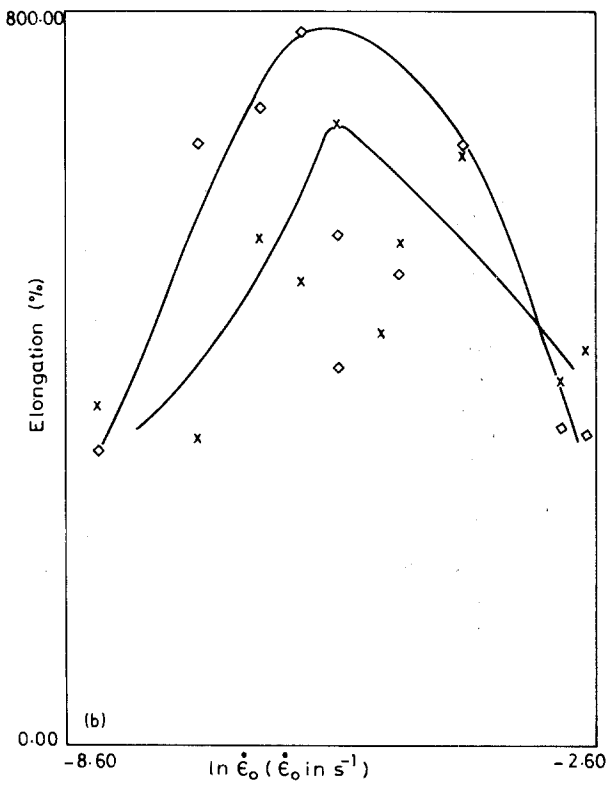
Figure 5 A double logarithmic plot of the steady state true stress, σ_s,–steady state true strain rate, $\dot{\epsilon}_s$, relationship for the different materials tested. (△) A_U, (□) A_S, (×) B_L, (◇) B_T, (○) C_L, (◆) C_T.

method [3, 4]. This point is illustrated in Table II by considering, as an example, the case of Alloy A with an unstabilized microstructure. (In passing, it is also noted that the approximately 900% elongation obtained here in the Al–Ca–Zn alloy represents the highest value for this material that has been reported to date.)

In the absence of repeated testing, any of the specimens that do not exhibit the correct variations in their mechanical properties could have been chosen for further study. Then, conceivably some unreliable conclusions regarding the operating mechanisms might have resulted.

Finally, there is a need to understand why specimens taken from the same sheet displayed such a significant scatter in the mechanical properties. A fractographic study of this aspect will be described elsewhere.





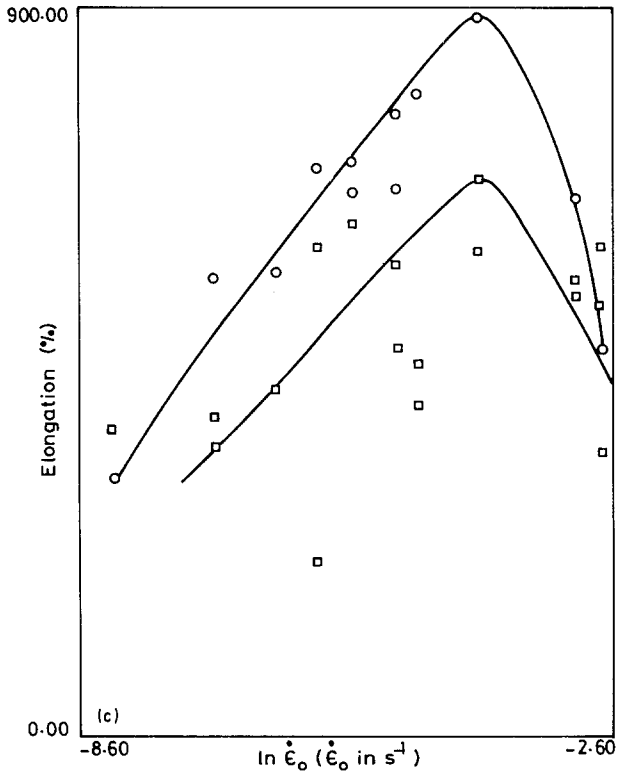


Figure 6 The elongation to fracture–true initial strain rate relationship for the three alloys: (a) laboratory-made alloy (A); (b) Supral 100 (B); (c) Al–Ca–Zn alloy (C). (a) (△) A_U, (□) A_S; (b) (×) B_L, (◇) B_T; (c) (□) C_T, (○) C_L.

TABLE II Correlation between the elongation to fracture and the directly measured value of m. Experimental Al–Cu–Zr alloy, unstabilized microstructure (A_U)

Steady state true strain rate (s^{-1})	Elongation at fracture[a] (%)	m
3.74×10^{-4}	108.4	0.25
8.03×10^{-4}	155.3	0.42
1.39×10^{-3}	168.2	0.42
2.14×10^{-3}	184.5	0.48
8.03×10^{-3}	169.7	0.33
0.0214	156	0.19
0.0372	148	0.19

[a] From Fig. 6a and b it is seen that the elongation to fracture decreases from around 780% to about 185%, as the grain size is increased from 2.2 μm to 20.2 μm (see text). But the mechanisms of flow are not significantly altered by this change in grain size at strain rates clearly to the left of the strain rate for which the elongation is maximum [6].

4. Conclusions

The following conclusions have resulted from the present study on one laboratory-made and two industrially prepared alloys.

1. Considerable scatter may be present in the mechanical properties of a superplastic alloy even when all the specimens are taken from a single sheet.

2. Accepting specimens for fundamental studies because their data fall on the mean log stress–log strain rate curves may not always lead to reliable conclusions.

3. Stress–strain curves on a linear scale of different samples should be compared for the reproducibility of results before subjecting the specimens to texture/microstructural analysis.

4. In the present experiments, the elongation at fracture could only be related to the local value of the strain-rate sensitivity index, m, and not the mean value obtained from log stress–log strain rate plots.

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