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Effect of superplastic deformation on the grain size and tensile properties of Al–6.2Zn–2.5Mg–1.7Cu (7010) alloy sheet

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An Al–Zn–Mg alloy (7010) was cold-rolled and annealed to produce a small recrystallized grain size, and superplastically deformed in the temperature range 475 to 520° C at strain rates $\dot{\epsilon} = 1.1 \times 10^{-5}$ to 2.8×10^{-3} sec^{-1}. At 500° C and $\dot{\epsilon} = 2.8 \times 10^{-5}$ sec^{-1} superplastic elongations up to 350% were obtained, but above about 60% elongation the residual room-temperature tensile properties after heat treatment decreased due to increasing grain-boundary cavitation. Grain growth rates were increased by superplastic strain.

1. Introduction

In recent years superplastic forming of metals has become a commercially attractive process. The potential of superplastically formed titanium structures in the aerospace industry has been recognized in both the USA [1] and the UK [2]. Significant cost and weight savings have been reported for such structures compared with conventional riveted structures [3].

Aluminium alloys, unlike titanium alloys, tend to develop cavities during superplastic deformation [4–7] and this has limited the application of high-strength superplastic alloys based upon the Al–Zn–Mg system (7000 series alloys). Although the development of cavities is well documented in many alloy systems [8] there is a dearth of data relating cavitation to mechanical properties. Some experiments were therefore carried out on an Al–Zn–Mg alloy (7010) to determine the relationship between superplastic strain, cavitation and residual mechanical properties. The results of these experiments are described in this paper.

2. Experimental technique

Aluminium alloy 7010 containing 6.2% Zn, 2.5% Mg, 1.7% Cu, 0.4% Zr, 0.11% Fe and 0.07% Si in the form of 50 mm thick plate was homogenized for 88 h at 465° C and cold-water quenched (CWQ). The plate was rolled at 450° C to 4.9 mm and cold-rolled to sheets of thickness 1.63 to 1.75 mm. Isothermal anneals in a salt bath, followed by a CWQ, were carried out to produce a small recrystallized grain size. Annealing temperatures were 480°, 500° and 520° C for times up to 21 h. The mean linear-intercept grain diameter (d_{MLI}) was measured in two planes (L–ST and T–ST) and in the two principal directions in each plane.

Test pieces with a 30 mm long × 5 mm wide gauge length cut from the longitudinal and transverse directions of sheet were pulled in uniaxial tension at 475°, 500° and 520° C at cross-head speeds in the range 0.02 to 5.0 mm min^{-1} (initial strain rates $\dot{\epsilon}_{\text{I}}$ were 1.1×10^{-5} to 2.8×10^{-3} sec^{-1}). Two types of hot tensile test were performed: a stepped cross-head speed test to determine the strain-rate sensitivity (m) and constant cross-head speed test to determine the superplastic elongation to fracture.

To measure the effect of superplastic strain on grain size, cavitation and room-temperature tensile properties, transverse test-pieces were deformed at 500° C and $\dot{\epsilon}_{\text{I}} = 2.8 \times 10^{-5}$ sec^{-1} to various true areal strains up to 1.2 (350% elongation). After deformation, and without remachining,

TABLE I Effect of annealing on the mean linear intercept grain size d_{MLI}

Temperature (°C)	Time at temperature (h)	Position in sheet	Mean linear intercept d_{MLI} (μm)				Grain aspect ratio* for T–ST plane
			L–ST plane		T–ST Plane		
			L direction	ST direction	T direction	ST direction	
480	0.17	Surface	12.1	9.4	12.6	8.8	
		Centre	12.9	9.05	14.2	9.6	1.47
	1	Surface	11.1	10.3	11.9	10.7	
		Centre	12.6	10.2	12.6	10.2	1.24
	21	Surface	14.0	11.6	16.6	13.2	
		Centre	15.6	11.6	16.6	13.2	1.26
500	0.17	Surface	11.0	10.0	10.9	9.1	
		Centre	12.0	9.7	12.6	9.3	1.29
	1	Surface	16.5	12.6	14.4	12.1	
		Centre	16.5	10.6	15.5	11 5	1.35
	21	Surface	22.9	20.8	24.0	17.5	
		Centre	22.0	17.5	25.9	18.7	1.39
520	0.17	Surface	16.0	14.0	18.0	15.0	
		Centre	14.5	13.0	17.0	13.0	1.31
	1	Surface	25.0	24.0	17.9	14.2	
		Centre	19.5	16.0	16.0	14.6	1.11
	2	Surface	27.0	24.5	17.7	15.75	
		Centre	25.0	22.0	15.7	14.2	1.1

*For definition see text.

some test pieces were re-solution heat treated (SHT 475° C for 10 min; CWQ; aged 24 h at 120° C and 10 h at 172° C) and tested at room temperature at a cross-head speed of 1.27 mm min^{-1}. Cavitation was monitored by density measurements made on sections 20 to 25 mm long cut from the gauge length [7] of the test pieces. Comparisons were made with control specimens cut from the test-piece head.

3. Effect of isothermal anneals and superplastic strain on grain size

Ideally a small equiaxed and thermally stable grain size is required for superplasticity [9]. A series of annealing treatments was therefore carried out to determine the optimum heat treatment for the cold-rolled sheet prior to superplastic deformation.

The mean linear intercept grain diameters (d_{MLI}) after isothermal anneals are listed in Table I. Logarithmic grain growth was obtained according to the equation

$$d_{MLI} = A \log t + C \tag{1}$$

The constants A and C are listed in Table II. For anneals at 500° C the grain growth rate was slightly greater in the T direction than in the ST direction.

At 480° C small recrystallized grains resistant to

TABLE II Constants in Equation 1 relating the mean linear intercept grain size d_{MLI} to time at temperature

Conditions		Constants in Equation 1 (d_{MLI} in μm, t in h)			
		T direction		ST direction	
		A	C	A	C
After annealing at 500° C	Centre of sheet	6.47	16.9	4.74	12.63
	Surface of sheet	6.29	15.31	4.74	13.18
After superplastic deformation at 500° C	$\dot{\epsilon}_1 = 2.8 \times 10^{-4}$	8.28	21.9	9.47	14.5
	$\dot{\epsilon}_2 = 2.8 \times 10^{-5}$	20.23	10.48	22.38	−2.56
	$\dot{\epsilon}_3 = 1.1 \times 10^{-5}$	21.11	12.23	24.74	−10.25

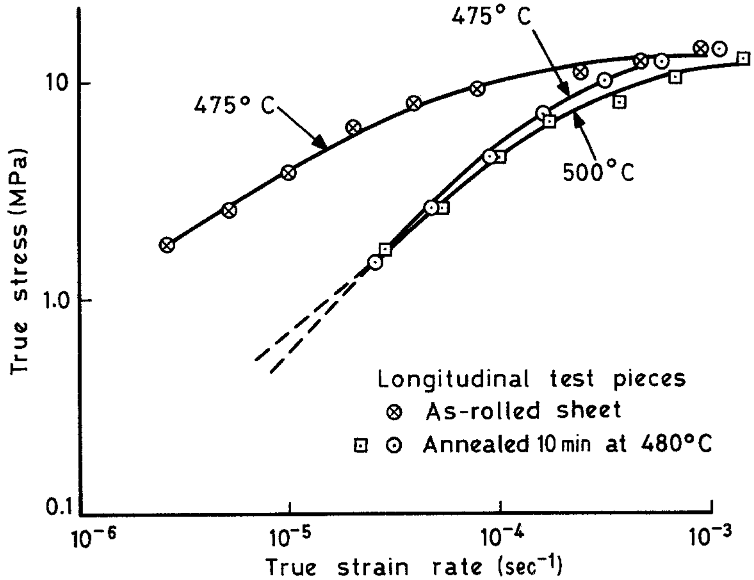

Figure 1 Plots of true stress against true strain rate, derived from stepped strain-rate tests.

growth were obtained, e.g. 8.8 to 14.2 μm diameter after 10 min and 11.6 to 16.6 μm diameter after 21 h (Table I). Temperatures above 480° C produced a coarser grain structure, and below 480° C recrystallization was incomplete. The grain aspect ratio

$$\left(\frac{d_{\mathrm{MLI}} \text{ in the } T \text{ direction}}{d_{\mathrm{MLI}} \text{ in the } ST \text{ direction}}\right)$$

could not be reduced significantly except at 520° C (Table I). A standard annealing treatment selected for all test pieces prior to superplastic deformation was therefore 10 min at 480° C to produce an aspect ratio of less than 1.5.

4. Effect of temperature and strain rate on flow stress, strain-rate sensitivity and elongation

Plots of flow stress against strain rate for annealed sheet in the L and T directions are shown in Figs. 1 and 2; the curves were similar at 475° and

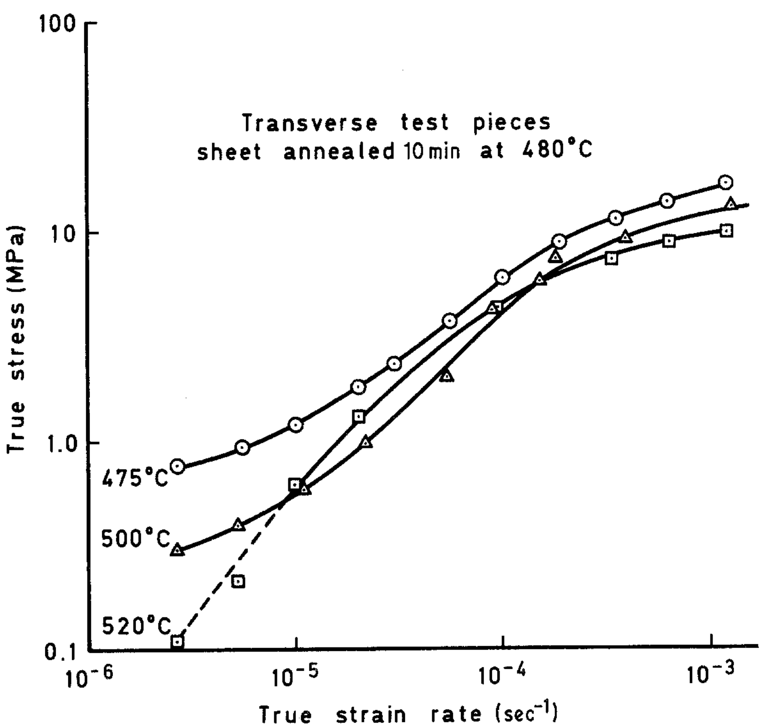

Figure 2 Plots of true stress against true strain rate, derived from stepped strain-rate tests.

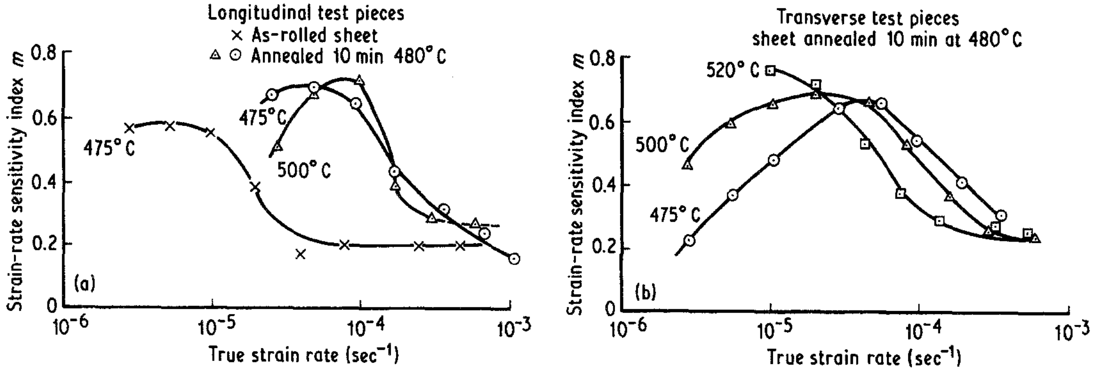

Figure 3 Effect of strain rate and test temperature on the strain-rate sensitivity index m determined in stepped strain-rate tests; (a) longitudinal test pieces, (b) transverse test pieces.

500° C. Much higher flow stresses were obtained for as-rolled sheet in the L direction at 475° C (Fig. 1). Since grain coarsening was slow at this temperature, the high value of flow stress is attributed to the larger aspect ratio of the grains and the higher dislocation density. In the T direction at 520° C at certain strain rates the flow stress could be greater than at 500° C (Fig. 2); this was attributed to more rapid grain growth at 520° C.

Plots of $m = \mathrm{d}(\ln \sigma)/\mathrm{d}(\ln \dot{\epsilon})$ against strain rate, obtained from stepped cross-head speed tests, are shown in Figs. 3a and b. With increasing temperature the maximum m values occurred at higher strain rates for longitudinal test pieces ($m = 0.70$ at 475° C, 0.72 at 500° C) but at lower strain rates for transverse test pieces ($m = 0.66$ at 475° C, 0.69 at 500° C, 0.72 at 520° C). The longitudinal test piece tested in the as-rolled condition at 475° C produced a maximum m value of 0.6, but at a strain rate less than $10^{-5}\,\mathrm{sec}^{-1}$ (Fig. 3a).

The effect of test temperature and strain rate on the superplastic elongation values for transverse test pieces is shown in Fig. 4; elongation values at all temperatures were greatest at strain rates less than $10^{-4}\,\mathrm{sec}^{-1}$. In tests at $\dot{\epsilon} = 8.5 \times 10^{-5}\,\mathrm{sec}^{-1}$ longitudinal test pieces produced a slightly higher elongation than transverse test pieces; this agrees with the higher m values for the L direction (Fig. 3b). The largest elongation value obtained in the present test programme was 350% at 500° C and $\dot{\epsilon} = 2.8 \times 10^{-5}\,\mathrm{sec}^{-1}$. A much lower elongation value was obtained for the as-cold-rolled sheet under these conditions; m values for this material (Fig. 3a) suggest that very low strain rates ($< 10^{-5}\,\mathrm{sec}^{-1}$) would be required for higher elongations.

5. Effect of superplastic strain on cavitation and room-temperature tensile properties

Transverse test pieces were pulled at 500° C and $\dot{\epsilon} = 2.8 \times 10^{-5}\,\mathrm{sec}^{-1}$ to various strains up to 350% elongation (1.2 strain). In each test piece the

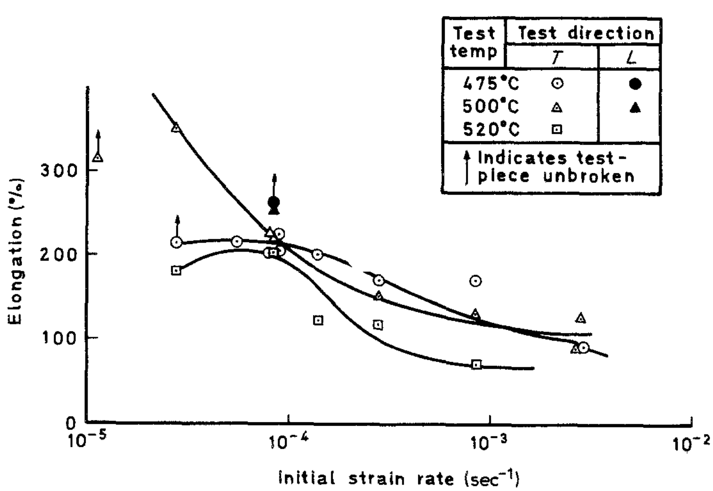

Figure 4 Plot of elongation against initial strain rate in tests at constant cross-head speed.

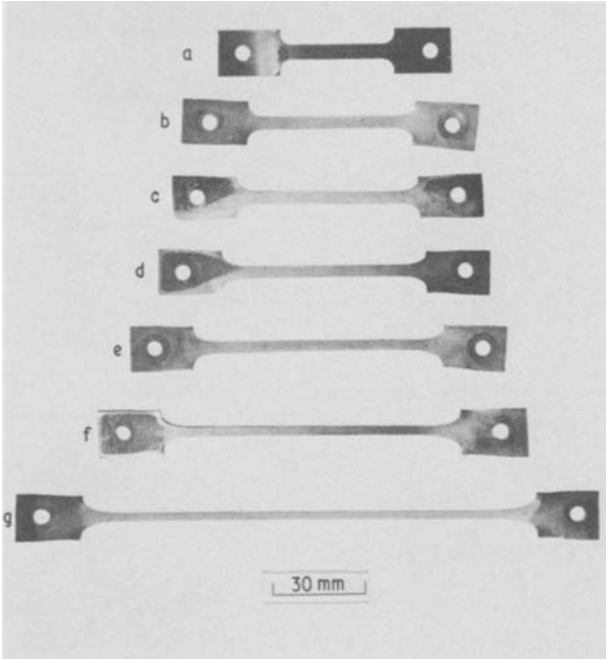

Figure 5 Transverse tensile test pieces after superplastic deformation at an initial strain rate of 2.8×10^{-5} sec^{-1} at 500° C; (a) 0%, (b) 45%, (c) 60%, (d) 80%, (e) 120%, (f) 166%, (g) 350%.

cross-section was uniform along the gauge length, as shown in Fig. 5. The void volume in the gauge length is plotted against superplastic strain in Fig. 6b; a significant increase in void volume occurred after about 60% elongation (0.47 strain) and increased linearly with strain at a rate of 0.06 vol% per 1% elongation.

The tensile properties after reheat treatment of the as-formed test pieces are given in Table III and plotted against superplastic strain in Fig. 6a. A reduction in strength and ductility coincided with an increase in the cavitation, and very low ductility values were obtained above about 100% elongation (0.70 strain) which corresponded to about 4% void volume.

6. Effect of superplastic strain on grain growth

The grain diameter is plotted against superplastic strain ϵ at 500° C in Fig. 7. The rate of increase in grain diameter, $\Delta d_{\mathrm{MLI}}/\Delta\epsilon$ was particularly large at the lower two strain rates, when $\epsilon < 0.3$ and was greater in the T direction (the tensile axis) than in the ST direction. In the strain range

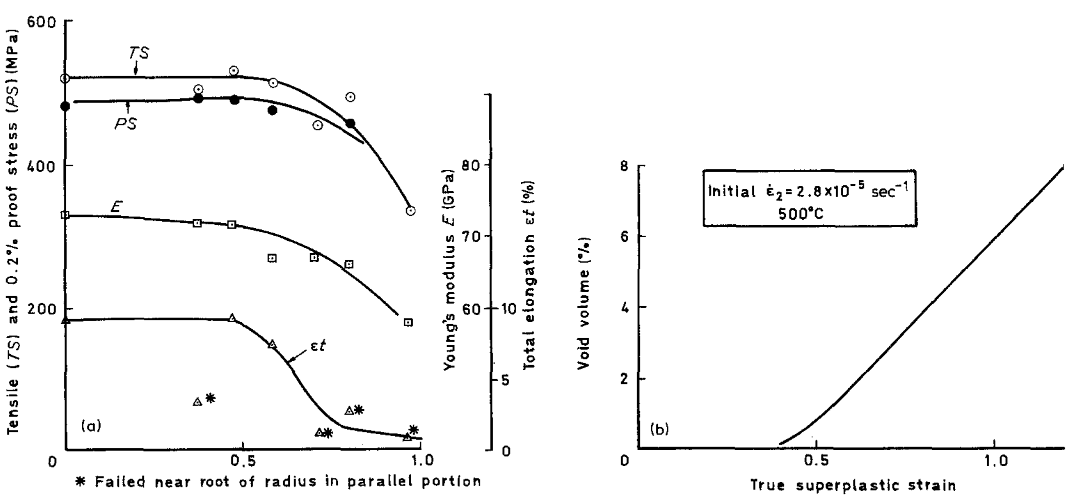

Figure 6 Effect of superplastic strain on (a) tensile properties, (b) void volume.

TABLE III Effect of superplastic strain on room-temperature tensile properties

Elongation (%)	0	45	60	80	103	123	166
True strain	0	0.37	0.47	0.58	0.71	0.8	0.98
Tensile strength (MPa)	521	508	532	515	457	496	335
0.5% proof stress (MPa)	492	–	505	491	–	475	–
0.2% proof stress (MPa)	484	495	492	477	–	460	–
0.1% proof steess (MPa)	470	478	476	462	445	446	338
Young's modulus (GPa)	73	72	72	67	67	66	58
Elongation (% on 24 mm)	9.2	3.3*	8.8	7.5	1.2*	2.9*	0.8
Uniform elongation (%)	9.2	0.32	7.9	6.7	0.11	2.0	0.12

*Test piece failed near root of radius in parallel portion outside gauge length.

$\epsilon = 0.3$ to 1.2, d_{MLI} increased linearly with superplastic strain.

Plots of grain diameter against time at 500° C for isothermally annealed sheet and for superplastically deformed sheet are compared in Fig. 8. Comparisons made after the same time at temperature, e.g. 1.7 h (*A* in Fig. 8), show that the grain diameter in sheet superplastically deformed at $\dot{\epsilon} = 2.8 \times 10^{-4}$ sec^{-1} was slightly larger, and was increasing more rapidly with time, than in the isothermally annealed sheet. The superplastic strain after 1.7 h at this strain rate was 0.76 (113% elongation). A similar grain diameter was obtained after 7 h in sheet superplastically deformed at the lower strain rate $\dot{\epsilon} = 2.8 \times 10^{-5}$ sec^{-1} (*B* in Fig. 8); however, the strain was only 0.25 (28% elongation).

When comparisons are made at the same strain of 0.76, the corresponding times at the two lowest strain rates are indicated by *C* and *D* in Fig. 8. The conditions at positions *A* to *D* in Fig. 8 are summarized in Table IV. These data indicate that when the grain diameters are compared in cold-rolled and isothermally annealed sheet, and in annealed and superplastically deformed sheet, the diameters were greater in the superplastically deformed sheet. For superplastic sheet given the same superplastic strain (positions *A*, *C*, *D* in Fig. 8), the grain diameter increased with time at temperature and increased much faster than in the cold-rolled and isothermal sheet. Thus the dynamic conditions in the superplastically deforming sheet appeared to lead to greater grain growth rates.

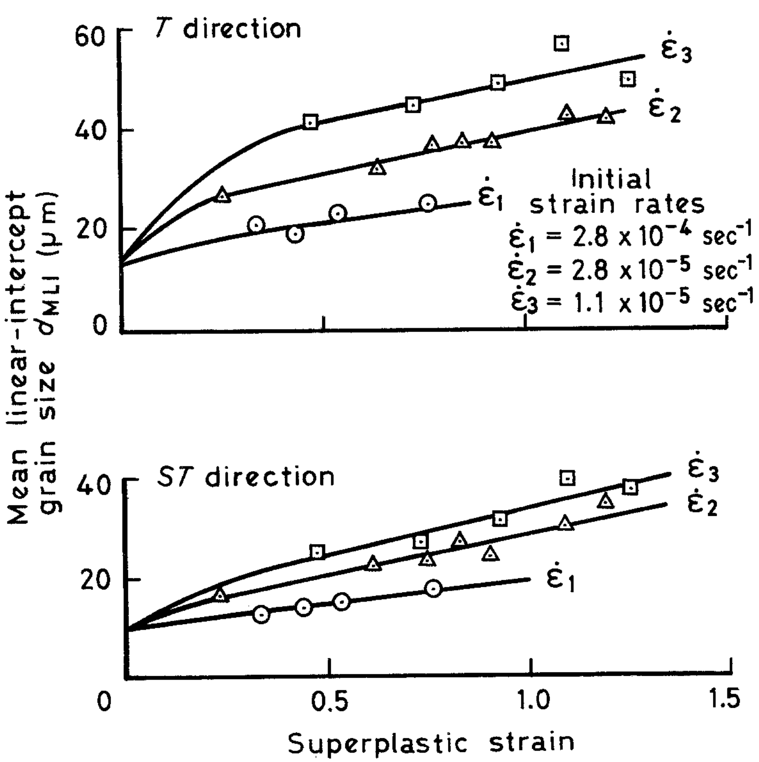

Figure 7 Effect of superplastic strain at 500° C and initial strain rate on grain size in the gauge lengths of transverse test pieces.

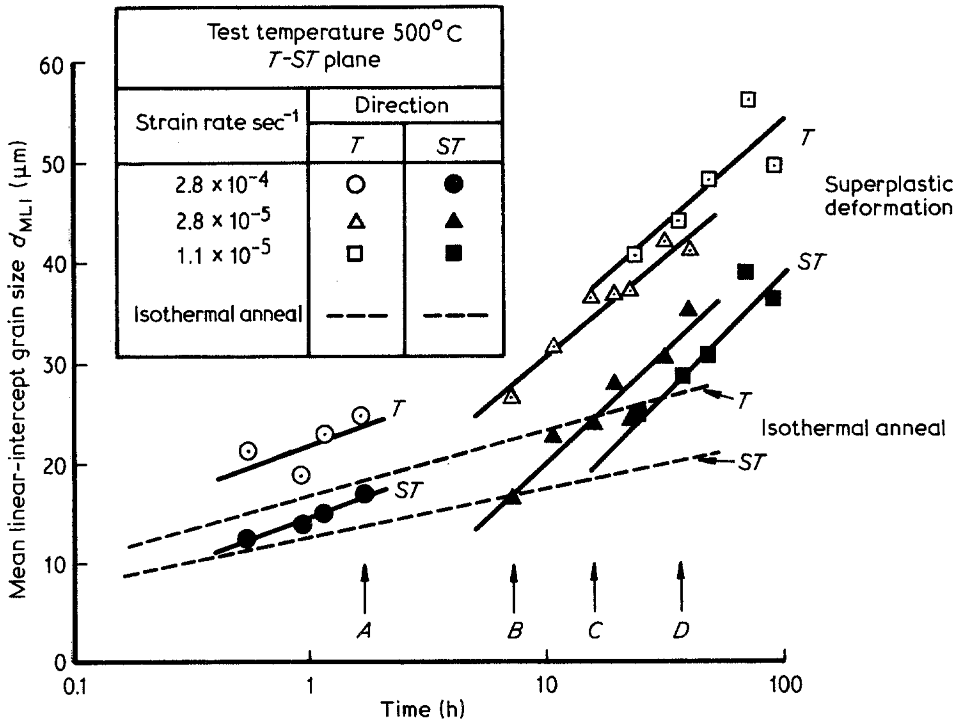

Figure 8 Effect of time at temperature and initial strain rate on grain size during superplastic deformation.

7. Discussion

The results show that although superplastic elongation values of up to 350% are possible in the transverse direction of the 7010 alloy, the tensile properties are adversely affected above about 60% elongation by the onset of cavitation. The results are qualitatively similar to those reported for the 7475 aluminium-alloy given a prior thermomechanical treatment to increase superplastic formability [4]. The grain sizes in the 7010 and 7475 alloys were initially similar with a lower aspect ratio in the 7010 alloy (1.5) compared with 7475 (1.8), but grain growth appeared to be greater in the 7010 alloy.

Other important differences between the alloys were the flow stresses and *m*-values under the optimum superplastic conditions ($\dot{\epsilon} = 2.0 \times 10^{-4}\,\text{sec}^{-1}$ and 516° C for 7475 [4] and ($\dot{\epsilon} = 2.8 \times 10^{-5}\,\text{sec}^{-1}$ and 500° C for 7010). The flow stresses were similar in the *L* directions but much higher for the 7010 in the *T* direction, while the *m* values were much lower in the 7010; values were 0.4(*L*) and 0.62(*T*) for 7010, and 0.9(*L*) and 0.85(*T*) for 7475. Thus significantly higher superplastic elongations have been reported for 7475 (up to 1200% [10]) compared with those obtainable for 7010 or 7075 aluminium-alloys with lower maximum forming temperatures.

TABLE IV Effect of time and strain on grain size in isothermally annealed and superplastically deformed sheet at 500° C

Position on time scale in Fig. 8	Equivalent time and strain rate	Elongation (strain)	Comments
B	7 h 2.8×10^{-5} sec^{-1}	28% (0.25)	Same grain size, different strain
A	1.7 h 2.8×10^{-4} sec^{-1}	113% (0.76)	
C	15 h 2.8×10^{-5} sec^{-1}	113% (0.76)	Same strain, grain size increasing in order *A* – *C* – *D*
D	36 h 1.1×10^{-5} sec^{-1}	113% (0.76)	

The behaviour of the 7000 series of alloys differs from that of alloys such as Al–6Cu–0.5Zr (SUPRAL) and Al–Mg–Li [11, 12, 13], which exhibit dynamic recystallization during superplastic deformation; these alloys tend to retain a fine grain size and low flow stress to large superplastic strains.

Cavitation in the 7000 series of alloys is characterized by a wide variation in cavity size and number within one sheet, and between different batches of sheet. For example for 7475 alloy deformed to 1.5 strain at $\dot{\epsilon} = 2 \times 10^{-4}\,\text{sec}^{-1}$ and 516° C, void volumes reported were 0.25, 1.5 and 4% [4, 10, 14]. Although the prior processing route developed for 7475 appeared to be associated with a low rate of cavitation occurred at about the same strain of 0.5 (60% elongation) in both 7010 and 7475 alloys. Above 1.1 strain the rate of cavitation was similar in 7010 at 0.6 vol% per 1% elongation, and in 7475 at 0.02 vol%per 1% elongation.

The differences in cavitation at low strains may be partly caused by the sampling techniques used in the two programmes. For 7475 a 5 x 5 mm area [14] was cut from a region adjacent to the room-temperature test piece, and was solution-treated and water-quenched before the density measurements were made. In the present work density measurements were made on sections 20 to 25 mm long cut from the gauge length of superplastically formed test pieces in the as-formed state. The advantages of the present method were a larger sample size, which favoured greater accuracy in the density measurements, and less sensitivity to variation in cavitation along the gauge length; possible sintering of cavities during solution heat-treatment was also avoided.

Another effect difficult to quantify was that arising from the difference in aspect ratio, i.e. the width to length ratio of the gauge lengths of the superplastic test pieces. This was 42/52 for 7475 but 5/25 for 7010. Recent work on titanium by Inglebrecht indicates that during uniaxial superplastic deformation the R value (width strain/thickness strain) depends on the initial aspect ratio. In aluminium alloys this could affect the cavitation for a given uniaxial strain. However it is significant that although the magnitude of the cavitation differed in the two alloys 7010 and 7475, the reduction in tensile and fatigue properties coincided with the onset of cavitation at similar strains in both alloys.

A number of methods have been suggested for reducing cavitation, e.g. to annealing at high temperature to remove residual hydrogen [15], or applying a net positive pressure [16] across the sheet thickness during forming or a post hot-isostatic pressing operation, but the reliability, practicality and cost of these suggestions for complex components has yet to be determined. In practice it may be difficult to predict precisely the void volume (and hence the mechanical properties) of high-strength 7000 series alloy sheet after superplastic forming into complex shapes. Until further data is available, the use of superplastically formed components in highly loaded structures will be limited.

8. Conclusions

1. A small, relatively stable grain size (8 to 15 μm) was produced in 7010 Al–Zn–Mg alloy sheet by cold-rolling and annealing for 10 min at 480° C.

2. This alloy was superplastic at temperatures in the range 480° C to 520° C and strain rates between 10^{-5} and $10^{-4}\,\text{sec}^{-1}$.

3. The elongations produced under superplastic conditions were greater for stress in the longitudinal direction than in the transverse direction. The maximum elongation in the transverse direction (350% at 500° C and $2.8 \times 10^{-5}\,\text{sec}^{-1}$) was limited by cavitation.

4. Grain growth rates during superplastic deformation at 500° C were greater than during isothermal annealing.

5. A reduction in tensile properties after superplastic deformation and re-heat treatment coincided with the onset of cavitation during superplastic deformation, occurring at strains between 0.5 and 0.7.

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