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An analysis of grain-growth data in duplex materials on static annealing and during superplastic deformation

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An analysis of data on grain growth in several duplex materials subjected to superplastic deformation (SPD) and to static annealing (SA) is presented. Also, for comparison, data of a single-phase alloy and a particle-strengthened one were considered. The theoretical equation $D^n - D_0^n = Kt$ was employed, where D and D_0 are the instantaneous and initial average grain size, respectively, t is the deformation or annealing time, n is the kinetic exponent and K is the rate constant. When analysing the selected D–t data sets, the fact that D_0 values were generally not small enough to be negligible in the above equation was taken into account. It is concluded: (i) for a given duplex alloy, the coarsening mechanisms acting during SA and SPD are different, the kinetics being enhanced by concurrent deformation, and (ii) whatever the alloy, the value for n for grain growth under SPD is near to 2, (as for normal grain growth in single-phase materials).

1. Introduction

The theoretical models developed for steady-state grain and particle growth during static annealing predict a coarsening law of the type [1]:

$$D^n - D_0^n = Kt \qquad (1)$$

where D and D_0 are the instantaneous and initial average grain (or particle) sizes, respectively, t is the annealing time, n is the kinetic exponent and K is a rate constant. According to these models different integer values of the kinetic exponent n ($2 \leqslant n \leqslant 5$) have been associated with various controlling mechanisms for grain growth in single-phase materials and for particle coarsening. When second-phase particles are present, the motion of matrix grain boundaries during static annealing (SA) is inhibited by a retaining force due to particles (Zener effect), and normal grain growth terminates early at a limiting grain size [1–4]. Then, as the particle and grain growth are now linked, in steady state the grain-size/particle-size ratio remains constant (unless abnormal grain growth takes place) and the grains should coarsen with the same kinetics of the acting Ostwald ripening process. Moreover, the role of the particle volume fraction on the particle-coarsening rates when lattice diffusion ($n = 3$) or grain-boundary diffusion ($n = 4$) is controlling, has been analysed by Ardell [1, 5, 6]. In relation to Equation 1, he concluded that n is unaffected while K increases as the particle volume fraction increases. Thus, in two-phase materials, as is the case of most superplastic alloys, it can be expected that the grain-growth kinetics will be similar to those of second-phase particles in a matrix, at least for the above mentioned mechanisms [4, 7]. On the other hand, during superplastic deformation (SPD), grain growth is not only the result of the annealing time at the test temperature, but is also enhanced by deformation [4, 7–9]. It has been assumed by some authors [7–9] that if the controlling mechanisms for coarsening are the same, then the same types of laws as for static annealing might apply for describing strain-enhanced grain growth.

When analysing grain-growth data with the purpose to produce an intepretation of the controlling mechanism involved, within the frame of the coarsening theories available, it is important to evaluate properly the kinetic exponent n in Equation 1. Many inconsistencies in the experimental n values reported in the literature have been claimed [7, 10, 11] to arise when D_0 is unjustifiably neglected and the following expression is used for the analysis of D–t data:

$$D^{n'} = K't \qquad (2)$$

where n' is the actual value determined. D_0 can only be neglected in Equation 1 for a given data set if the quantity $(D_f/D_0)^n$, where D_f is the final grain (or particle) size, is large.

Recently, two simple and reliable procedures for analysing D–t data, which do not require the simplifying assumption that D_0 can be neglected in Equation 1, have been discussed. They will be referred to as Methods 1 and 2. Method 1 [10] is based on the representation of D^n against t for different values of n, from which the n value resulting in the best straight

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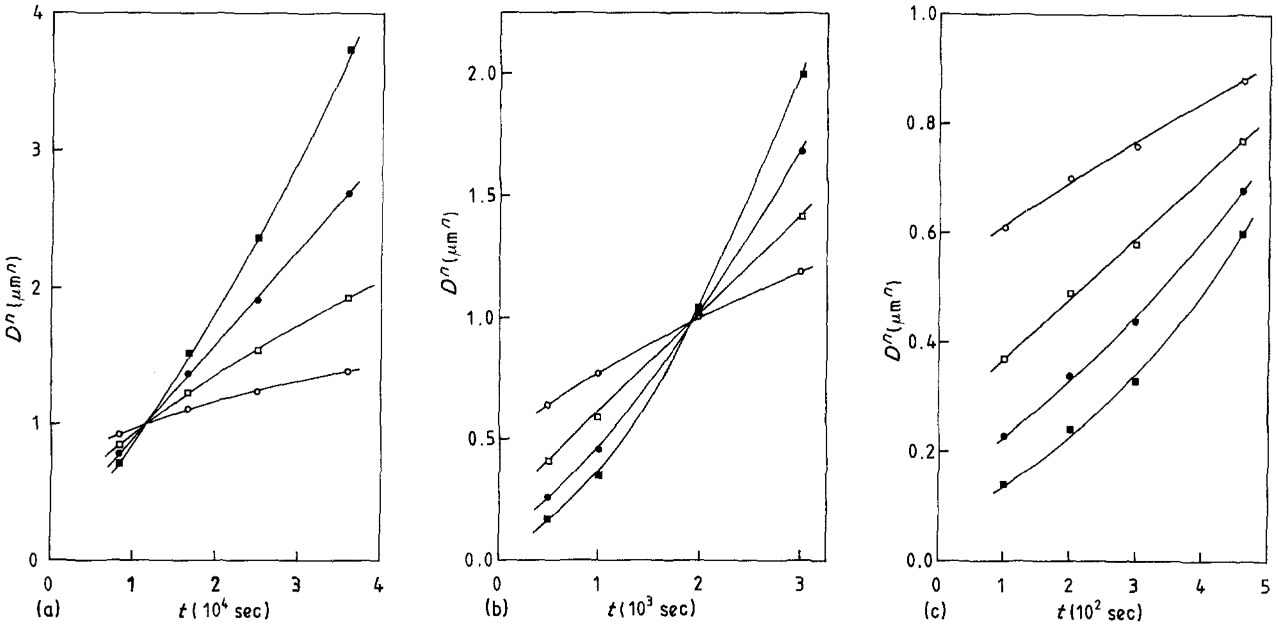

Figure 1 D^n–t plots corresponding to grain growth under superplastic deformation of a Zn–22% Al alloy [7], at 495 K and for different strain rates. (a) $\dot{\varepsilon}_0 = 3 \times 10^{-5}\,\mathrm{sec}^{-1}$, (b) $\dot{\varepsilon}_0 = 1 \times 10^{-3}\,\mathrm{sec}^{-1}$, (c) $\dot{\varepsilon} = 1 \times 10^{-2}\,\mathrm{sec}^{-1}$. n = (○) 1, (□) 2, (●) 3, (■) 4.

line is chosen. Whereas, according to Method 2 [11], if the experimental data are analysed as dD/dt against D on a log–log scale, a straight line should result, with its slope being given by $(1 - n)$. For both procedures, once the kinetic exponent is known, the value of the rate constant K can then be easily calculated.

In this paper an attempt is made to re-analyse, using Equation 1, quantitative data for grain growth under SPD and SA available in the literature for different duplex superplastic alloys. The following materials were considered: a Zn–22% Al alloy [7]; a Ti–6% Al–4% V alloy [12–14]; and an Fe–25.7% Cr–6.6% Ni stainless steel [15]. Also, for comparison, the coarsening data for a single-phase Sn–1% Bi alloy [9] and those for a particle-strengthened Cu–2.8% Al–1.8% Si–0.4% Co alloy (Coronze 638) [16] were taken into account. For this analysis, the above-mentioned Methods 1 and 2 were employed.

2. Data selection and analysis procedure

Among the many quantitative SPD coarsening experimental data available in the literature [4, 7, 9, 12–26], only some of them [7, 9, 12–16] were suitable for this study. In fact, the analysis was limited to those D–t data sets consisting of at least four experimental points with a reasonably low scatter that could correspond to grain growth in superplastic Regions I or II. The experimental conditions, temperature and deformation rate wherever pertinent, corresponding to the data sets selected for the alloys considered, are indicated in Table I. In all the studies included, the average grain size, or simply the grain size, was given as a function of the mean lineal intercept (MLI) for one or two phases, as shown in Table I. For the SPD data, t is the deformation time. When graphically analysing the selected D–t data by Method 1, a best straight line in the D^n–t space could be visually chosen with an absolute accuracy for n better than 0.5. The n, K and R results reported in Table I were numerically obtained by using Method 2 with least square analysis; R being the corresponding correlation coefficient. It should be noted that because of the nature of the SPD data set presented in [13] for the Ti–6Al–4V alloy, those data could only be treated by Method 2.

3. Results

D^n–t plots presently derived from the SPD data at 495 K for the Zn–22% Al alloy are shown in Fig. 1, for integer values of n in the range $1 \leqslant n \leqslant 4$. In each figure, the n value for which the data best fit a straight line can be easily detected and leads to n = 2 to 3. The n, K and R values calculated through Method 2 and associated with Equation 1 for these SPD experiments are shown in Table I. From this Table and Fig. 2 it can be appreciated that under SA for the temperatures

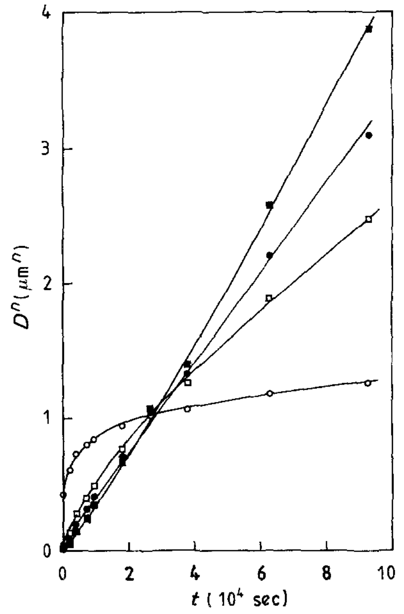

Figure 2 D^n–t plot corresponding to grain growth under static annealing of a Zn–22% Al alloy [7], at 495 K. n = (○) 1, (□) 4, (●) 5, (■) 6.

TABLE I Values of the kinetic exponent n and of the rate constant K, obtained from the analysis of grain-growth experimental data for different alloys under superplastic deformation (SPD) and static annealing (SA), using Method 2

Alloy	Reference	Experimental conditions		$\dot{\varepsilon}_0$ (sec^{-1})	$\dot{\varepsilon}$ (sec^{-1})	$\dot{\varepsilon}_c$ (sec^{-1})	Grain size definition	n	K (μm^n sec^{-1})	R
		SPD/SA	Temp. (K)							
Zn–22Al	[7]	SPD	495	3×10^{-5}			MLI (α and β)	3.0	6.9×10^{-5}	-0.98
		SPD	495	1×10^{-3}				1.8	3.7×10^{-4}	-0.94
		SPD	495	1×10^{-2}				1.8	1.0×10^{-3}	-0.43
		SA	472					4.7	1.0×10^{-5}	-0.82
		SA	495					4.8	3.4×10^{-5}	-0.94
		SA	523					4.9	1.1×10^{-4}	-0.97
Ti–6Al–4V	[12]	SPD	1200		2×10^{-4}		1.68 MLI (α and β)	2.3	4.2×10^{-2}	-0.91
ø̈}		SA	1200					4.8	1.0×10^{-3}	-0.90
	[13]	SPD	1173		2×10^{-4}		MLI (α and β)	2.5	3.1×10^{-1}	-0.95
		SA	1223					3.2	2.4×10^{-1}	-0.89
	[14]	SPD	1213		2.3×10^{-4}		MLI (α)	2.1	1.5×10^{-2}	-0.69
Fe–25.7Cr–6.6Ni	[15]	SPD	1273		1×10^{-4}		MLI (α and β)	2.2	9.6×10^{-5}	-0.98
		SA	1273					4.5	4.4×10^{-1}	-0.95
Sn–1Bi	[9]	SPD	293			4.5×10^{-7}	MLI	2.7	1.0×10^{-3}	-0.75
		SPD	293			1.7×10^{-6}		1.8	1.2×10^{-4}	-0.76
		SPD	293	1.7×10^{-4}				1.9	2.6×10^{-3}	-0.58
		SA	293					2.0	8.6×10^{-5}	-0.91
Cu–2.8Al–1.8Si–0.4Co	[16]	SPD	823	1.4×10^{-5}			MLI (α)	2.0	1.1×10^{-4}	-0.93
		SPD	823	5.6×10^{-5}				1.8	2.2×10^{-4}	-0.88
		SPD	823	2.8×10^{-4}				2.5	2.1×10^{-3}	-0.99

R = correlation coefficient as resulting from Method 2 in least square analysis. MLI = mean lineal intercept. $\dot{\varepsilon}_0$, $\dot{\varepsilon}$ and $\dot{\varepsilon}_c$ correspond to initial strain rate, constant strain rate and creep strain rate, respectively.

of 472, 495 and 523 K, a grain growth kinetics with approximately $n = 5$ takes place.

From the above results it can be noted that the n values obtained by the graphical procedure (Method 1) are in good agreement with those obtained by the analytical one (Method 2). This agreement was also found for the other materials analysed. Accordingly, and in benefit of brevity, for the other alloys, only the results obtained by Method 2 are presented.

The n, K and R values calculated for the Ti–6% Al–4% V and Fe–25.7% Cr–6.6% Ni microduplex alloys are also shown in Table I. These n values are similar to those obtained above for the Zn–22% Al alloy. In fact, the SPD data sets for the titanium alloy (reported by different authors, for similar conditions of strain rates and temperature) as well as that for the microduplex steel lead to n values near to 2, between 2.1 and 2.5. Moreover, the values under SA obtained for both alloys are again significantly larger than 2. For the microduplex steel, $n = 4.5$ was obtained, while the n values derived for the titanium alloy was 4.8 in one case (1200 K, [12]) and 3.2 in the other (1213 K, [13]).

The results of the analysis corresponding to the single-phase Sn–1% Bi alloy studied by Clark and Alden [9], see Table I, lead to $n = 2$ for SA and to $n = 2$ to 3 for SPD. The results for the Cu–2.8% Al–1.8% Si–0.4% Co alloy are also given in that table. For the three SPD data sets considered, n values close to 2 were obtained, while K increases as $\dot{\varepsilon}$ increases. In this copper alloy no measurable grain growth was found during SA at the temperature and for the times of interest [16].

4. Discussion

Senkov and Myshlyaev studied the superplastic Zn–22% Al alloy and have recently published [7] the most complete data set for grain growth in duplex alloys under SPD. They verified that the distribution of α and β grain sizes after SA or SPD practically coincided, provided the grain sizes were normalized with respect to the corresponding mean. This observation indicated that the experiments were performed under steady-state coarsening conditions. It was also detected that at a given instant of annealing the α-phase size/β-phase size ratio remained approximately constant. They also showed that the $D-t$ data for SA satisfactorily fitted an $n = 4$ or $n = 5$ kinetics. Moreover, Senkov and Myshlyaev, fully aware of the limitations of processing grain-growth data using Equation 2, nevertheless found it expedient to analyse their SPD data by using the simplified expression, as in Equation 2, thus actually obtaining an n' value close to 4 (1/0.28). On the grounds of such results and critically evaluating the possible grain-growth mechanisms among those treated in the literature, they proposed that the same controlling mechanism, diffusion along grain boundaries, was operative for SA and SPD. So, assuming a common mechanism with $n = 4$, they finally separated in the SPD data the grain-growth contribution due to SA from that due to deformation. Unfortunately, the use of Equation 2 for the analysis of the SPD data sets considered, leads to an unacceptable error in the estimation of the pertinent true n value. In effect, from the present analysis of the data for the Zn–22% Al alloy [7] it is deduced that the n values associated with SA ($n \simeq 5$) are significantly different from those corresponding to SPD ($n \simeq 2$ to 3), see Table I and Figs 1 and 2. This result implies that for the said material the rate-controlling grain-growth mechanisms during SA and SPD are different.

The n values obtained here for the Sn–1% Bi alloy studied by Clark and Alden [9], see Table I, are in good agreement with the values reported by those authors, who have shown that their experimental data reasonably verified a coarsening kinetic with $n = 2$, under SA and SPD (for $1.7 \times 10^{-4}\,\text{sec}^{-1} < \dot{\varepsilon} < 1.7 \times 10^{-6}\,\text{sec}^{-1}$) conditions. In view of this last observation and of the similarities of grain-size and grain-type distributions in deformed and undeformed samples detected in that work, Clark and Alden [9] concluded that during deformation a normal process of grain growth takes place. They also suggested that the possible mechanism for strain-enhanced coarsening is the production of excess vacancies in the grain-growth region as a consequence of grain-boundary sliding, leading to increased grain-boundary mobility. Later Kaibyshev and co-workers [25, 26] arrived at similar conclusions when studying grain-growth kinetics under SPD of a Zn–0.4% Al alloy, which is similar in microstructure to a single-phase one. They also detected experimentally that the vacancy concentration increases with increasing strain rate. Moreoever, there was reasonable agreement between their experimental results and an expression such as Equation 1, where K was predicted to be proportional to the vacancy concentration. It was suggested [25, 26] that the biggest increase of vacancy concentration should occur in the strain-rate range where the contribution of grain-boundary sliding to the total deformation is greatest.

The n values derived here for grain growth under SA in different duplex alloys, see Table I, indicate, within the framework of coarsening theories available [1], that boundary-diffusion ($n = 4$) or dislocation-pipe-diffusion ($n = 5$) coarsening controlling mechanisms are operating. On static annealing of two-phase materials, usually a kinetics with $n = 4$ has been observed [8, 27]. On the other hand, for those duplex materials examined here, a strain-enhanced grain growth with a kinetic exponent close to $n = 2$ was detected, see Table I. A possible explanation for this behaviour under SPD is that, as a consequence of grain-boundary sliding, the two closely related processes of grain-boundary migration and boundary diffusion are enhanced, possibly by an increasing vacancy concentration near the sliding zones. Moreover, as the grain boundaries then act as fast-diffusion paths, the grain-boundary migration process becomes the controlling step for grain growth, as for normal grain growth in single-phase materials. It can then be expected that for any duplex alloy submitted to SPD, within an appropriate range of strain rate, temperature and grain size, the grain-growth mechanism will be the same and n a constant, while increases in strain rate and/or temperature will lead to larger values of K. This suggestion, which has already been proposed for the case of single-phase superplastic materials [8, 26], finds support in the experimental evidence presented in Table I. Incidentally, such a suggestion also seems to apply to the particle-strengthened Cu–2.8% Al–1.8% Si–0.4% Co alloy, see Table I.

Outside the range of experimental conditions referred to above, other controlling coarsening mechanisms could possibly operate. Thus, the SPD tests with slower strain rate analysed here for the Zn–22% Al and Sn–1% Bi alloys, where an n value close to 3 was detected (Table I), might well correspond to a diffusion-controlled coarsening mechanism [1]. On the other hand, in the Ti–6% Al–4% V alloy deformed under a constant strain rate of $2.3 \times 10^{-4}\,\text{sec}^{-1}$ [14], grain growth was detected at a deformation temperature of 1213 K (see Table I), while grain refinement associated with dislocation activity (dynamic recrystallization) was observed at 1033 K. Cope and Ridley [14] remarked also that grain refinement may be possible at any temperature in the superplastic range, provided the strain is sufficiently high. Another situation where Equation 1 cannot be applied was reported by Kashyap and Mukherjee [15] who observed that grain size decreased in the early stages of the superplastic deformation ($\varepsilon < 0.04$) of the duplex steel presented in Table I.

5. Conclusions

1. For a given duplex alloy, the coarsening mechanisms acting during static annealing and superplastic deformation are different.
2. Whatever the alloy, the value of the kinetic exponent n for grain growth under superplastic deformation is near to 2 (as for normal grain growth in single-phase materials).

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