MICROSTRUCTURAL DEVELOPMENT IN A SUPERPLASTIC P/M 7064 ALLOY

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Abstract

A powder-metallurgy (P/M) alloy based on 7064 was processed to a highly superplastic condition capable of tensile elongations of 1400% and microstructural development was characterized by transmission electron microscopy (TEM). The alloy was found to recrystallize during heating to the superplastic deformation temperature. Grain-specific orientation measurements via TEM were used to obtain grain boundary misorientations in the recrystallized condition (with no tensile strain) and in the recrystallized and superplastically deformed condition (with a true strain ϵ=1). Superplastic deformation caused substantial changes in the types of grain boundaries present, with several boundary types in the annealed material being replaced by their twin-modified counterparts in superplastically deformed material. This occurred simultaneously with the appearance of twin-related boundaries (of first and second generation) and the $\Sigma 7$ misorientation. Twinning processes are active during SPD of this alloy and serve to alter the boundaries available for such structure dependent processes as grain boundary sliding.

Introduction

Many fine-grained alloys are superplastic when deformed within certain ranges of temperature and strain rate. The fine, equiaxed grains present in these alloys are usually developed during the recrystallization of rolled sheet. Recrystallization can occur during an anneal prior to deformation, as for 7075 [1,2], or during the early stages of superplastic deformation (SPD), as reported for Supral [3-6]. In either case, grain boundaries arise which are capable of supporting superplastic deformation mechanisms such as grain boundary sliding (GBS) and the associated accommodation processes.

Studies of GBS rates have shown a dependence on grain boundary structure [7-9], and it has been suggested that this is related to the rate of dissociation of trapped lattice dislocations into grain boundary partials. The questions as to what grain boundary structural characteristics are beneficial for SPD and how they develop can probably best be addressed by careful study of highly superplastic materials. A full description of grain boundary structure requires values for the five degrees of freedom of the boundary, rigid body translations and relaxation effects [10]. Studies involving polycrystalline materials, however, where large numbers of grains are considered, are usually restricted to the three orthogonal angles describing the relative crystallographic orientation of neighboring grains. This grain-to-grain misorientation can be expressed as Euler angles, angle/axis pairs or 3 x 3 rotation matrices (of direction cosines) and then compared to special geometric relationships, e.g. coincident site lattices (CSL's). Large numbers of boundaries can be evaluated via computer assisted analyses of Kikuchi patterns which can be obtained by convergent beam technique in the transmission electron microscope or by backscattering in the scanning electron microscope.

Recrystallization processes may result in the appearance of specific types of grain boundaries. A reorientation of ~40°/<111> is frequently found for recrystallization nuclei in pure aluminum, under a variety of conditions [11]. This is close to the special relationship of 38.2°/<111>, which is the $\Sigma 7$ CSL, and the recrystallization reorientation is often referred to as $\Sigma 7$. A recently proposed theory of texture development attributes the final orientations present in recrystallized material to multiple twinning or "twin chain" effects. In this scheme a twin (which can be described as 60°/<111> or a $\Sigma 3$ CSL) combines in multiples to form higher generation twins, viz.:

$\Sigma 3^2 = \Sigma 9$, a second generation twin; $\Sigma 3^3 = \Sigma 27$, a third generation twin;

note also that, for appropriate choice of rotation matrices,

combinations can produce $\Sigma 9 \times \Sigma 3^{-1} = \Sigma 3$, etc.

An important consideration of the twin chain argument is that the successive twins cannot form on random {111} planes. Instead, the active twinning variants must be biased in order to achieve the final orientation [12]. The multiplication of Σ values can be applied to other orientations than twins. An example of this can be used to explain the absence of a $\Sigma 7$ reorientation when comparing textures from rolled and fully recrystallized material. The $\Sigma 7$ boundary, while prevalent in the early stages of recrystallization, is not stable with respect to further orientation change, and this, presumably, is due to twin modification of the recrystallization front; a $\Sigma 7$, for instance, could be modified to a $\Sigma 21$a by twinning [11].

The categorization of grain boundaries as CSL's requires a discrimination criterion for the allowable deviation from coincidence. The criterion proposed by Brandon [13] is widely used, and allows a deviation of $15°/\Sigma^{1/2}$. This has been criticized as too restrictive by some researchers [11], and deviations of 5 - 10° have been allowed in some studies. An important consideration is the frequency of random occurrence of CSL's with a given criterion, and this has been discussed by a few authors for the case of the Brandon criterion.

In this paper we discuss microstructural development in a highly superplastic aluminum alloy. The properties and rheological behavior of this material have been reported previously [14,15]. An earlier study showed the change in several microstructural parameters as a function of processing and SPD [14]. Here we are primarily concerned with identification of the grain boundary types present during SPD, and how they differ from those which are due solely to recrystallization. From this information we hope to develop a better understanding of the microstructural dynamics of SPD.

Procedure

The material used for this study was produced from rapidly solidified powder which was consolidated, extruded and warm rolled. The details of the processing were reported earlier [14].

All of the processing subsequent to solidification was conducted at temperatures less than 425°C to minimize coarsening of the Al_3Zr phase. The composition of the alloy is shown in Table I.

Table I. Alloy Composition

Zn	Mg	Cu	Zr	Fe	O	Si	Al
7.00	2.44	1.91	0.42	0.09	0.09	0.05	Bal.

The extruded material was rolled in a room-temperature mill with intermediate anneals at 250°C. Details of the processing were reported previously [14]. Specimens were given a rolling reduction of 84% prior to heating to the superplastic test temperature of 470°C. Two samples were prepared for TEM study. The sample designated as annealed was subjected to the thermal portion of the test cycle, but was not strained. The superplastically deformed sample received identical treatment, with the addition of tensile elongation to a true strain $\epsilon = 1$.

Transmission electron microscopy samples were prepared by electropolishing in a 1:3 mixture of nitric acid and methanol at -30°C. Convergent beam Kikuchi patterns were obtained in microprobe mode in a Philips 400T microscope at 120 kV, with a probe size of 0.2 μm. Areas selected for misorientation studies were photographed at several different tilt angles to enhance grain contrast. The locations of grain boundaries were determined by comparison of these photographs and traced on a micrograph taken at zero degrees tilt. The region of interest was then re-examined, with the goniometer stage locked at zero degrees. Diffraction patterns were obtained from each of approximately 50 contiguous grains for each condition and catalogued accordingly. The patterns were digitally input to a PC, and analyzed with programs developed at The Ohio State University [16]. An additional subroutine developed at the Rockwell International Science Center [14] was used to determine deviation of misorientations from ideal CSL values. Using the catalogue of grain orientations, boundaries between adjacent grains were categorized as the nearest CSL within a maximum allowable deviation of six degrees

The frequencies of occurrence for CSL's when random processes determine grain-to-grain misorientation have been evaluated when the allowable deviation from exact coincidence is given by Brandon's criterion ($15°/\Sigma^{1/2}$)[13]. The use of a constant 6° criterion will result in different frequencies and these are summarized in Table II. Uncorrected frequencies are given in column five and these values were calculated using the relation [17,18]

$$F^{\Sigma} = \frac{\lambda_{\Sigma} N_c^2}{\pi N(g_{\Sigma})} (\omega_{\Sigma} - \sin \omega_{\Sigma})$$

where F^{Σ} is the frequency of occurrence for the misorientation represented by Σ, λ_{Σ} is a factor accounting for switching symmetry in the misorientation ($\lambda_{\Sigma} = 1$ for all Σ except $\Sigma 39b$ for which $\lambda_{\Sigma} = 2$), N_c, the order of the symmetry subgroup, is 24 for cubic crystals, and $N(g_{\Sigma})$ is the group multiplicity number [17,18]. The angle ω_{Σ} is the allowable deviation (= 6° or 0.105 rad) from exact coincidence. The deviation criterion defines a sphere centered on the exact CSL and a given misorientation may fall within the allowed deviation criterion for more than one CSL. Hence, these frequency values must be reduced to account for such overlap, and the corrected values are included in the sixth column of Table II. The occurrence of a CSL with higher than random frequency implies a distinct mechanism for formation of such a boundary.

Table II. Frequencies of Occurrence of CSLs (Σ=1-39) for 6° Allowed Deviation

Σ	λ_Σ	$N(g_\Sigma)$	$(\omega_\Sigma - \sin\omega_\Sigma)$	$F^\Sigma \times 100\%$ (6°-uncor.)	$F^\Sigma \times 100\%$ (cor. for overlap)
1	1	24	$1.9129 \cdot 10^{-4}$	0.0146	0.0146
3	"	6	"	0.5845	0.5654
5	"	4	"	0.8768	0.8055
7	"	3	"	1.1691	0.9228
9	"	2	"	1.7536	1.5768
11	"	2	"	1.7536	1.6512
13a	"	4	"	0.8768	0.4717
13b	"	3	"	1.1691	0.7091
15	"	1	"	3.5073	3.4087
17a	"	4	"	0.8768	0.6761
17b	"	2	"	1.7536	1.7536
19a	"	2	"	1.7536	1.1604
19b	"	3	"	1.1691	0.7893
21a	"	3	"	1.1691	0.5499
21b	"	1	"	3.5073	3.1969
23	"	1	"	3.5073	3.0787
25a	"	4	"	0.8768	0.5839
25b	"	1	"	6.5073	3.1057
27a	"	2	"	1.7536	1.2268
27b	"	1	"	3.5073	3.5069
29a	"	4	"	0.8768	0.8481
29b	"	1	"	3.5073	3.3197
31a	"	3	"	1.1691	0.8595

31b	"	1	"	3.5073	3.1787
33a	"	2	"	1.7536	1.5118
33b	"	1	"	3.5073	3.0433
33c	"	2	"	1.7536	1.6526
35a	"	1	"	3.5073	3.4066
35b	"	1	"	3.5073	2.9093
37a	"	4	"	0.8768	0.3921
37b	"	1	"	3.5073	3.4084
37c	"	3	"	1.1691	0.8147
39a	"	3	"	1.1691	0.7089
39b	2	1	"	7.0145	6.9051
Total:				71.9136	62.7128

Although such a broad method of associating boundaries with CSL misorientations may not be a rigorous means of determining the boundary structure, we contend that it does provide a simple method of associating boundaries with distinct formation mechanisms, such as those represented by the $\Sigma 7$, $\Sigma 3$ and $\Sigma 9$ boundaries which are attributable to recrystallization, twinning and twin chains, respectively. An increase in deviation in the superplastically deformed material would be expected subsequent to formation due to grain rotation, and the amount of deviation may be related to the amount of strain after nucleation of recrystallization.

Results

Table III lists the observed CSL's (within the 6° criterion) in each condition, as well as the amount of deviation ($\Delta\theta$) and f_R, the frequency vs. random (rounded to the nearest integer). Only CSL's with misorientation greater than 20° were considered for comparison, up to $\Sigma 39b$. The boundaries were designated by the grain numbers from the catalogue of diffraction patterns.

Table III. Grain Boundaries Near CSL's, $\Sigma \leq 39$, $\Delta\theta \leq 6°$

Σ	θ/ h k l	$\epsilon = 0$			$\epsilon = 1$		
		Boundary	$\Delta\theta$	f_R	Boundary	$\Delta\theta$	f_R
3	60.00°/1 1 1				40/41	1.0°	2
5	36.87°/1 0 0	17/18 17/47 33/42	5.7° 4.3° 3.6°	4			

7	38.21°/1 1 1				11/13 13/14 14/18	3.7° 5.8° 3.5°	3
9	38.94°/1 1 0				10/11 18/21	5.0° 3.9°	1
11	50.48°/1 1 0	15/16 15/18	6.0° 6.0°	1	39/41	2.6°	
13a	22.62°/1 0 0	36/37	5.6°	2			
13b	27.80°/1 1 1	15/22 18/49 37/39	2.2° 4.4° 5.4°	3			
15	48.19°/2 1 0				8/10 44/45	3.6° 3.2°	1
17a	28.07°/1 0 0	16/18	4.9°	2			
17b	61.93/°2 2 1	10/13	5.4°	1			
19a	26.53°/1 1 0				7/8 24/39	5.6° 4.7°	2
19b	46.83°/1 1 1	17/28 19/20	4.5° 3.2°	3	11/26 23/39	2.5° 4.0°	3
21a	21.79°/1 1 1	14/22	5.4°	2			
21b	44.40°/2 1 1	3/16 11/12 13/26 26/27 27/34	5.7° 4.5° 5.3° 5.1° 3.4°	2	35/36	4.0°	
23	40.45°/3 1 1	10/12 13/27	4.0° 6.0°	1	24/25	2.0°	
25b	51.68°/3 3 1	13/14	1.3°		27/31	3.9°	
27a	31.58°/1 1 0				43/45	5.6°	1
27b	35.42°/2 1 0	5/15 15/1	5.1° 4.3°	1	32/35	3.5°	
29b	46.39°/2 2 1	29/31 34/35	5.6° 4.6°	1	5/7 18/22 19/22 25/28 25/39	4.7° 4.8° 4.6° 2.3° 2.2°	2
31b	52.19°/2 1 1				9/10 25/26 26/27	4.8° 3.8° 3.9°	1
33a	20.05°/1 1 0	42/43	3.9°	1	14/15	5.8°	1
33b	33 55°/3 1 1	20/22 20/49 21/31 22/23 23/24 30/42	2.8° 5.9° 2.6° 2.3° 3.6° 5.8°	2	5/6 39/40	1.0° 3.1°	1
33c	58.98°/1 1 0				6/10	3.5°	

35a	34.04°/2 1 1	13/25	4.7°		20/21 21/22 21/45 26/29 34/45	6.0° 2.7° 5.3° 4.8° 4.3°	2
35b	43.23°/3 3 1	2/47 12/27 18/28 29/30 35/37 36/48	3.6° 5.8° 4.5° 4.1° 5.9° 5.9°	2	14/24 19/23 32/37	5.2° 5.2° 5.8°	
37b	43.13°/3 1 0				4/5	3.5°	
39b	50.13°/3 2 1				3/14 9/26 14/23 25/39 30/31 32/36 36/37	4.2° 4.2° 4.9° 2.2° 2.1° 2.7° 5.7°	1

Discussion

Examination of the data on near-CSL boundaries presented in Table III reveals a characteristic pattern of absences in the columns for both the annealed ($\epsilon = 0$) and the strained ($\epsilon = 1$) conditions. The CSLs observed with greater than random frequency in either one of these conditions tend to be missing in the other. Also, the absences in one column correspond to the presence of twin-related counterparts in the other column. Thus, Σ5, Σ13a, Σ13b, Σ21a and Σ21b are all present the annealed condition but not in strained material. In contrast, Σ3, Σ7, Σ9, Σ15 and Σ39 are all seen to present upon straining but are absent in the annealed condition.

The Σ7 reorientation has been associated with the early stages of recrystallization in Al but is evidently unstable and appears to be replaced by its twin-modified counterparts, i.e. Σ 21a and Σ21b, later in the process [11]. Both of these latter boundary types have frequencies of occurrence ~2 × random in the annealed material, which has experienced prolonged heating at the superplastic deformation temperature and therefore represents material in the later stages of recrystallization.

In the deformed material, the components that lead to the formation of Σ21a and Σ21b in the annealed condition, namely Σ7 and Σ3, evidently reappear. It is recognized that this assumes theΣ7 reorientations were present in annealed material during the early stages of annealing but were replaced in the course of annealing by their twin-modified counterparts, i.e. Σ21a and Σ21b. Furthermore, Σ5 in the annealed condition appears to be replaced by a twin-modified counterpart, Σ15, while Σ13 is replaced by Σ39. Thus, just as Σ7 is unstable during annealing to produce recrystallization [11] we find that Σ21, Σ5 and Σ13 appear to be unstable during superplastic deformation and are replaced by their twin-modified counterparts, Σ7, Σ15 and Σ39. The details of the mechanism(s) for these changes were not discerned in this work. It is possible that dissociation of boundaries into components of differing mobilities during straining could result in the formation of both twin reorientations (i.e. Σ3 and Σ9) and twin-modified orientations. Indeed, Σ3 boundaries are present in the deformed material with a frequency of occurrence of 2×random and Σ9 boundaries are also seen. Other possibilities may include shear transformation during superplastic deformation.

Conclusions

Twinning processes are active during superplastic deformation of this P/M alloy. The absence of Σ21 boundary misorientations, coupled with the re-emergence of Σ7 and the presence of Σ3 misorientations in superplastically deformed material, suggests that twinning processes are involved in microstructural evolution during elevated temperature straining of this alloy. Other CSLs, including Σ5, Σ13a and Σ13b, are also replaced by their twin-modified counterparts (Σ15 and Σ39). Such twinning processes in association with hot deformation alter the grain boundary types present in the material and this may facilitate superplastic deformation by slip-accommodated grain boundary sliding.

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