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Misorientation texture of post-recrystallized α-brass

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Studies of annealing textures in the past were mainly based on X-ray diffraction for macrotexture and electron diffraction for microtexture. However, the application of the electron back-scattering (EBS) technique now provides a powerful tool to obtain grain-boundary misorientation data (the grain misorientation texture or GMT) and study the change in grain-boundary structure during annealing. The change in the GMT of cold-rolled brass annealed at two different temperatures is reported. The proportion of CSL boundaries was found to increase with annealing temperature. The significance of this finding with regards to an understanding of the texture transition and the grain-growth phenomenon is discussed.

1. Introduction

Annealing is an important part of the processing of cold-worked engineering metals and alloys. The physical changes that occur during annealing are of great technological importance in the control of structures and hence properties. α-brass is well known for its ductility and its wide commercial usage in press-formed artefacts. Scientifically, α-brass represents a wide range of substitutional face-centred cubic low stacking fault energy alloys which gives the well-known alloy-type rolling texture at large rolling strain and the brass-type recrystallization texture upon annealing. Annealing of heavily rolled brass at low temperature produces the characteristic {2 3 6} ⟨3 8 5⟩ primary recrystallization texture [1]. Good drawability, however, has been shown to be associated with the development of the {1 1 0} ⟨1 1 2⟩ texture component at the expense of grain coarsening [2].

The origin of these annealing textures has been a subject of much debate in the literature [3]. Analysis of the texture changes in the past has relied heavily on various X-ray techniques which yield information about the statistical distribution of the grain orientations in a two-dimensional pole figure or three-dimensional crystallite orientation distribution function (CODF). In order to obtain the fullest possible picture of the interrelationships between the texture, microstructure and properties, it is necessary to collect data on a grain-specific basis, in addition to more conventional macrotexture measurements. Recently, a technique for determining a crystal orientation from its electron back-scattered (EBS) diffraction pattern in a scanning electron microscope has been developed. It is a powerful research technique for the collection of microtextural data from which the proportion and distribution of grain boundaries which possess special properties can be obtained [4–7]. In the present paper, the change in the grain misorientation texture (GMT) of α-brass during annealing from 400–600 °C is reported.

2. Experimental procedure

A commercial hot-rolled Cu–30 Zn brass plate 9 mm thick with initial grain size of 30 μm was cold rolled 90% in thickness on a 2 high mill of 127 mm roll diameter. The direction of the cold rolling was along the original hot band rolling direction and the rolled specimens were annealed in a salt bath for 1 h at various temperatures between 300 and 600 °C. Incomplete pole ordinary {1 1 1} pole figures were obtained by the Schultz method. The annealed grain sizes were determined from the longitudinal section of the specimens by a standard intercept method.

Specimens for the electron back-scattering experiment were prepared from the rolling plane sections which were mechanically polished and etched in ferric chloride solution to reveal the grain boundaries. Microtextural data were obtained through a computerized electron back-scattering system. To maximize the back-scattered signals, the specimens were tilted so that the electron beam made an angle of 20° with the specimen surface. The crystallographic orientation of each grain was determined by indexing the Kikuchi pattern and was automatically calculated and recorded. The program could then be used to calculate the misorientation axis/angle pair between consecutive numbered grains or between selected grains. All 24 crystallographically equivalent axis/angle pairs were calculated and the lowest axis/angle pair was outputted. A separate program was used to calculate the coincident site lattice (CSL) value (i.e. the reciprocal of density of common lattice sites) of neighbouring grains.

3. Results

The experimental {1 1 1} pole figures of the 90% cold-rolled brass annealed at 400, 500 and 600 °C are shown in Fig. 1. A moderately strong {2 3 6} ⟨3 8 5⟩ primary recrystallization texture was found at 400 °C (Fig. 1a). At 500 °C the primary recrystallization texture was found to be weakened, and the {1 1 0} ⟨1 0 0⟩ and {1 1 0} ⟨1 1 2⟩ texture components began to emerge (Fig. 1b). Upon annealing to 600 °C (Fig. 1c) the {1 1 0} ⟨1 1 2⟩ component further strengthened and became the strongest component.

Fig. 2 shows the change in grain size with temperature. Throughout the annealing an abundance of straight-sided annealing twins were observed. These twin boundaries were not counted in the grain-size measurement. The average increase in grain size obtained from 300–400 °C was small in the range 10–15 μm, but a rapid increase was observed from 400 °C upwards. These changes corresponded with the replacement of the primary recrystallization texture by the growth texture over the temperature range being studied.

The {1 1 1} pole figure determined from the electron back-scattering pattern (EBSP) of 100 random but contiguous recrystallized grains at two annealing temperatures (i.e. 400 and 600 °C) are plotted in Fig. 3. The pole figure thus determined is not symmetrical to the specimen axis as the symmetrical orientations are not shown. Despite the small number of orientations sampled compared with the averaged X-ray texture, some similarity could still be recognized. The clustering and strengthening of the {1 1 0} plane intensity was obvious as the annealing temperature was increased from 400 °C to 600 °C. Fig. 4 show the distributions of the misorientation axis of the lowest axis/angle pair solutions (i.e. the pair which gives the minimum angle of rotation) in the standard unit sterographic triangles. A difference exists between the two distributions at 400 and 600 °C. In general, there is a preference for misorientation axes around ⟨1 1 1⟩ and ⟨1 1 0⟩ poles. The results of annealing at 600 °C (Fig. 4b) show some clustering of poles around ⟨2 2 1⟩ and ⟨2 1 0⟩ positions which are almost absent at 400 °C (Fig. 4a). An analysis of the grain boundaries possessing coincident site lattice (CSL) has been made. Only those CSL values below $\Sigma = 35$ are included providing the ratio $V/V_m < 1$, where V is the volume of the superstructure cell and V_m is the volume of the lattice cell. Using these criteria, 23% of boundaries can be classified as CSLs for the 400 °C anneal rising to 35% for the 600 °C anneal. From a χ-square test there is a significant difference in the percentage of CSL boundaries in the two distributions and the probability of this finding being due to chance is less than 0.08. The types of CSL boundaries and the deviations

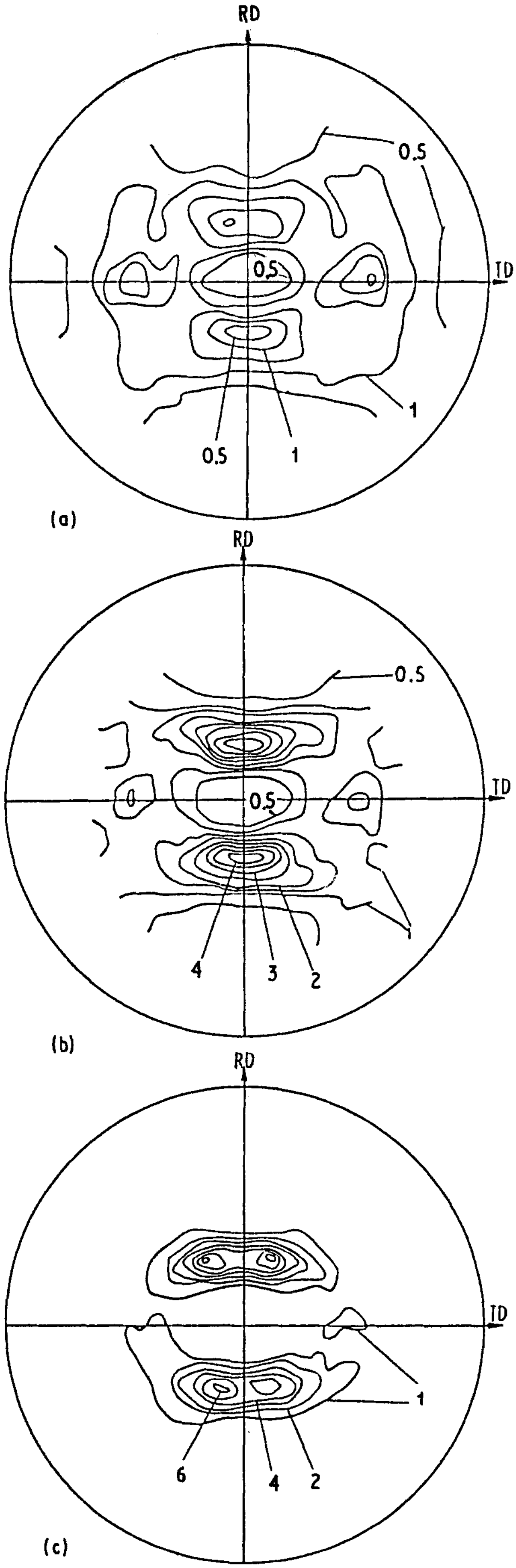

Figure 1 Experimental {1 1 1} pole figures of 70/30 brass cold rolled 90% and annealed for 1 h at (a) 400 °C, (b) 500 °C, and (c) 600 °C.

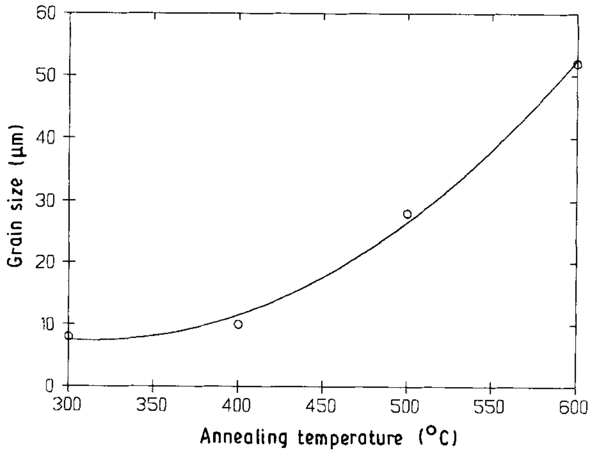

Figure 2. Grain size of the 90% cold-rolled 70/30 brass annealed at each temperature for 1 h.

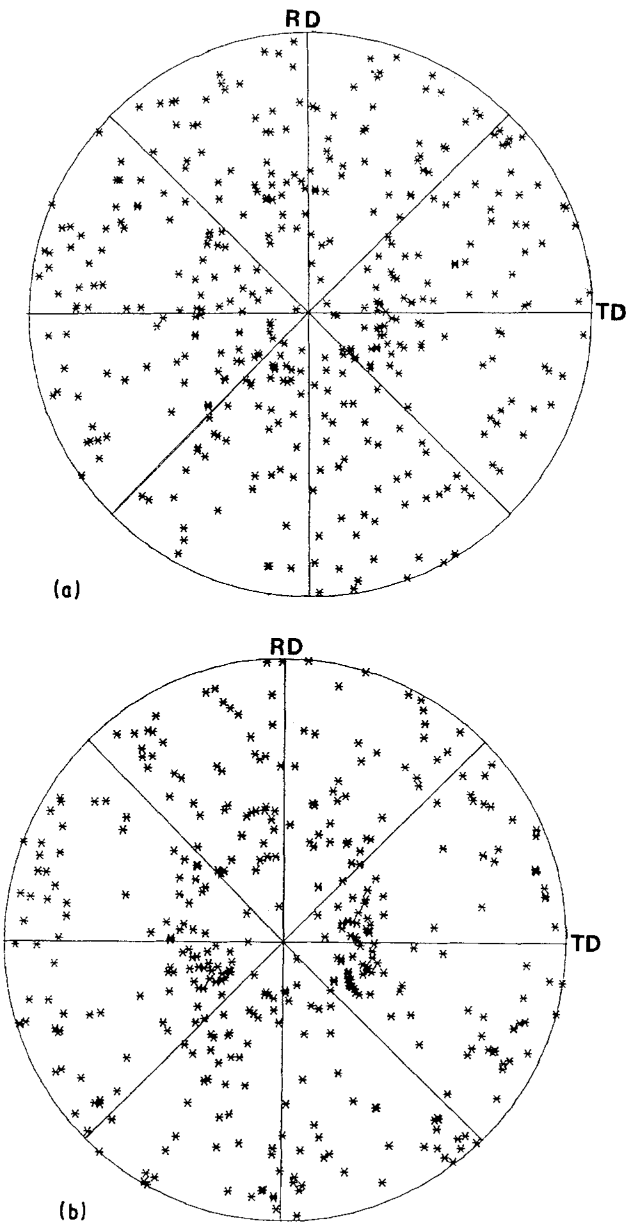

Figure 3 {1 1 1} pole figures of 100 random grains determined from the EBSP technique for a 70/30 brass cold rolled 90% and annealed for 1 h at (a) 400 °C, and (b) 600 °C.

from exact CSL matching (V/V_m ratio) are shown in Table I.

A mixture of large and small grains could be observed in brass samples annealed at 600 °C. The smaller grains were about one-third to one-quarter the size of the larger ones. The proportion of non-random boundaries between small/small (SS) and large/small (LS) grains in several grain assemblies (i.e. a large grain plus the small grains which surround it) has also been studied. Fig. 5 shows a scanning electron micrograph of a typical grain assembly of the brass annealed

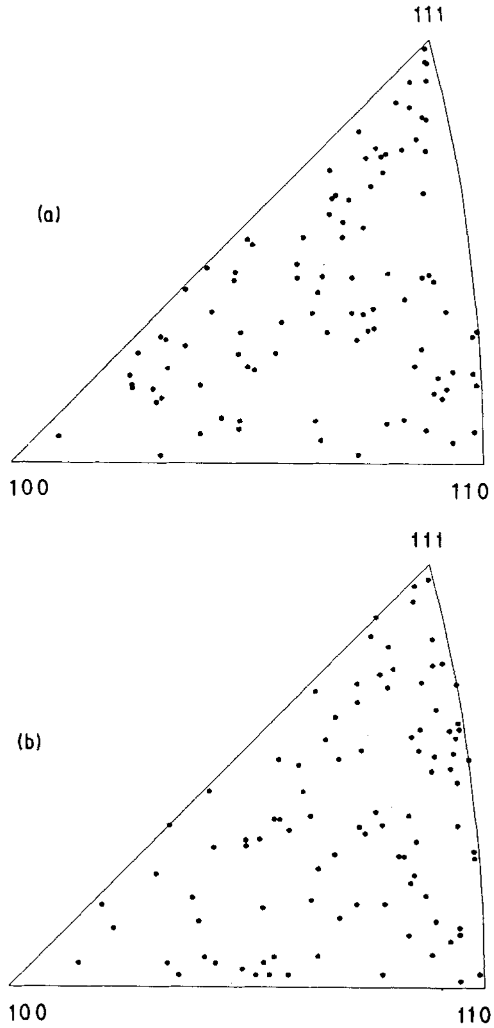

Figure 4 Grain misorientation texture (GMT) plots of the misorientation axes of the lowest axis/angle pair solutions of 100 random but contiguous grains in a standard unit sterographic triangle for the 70/30 brass annealed for 1 h (a) 400 °C, and (b) 600 °C.

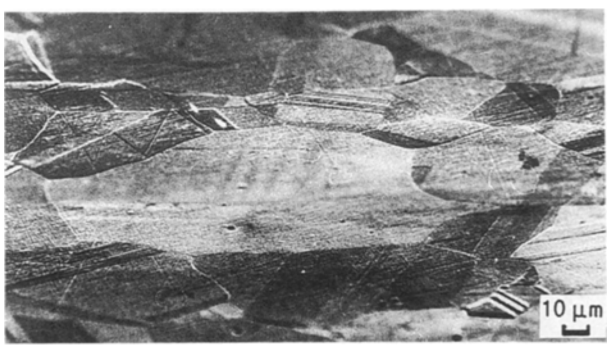

Figure 5 Scanning electron micrograph of a grain assembly (i.e. a large grain plus the surrounding small grains) of the 70/30 brass annealed at 600 °C. Of the 21 small/small grain boundaries 10 were found to be CSL boundaries while no CSL boundaries could be found between the large and the small grains.

TABLE I Distributions for the CSL boundaries for brass annealed at 400 and 600 °C

Temperature (°C)	Axis	Σ	V/V_m	Remarks	Axis	Σ	V/V_m	Remarks
400	111	3	0.04	a	984	17	0.82	
		3	0.36	a				
		3	0.74	a	110	19	0.22	
		3	0.04	a		9	0.95	b
		3	0.91	a				
		3	0.48	a	100	5	0.38	
		3	0.07	a				
		3	0.60	a	311	33	0.46	
		7	0.87	d				
		19	0.23		331	25	0.39	
		13	0.86					
		7	0.87		950	27	0.53	
						27	0.56	
	932	23	0.96					
		23	0.96		732	23	0.98	
600	111	3	0.73	a	110	9	0.94	b
		3	0.47	a		9	0.93	b
		3	0.51	a		9	0.73	b
		3	0.19	a		9	0.80	b
		3	0.54	a		9	0.60	b
		3	0.43	a		9	0.60	b
		3	0.70	a		11	0.88	
		7	0.54	d				
		7	0.77	d	210	15	0.92	c
					or			
		21	0.73		940	15	0.90	c
						27	0.20	c
	221	17	0.61			27	0.45	c
		17	0.96			27	0.49	c
		29	0.45					
					331	25	0.09	
	328	23	0.89					
					765	21	0.79	
	430	19	0.90					
					711	− 5	0.94	
	810	5	0.65					
					775	7	0.86	
	921	5	0.92			31	0.76	

[a] ⟨1 1 1⟩ 60° first order twin relationship.
[b] ⟨1 1 0⟩ 38.94° second order twin relationship.
[c] ⟨0 1 2⟩ 35.43° third order twin relationship.
[d] ⟨1 1 1⟩ 38.21° maximum growth rate orientation (MGRO) relationship in the literature [8, 9].

at 600 °C. The grains appeared distorted due to the tilting of the specimen in the microscope. The SS group of grains were all found to have a higher proportion of CSL boundaries than the LS group of grains.

4. Discussion

Examination of the grain misorientation texture (GMT) change of these α-brass samples shows a large proportion of the boundary types may be classified as CSL or near CSLs, and a breakdown of the CSL proportion indicates that there is an increasing preference for twin boundaries for samples annealed from 400–600 °C. In addition to the $\Sigma = 3$–60° ⟨1 1 1⟩ first order twin relation, boundaries of the type $\Sigma = 9$–38.94° ⟨1 1 0⟩ and $\Sigma = 15$–31.59° ⟨2 1 0⟩ are also abundant in the brass sample annealed at 600 °C. These Σ9 and Σ15 CSL boundaries correspond to the second and third order twin relationships to the twin origin. The {1 1 0} ⟨1 1 2⟩ growth texture (Fig. 1c) may be obtained as a second order twin (i.e. $\Sigma = 9$–38.94° ⟨1 1 0⟩) of the {1 1 0} ⟨0 0 1⟩ Goss component which is present in the primary recrystallization texture. The importance of twinning to the development of annealing textures has long been recognized [9–11]. However, the mechanism of twin formation is still a matter of dispute. Wilbrandt and Haasen [12] and Berger *et al.* [13] suggested that the new orientations are generated by a selection process favouring coincident orientations associated with a small grain-boundary energy. Stuitze and Gottestein [14] emphasized the importance of grain-boundary mobility. In the process of multiple twinning, the evolution of an annealing texture in low stacking fault energy materials is accounted for by the growth selection among the lower order twins having the maximum growth rate orientation (MGRO) with respect to the matrix grains (i.e. grains possessing a 30–40° ⟨1 1 1⟩ rotation relationship) [9]. An analysis of the misorientation of the lower order twins of the {1 1 0} ⟨0 0 1⟩ with the primary recrystallization texture {2 3 6} ⟨3 8 5⟩ (Fig. 3a) reveals that only the {1 1 0} ⟨7 4 4⟩ second order twin can be classified as a near CSL boundary of the type $\Sigma = 7$–$38.21°$ ⟨1 1 1⟩ with $V/V_m = 0.90$. Although the relationship between the Σ value and the grain-boundary energy is complex, it is generally accepted that CSLs with a fairly low Σ value have lower energies than random boundaries. The increase in the proportion of CSL boundaries with annealing temperature and hence the overall reduction in interfacial energy is an important factor during the growth stage. Based on the energy consideration alone, the {1 1 0} ⟨1 1 2⟩ second order twin should then be favoured compared with other twins during the growth process.

The kinetics of grain growth in α-brass have been studied by various workers [15, 16]. Different stages of grain growth with annealing time and temperature were observed. Brickenkamp and Luke [17] reported two stages of grain growth in 70/30 brass cold rolled 95% and isothermally annealed at 800 °C. The change in the annealing texture of the cold rolled brass from {2 3 6} ⟨3 8 5⟩ to {1 1 0} ⟨1 1 2⟩ was observed to accompany a more rapid increase in grain size. Similar changes in the grain-growth kinetics with rapid texture change during normal grain growth have also been observed in an Al–Mn alloy [18]. Such a variation was argued by Simpson *et al.* [19] to be caused by a change in the growth process and grain-boundary structure with increasing annealing temperature.

The grain-specific texture data obtained from the EBS technique provide useful topological information of the grain-growth process. From the study of several grain assemblies, the boundaries between small grains are more likely to be CSL boundaries than those between large and small grain. Anomalous grain growth has not been reported previously in α-brass. Any anomalous grain growth will stagnate because boundaries between two small grains have lower energy than those between the large and small grains. This observation is in agreement with the GMT results for grain boundaries in a Nimonic PE 16 Alloy [20]. The idea of a two-stage grain growth analogous to the relation between recovery and recrystallization has been discussed by Randle and Ralph [6]. The first stage of grain growth involves "boundary recovery and recrystallization" while the second stage involves an actual increase in grain size. The small increase in the recrystallized grain size of the brass annealed at from 300–400 °C may well represent the grain-growth incubation period required [21]. A full picture of macroscopic texture changes during post-recrystallization annealing needs to encompass not only a specific microtextural distribution of orientations but also their grain-boundary misorientation data (the grain misorientation texture).

5. Conclusion

The change in grain misorientation texture (GMT) of α-brass annealed at two different temperatures has been studied. There is an increase in the proportion of CSL boundaries with increasing annealing temperature. The number of twin boundaries (up to third order twins) accounts for a significant proportion of the CSL boundaries. The change in the grain size of the annealed brass is accompanied by a change in the annealing texture whose explanation may be based on a selective growth of the low-order twins which have special CSL boundaries with the primary recrystallization texture components.

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