

Rolling and Annealing of Fine Grained 70 : 30 Brass

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The rolling textures and microstructures developed in fine grained (2–5 μm diam.) 70:30 brass are different to those found in coarser grained material. The $\{111\}\langle uvw \rangle\gamma$ fibre usually found at medium reductions is not developed but the normal $\{110\}\langle 112 \rangle$ texture still emerges at higher reductions. The microstructures are related to these changes. Although twinning is a deformation mode at low reductions the volume of twins is never large and the twin alignment characteristic of normal brass at $\sim 70\%$ reduction does not occur. The pattern of shear band development is changed and large areas of the microstructure are featureless at high reductions. Hardness values show an unexpected rise between 60 and 90% reduction and this is attributed to a Stage IV regime of strain hardening. After annealing at 300 and 900°C the textures are typical of those found in coarser grained material.

KEY WORDS: 70:30 Brass, fine grained, rolling texture, annealing texture, microstructure.

INTRODUCTION

There has been much interest recently in fine grained materials because they possess an inherent high degree of formability and enhanced strength. Much of this work has been concerned with aluminium and its alloys and much of it has been aimed at producing superplastic behaviour (Sherby and Wadsworth, 1984).

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The nature of the deformation processes in these fine grained materials has been extensively studied in the high temperature, low strain rate regimes typical of superplasticity (Suery and Baudelet, 1982) but there is surprisingly little information about more normal conditions based on room temperature deformation at strain rates typical of industrial forming processes. Fine grained materials are usually prepared by thermomechanical treatments based on rapid heating of a two phase material to a high final recrystallization temperature (Wert, 1982). In such cases the deformation and subsequent annealing processes are complicated by the second phase particles and any initial study would be simplified if single phased material could be used. It has been shown recently that satisfactory grain refinement can be achieved in 70:30 brass by rolling to 80% reduction and then heating rapidly to 900°C for a very short time (<5 sec) (Malin and Yeung, 1988). The deformation processes in normal 70:30 brass have been studied intensively for many years by many methods and are well understood (Duggan *et al.*, 1978; Fargette and Whitwham, 1976). The most notable feature is the occurrence of mechanical twinning and this is reflected in the general microstructure, the nature of the inhomogeneities of deformation, the pattern of texture development and the subsequent annealing behaviour. The possibility of mechanical twinning in this material was first suggested by Vogel (1921) more than 50 years ago but its role in texture development was first appreciated by Wassermann (1963) who attributed the well known brass texture $\{110\}\langle 112 \rangle$ to a twinning process. In the intervening years many microstructural investigations (Hutchinson *et al.*, 1979) have shown that twinning is, indeed, a feature of the deformation of α -brass and other low SFE materials and although the scale of the twinning and the pattern of texture development are not quite those predicted by Wassermann his proposal was a major intuitive achievement. This paper which describes the properties, microstructure, texture and annealing behaviour of a rolled fine grained 70:30 brass is dedicated to his memory.

EXPERIMENTAL

A 2 mm thick sheet of fine grained 70:30 brass with grain diameter 2–5 μm was produced as outlined above and cold rolled to 88%

reduction. Specimens for examination were removed at intervals during rolling. Vickers hardness tests were made with a 200 g load; microstructural work was carried out on longitudinal sections and textures studies used normal midplane sections and 111 pole figures. In selected cases these were supplemented by ODF measurements.

A fully automated texture goniometer was used to prepare four incomplete pole figures ($\alpha_{\max} = 85^\circ$) from each such specimen. Three dimensional orientation distribution functions (ODF) were then calculated using the series expansion method of Bunge (1969) with spherical harmonic functions. These were corrected for ghost errors, truncation errors and symmetry effects and the results expressed in the form of a component analysis in which single Gauss-type components were fitted to the experimental peaks (Lucke *et al.*, 1981; Hirsch and Lucke, 1987). In this way the volume fractions (M_i), scatter widths (PSI) and true densities ($S(g)$) of selected components were established. Finally the observed densities in each of the significant orientation fibres of fcc rolled textures (β , α and τ) (Hirsch and Lucke, 1988) were determined.

Annealing was carried out in salt baths. The thickness of the specimen material was such that <1 sec was required to reach the bath temperature. Specimens were examined after annealing at 300 and 900°C.

RESULTS

A Rolled material

1. *Hardness.* Hardness values for the deformed fine grained brass are shown in Figure 1 together with values from a coarse grained specimen ($\sim 100 \mu\text{m}$ diam.). As expected the fine grained material hardened more rapidly and more extensively and the apparent saturation value was retained to higher strains. The most interesting feature, however, is the inflection in the hardening curve at $\sim 65\%$ reduction and the subsequent increase in strain hardening rate. This behaviour was not observed in the coarser grained material.

2. *Microstructure.* The limited resolution of optical microscopy does not permit detailed investigation of fine grained specimens and only multigrain features could be detected. Shear bands of this type

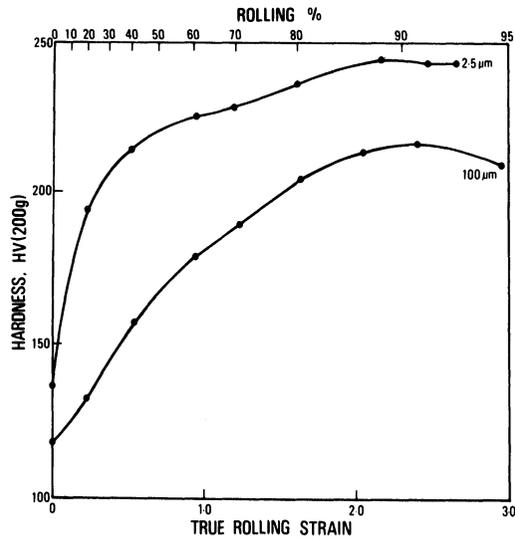


Figure 1 Hardness of cold rolled, fine grained brass.

were evident at 80% reduction. Both positive and negative bands were present but the bands were much finer than those normally observed and less frequent than usual (Figure 2).

Electron microscopy showed that twinning was a major deformation mode during rolling to 20% reduction (Figure 3). The twins

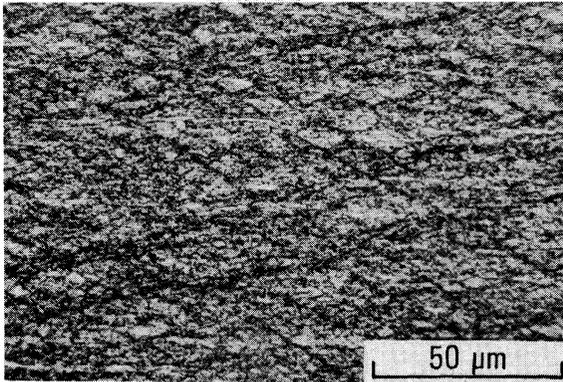


Figure 2 Longitudinal section of cold rolled (80%), fine grained brass.

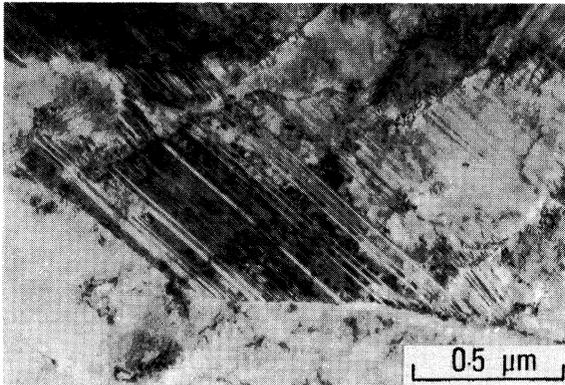


Figure 3 Longitudinal section of cold rolled (20%), fine grained brass.

were much finer ($<0.01 \mu\text{m}$ thick) than those normally seen and occurred more frequently. There was little evidence of local orientation changes arising from features such as deformation bands and the twins were usually continuous and straight right up to the grain boundaries. The diffraction patterns from even the most heavily twinned grains were very sharp and showed no evidence of orientation spreading. After 40% reduction the microstructure was much more confused. Twinned areas could still be detected and twin alignment with the rolling plane had begun but no clear structure was seen in many of the grains (Figure 4). This trend

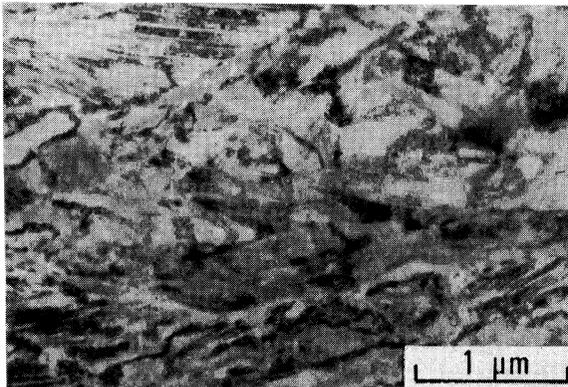


Figure 4 Longitudinal section of cold rolled (40%), fine grained brass.

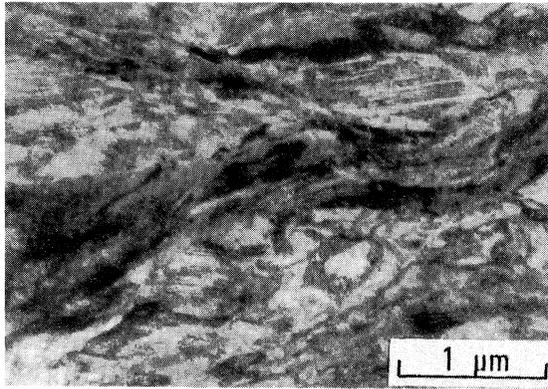


Figure 5 Longitudinal section of cold rolled (60%), fine grained brass.

which contrasts strongly with the pattern of microstructure development in normal specimens continued with further rolling. The first shear bands appeared after 60% reduction (Figure 5); the edges of these bands were less clearly defined than usual and the internal structure was more diffuse. The bands crossed many grains and were $<0.5 \mu\text{m}$ thick; the crystallite volumes within them were $\sim 0.15 \mu\text{m}$ thick. At this stage most of the structure apart from the twinned volumes was featureless and irresolvable. Diffraction patterns from such regions were essentially polycrystalline in nature although there was a tendency to favour $\langle 110 \rangle$ and $\langle 111 \rangle$ patterns in the TD sections examined. The number of shear bands increased with further rolling to 80 and 88% reduction. Occasional remnants of twinned areas were present but the structure otherwise was almost featureless (Figure 6). The shear bands were very wavy and an elongated internal structure could be seen in many of them.

3. Texture. Experimental 111 pole figures from specimens rolled to various reductions are shown in Figure 7 and the corresponding corrected ODF's in Figure 8. Selected details of the component analysis are given in Table 1. The skeleton lines for the β , α and τ fibres are shown in Figure 9. This very extensive texture data is summarized below. The notations used hereafter are those proposed by Hirsch and Lucke (1988).

Starting material. The fine grained starting material had a very

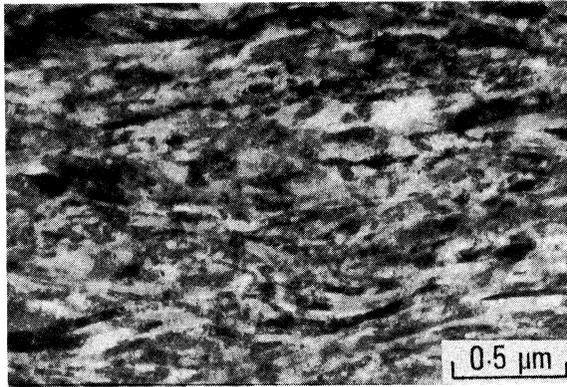


Figure 6 Longitudinal section of cold rolled (80%), fine grained brass.

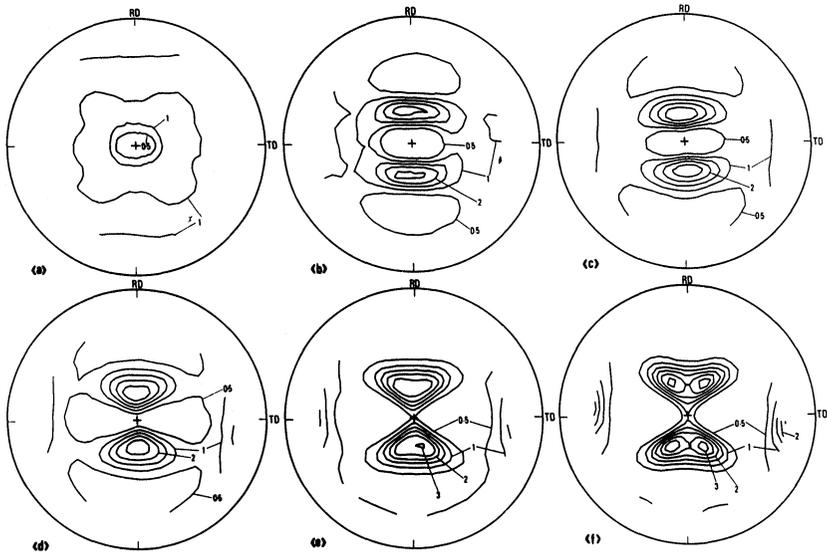


Figure 7 111 Pole figures of cold rolled, fine grained brass. (a) 0%, (b) 20%, (c) 40%, (d) 60%, (e) 80%, (f) 88% reduction.

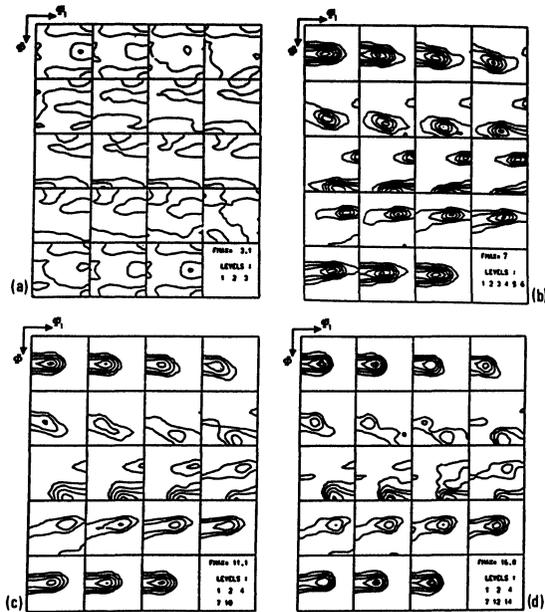


Figure 8 True orientation distribution functions of cold rolled, fine grained brass. (a) 0%, (b) 40%, (c) 80%, (d) 88% reduction.

Table 1 Volume fractions, M_i , of rolling texture components

Component*	Indices		Euler angles			M_i (%) after rolling				
	Plane	Direction	°	°	°	0	20	40	80	88
<i>G</i>	011	100	0	45	0		3.1	6.1	3.3	4.6
<i>B</i>	011	211	34	45	0	2.4	11.5	10.1	16.1	26.7
<i>B/G</i>			15-18	45	0	0.9	6.2	15.0	13.5	16.6
<i>C</i>	112	111	90	30	45		4.9		5.6	1.4
<i>T</i>	258	121	55	30	10	10.1				
<i>TC</i>	255	511	16-21	48-49	60-65				7.7	5.5
<i>S</i>	123	523	55-65	33-39	65		11.2	14.9	24.5	33.0
<i>W</i>	001	100	0	0	0	2.7				
W_{RD}	025	100	0	19	0	1.6				
<i>P</i>	011	111	55	45	0		4.6			
$\sim P$			45-72	45	0	4.9		4.9	4.2	
rot. <i>G</i>	011	011	90	45	0	1.3				
$\sim C$			81	30	40	3.4		6.7		
<i>C/S</i>			66	33	55	3.0				
<i>B/S</i>			50	44	80	3.2				
Background			45	45	45	66.2	58.2	42.4	24.8	12.4

* The symbols used are those proposed by Hirsch and Lucke (1988).

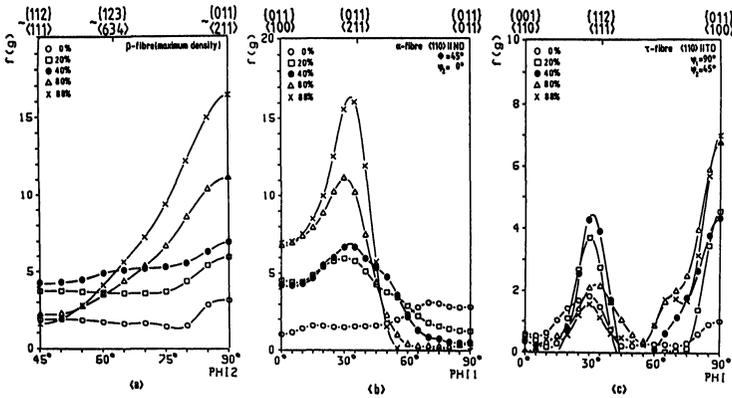


Figure 9 Orientation densities ($f(g)$) of orientations present in various fibres of cold rolled, fine grained brass. (a) β , (b) α , (c) τ fibres.

weak texture with minor scattered orientation concentrations at $\{110\}\langle 011\rangle$ (rot.G), near $\{258\}\langle 121\rangle$ (T), near $\{011\}\langle 388\rangle$ and near the cube orientation (W). The values of the orientation density, $f(g)$, in the β fibre were approximately constant over most of the fibre. The strongest individual component was $\{258\}\langle 121\rangle$ with an M_i value of 10.1% but most of the texture was made up of minor orientations which are represented in the Background value of $\sim 66\%$. This texture is typical of that found in 70:30 brass of more normal grain size after moderate rolling and annealing.

20% Reduction. The texture was characteristic of that normally found in fcc materials after small reductions. Orientation concentrations had begun to develop along both the α and β fibres and the major component of the texture $\{110\}\langle 211\rangle$ occurred at their intersection with an M_i value of 11.5%. The common fcc rolling texture component, S, at $\{123\}\langle 634\rangle$ was also prominent at 11.2% but Background orientations still accounted for $\sim 58\%$ of the texture.

40% Reduction. The texture was again typical of that found in coarser grained 70:30 brass. The minor orientation concentrations observed in the skeleton fibres after $\sim 20\%$ reduction had continued to develop and the strongest values occurred at S and near the B and G orientations. The Background intensity was reduced to $\sim 42\%$.

80% Reduction. The texture was generally typical of that found in brass at this level of rolling except that the normal strong γ fibre was completely absent and the $\{112\}\langle 111\rangle$ C component was still observed. Normally this component would disappear from the texture at ~ 70 – 75% reduction. The α and β skeleton lines show the normal increase of the B component (now $\sim 30\%$ with B/G) with major scattering along the β fibre towards the S component with $\sim 24\%$. The texture was now quite sharp and the contribution of random orientations was reduced to $\sim 25\%$.

88% Reduction. The texture continued to sharpen with further rolling and the M_i value for Background orientations was reduced to $\sim 12\%$. The strength of the β fibre orientations increased with S orientations now accounting for $\sim 33\%$ of the volume and B and B/G orientations $\sim 43\%$. Once again the γ fibre was completely absent.

B Annealed material

Details of the grain size developed after annealing at 300°C and 900°C are given in Table 2. In general nucleation occurred uniformly and the microstructural changes were similar to those described elsewhere.

Selected 111 pole figures from material annealed at 300°C and 900°C for various times and after various reductions are given in Figure 10 and ODF's that illustrate the various effects in Figure 11.

Table 2 Grain size of rolled and annealed fine grained brass

Reduction	Temperature	Time	Grain size	Remarks
40%	300°C	3 h	$\sim 5 \mu\text{m}$	somewhat variable
80	300	3 h	~ 5	uniform
88	300	3 h	~ 5	uniform
40	900	2 s	< 5	
		10	25	
80	900	2 s	10–15	
		10	~ 35	
88	900	2 s	15–20	
		5	~ 35	
		10	~ 60	

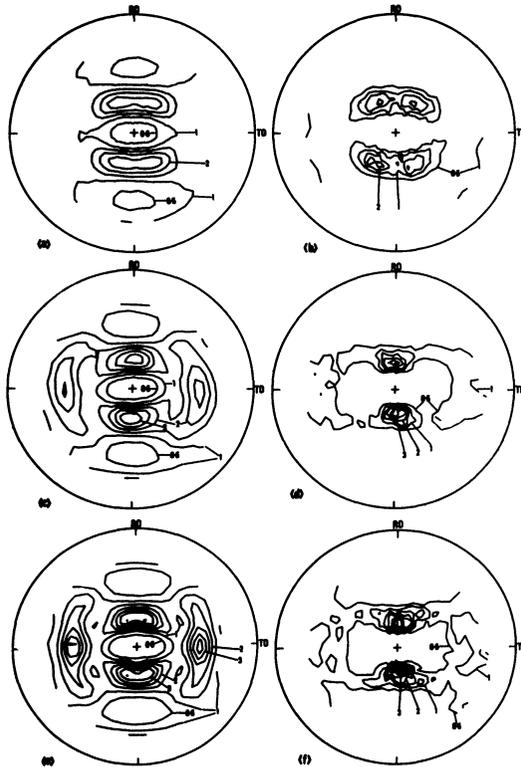


Figure 10 111 pole figures of annealed, fine grained brass. (a) 40% reduction, 300°C, 3 hours. (b) 40% reduction, 900°C, 10 sec. (c) 80% reduction, 300°C, 3 hours. (d) 80% reduction, 900°C, 10 sec. (e) 88% reduction, 300°C, 3 hours. (f) 88% reduction, 900°C, 5 sec.

Table 3 gives details of the texture component analysis. The significant aspects of the data are summarized here.

(i) *Annealing at 300°C*. The typical low strain weak texture observed in the specimen rolled 40% showed mainly α fibre orientations (G, B and $P, \{110\}\langle 111 \rangle$) with $\sim 55\%$ Background orientations. The textures developed in 80 and 88% rolled material were also rather weak with 55 and 38% Background material respectively. The most prominent component occurred near $\{4, 4, 11\}\langle 11, 11, 8 \rangle$ (D) but there was marked scattering towards S and also around the normal direction. The well known brass

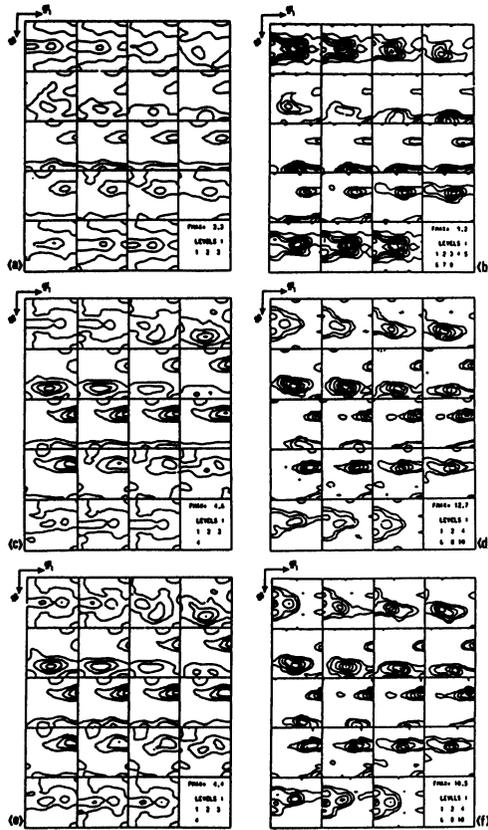


Figure 11 True orientation distribution functions of annealed fine grained brass. (a) 40% reduction, 300°C, 3 hours. (b) 40% reduction, 900°C, 10 sec. (c) 80% reduction, 300°C, 3 hours. (d) 80% reduction, 900°C, 10 sec. (e) 88% reduction, 300°C, 3 hours. (f) 88% reduction, 900°C, 5 sec.

recrystallization component, BR , at $\{236\}\langle 385\rangle$ was not observed but this is not unusual after only 88% reduction.

(ii) *Annealing at 900°C*. The textures of these specimens were very much sharper. After only 40% reduction the background value of M_i was reduced to $\sim 3\%$ and a stronger peak with $M_i = 35\%$ occurred near $\{110\}\langle 855\rangle$ which is only 5° from the major B component of the rolling texture. A second peak with $M_i = 21\%$ occurred in an RD rotated cube orientation (W_{RD}) at $\{058\}\langle 100\rangle$.

Table 3 Volume fraction, M_i , of texture components in annealed fine grained brass

Component*	40% reduction		80% reduction		88% reduction	
	300°C	900°C	300°C	900°C	300°C	900°C
<i>G</i>	1.6		2.1		2.5	
<i>B</i>	7.3		5.7			2.0
<i>B/G</i>	3.4	8.3	3.9	16.0		4.3
<i>C</i>	2.4			18.1		27.6
<i>S</i>	6.2	14.8	9.1	38.1		23.8
<i>W</i>	4.8		0.4		1.9	
W_{RD}	2.5	20.3		18.8	4.3	18.7
<i>P</i>	8.0				4.5	
$\sim P$			4.3			
rotated <i>G</i>	7.2					
$\sim C$		1.9				
<i>D</i>			6.8		14.9	
S_{ND}		35.2	11.9		15.7	13.9
$\sim B$		5.1			17.8	8.8
15°, 21°, 15°	0.5					
57°, 31°, 0°		12.4				
Background	56.0	2.0	55.5	9.1	38.3	0.7

* The symbols used are those proposed by Hirsch and Lucke (1988).

For material rolled to 80 and 88% reduction the textures were characteristic of copper type rolling textures rather than of annealed brass. No BR components were detected and the major features were strong peaks at the near the *C* and *S* orientations. The rotated cube orientation just described was also prominent. The most obvious feature of these high temperature textures is the apparent retention of rolling texture components after annealing. The components involved are different at 40% reduction, where α -fibre orientations are dominant to those found after higher reductions where β orientations emerge.

DISCUSSION

The pattern of microstructural and texture development in fine grained brass is different to that in larger grain sized material. At low levels of reduction deformation twinning is more profuse and the twins are very much finer than usual ($<0.01 \mu\text{m}$ rather than $0.02\text{--}0.05 \mu\text{m}$) (Duggan *et al.*, 1978). By 40% reduction, however,

the volume of twinned material is less than usual and much of the structure is featureless and irresolvable. The commonly observed rotation of the twins into alignment with the rolling plane does not occur. The shear bands are also finer than usual and much more wavy but they develop at the usual strain levels and with the usual frequency. These microstructural differences are only partly understood. Whether or not deformation occurs by slip or twinning or both is determined in part by orientation and the fine grained brass used here had an initial diffuse orientation concentration near $\{011\}\langle 011\rangle$ (see Figure 8a at 70–90°, 45°, 0°). The theoretical analysis of Chin *et al.*, (1969) has shown that in orientations near $\{011\}\langle 011\rangle$ twinning is the preferred mode and experiments with a low SFE, Fe–Co alloy showed that twin components were already present in the pole figure of a suitable crystal after only 10% reduction. The size of deformation twins is controlled principally by SFE, deformation temperature and strain rate but Baker and Peters (1967) have observed that the twin thickness in a fine grained ($\sim 1\ \mu\text{m}$) Cu-10Sn alloy was less than that of 30–50 μm material. Grain size effects have also been examined recently by Nourbakhsh and Vujic (1986) in plane strain experiments with 70:30 brass. Twinning was more prevalent at lower strains but twin sizes were not reported.

The featureless nature of most of the microstructure at low-medium reductions is not understood. Such volumes occur sparsely in larger grained material where they are usually associated with the orientation $\{011\}\langle 100\rangle$ (Duggan *et al.*, 1978). It might be expected that deformation of a fine grained material would result in an increased production of grain boundary inhomogeneities and so lead to structural confusion but in the 20% rolled specimens the grains were distinguished by a greater than usual uniformity and an apparent absence of both internal and grain boundary inhomogeneity. Despite this the major feature of the microstructure at reductions $>40\%$ is its featureless nature. The volume of twinned material decreases rather than increases and twin alignment does not occur. These effects are readily seen in the texture where the strong γ fibre of low SFE materials does not develop. Instead elements of the copper type rolling texture persist almost to 80% reduction. It must be concluded that twinning is not a major deformation mode in fine grained brass at medium strain levels (say

30–75% reduction) and that it is replaced by slip. Such a conclusion is incompatible with some aspects of current view of both microstructure and texture development. It is usually argued that shear bands develop because an aligned microstructure (deformation twins in low SFE materials) is incapable of further homogeneous deformation. In this work the first shear bands develop in a twin-free featureless microstructure. Except for their fineness and waviness they occur in the usual fashion and at the usual strain levels. Waviness in shear bands has been discussed only by Yeung and Duggan (1986) who pointed out that wavy bands required hardness differences in alternating lamellar regions of the structure. The creation of the necessary regions would require cooperative behaviour of at least several grains in the present material and no evidence for such behaviour is apparent in the microstructure.

Despite these very marked differences the brass texture finally emerges in the fine grained material between 80 and 85% reduction and in so doing presents difficulties with respect to current theories of rolling texture development in low SFE materials. In general these attempts to relate texture development with microstructure. In the relatively simple theory of Hutchinson *et al.* (1979) normal slip processes at low strains produce similar textures in all fcc metals but as twinning becomes more significant and as twin rotation occurs the γ fibre develops in low SFE materials. Further slip is precluded in this very restrictive structure and shear bands form so that the aligned twinned volumes are destroyed. Normal slip processes are then resumed in the very small structural units of the shear bands and these lead to the final brass texture. The recent and very detailed explanation from Hirsch and Lucke (1988) is based on similar concepts but is much more precise both crystallographically and quantitatively. In particular it is shown that the final post shear band stage of texture development begins with an accumulation of B and G orientations in the shear bands and that homogeneous slip in the fine crystallites re-establishes the β fibre with G orientations rotating to the stable B position. The difficulty with the present fine grained brass is the absence of an aligned structure and the development of the brass texture at a lower strain than usual. Here a suggestion of Hirsch and Lucke (1988) may be relevant. They point out that limited twinning occurs at higher reductions in low zinc brasses without the development of a major aligned, lamellar

structure and that slip therein would lead to the observed accumulation of G and hence B orientations. It is speculated that a similar effect might occur in copper at very high reductions ($>97\%$). At the reductions involved a major feature of the microstructures is their fineness. If very fine structural units are significant to the development of brass texture components in medium SFE materials it is possible that in 70:30 brass a small initial grain size would remove the necessity for twin alignment and lead to the observed development of the brass texture at lower than usual reductions.

Figures 10 and 11 show that reduction of the initial grain size to 2–5 μm does not drastically change the recrystallization texture of 70:30 brass. If annealing takes place at 300°C the major difference is the appearance of greater than usual concentrations of rolling texture orientations such as D . These orientations develop during homogeneous deformation and their carry over into the recrystallization texture confirms that microstructural inhomogeneity has been reduced in volume as shown above. This is further confirmed by the absence of bimodal grain size distributions in the annealed specimens. These distributions which are not unusual in annealed brasses have their origin in selective nucleation at inhomogeneities and associated differences in growth rate.

For material annealed at 900°C the results confirm recent work on very high temperature annealing (Yeung *et al.*, 1988) which has shown that retention of rolling texture components is a major feature of the texture. Once again the orientations involved are those associated with homogeneous deformation processes. Table 2 indicates that grain growth is rapid at 900°C and raises the possibility that the textures shown in Figures 10,11 and described in Table 3 contain significant growth contributions. Pole figures from specimens annealed for 2 and 4 sec have proved to be virtually identical to that in Figure 10 and, therefore, negate this possibility.

Although the hardness results of Figure 1 do not provide normal stress-strain data hardness can be related to an effective flow stress and it is apparent that the usual strain hardening behaviour of 70:30 brass has been changed in the fine grained material. The marked inflection at $\sim 65\%$ reduction is not typical of normal hardness data and the hardness remains saturated. Similar inflections to a higher strain hardening rate in 70:30 brass have been reported recently by

Blanchard *et al.* (1963) and Nourbakhsh and Vujic (1986) although the inflection occurred at considerably higher strains (4.5 and 2.2 respectively) and saturation did not occur. It seems clear that a Stage IV regime of strain hardening is being observed in each case even though the present work entailed a two stage straining technique (rolling + hardness testing). Such techniques do not normally detect Stage IV effects (see for example Cairns *et al.* (1971) or the 100 μm brass of Figure 1). Stage IV hardening has attracted considerable recent attention and particularly since the detailed review of Gil Sevillano *et al.* (1980). It is common to find an increasing flow stress even at strains of 8 or more (e.g. Nuttall and Nutting, 1978; Hughes *et al.*, 1986) and the effect is observed in both bcc and fcc metals and over a wide range of SFE. In some cases, however, and as reported here, the increase is limited and a later saturation or even a decrease is observed (Zehetbauer and Trattner, 1986). It has been argued that while the decreasing strain hardening rate during Stage III is due to recovery effects the linear strain rates typical of Stage IV are due to the creation of non-recoverable dislocation substructures. Zimmer *et al.* (1983) have suggested that this structure might be associated with the original grain boundaries of the material which is obviously relevant to the present work but both Hughes *et al.* (1986) with Ni-Co alloys and Nourbakhsh and Vujic (1986) with 70:30 brass have shown that grain size has no significant effect on Stage IV behaviour. In the former work the required substructure was believed to consist of bundles of elongated subgrains (or microbands) in medium SFE materials and bundles of twins in low SFE alloys. No specific conclusion about the structure was drawn in the latter work but it was reported that in normal (90 μm) brass the transition was associated with the appearance of sample sized shear bands. A similar correlation did not occur in a 4 μm grain sized brass. It seems, in summary, that a change to small grain size leads to an introduction of Stage IV behaviour even during a two stage testing programme and that this is unaccompanied by any significant change in structure other than the introduction of large volumes of featureless material.

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