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Effects of Grain Size on Rolling and Annealing Textures in 82%–92% Cold Rolled α-Brass

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Dedicated to the memory of Professor Günter Wassermann

It is shown that the initial grain size in α-brass affects rolling texture formation by the large scale arrangement of developing shear bands, which destroys {111} oriented material when the grain size is small. This is likely to be a general phenomenon in low SFE materials. On annealing at 300°C in the intermediate rolling reduction range (i.e. 82%–92%), the recrystallisation texture changes from {236}⟨385⟩ to {110}⟨110⟩ with increasing initial grain size. At 600°C the texture becomes {110}⟨112⟩ at grain sizes greater than 200 μm. Nucleation sites of {110}⟨110⟩ crystals have been characterised and conditions favouring the formation of {110}⟨112⟩ have been identified.

KEY WORDS: Brass, rolling texture, recrystallization texture, grain size, pole figure measurement, TEM investigation.

INTRODUCTION

Wassermann, among his many contributions which have helped shape our present understanding of textures, held that the "normal" texture formed during the rolling of fcc metals and alloys is that exhibited by copper and aluminium. The other fcc rolling texture, the so-called "alloy" texture developed in α-brass and silver, he

argued, arose because of the intervention of deformation twinning (Wassermann, 1963). At the time these views were heavily criticised but with the improvement in measurement techniques and in particular the application of electron microscopy to texture studies, the situation has been transformed. Combined X-ray and electron microscope analysis have conclusively demonstrated that α-brass froms a "copper-type" texture after intermediate deformation which changes to the "alloy-type" at high rolling strains. The mechanism by which the texture transformation occurs depends upon the coupled rotation of laminar twin/matrix elements formed early in the deformation, and shear banding. The classical $\{110\}\langle 112\rangle$ "alloy-type" texture arises from crystallite rotation in the shear band structure (Gil Sevillano *et al.*, 1977; Hutchinson *et al.*, 1979). Thus, although some of the details of Wassermann's theory of the formation of the $\{110\}\langle 112\rangle$ deformation texture found in low stacking fault energy metals and alloys are no longer deemed necessary nor correct, his essential insights into the nature of the texture transition have been vindicated.

The primary recrystallisation texture of α-brass has been mostly studied after heavy rolling reductions (i.e. >90%) and has been variously described as $\{113\}\langle 121\rangle$ (Schmidt and Boas, 1925; Wilson and Brick, 1945}, $\{225\}\langle 734\rangle$ (Beck and Hu, 1952), $\{326\}\langle 634\rangle$ (Lücke and Schmidt, 1972), or $\{236\}\langle 385\rangle$ (Schmidt and Lücke, 1975). In terms of CODF measurements, the primary recrystallisation texture comprises a complex orientation tube with a maximum intensity at $\{236\}\langle 385\rangle$. At low rolling strains (40–50% reduction), the recrystallization texture is a weak $\{110\}\langle 112\rangle$ retained rolling texture which has been exploited in the commercial production of non-earring brass (Schmidt and Lawley, 1972). The effect of moderate-to-heavy rolling reduction (60–90%) on the subsequent recrystallisation texture has been comparatively less studied. Wilson and Brick (1945), using X-ray film techniques reported a gradual perfection of $\{113\}\langle 112\rangle$ over this intermediate degree of rolling reduction. Gokyu *et al.* (1968), showed by inverse pole figures a moderate $\{110\}$ component below 90%. At higher reductions the $\{113\}$ component increased rapidly. The $\{110\}$ rolling component was assumed to be associated with the $\langle 112\rangle$ rolling direction.

On increasing the annealing temperature (i.e. to 600°C or

greater) the texture of α-brass is frequently reported as $\{110\}\langle 112\rangle$ (Wilson and Brick, 1945; Burghoff and Bohlon, 1942; Rosi and Alexander, 1950; Roberts, 1966; Horiuchi *et al.*, 1967; Baukloh *et al.*, 1970; Rellick and Lawley, 1975). Perovic and Karastozkovic (1980) described their pole figures obtained after 600°C annealing as $\{113\}\langle 332\rangle$. Recent work by Brickenkamp and Lücke (1981) who used CODF techniques described the main growth component of 70/30 brass rolled 95% and annealed at 800°C as $\{197\}\langle 211\rangle$.

There is less published work on the effect of high temperature annealing of moderately deformed α-brass. The texture of 68/32 brass rolled 85% and annealed at 650°C using early X-ray film techniques revealed a $\langle 110\rangle$//ND fibre texture which was described (Philips and Samans, 1942) as a combination of $\{011\}\langle 011\rangle$ and $\{011\}\langle 511\rangle$. $\{011\}\langle 011\rangle$ has been reported (Burghoff and Bohlon, 1942; Baukloh *et al.*, 1970) to occur at some intermediate stage of grain growth, which is replaced by the $\{110\}\langle 112\rangle$ components between 600°C and 800°C.

Comparatively little work has been done on the effects of initial grain size on rolling and annealing textures in α-brass. The early work of Wilson and Brick (1945) was quite comprehensive but the experimental techniques were relatively insensitive. Ozturk *et al.*, (1981) found only small effects when the grain size was changed from 20 μm to 200 μm. Duggan and Lee (1986) reported the formation of $\{110\}\langle 110\rangle$ in α-brass of grain size 3000 μm cold rolled 80% and annealed at 300°C.

This paper reports the effects and seeks to provide explanations for the role that initial grain size plays in the formation of rolling and annealing texture in α-brass, and is dedicated to the memory of Günter Wassermann, one of the giants of texture research.

EXPERIMENTAL

Commercial hot-rolled 70/30 α-brass plate of 9 mm thickness with an initial grain size of 30 μm was the starting material. Three other grain sizes 250 μm, 1000 μm and 3000 μm were obtained by annealing the hot-rolled plate at 800°C for various times. The specimens were cold rolled to reductions of 82% and 92% as this was the critical deformation range over which the $\{110\}\langle 112\rangle$

rolling texture starts to develop strongly. Rolling was done at room temperature on a 2 high mill of 127 mm roll diameter without end to end reversal between passes. The direction of cold rolling was along the original hot band rolling direction. Between 6 and 12 passes were used to achieve the deformation and after each pass the specimens were quenched to approximate isothermal deformation. Heat treatments were carried out in a salt bath within a temperature variation of 1°C.

During the course of the work it became apparent that higher zinc brasses might be important in determining what was happening and so this variable was also introduced. 65/35 brass was prepared to have initial grain sizes of 60 μm and 500 μm and processed in the ways outlined above.

Incomplete ordinary (111) pole figures were determined by the Schultz reflection method on a Siemens three circle goniometer with filtered Cu Kα radiation. 25 mm squares were cut from the brass sheet and 10% of the thickness was removed by etching in a 40 vol. pct., nitric acid solution. The random intensity level was measured from a compacted and sintered brass powder specimen. For specimens annealed at 600°C, the chart traces were spiky with an overall reduction in intensity due to extinction. This effect was removed by compression of 1%–2% in the thickness direction. Foils for transmission electron microscopy (TEM) were prepared by electropolishing in methanol—nitric acid (2:1 at −30°C and at 8V) and examined using a Philips EM 300 at 100 KV.

RESULTS AND DISCUSSION

The hot band textures were weak $\langle 110 \rangle$//ND fibre textures. Following cold rolling, textures were measured at the surface, quarter and half thickness levels to check through-thickness texture variations. These were small at 82% and after 92% cold reduction, any through-thickness variation in texture was found to be undetectable in all but the largest grain sized material. The primary cause of this effect in large grain sized material was that relatively few grains were involved in the measurements. An attempt to cope with this problem as it occurred in the 3000 μm grain sized brass is described below.

(a) Effect of initial grain size on deformation structure and texture

Figures 1 and 2 show the effects of grain size on rolling and annealing textures. The rolling reductions shown are 82% and 92% because it is in this range that grain size has most effect on the formation of $\{110\}\langle 112\rangle$ rolling textures and $\{110\}\langle hkl\rangle$ annealing textures. As the grain size increases to 1000 μm there is a significant increase in $\{111\}$ after 82% reduction, Figure 1a, d and g, while $\{110\}\langle 001\rangle$ is relatively unchanging. After 92% rolling of material in the 30 μm–1000 μm range $\{110\}\langle 112\rangle$ is developed and $\{111\}\langle hkl\rangle$ declines Figure 2, but the pattern of increasing $\{111\}$ with increasing grain size is maintained however, Figures 1 and 2.

The cold rolled texture of the 3000 μm grain sized material appears strange. This is due to the rather small number of crystals taking part in the texture measurement. In order to investigate this further a plate of large grain size brass 150 mm × 100 mm was carefully divided into 25 mm × 25 mm squares, labelled and sawn. Textures were determined for each piece and two clearly identifiable textures types were found. Type A texture, a very strong $\{111\}\langle hkl\rangle$ of strength 6 × random accounted for 67% of the area measured, and Type B, a single variant $\{110\}\langle 112\rangle$ of the kind shown in Figure 1j accounted for 20% of the area. The remaining 13% of the area had a strong texture of irrational indices close to $\{110\}\langle 112\rangle$. In all cases the 111 intensity was high, averaging 4–5 × random. Thus, as far as $\{111\}$ is concerned the 3000 μm grain sized brass fits the pattern shown in the smaller grain sized materials, i.e. as grain size increases the strength of $\{111\}$ increases.

The effect of grain size on rolling texture is remarkable even when the sampling problem is taken into account. The origin of the $\{111\}$ texture in rolled FCC, low SFE metals and alloys has been shown to reside in the coupled rotation of twin/matrix lamellae, formed earlier in the deformation, until the twin planes lie approximately parallel to the rolling plane (Hutchinson *et al.*, 1979). This lamellar structure is a necessary precursor to shear banding, (Duggan and Yeung, 1984; Morii *et al.*, 1985). The grain size effect can be explained using the observations of Yeung and Duggan (1986). In their work on flow localisation they noticed that a wavy or braided pattern of deformation was set up which was

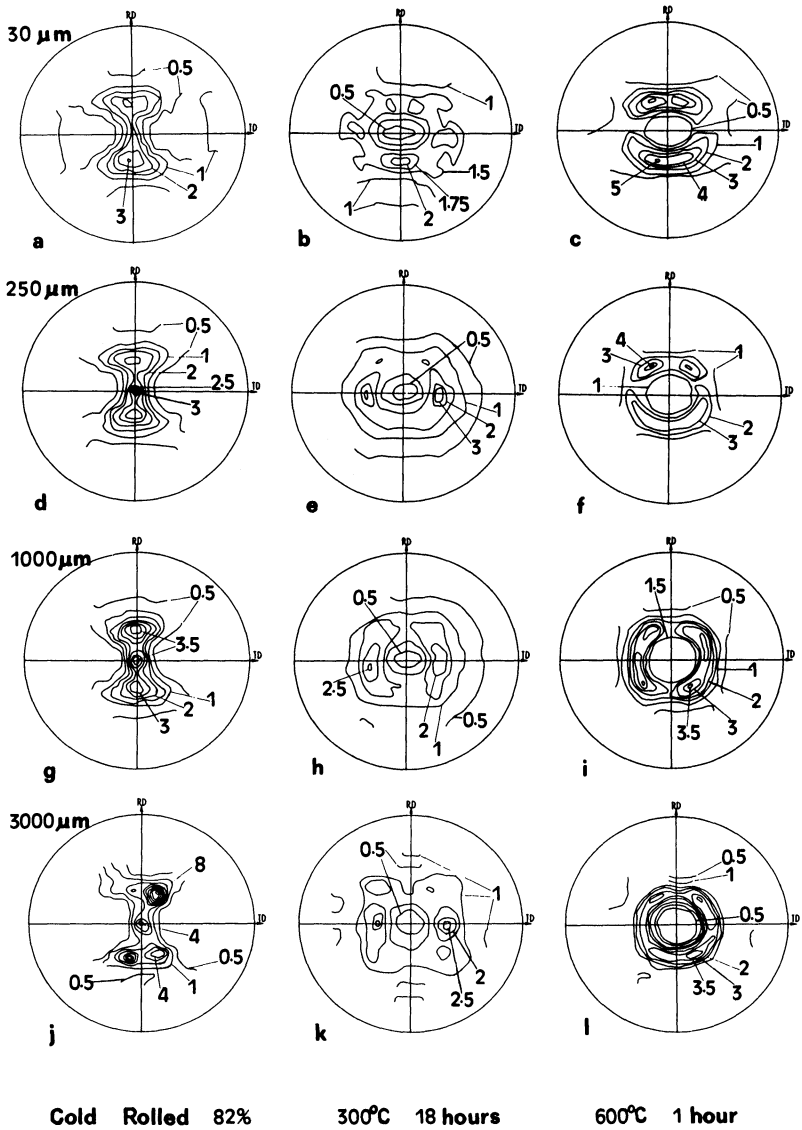


Figure 1 (111) pole figures of 70/30 brass of different grain sizes after cold rolling 82%, annealing at 300°C for 18 hours, or annealing at 600°C for 1 hour.

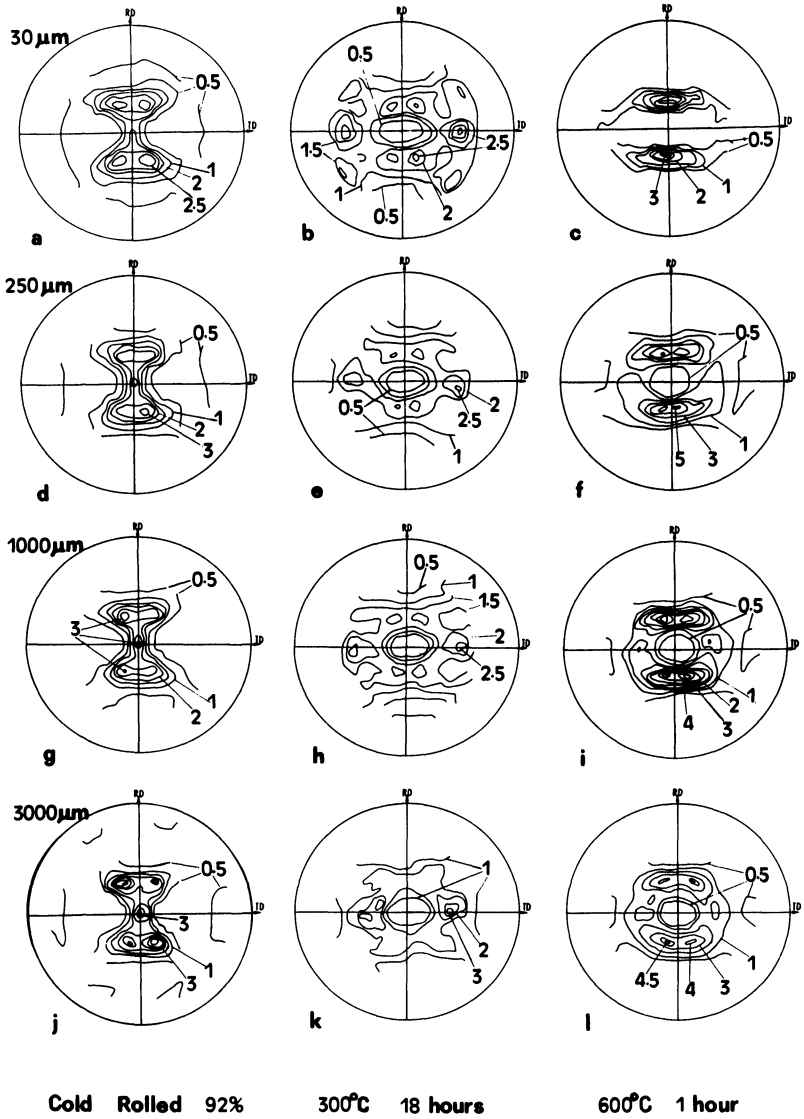


Figure 2 (111) pole figures of 70/30 brass of different grain sizes after cold rolling 92%, annealing at 300°C for 18 hours, or annealing at 600°C for 1 hour.

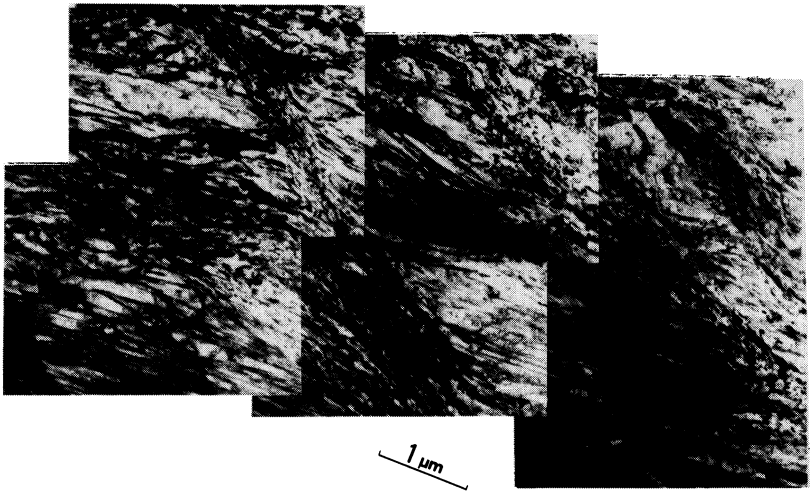


Figure 3 Electron micrograph from the longitudinal section showing wavy deformation structure in α-brass (65/35) of 60 μm initial grain size after 82% cold rolling. The scale marker is parallel to RD.

especially well developed when the grain size was small. A good example of this structure is shown in Figure 3 taken from the longitudinal section of 65/35 brass of grain size 60 μm after rolling 82%. The texture is identical to that shown in Figure 1a and the structure is of deformation twins bounded by a distinguishable envelope of material which, when it makes 35° to the rolling plane is identical to what is elsewhere described as shear bands. In larger grain sized brass however this wavy structure is rarely developed. Figure 4 shows a montage taken from 70/30 brass of 3000 μm grain size after rolling 82% and slight annealing. Comparison of such deformation microstructures led to the suggestion (Yeung and Duggan, 1986) that the wavy structure arises from the elongation and necking of individual grains when the grain size is small. This suggestion can be taken further. Comparison of Figures 3 and 4 with the textures show how 111 intensity is affected by grain size, for it is clear that the process responsible for producing the wavy structure is also responsible for reducing 111 intensity. The wavy structure subdivides the twin lamellae with material of different orientation, thus reducing {111}. Therefore it is clear that initial

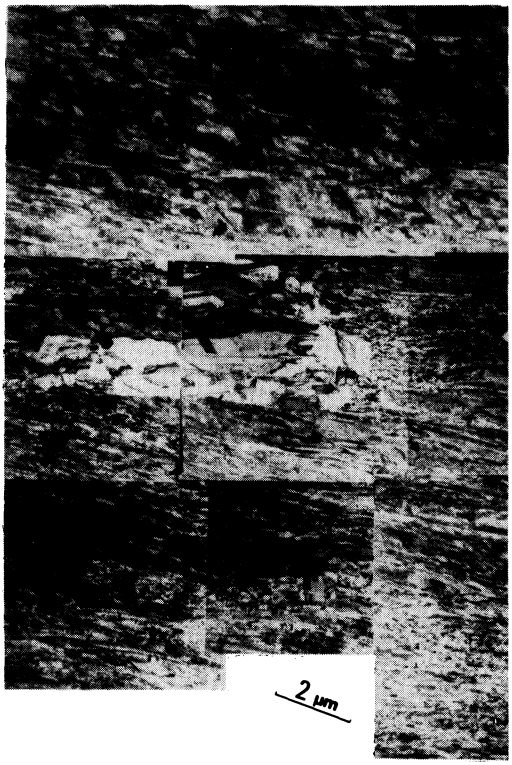


Figure 4 Electron micrograph from the longitudinal section of α-brass (70/30) of initial grain size 3000 μm after 82% cold rolling and annealing for 30 seconds at 300°C. The scale marker is parallel to RD.

grain size affects both structure and texture development in α-brass and in all such low SFE materials. After 97% cold rolling, the effect of initial grain size on deformation texture is not discernible in the (111) pole figures of the accuracy used here (Lee, 1986).

(b) General Comments on Annealing Textures

The classical high strain, low temperature primary recrystallisation texture described by the ideal orientation {236}⟨385⟩ begins to form in small grain sized brass after 82% reduction, Figure 1b and strengthens after 92% rolling, Figure 2b. Further rolling produced a

very strong {236}⟨385⟩ texture after 300°C annealing, which agrees with other workers. Grain size becomes less important at higher strains, Figure 2a, b & c all show evidence of {236}⟨385⟩ development. The mean grain sizes found at the end of primary recrystallisation were all less than 20 μm. The most important effect of initial grain size on annealed grain size was the degree of inhomogeneity found in the annealed grain size distributions. This was very large in 1000–3000 μm material.

At 600°C the 30 μm grain sized brass develops {110}⟨112⟩ after 82% cold rolling which rotates towards {113}⟨332⟩ at 92% reduction, Figure 1c and 2c. Larger grain sizes prevent the formation of {113}⟨332⟩ until rolling reduction reaches 95%, Figures 1 and 2 and Lee (1986). It is thus clear that only at the heaviest rolling strains, i.e. >95%, that initial grain size is relatively unimportant, and this fact must be borne in mind when other workers results are being considered.

(c) Origin of {110}⟨110⟩ Annealing Texture

Inspection of Figure 1 shows that {110}⟨110⟩ is formed in all grain sizes of 250 μm and above after 82% rolling and annealing at 300°C. It is only found in material of grain size 3000 μm after 92% reduction. In this latter material {110}⟨110⟩ forms from all deformation texture types, i.e. A and B and the irrational index texture.

The idea that short-time, high temperature annealing might be equivalent to annealing for longer times at lower temperatures was investigated. In all cases where {110}⟨112⟩ formed at 600°C, an earlier stage of annealing at 600°C showed {110}⟨110⟩ to be also present.

Texture control by using the variables of initial grain size, rolling strain, annealing temperature and time was investigated. By adjusting the annealing time at 600°C, in larger grain sized brass after 92% cold rolling, it was possible to produce a balanced mixture of {110}⟨110⟩ and {110}⟨112⟩, Figures 5b–d. It is thus clear that {110}⟨110⟩ is formed at both 300°C and at 600°C, while {110}⟨112⟩ is found only at higher temperatures. Doubling the time of annealing at 300°C did not produce any evidence of {110}⟨112⟩. It is also clear that the behaviour of 30 μm brass is quite different, the

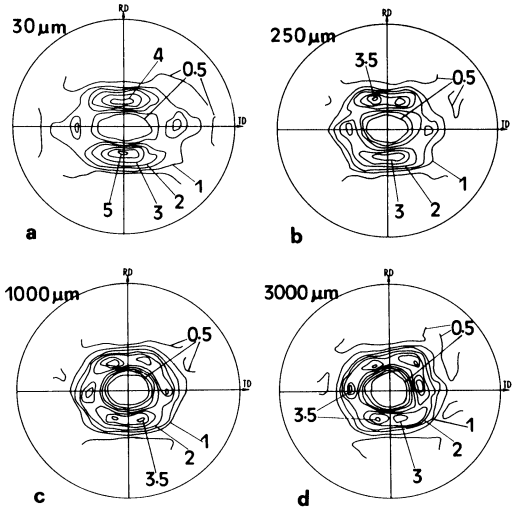


Figure 5 (111) pole figures of 70/30 brass of different initial grain sizes after 92% reduction and annealing at 600°C for 2 minutes.

{236}⟨385⟩ texture is beginning to form directly at 600°C, Figure 5a. This texture is dealt with elsewhere (Duggan and Lee, 1987).

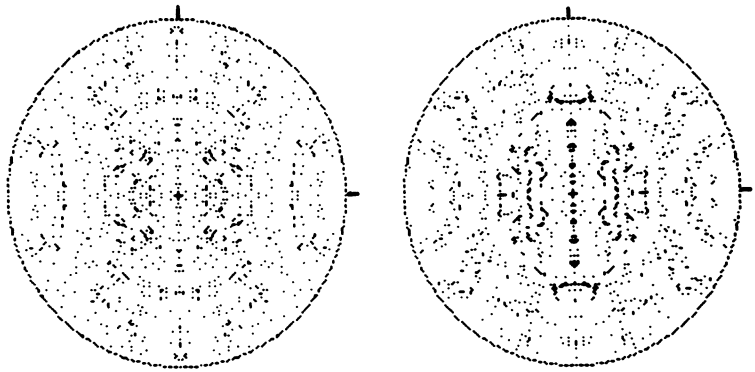
An exhaustive TEM investigation of the earliest stages of nucleation in materials which developed {110}⟨110⟩ was pursued with unexpected results. As expected, nucleation was copious in shear bands, but a new site was discoverred in brass of 3000 μm initial grain size. Long and relatively thick (~3 μm) vein-like structures varying in inclination from 10° to 0° with respect to the rolling direction were found. These produced infrequent but large recrystallized grains, Figure 4. Careful observation did not allow these vein-like structures to be distinguished from grain boundary regions which had precisely the same structure. Not all grain boundaries had such structures adjacent to them and because of this latter point, the structures will be called veins in this paper.

After 30 seconds annealing at 300°C vein nuclei were large (typically 3 μm wide by 10 μm long) and extensively twinned, Figure 6, compared with recrystallised grains formed in shear bands, Figure 7, which were of diameter 1–2 μm. Roughly one vein nucleated grain was found in each good foil, compared with ten

Figure 6 Electron micrograph from the longitudinal section showing a recrystallised grain of orientation {110}⟨110⟩ nucleated in a vein. The upper grain with equilateral twin traces has the orientation {114}⟨110⟩. Material is 70/30 brass of initial grain size 3000 μm cold rolled 82% and annealed at 300°C for 30 seconds, the scale marker is parallel to RD.

nucleated in shear bands. Selected Area Diffraction (SAD) techniques could be usefully employed with crystals of ~1 μm diameter or larger and this showed that almost all grains nucleated in veins belonged to the texture {110}⟨110⟩ plus various twins. An example is shown in Figure 6, and in addition the grain shown in Figure 4 is also close to {110}⟨110⟩. Shear band nucleated grains were substantially randomly oriented after 82% rolling reduction in both 30 μm and 3000 μm grain sized material, Figure 8. The relative infrequency of {110}⟨110⟩ oriented grains implies that the texture cannot be very strong unless these grains are relatively large.

Figure 7 Electron micrograph from the longitudinal section of 70/30 brass cold rolled 82% and annealed at 30 seconds of 300°C, showing small recrystallised grains formed in a shear band. Initial grain size 30 μm; scale marker parallel to RD.



(a) 3000 μm 30 seconds (b) 30 μm 1 minute

Figure 8 (111) pole figures derived from SAD measurements of 100 recrystallised grains formed in shear bands in α-brass after 82% rolling and annealing for 30 seconds at 300°C. (a) initial grain size 3000 μm (b) initial grain size 30 μm.

In summary, the evidence presented here supports the notion of a particular site, a special location within a vein, which forms viable nuclei of the orientation $\{110\}\langle 110\rangle$. The formation of these sites depends on both grain size and rolling strain, occurring in small grain sizes at low reductions (i.e. 30 μm and 65% rolling (Lee, 1986)) and persisting in large grain sized material after 92% reduction. The origin of these vein structures is not known but is probably related to grain-to-grain inhomogeneity strain discussed by Ozturk and Davies (1981). Crystallite rotation within the veins leads to occasional $\{110\}\langle 110\rangle$ oriented nuclei.

(d) Formation of $\{110\}\langle 112\rangle$ at 600°C

It has not been possible in this study to isolate the source of $\{110\}\langle 112\rangle$. However it is timely given the present stage of debate (Plege, 1986; Abbruzzese and Lucke, 1986) to present relevant observations made in this investigation. An important point is that the description $\{110\}\langle 112\rangle$ is not accurate for the pole figures shown in Figures 1, 2 and 5. In most cases $\{110\}\langle 114\rangle$ is at the centre of the spread, the limits of which are $\{110\}\langle 112\rangle$ and $\{110\}\langle 001\rangle$. Careful examination of published pole figures shows

the same large spread for this high temperature component. For ease of comparison the description {110}⟨112⟩ will be used throughout and be understood to include a spread unless otherwise specified.

In summary, the experimental facts are:

(i) {110}⟨112⟩ is formed only at 600°C. There is no combination of strain and grain size which produced this texture at lower temperatures. An experiment in which material cold rolled 92% and annealed at 400°C and 500°C for one hour failed to produce {110}⟨112⟩. At 400°C the texture was {236}⟨385⟩ and at 500°C a mixture of {110}⟨001⟩ + {236}⟨385⟩ was formed.

(ii) {110}⟨112⟩ is not formed directly from the deformed material by nucleation involving dislocation rearrangement, but is a growth texture. It can form from {236}⟨385⟩, Figures 2e, f and 5b and from {110}⟨110⟩, Figures 2k, l and 5d. This might be misleading as the spread along the TD when {236}⟨385⟩ was involved always included a reasonable level of {110}⟨110⟩. In other words {236}⟨385⟩ had to be weak and to include {110}⟨110⟩ in order to give rise to {110}⟨112⟩. Further evidence that {110}⟨112⟩ is a growth texture is furnished by an experiment in which the materials annealed at 300°C were reheated to 600°C. The 600°C textures were {110}⟨112⟩. This result was identical to that obtained by Horiuchi *et al.*, (1967), and they also insisted that {110} ⟨110⟩ was present alongside the more usual low temperature brass texture.

(iii) The orientation {110}⟨114⟩ is present in the primary recrystallisation texture. This is a twin of {110}⟨110⟩ and a good example is shown in Figure 6. In this micrograph the twin {110}⟨114⟩ oriented grain is bulging into the laminar deformation structure, and has formed a stepped interface. A simulation involving five crystals oriented within 5° of {110}⟨110⟩, when twinned gives the orientation spread shown in Figure 9. This simulation result shows patterns of pole density which are similar in orientation to the 600°C pole figures shown in Figures 1 and 2. It is tempting to think that such twins, which belong to the {110}⟨112⟩ spread, consume their parent matrices at high temperature, but this implies a temperature moderated boundary mobility in coherent interfaces, a process for which no model presently exists.

(iv) Figure 10 shows that increasing the zinc content of α-brass to

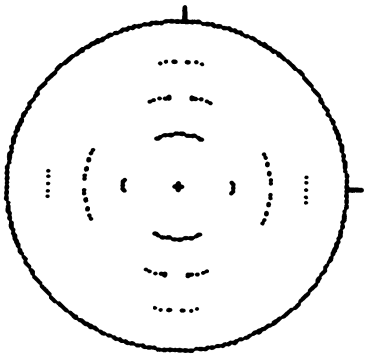


Figure 9 (111) pole figure of first order twins derived from five orientations within 5° of {110}⟨110⟩. The original five orientations are also included.

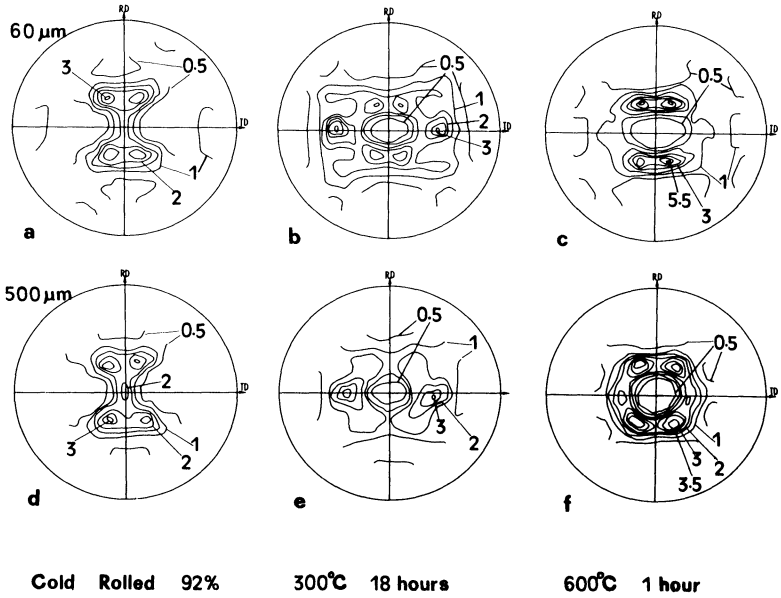


Figure 10 (111) pole figures of α-brass (65/35) of different initial grain sizes after cold rolling, annealing at 300°C for 18 hours or at 600°C for 1 hour.

35% significantly slows texture development. The grain size effects are identical to those described above but texture development is delayed. One hour at 600°C is equivalent to two minutes at this temperature in 70/30 brass, Figure 5.

(v) oriented growth theory is normally adduced to explain the development of {110}⟨112⟩ from the {236}⟨385⟩ primary recrystallisation texture, because both textures lie within the classical 30°–40° ⟨111⟩ orientation relationship. However, no such relationship exists between {110}⟨110⟩ and {110}⟨112⟩ and so this explanation is suspect.

CONCLUSIONS

In this work it has been demonstrated for brass that initial grain size affects both deformation and annealing textures. In particular it is shown that:

(i) As grain size decreases the level of {111} in the cold rolling range 80%–92% decreases. The reason for this lies in the development of a wavy shear band structure characteristic of small grain sizes. This structure has a different texture to the twin lamellae and so {111} is reduced.

(ii) The 300°C recrystallisation textures formed after rolling 82%–92% change from {236}⟨385⟩ when the initial grain size is 30 μm, to {110}⟨110⟩ as grain size increases. A particular type of structural defect is associated with the nucleation of {110}⟨110⟩.

(iii) Nuclei formed in shear bands of all grain sizes investigated after rolling 82%–92% are fairly randomly oriented.

(iv) The recrystallisation texture formed at 600°C is {110}⟨112⟩ when the grain size is greater than 200 μm. Increasing zinc content slows the rate of development of this texture.

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