

LA-UR-95-1885

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## Title:

THE DEVELOPMENT OF CUBE AND NON-CUBE RECRYSTALLIZATION TEXTURES

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## Submitted to:

For publication and oral presentation at 16th RIS0 International Symposium, Roskilde Denmark, September 4-8, 1995

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## THE DEVELOPMENT OF CUBE AND NON-CUBE RECRYSTALLIZATION TEXTURES

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### ABSTRACT

The development of recrystallization textures in cold rolled copper has been characterized using an electron backscatter pattern (EBSP) technique. Cube oriented grains exist in materials that have been annealed after a series of rolling strains, between 1.0 and 4.5 (von Mises strain). The strength and sharpness of these cube textures increases with increasing strain thus replacing the random texture produced by recrystallization of moderately deformed copper. The preferential formation of the cube texture is attributed to the homogenization of stored energy gradients adjacent to randomly oriented nucleation sites. This process, coupled with the development of the microstructure adjacent to deformed cube sites, favors nucleation and growth of cube grains over randomly oriented grains.

### 1. INTRODUCTION

The topic of recrystallization texture development has been controversial for years since the development of oriented nucleation and oriented growth models (Doherty, Gottstein, Hirsch, Hutchinson, Lücke, Nes and Wilbrandt 1988). The formation of preferred textures, such as cube textures in fcc metals, has been extensively investigated by X-ray, microstructural and calorimetry analyses. The lack of a conclusion as to the mechanisms that control cube preference, however, indicate that future investigations must approach the question of texture development from an even more microscopic level. Over the last several years new techniques for examining deformation and recrystallization textures have evolved, particularly the use of automated EBSP systems (Adams, Wright and Kunze 1993, Wright 1993). This system permits measurement of recrystallization textures as well as deformation textures (with some limitations) via a point by point collection of individual orientations. A particular advantage of this technique is that it produces local texture information from which local microstructural information may be deduced. This technique has been used extensively to study the development of deformation, recovery and recrystallization textures and microstructures in aluminum and its alloys (Skjervold 1993, Samajdar 1994). Measurements of partially recrystallized copper have showed the preferential development of cube textures at the earliest stages of recrystallization, even in materials that once fully recrystallized, exhibit random recrystallization textures (Necker 1992).

The use of automated systems provides information far in excess to X-ray analysis. Over several hours, texture data equivalent to X-ray data can be collected. However the analytical software allows for the evaluation of misorientations within grains, misorientations between grains, quantities of particular misorientations, spatial and size distribution of orientations and volume fraction analysis of orientations. The misorientation and orientation analyses can be coupled such that misorientations around a given orientation throughout the material can be determined, and visa versa. This technique lends itself to the characterization of recrystallization from the earliest stages through grain growth.

## 2. EXPERIMENTAL PROCEDURE

Oxygen free electronic (OFE) copper plate, 25.4 mm thick, with no impurity greater than 6 ppm except oxygen (10 ppm), was rolled to von Mises equivalent strains of 1.0, 1.5, 2.0, 2.6, 3.5 and 4.5 (a range of 58% to 98% thickness reduction). The initial grain size was 40-45  $\mu\text{m}$ . Material from each strain level was annealed in a silicone oil bath at temperatures chosen in order to produce a fully recrystallized structures over a one to two hour period. The annealing temperatures chosen were 100°C, 125°C, 150°C, 175°C, 200°C and 225°C, respectively. Sections of the rolled and recrystallized materials, approximately 10mm by 15 mm, were metallographically prepared at the half thickness rolling plane and along the long transverse section for Orientation Imaging Microscopy (OIM). This automated EBSP system consists of an ISI-200B scanning electron microscope (SEM), a low level light camera, a Custom Camera Designs, Ltd. camera control unit, a Hamamatsu image processing box and a Silicon Graphics work station to drive the SEM stage motors and collect the data. The stage is moved through a hexagonal grid system, the grid size being determined by the microstructure size. Typically a minimum of four to six measurements per average grain size are required. All deformed samples were measured using a 2  $\mu\text{m}$  step size. The electron backscatter pattern is captured by the low level light camera and the pattern image is processed to improve image quality before being fed into the computer. The software analyzes the pattern and calculates the orientation. This full cycle averages 1.1 seconds per pattern. The data analysis of the compiled orientations was performed on a MacIntosh Quadra 800 using software developed by S.I. Wright at Los Alamos National Laboratory. This software allows for the analysis of both local texture and microstructural information.

## 3. RESULTS AND DISCUSSION

Measurements of recrystallization textures of copper strained from 1.0 to 4.5 by X-ray reflection techniques shows a transition from a random texture ( $\epsilon=1.0, 1.5$ ) to a steadily strengthening cube texture ( $\epsilon=2.0, 2.6, 3.5$ ) to a texture virtually 100% cube when  $\epsilon>3.5$ . Cube is defined as all orientations with a 20° misorientation of  $\langle 100 \rangle \{001\}$ . The same transition is apparent through EBSP analysis. The texture of the material recrystallized after  $\epsilon=1.5$  is a random texture (Fig. 1a). The pole figures are produced by plotting each orientation as a dot, therefore regions of the pole figure that show a collection of very closely spaced points represent data from larger than average grains. Larger grains contain more measured points and since there is always a variation of orientation within a recrystallized grain, these collections of closely oriented points will correspond to the larger points on the pole figures. Cube texture represents approximately 10% of the texture in the copper recrystallized at  $\epsilon=2.0$  and approximately 38% after recrystallization of material at  $\epsilon=2.6$  (Fig. 1b). Notice that the only regions in Fig. 1b that show closely spaced points are in the cube and twin-of-cube positions. Away from the cube and twin-of-cube orientations, the pole figure contains evenly distributed points. Therefore the non-cube related texture is random and those random orientations are smaller than the average cube and twin orientations. Non-cube orientations become smaller and smaller with increasing prior strain. The orientation map of this material, Fig. 2, shows that the cube regions are the dominant features with all other orientations being smaller. By  $\epsilon=4.5$  the only feature apparent in the pole figures is the cube orientation.

## Development of recrystallization textures

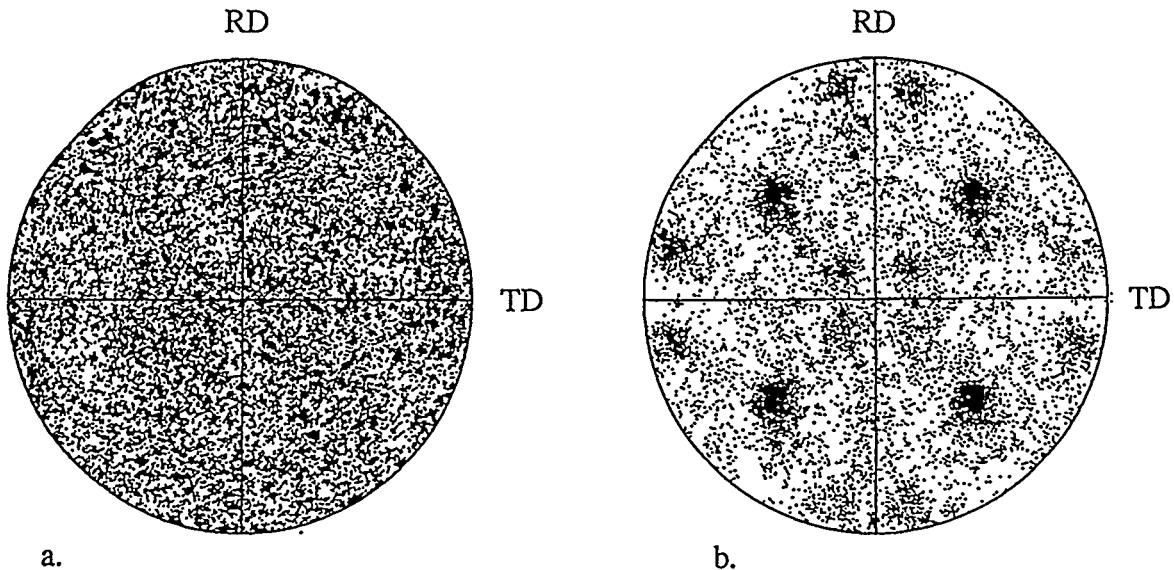


Fig. 1a - 1b. (111) recrystallization pole figures at a)  $\epsilon=1.5$  and b)  $\epsilon=2.6$

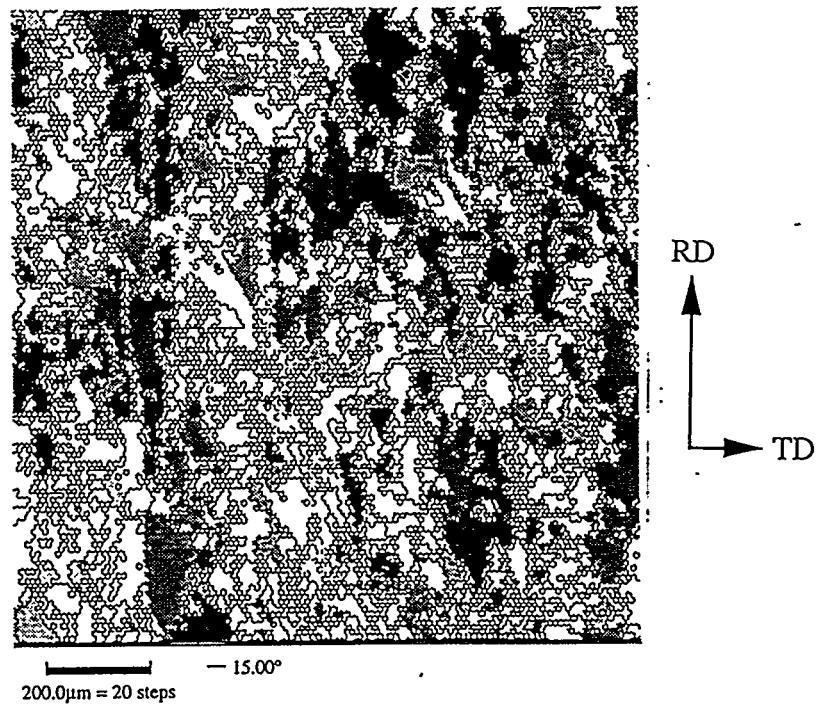


Fig. 2. Orientation map of copper recrystallized at  $\epsilon=2.6$ . Darker shading indicates cube orientations closer to ideal  $<100>\{001\}$ . Boundary line indicate all misorientations  $>15^\circ$ .

Even the twin orientations are evenly scattered indicating they are smaller. Quantitative analysis of the cube and directly related twin-of-cube orientations are in Fig. 3a and 3b. Each is plotted as a function of the misorientation of cube from the ideal  $<100>\{001\}$ .

There are three points that are vividly made in these two figures. The spread of the recrystallized cube orientations about the ideal  $<100>\{001\}$  decreases with increasing strain.

Material at  $\epsilon=4.5$  and recrystallized produces cube oriented grains at a volume fraction of 96% (within  $20^\circ$ ) and 60% of these orientations are within  $5^\circ$  of  $<100>\{001\}$ . This trend means that the preferred cube nucleation sites become more closely aligned with  $<100>\{001\}$ .

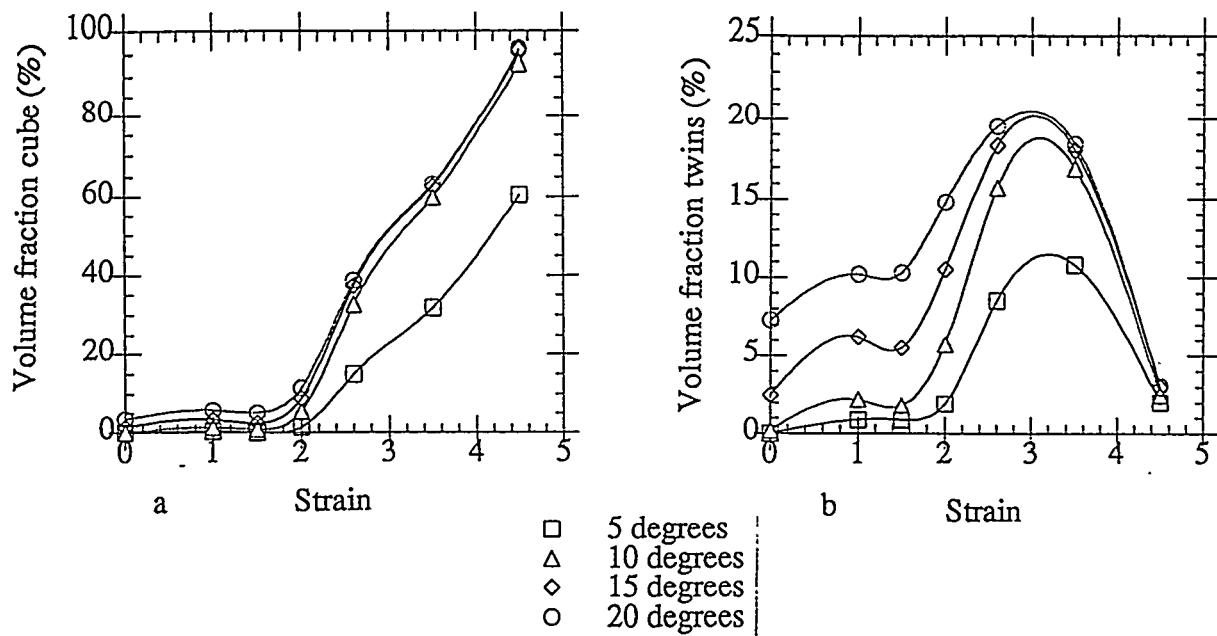


Fig. 3a and 3b. Volume fraction (%) a) cube orientation and b) twin-of-cube orientation as a function of prior strain and level of misorientation from the ideal  $<100>\{001\}$

This change in cube orientation preference could be explained by one of two processes: 1) the deformation process forms regions more closely aligned with  $<100>\{001\}$ , and/or 2) the microstructure around cube orientations develop to provide a more favorable environment for nucleation and growth. Secondly, coincident with the sharpening of the cube texture is the strengthening of the cube texture from 6% to 96% of the texture. Note also the marked decrease in twin-of-cube intensity at large rolling strains.

The development of sharp, strong cube textures can be analyzed from two viewpoints. Most experimentalists are primarily interested in the cube orientation perspective - why does the cube orientation dominate the texture? Although not pursued as strongly, the question of why all those other nucleation sites do not foster recrystallization is as important as the former question. For nucleation and growth to occur there must be regions that are relatively strain free with a sufficient local stored energy gradient to pull the a mobile boundary forward. Deformation provides the stored energy however the complicated networks of dislocations are not evenly distributed. This process yields the stored energy gradients. With increasing strain some regions become saturated with dislocations forcing other regions to accommodate the increasing strain. The stored energy becomes more evenly distributed. This process has a two-fold effect - there are fewer "relatively strain free" regions and the local stored energy gradients diminish. In the case of heavily cold rolled copper, the sites that still have a sufficient driving force for recrystallization are the cube sites. By  $\epsilon=4.5$  recrystallization is no longer a competitive process since 99% of the texture is either cube or twin-of-cube.

The third point, from Fig. 3b, is the dramatic shift in the volume of twins. The degree of annealing twinning is substantial at all levels of strain with the twin boundaries,  $60^\circ\text{-(111)}$  and  $38^\circ\text{-(110)}$  making up as much as 40% of all boundaries (boundary defined as  $>15^\circ$  misorientation). At the lower strains the volume of twin orientations directly related to cube grains is larger than the volume of cube oriented material. As the level of strain is increased the volume of twins related to cube increases, because the volume of cube increases. However the ratio of cube volume to twin volume decreases until the final texture is dominated by cube orientations and most twins are no more than 5-10  $\mu\text{m}$  in any dimension. There currently is no explanation for this trend.

Misorientation distributions are very similar for the materials strained 1.0-2.6 (Fig. 4), exhibiting peaks at  $60^\circ\text{-(111)}$  and  $38^\circ\text{-(110)}$  surrounded by an otherwise random distribution.

Materials strained to 3.5 and 4.5 show the same peaks for the twin orientations however the quantity of randomly distributed misorientations diminishes because no other orientations exist except cube and twin-of-cube. Misorientation analysis between cube-neighbor orientation pairs and non-cube-neighbor orientation pairs shows that there is approximately equal amounts of twinning for cube and non-cube orientations however the actual amounts are difficult to quantify because of the very extensive twinning throughout the material. The high density of twins also precludes reliable grain size analysis.

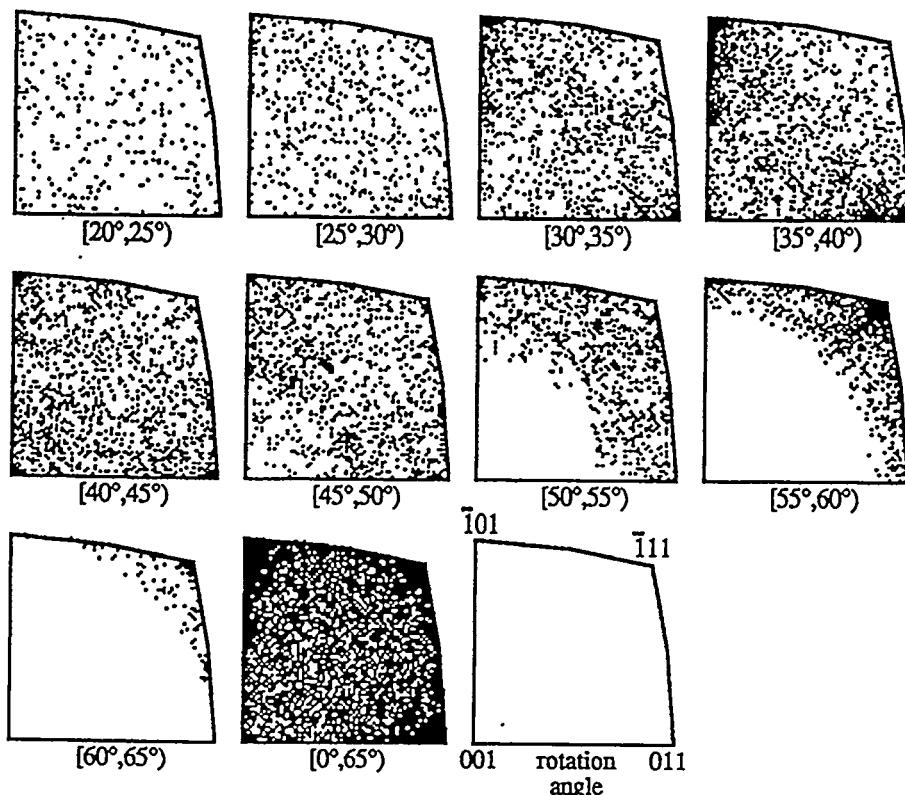


Fig. 4. Misorientation distribution of all misorientations  $>20^\circ$  in material at  $\epsilon=2.0$ , fully recrystallized.

Some very surprising results have been derived from the EBSP work on deformed material. The patterns of highest image quality come from regions with the highest levels of lattice order. If the electron beam strikes a grain boundary the pattern image quality is poor and if the beam falls on highly strained material the pattern most often can not be recognized. The latter finding would appear to allow one to differentiate between deformed and recrystallized regions by the quality of the image. This distinction is a function of the strain level and the initial size of the grains. Approximately 55% of the grid points produced identifiable patterns in the material at  $\epsilon=1.0$ . These orientations produce the classic eye-glass shape texture in a (111) pole figure. Subsequent deformation brings prior grain boundaries into closer proximity and strains the lattice significantly. So by  $\epsilon=4.5$  one would assume that it would be all but impossible to find a identifiable pattern in deformed material. However, the material at  $\epsilon=4.5$  did produce some identifiable patterns which formed the deformation texture. The orientation map shows a number of single points ( $2 \mu\text{m}$ ) but also several regions where orientations were measured over a  $4\text{-}6 \mu\text{m}$  area. These regions are too large to be remnants of the rolling microstructure therefore these regions must have been produced by recovery. This result can be explained by the processing technique. The material had been rolled from a  $25.4 \text{ mm}$  plate to a  $500 \mu\text{m}$  sheet. Most of the work done to roll the copper is not stored but heats the copper, to the point where the material could not be handled comfortably with bare hands. Note that the most heavily

rolled material recrystallizes at 100°C so it certainly could have recovered. There are a few points that are within 10-20° of cube and they are single or paired points. Recovery appears to occur throughout the material, not only in cube regions and there are certainly other orientations with a size advantage over the cube regions. Additionally, several of these larger regions have already undergone twinning. But these orientations do not grow because they are not found in the fully recrystallized state. The reason these small regions may not be found by EBSP in the fully recrystallized samples is that the grid size used to measure the recrystallized material is 10  $\mu\text{m}$ , making it unlikely that the electron beam would fall directly on these non-cube related regions.

#### 4. CONCLUSIONS

The development of strong and sharp cube recrystallization textures is attributed to the homogenization of stored energy gradients with increasing strain. This process changes the microstructural environment around previously active nuclei making them less active or inactive. At  $\varepsilon=1.0$  there are deformed cube related regions with the capacity to nucleate and grow. These regions are typically not very closely aligned with  $\langle 100 \rangle\{001\}$ . At this strain there may be deformed cube regions with orientations more closely aligned to  $\langle 100 \rangle\{001\}$  but do not have the local microstructural environment for nucleation and growth. Deformed cube regions with orientations more closely aligned to  $\langle 100 \rangle\{001\}$  either form by increasing strain and/or develop the microstructural environment allowing them to be active nuclei.

Successive rolling passes without cool-down time between passes can lead to dynamic recovery of relatively pure copper. Recovery occurs within deformation texture components as well as in cube oriented regions.

Analysis of recrystallization texture development by EBSP offers a significant improvement over other characterization techniques, yielding correlatable texture and microstructure information.

#### ACKNOWLEDGMENTS

The authors would like to thank S.I. Wright for developing and constantly upgrading the EBSP software, the basis of our analytical research.

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