Joint investigation of the local material rotation and lattice spin in a cube {100} <001> oriented single crystal

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Abstract. Cube {100} <001> oriented single crystals of Al 1% Mn were compressed in channel-die. Their lateral faces were covered with transferable carbon grids with a step of 100µm. At a deformation of about 0.3, the vertical bars of the grids show undulations whose characteristic length is of the order of the millimetre and which become sharper and smaller as the deformation proceeds. Fiducial golden grids with a step of 20µm remain largely unaffected. This shows that the investigated heterogeneity is typical of the mesoscopic scale and has no directly related patterns at the macroscopic and microscopic level. Microfocussed X-rays were used to measure the crystallographic rotations during the process. The investigated spot was a few 0.1 mm². At a deformation of 0.6, the lateral faces of the crystal undergo a split into two Cube orientations each rotated of about 15° around the transverse axis. This is put in relation with the undulations of the bars. At 0.9 an additional rotation around the longitudinal axis appears. The local material rotation and the lattice spin at the mesoscopic scale are interpreted in accordance with previous analyses of the evolution of the Cube texture based on EBSD and the observation of the traces of slip systems.

1. Introduction

Among all the crystallographic orientations, Cube displays a particular behaviour when submitted to plane strains [1]. When compressed in channel-die, four slips systems of a Cube single crystal are equally stressed but locally, only two of them are simultaneously active. So the crystal splits up into deformation bands parallel to the vertical lateral faces, those in contact with the walls of the die. The width of these bands ranges typically from 50 to 200µm [2] that is, for metals, the mesoscopic scale.

Now, the results above were obtained by observing the traces of the slip systems, electro-litho-depositing fiducial golden grids with a step of 5 µm in a scanning electron microscope and performing EBSD analyses. Other techniques have appeared recently. One of them is the transferable carbon grids made of a polyester film covered by a photographic emulsion on which a laser beam impresses a grid. These grids are applied on to the test piece which has beforehand been covered with a film of glue and deform with it. The step can be 30 µm or wider. Another is the microfocus X-ray texture analysis, in which crystallographic information is gathered from an elliptic spot which ranges from a few tenths to a few hundreds of µm, according to the device, while classical X-rays work on square millimetres. This is documented in various papers, among them [3]. Table 1 sums up, for each scale, the means of investigation of the material rotation of the test piece and the spin of the crystal lattice. The aim of the present paper is to find if, by using carbon grids or microfocussed X-rays, the previous observations can be confirmed.
Table 1. Means of microstructural investigation relevant to each scale.

| Scale     | Material rotation            | Lattice spin         |
|-----------|------------------------------|----------------------|
| microscopic | Fiducial golden grids         | EBSD                 |
| mesoscopic | Transferable carbon grids     | Microfocussed X-rays |
| macroscopic | Grids drawn on the test piece | Classical X-rays     |

2. Experiments

Cube single crystals of Al 1%w Mn were cut into test pieces 10.3 mm high and 8 mm long (unchanging width: 7 mm). Their lateral faces were covered by transferable carbon grids forming initially squares of 100µm, the bars being around 35µm thick. They were compressed in channel-die at room temperature by steps of ε = 0.15. At each step the test pieces were released from the die, the Teflon™ wrapping which acts as a lubricant was changed and pictures of the grids were taken with a binocular magnifier, the magnification varying from X = 3 to X = 28.5. Some of these pictures can be seen in figure 1. The axes have been numbered 1 (elongation), 2 (transverse) and 3 (compression).

Figure 1. Carbon grids on the lateral faces of a test piece at various deformations. 
ε: logarithmic deformation. X: magnification.
At $\varepsilon = 0.30$, no shear or other heterogeneity can be spotted except on the edges of the test-piece, but the vertical bars show slight undulations whose characteristic length is of the order of the millimetre. At this stage, the strain hardening changes, becomes linear and the abovementioned deformation bands appear. On the lateral faces, at further steps of deformation, the horizontal rungs remain pretty much horizontal and get thinner and thinner. The bars widen and form sharper patterns whose characteristic length lessens as the height of the sample decreases. Such is the case at $\varepsilon = 0.60$; see figure 2, b). The phenomenon accentuates at $\varepsilon = 0.90$, where it takes in places the aspect of chevrons such as those of insert d). The shears they reveal affect several bars at a time and remain widely horizontal. At larger deformations, the grid deteriorates because of the friction.

Is this an essentially mesoscopic phenomenon? To answer this question, fiducial golden grids with 20 $\mu$m steps were deposited and similar compressions performed. The results can be seen on figure 2. On stretches of a few hundreds $\mu$m, the golden bars remain straight. Figure 1 a), for example, pictures part of one of the abovementioned undulations. Hence, these are typically mesoscopic. Conversely, the fiducial grids reveal heterogeneities of their own, spotted only at the microscopic scale: traces of slip systems, whose inclination with respect to axis 1 lies around 30°; coarse slip bands which form valleys in the initially polished surfaces, as on insert b); a greater activity of the slip systems with the growing deformation on insert c). Occasional shear bands can also be found [4].

![Figure 2](image)

**Figure 2.** Golden grids on the lateral faces of a test piece at various deformations.

Classical X-ray measurements on the lateral faces show that, after $\varepsilon = 0.30$, there is a split in the initial Cube orientation [4]. It rotates clockwise and anticlockwise around axis 2 and the spread gets wider with the deformation. What is the corresponding spatial organisation of the deformation? Microfocuss X-ray texture analysis with 100 $\mu$m foci was used in an attempt to investigate it.

Such an appliance is easily mounted on a classical rig, but the measurements take time: typically five hours for one point. In the case of the present work, Copper was used as the anode material and the X-rays scanned an ellipsoidal spot whose axes were about 250 $\mu$m horizontally and 150 $\mu$m vertically. Six points were chosen on the face at $\varepsilon = 0.60$; four grouped in half a square millimetre at the centre of the sample and two apart. The intensity was collected in a layer less than 30 $\mu$m deep under the surface of the test piece, hence, in only one vertical deformation band. The results throughout the face are remarkably uniform, especially at the centre of the test piece. Figure 3, a) shows the split already known through classical measurements. It is $\pm 15^\circ$ around the ideal Cube position. The poles are sharp and the scatter around each of them is of the order of 6°. Measurements were also done for $\varepsilon = 0.90$; see figure 3, b). The results are basically the same but an additional rotation around axis 1 is detected. All this shows a repartition between patches rotating clockwise and others counter clockwise, the pattern changing with a characteristic length of about 100 $\mu$m, at least in one direction. At larger deformations, the crystallography becomes confused.
3. Discussion

According to the analysis presented in [1], only one pair of slip systems is active locally. The systems work with equal slip rates and provoke a plastic spin $\omega$ in the axes of the crystal such that $\omega_{32} \neq 0$, $\omega_1 = \omega_2 = 0$. Now, the material rotation rate $\Omega$ and the lattice spin $r$ are linked by $\Omega = r + \omega$ [5]. This implies $\Omega_{13} = r_{13}$. The material rotation $\Omega_{13}$ is given by half the slant of the grids. It is determined by the boundary conditions applied to the test piece. The lattice rotation $r_{13}$ can be measured on the pole figures. At $\varepsilon = 0.60$, it is worthy of note that both are of the order of 15° and that the traces of the slip systems, initially inclined 45° with respect to axis 1, have moved towards 30°, that is, a rotation of 15° due to the tilt of lattice. The observations are much the same at $\varepsilon = 0.90$.

This can be interpreted as follows. The test piece is constrained horizontally but has vertical free faces. It evolves as a staking of horizontal layers with $\Omega_{13} = r_{13}$ of opposite signs. Their thickness follows the progress of the compression. The microfocussed X-rays investigate a spot which covers typically two sides of a chevron, hence the splits on figure 3. This scheme is compatible with the activity of either of the pairs of slip systems. No conclusion can be drawn on whether the parting into horizontal layers corresponds to a change in the pairs of active slip systems, but the continuity of the traces of the slip systems suggests that the same pair which is acting on the whole face.

References

[1] Wert JA, Liu Q and Hansen N 1995 Acta Mater. 43 4153-63.
[3] Basson F and Driver J 2000 Acta Mater. 48 2101-15.
[3] Darrieulat M, Zhani K, Fillit RY and Chenaoui A 2009 J. Eng. Mat. Tech. 131 1-1 1-9.
[4] Poussardin JY 2003 PhD dissertation, Ecole des Mines de Saint-Etienne (France).
[5] Havner KS 1992 Finite Plastic Deformation of Crystalline Solids (Cambridge University Press) 34-38.