X-ray diffraction extinction in strongly textured Ag, studied through the comparison of different order of reflections

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Integrated intensity ratios of second- to first-order X-ray reflections were measured from a strongly textured pure silver sample, oriented at the maximum pole density, using several wavelengths. This was done to determine whether extinction exists in strongly textured polycrystals, as suggested by pole figure measurements, where pole density maxima of the second-order reflection often exceeds those of the first-order reflection. The integrated intensities of the reflections were normalized using the corresponding integrated intensities from a powder sample. The dominant texture of the silver sample was [110]$< 011 >$, and the ratios were measured for the 111 and 222 reflection pairs.

All resulting ratios were larger than 1, indicating the presence of extinction, which affects first-order reflections more significantly than second-order reflections. Considering the larger number of possible reflections of a polycrystal compared to the few number of reflections of a single crystal, double diffraction between different grains is proposed here as the cause of the observed effect, similar to the well-known secondary extinction in single crystals.

To investigate whether a texture gradient could influence the results, EBSD observations were conducted on the sample. A heterogeneous texture was revealed at the edges, but these heterogeneities were not found to affect the results.

Keywords: X-ray diffraction extinction; polycrystals.

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1. Introduction

When comparing pole figures measured by X-ray or neutron diffraction for different orders of reflection from strongly textured polycrystals, the pole density maxima of the higher-order reflections often exceed those of the first-order reflections [1-3]. For example, from pole figures of the silver samples of this study measured in our laboratory, pole density maximum was 6.1 for pole figure 111, and 9.2 for pole figure 222, both measured by K_α Mo radiation, and a similar sample, annealed in oxygen atmosphere, exhibited maxima of 11.9 and 14.6 for pole figures 111 and 222 respectively, when measured using K_α Cu radiation. This has been interpreted as an indication of extinction, similar to that observed in single crystals. However, pole figures require several corrections such as defocusing, absorption, interpolation and normalization [4,5], and error propagation could be significant, so that the possibility exists that these corrections could lead to the observed differences. To minimize the need for corrections, a more direct procedure was devised, namely, to compare the first- and second-order theta/two theta reflections at pole density maxima, normalized against the corresponding reflections from a powder sample. Here, the only necessary correction is for defocusing, when the tilting angle of the maximum requires it, which is done by the normalization using the silver powder. The aim of this study is to apply this new procedure. Additionally, since different reflections and wavelengths are expected to have varying penetration depths, which could also contribute to the observed differences, a possible influence of a texture gradient near the surface was tested through a comparison with the microstructure observed by EBSD.

2. Sample preparation and measurements

A high purity silver sample, annealed at 600°C for four hours with a ramp rate of 300°C/h, 91.5$\%$ cold rolled, and annealed again for 4 hours through a 10°C/min ramp up to 800°C, was studied. Previous texture measurements conducted in our laboratory revealed that the dominant texture component was $\{110\} < 110 >$, as expected [6].

X-ray theta/two theta reflections were measured using a D8 Bruker diffractometer, equipped with a Huber Eulerian cradle to orient the samples at their pole density maxima. Slow scan speeds were employed, particularly for weak reflections, to ensure reliable statistical results. Copper, cobalt and molybdenum K_α radiations were used. Measurements were focused on the pole density maxima reflection pairs, 111 and 222. Due to texture heterogeneity, the tilt and azimuthal angles, χ and φ respectively, obtained for both diffraction orders did not coincide exactly, being the differences of about 1$\%$ from the average. Therefore, theta/two theta scans were performed at the optimal orientations for each maximum. Next, the same reflections were measured for a powder sample at the same angle χ, and the rates r_{hkl}, defined as:

$$r_{hkl} = \frac{I_{hkl}}{I^p_{hkl}}, \tag{1}$$

were calculated, where I_{hkl} is the integrated intensity of planes (hkl) of the sample, and I^P_{hkl} the corresponding intensity from the powder sample, both under the same conditions, except for the angle φ, which has no effect in the powder sample. Quantities r_{hkl} give the ratio of crystallites oriented according to the measurement condition, and if no extinction effect were present, they should be independent of the measured reflection order. Then, the quotients q_{hkl}, defined as:

$$q_{hkl} = \frac{r_{2h2k2l}}{r_{hkl}}, \tag{2}$$

were determined, which for the ideal case should be 1.0.

Optics used in all cases was as follows: A pinhole of 0.5 mm in diameter was used instead of the divergence slit, and the receiving and antiscattering slits were both 1 mm. Filters to suppress K_β radiation were used only when neighboring peaks were close enough to cause overlapping.

To evaluate areas under Bragg peaks, EVA software of the D8 diffractometer was used, which also subtracts background. In all cases the evaluated area comprised both X-ray components, namely $K_{\alpha 1}$ and $K_{\alpha 2}$.

3. Results and discussion

3.1. Results from X-ray diffraction

Peak areas obtained from these measurements for sample (I_{hkl}), and powder (I^p_{hkl}), as well as the ratios r_{hkl} and q_{hkl} are shown on Tables I to VI. Experimental errors (σ) are also included at the right of the values.

TABLE I. Primary results with cobalt radiation.

Reflection	I_{hkl}	σ_I	I^p_{hkl}	σ^p_I	r_{hkl}	σ_r
111 α	95.86	1.64	18.59	0.86	5.16	0.25
222 α	23.65	0.88	1.60	0.44	14.81	4.10

TABLE II. Final results with cobalt radiation.

Reflection	q_{hkl}	σ_q
111/222 (α)	2.9	0.86

TABLE III. Primary results with copper radiation.

Reflection	I_{hkl}	σ_I	I^p_{hkl}	σ^p_I	r_{hkl}	σ_r
111 α	1188.9	2.54	172.3	1.08	6.91	0.05
222 α	152.3	0.98	13.33	0.45	11.43	0.39

TABLE IV. Final results with copper radiation.

Reflection	q_{hkl}	σ_q
111/222 (α)	1.65	0.06

TABLE V. Primary results with molybdenum radiation.

Reflection	I_{hkl}	σ_I	I^p_{hkl}	σ^p_I	r_{hkl}	σ_r
111 α	3770.3	7.04	435.0	4.5	8.66	0.09
222 α	708.6	3.17	31.25	1.02	22.68	0.75

TABLE VI. Final results with molybdenum radiation.

Reflection	q_{hkl}	σ_q
111/222 (α)	2.61	0.08

3.2. Results from EBSD

Figure 1 shows an EBSD image of the sample's cross section, where a fine grained zone approximately 0.12 mm thick is observed at the bottom of the silver sample. Figures 2a) and b) display the texture in the middle and at the edge of the sample respectively, measured by the OIM technique. The microstructure is partially different between the upper and lower regions of Fig. 2a) by the amount of fine grains. To avoid problems related with these microstructure differences, the measurements of each pair of reflections were made in the same place of the sample.

Considering that the X-ray penetration depth in the sample ranges from 1.13 μm for reflection 111 with Co radiation, to 11.1 μm for reflection 222 with Mo radiation, it is realized, that the fine grained zone affects results to varying degrees

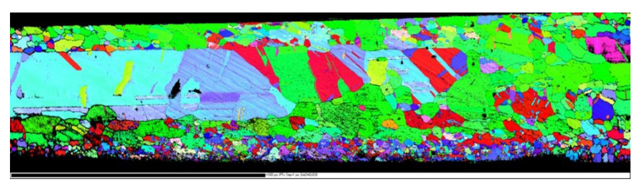

FIGURE 1. EBSD image of the sample cross section along the Transversal Direction (TD); the Normal Direction is vertical, and the Rolling Direction (RD) is horizontal.

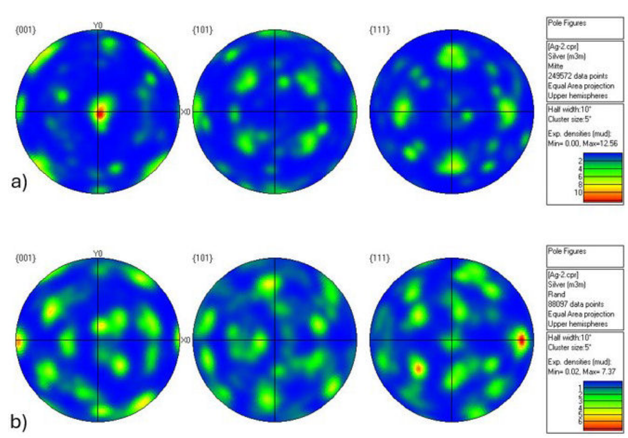

FIGURE 2. Pole figures of recrystallized silver sample. a) from the middle part, and b) from the bottom part of Fig. 1.

depending on wavelength. However, since all pairs of measurements were taken in the same sample location, the influence of texture heterogeneities on the results should be minimal.

4. Conclusion

In all three sets of measurements with different wave lengths, the maxima of second-order pole densities were consistently higher than those of the first-order. The effects of texture heterogeneities were minimized by measuring both orders of reflection and the powder, under the same conditions. Therefore, an extinction phenomenon is present, which affects more strongly the first-order reflections than the second-order reflections, similar to what is observed in single crystals. However, excluding primary extinction, since crystallites are not perfect enough as to cause it, extinction mechanisms in polycrystals are more akin to the secondary extinction. While it is true that crystallites of a polycrystal have broader orientation breaths than those of the coherent domains in single crystals, it is also truth that, in polycrystals, the incident and reflected rays have the possibility to be reflected by more planes than in single crystals, and this study indicates that, when the sample has a strong texture, this condition causes the extinction observed.

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