Scanning Low Energy Electron Microscopy for the determination of crystallographic orientation of polycrystalline metal grains

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ABSTRACT

We explore the possibility of a Scanning Electron Microscopical technique for the determination of crystallographic orientation, based on the measurement of the reflectivity of very low energy electrons. Our experiments are based on the concept that In the incident electron energy range 0–30 eV, electron reflectivity can be correlated with the electronic structure of the material [1], which varies with the local crystallographic orientation of the specimen.

The motivation for the development of this technique is to achieve a quick and high-resolution means for determining the crystallographic orientation of very small grains (<1µm) in a polycrystalline material. The key limiting factor is the cleanliness of the sample surface and also the geometrical setup of the experiment.

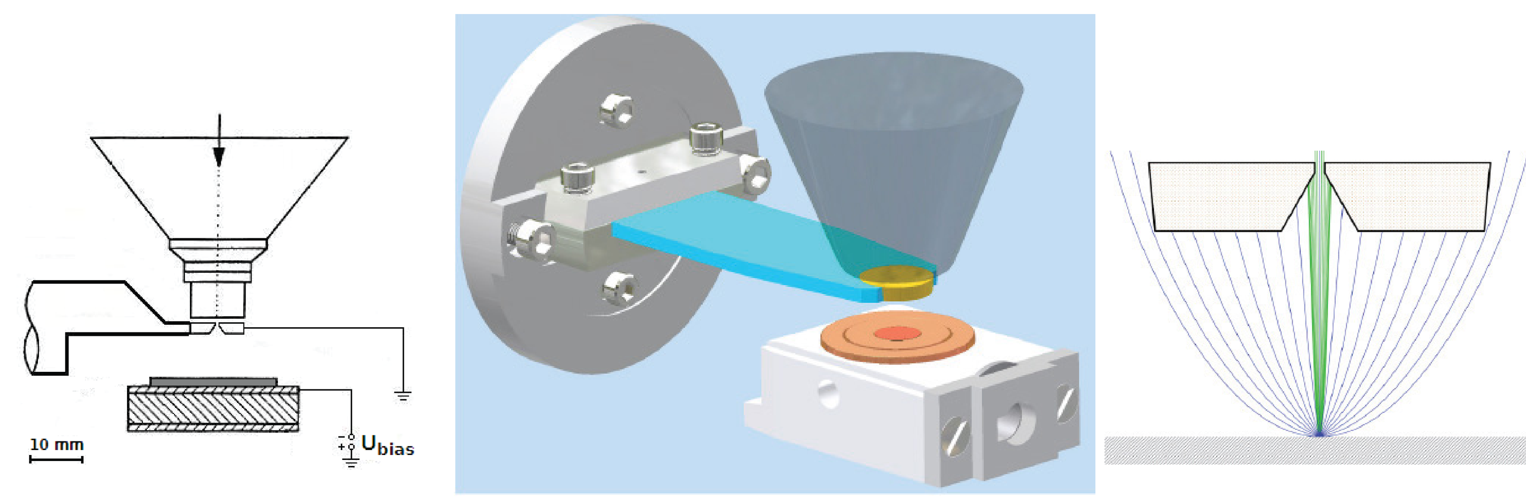


Figure 2: A schematic sketch (left) and a CAD model (center) of a Cathode Lens assembly [2]. Primary electrons are decelerated in the strong electrostatic field between the negatively biased sample and the grounded scintillation detector of Back Scattered Electrons (BSE, just below the objective lens). Signal electrons leaving the sample are accelerated back towards the BSE detector on parabolic trajectories. Since no in-lens detection is used here, a part of the signal electron bunch leaves through the central bore and is lost for detection (right).

EXPERIMENTAL SETUP

The experiment was performed in an **Ultra High Vacuum Scanning Low Energy Electron Microscope (UHV SLEEM-III)** of in-house design (Figure 1). It is equipped with a Cathode Lens assembly (Figure 2) which allows imaging at arbitrarily low incident electron energies (down to units of eV) without significant deterioration of resolution [1].

The samples, **Al and Cu poly- and single crystals**, were in situ cleaned in the Preparation Chamber of the UHV SLEEM-III by several cycles of Ar ion sputtering and heating to 450 °C. The cleanliness of their surface was verified by Auger Electron Spectroscopy.

They were then observed in the Main Chamber at a working pressure of $5{\cdot}10^{-8}$ Pa which ensures a very low rate of surface contamination. A series of images taken at incident electron energies between 0 and 30 eV with a step of 0.3 eV was made. The resulting series of images was then processed to obtain information about the reflectivity curves of the sample[1].

1 see poster of Knápek and Pokorná for detailed information

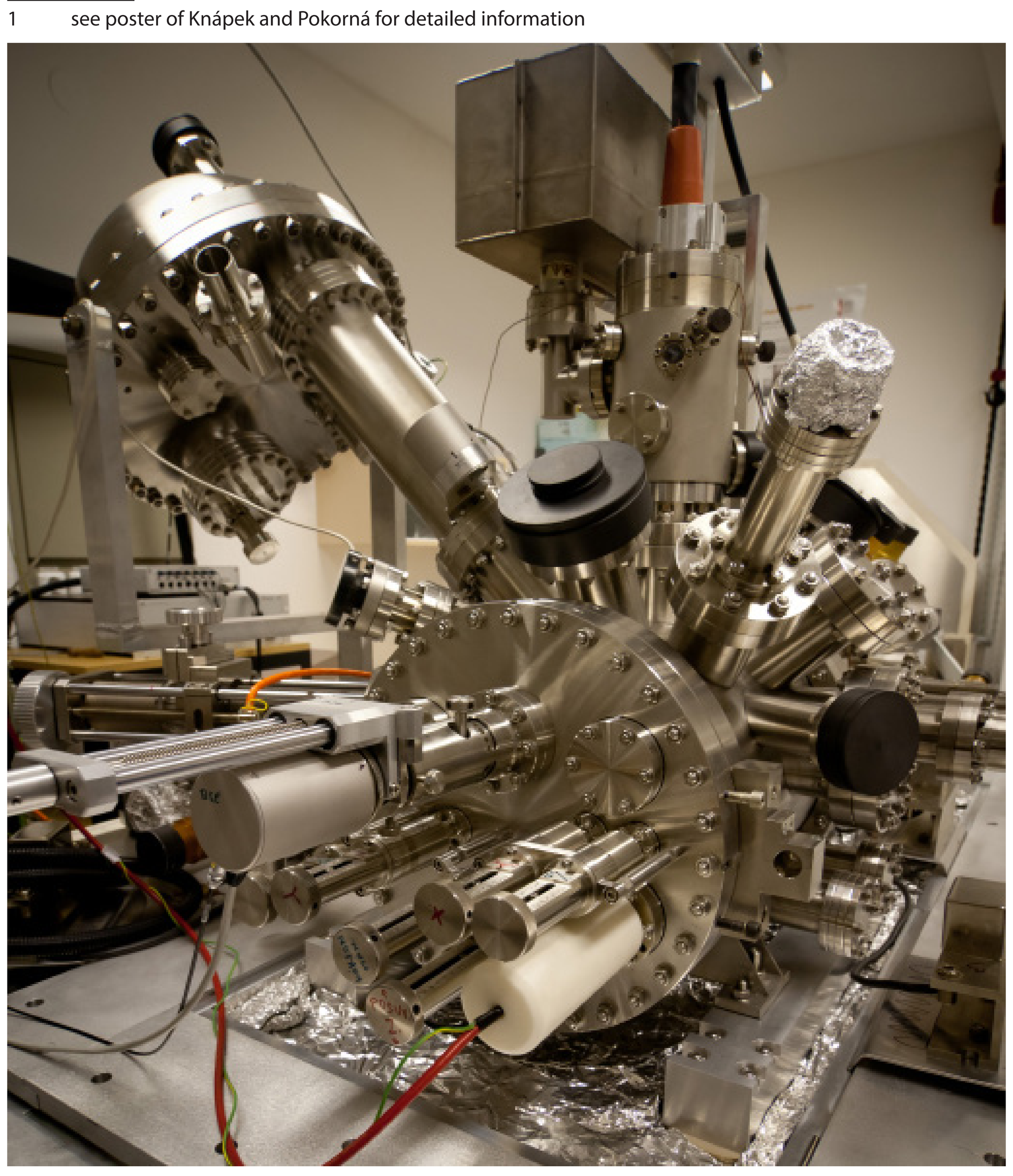


Figure 1: UHV SLEEM-III, an Ultra High Vacuum Scanning Electron Microscope equipped by a Cathode Lens allowing imaging at arbitrarily low incident electron energies (down to 0 eV).

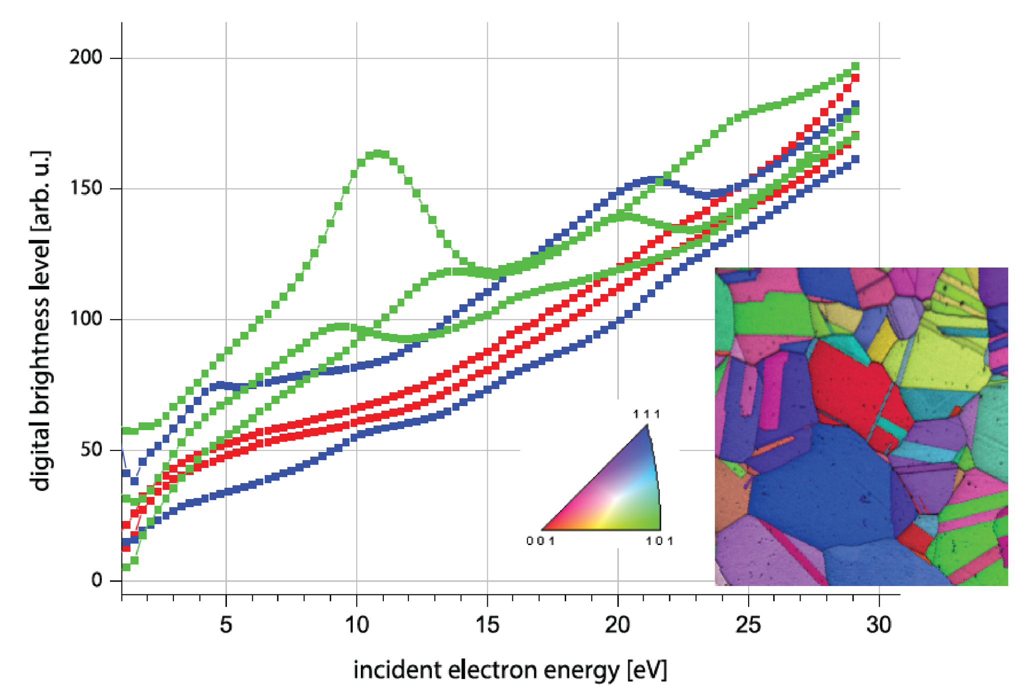


Figure 3: An example of reflectivity of selected grains in a Cu polycrystal . Grains close to the basic orientation (100) are denoted red; those close to (110) are green; and those close to (111), blue.

EXPERIMENTAL RESULTS

Reflectivity curves in the incident energy range 0 to 30 eV with a step of 0.3 eV were determined. In a polycrystalline sample, for each particular grain the reflectivity curve was obtained (Figure 3) and the crystallographic orientation was determined by Electron Back Scattered Diffraction (EBSD). Reflectivities of similarly orientated grains were compared.

Grains of similar orientation and similar location in the field of view exhibited a similar reflectivity curve. This is demonstrated eg by the red curves in Fig. 3 corresponding to the „red" grains close to orientation (100) in the centre of the field of view.

Grains of similar orientation but different location within the field of view exhibited different reflectivity curves (green and blue curves in Fig. 3, corresponding to „blue" (~Cu(111)) and „green" (~Cu(110)) grains in the sample).

A uniform single crystal exhibited a non-uniform image signal distribution depending on the azimuth and the distance from the image center (Figure 4). Different locations of the field of view exhibited different reflectivity curves. While this prevents a straightforward correlation between reflectivity and orientation (that is, the reflectivity of a grain would depend also on its position within the field of view and on its in-plane rotation), it allows to discern twin grains (Figure 5).

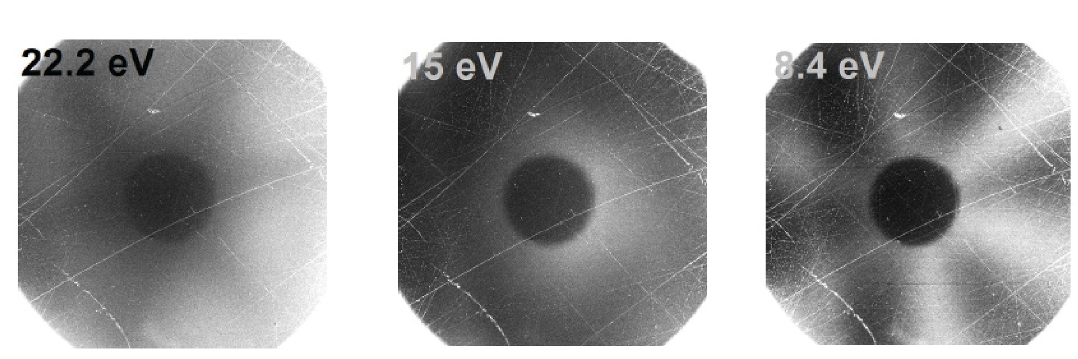
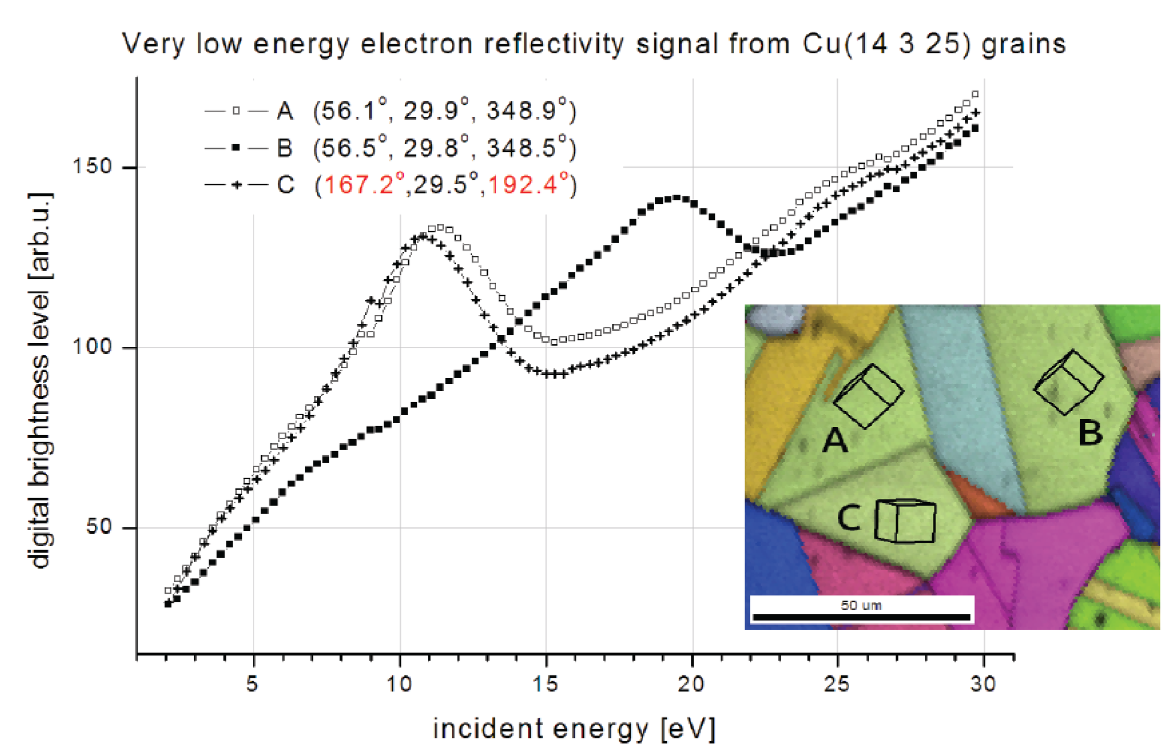


Figure 4: Non-uniform reflectivity of Al(111) single crystal.



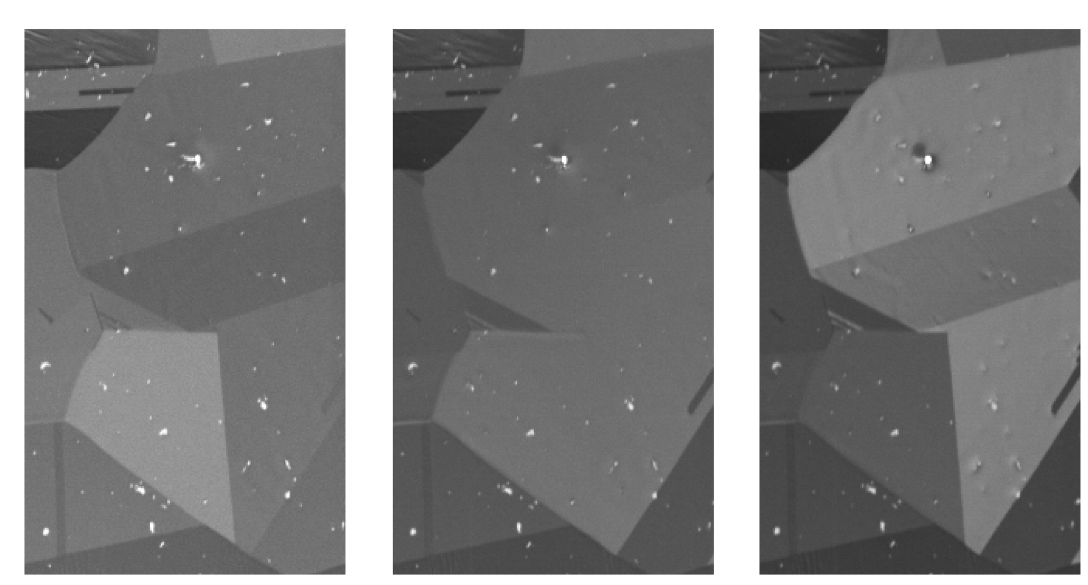


Figure 5: Twins (grains of an identical orientation but rotated about an axis perpendicular to the surface) in a Cu polycrystal. Reflectivity curve (above) and images at incident electron energies of 17.1 eV, 14.1 eV and 10.8 eV (below). FOV width = 50 µm.

CONCLUSION

Within the arrangement presented here we tested the ability to determine the local crystallographic orientation of grains in a polycrystalline sample from their very low energy electron reflectivity. Acquired data show that there is indeed an agreement which is however depedent also on the in-plane orientation of the grain. This is probably due to the strongly directional reflectivity distribution of the specimen – a result of diffraction effects – which also causes a portion of the signal to disappear in the central bore of the scintillation detector. This effect allows to discern between twin grains and is more pronounced in denser crystal faces. The state of the specimen surface, such as roughness or the presence of hydrocarbon adlayers and native oxide, is an influence on its own [3, 4].

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