Microelectrochemistry, EBSD and AME for single crystal experiments on polycrystalline surfaces

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In the last years the dependency of electrochemical properties of polycrystalline materials on the crystallographic orientation has been studied with great effort. For optical anisotropic materials like titanium and zirconium for example, Anisotropy Micro-Ellipsommetry (AME) has been applied to orientation determination of single grains down to 50 µm diameter [1]. Due to texture effects on polycrystalline materials, electrochemical measurements on single grains have been carried out using different types of microelectrodes [2], [3].

In this contribution, the crystalline orientations of single grains on technical titanium samples are determined by Electron Backscattering Diffraction (EBSD) and correlated to their electrochemical properties which have been studied with photo-resist micro electrodes. The results are compared to experiments which base on AME [1] for orientation determination. [2]

From the numerous different techniques for orientation analysis in the micrometer scale, EBSD has become one of the best alternatives for technical samples, because as a reflective method, it is not limited to thin substrates, as many X-ray methods for example. In contrast to AME, EBSD is not restricted to anisotropical lattice types and also the lateral resolution is 100 times higher.

Another difference between AME and EBSD is the fact that AME is only capable to determine one out of three Euler-angels of the crystallographic system; with the knowledge of one Euler-angle, the crystallographic plane cannot be determined properly and no calculation of Miller-indices is possible. For hexagonal systems only one crystallographic orientation can be determined properly by AME and that is the (0001) orientation with an Euler-angle $\Phi=0°$. Euler-angles with $\Phi=90°$ for example show two different crystal planes with different package densities, the (10-10) and the (11-20) orientation that cannot be distinguished by AME because they differ in the second Euler-angle φ_2 only which is not accessible by this method.

Micro electrochemical measurements with 50 µm photo-resist electrodes show that these two orientations, which have been determined by EBSD, show different corrosion behaviour, concerning their oxide growth velocity and interference colour of oxide films. The cyclovoltamogram in fig. 1 shows a grain with nearly (10-10)/ $\Phi=83°,\varphi_2=6°$ orientation and fig. 2 represents the typical i/U correlation of a (11-20)/ $\Phi=85°,\varphi_2=30°$ orientated grain. The (10-10) orientation shows a small overshoot in the first cycle up to 2V, whereas the (11-20) orientation has a lower current density in this range. Another difference can be observed at potentials above 8V, where the (10-10) orientation shows a slight, but continuous decrease in current density, which is not observed on (11-20) orientated grains. Although both grains have almost the same total charge flux between 0 and 10V, they show different oxide film thicknesses and interference colours

Further studies reveal an astonishing correlation between interference colours and crystalline orientation of single grains. This correlation allows to create an interference colour mapping of an oxidised titanium surface with which it is possible to estimate the crystallographic orientation through a normal microscope without using EBSD or AME. Therefore it is possible to evaluate orientation distributions of technical titanium samples, which can be a very important method for quality evaluation in industrial processes where the electrochemical properties of titanium play an important role, like electro catalysis or applications for dimension stable anodes (DSA).

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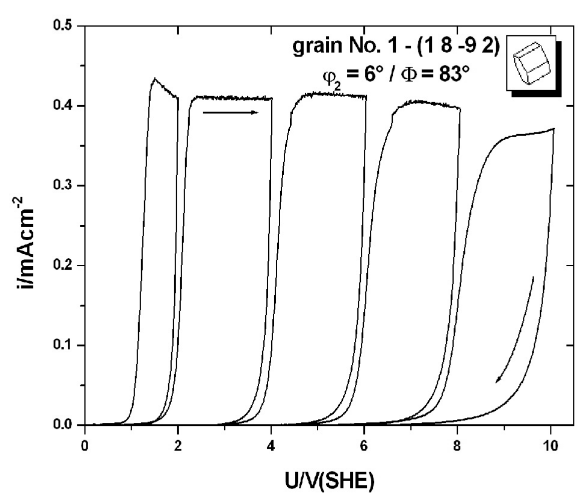

Fig. 1: *Current-time curve of a nearly (01-10) orientated grain in 0.5M H_2SO_4 with 50 mV/s and (U vs SHE)*

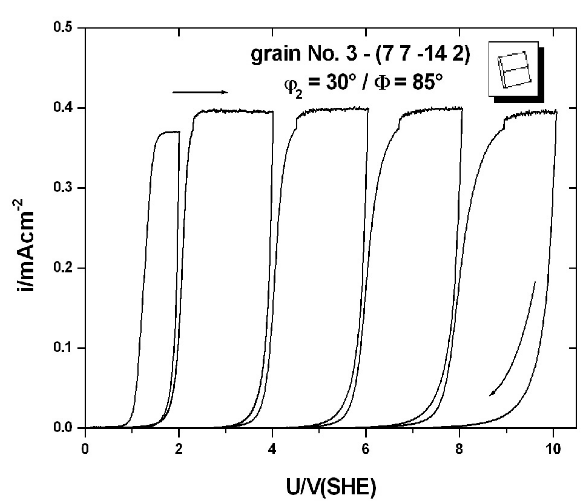

Fig. 2: *Current-time curve of a nearly (11-20) orientated grain in 0.5M H_2SO_4 with 50 mV/s and (U vs SHE)*