Microstructure of the ultra-fine grained Cu by UHV SLEEM

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Study of materials with ultra-fine structure belongs nowadays to fronted areas of research of the material engineering. Ultra-fine grained (UFG) materials are defined as polycrystals with very small grains of average grain sizes below 1 micrometer [1]. These materials are very attractive for many industrial applications (including aerospace, automotive, biomaterials, chemical sensors, construction, electronics, metal forming, etc.) because of their interesting mechanical and physical properties surpassing those of common materials. The superior mechanical properties include ultra high strength and superplasticity.

For bulk UFG materials homogeneous microstructure is very important in order to achieve advanced and unique properties. Microstructure of the UFG copper was investigated under ultra-high vacuum conditions by means of a scanning low energy electron microscope (UHV SLEEM) equipped with the cathode lens retarding the primary electrons and with an ion-gun for cleaning the sample surface. After examination of the specimen in the UHV SLEEM the grain orientation maps were obtained by the electron backscattered diffraction method (EBSD) in a conventional W – filament SEM.

Experiments presented here have been performed on the UFG copper. Commercial purity copper (99,98%) was processed by equal channel angular pressing (ECAP) method using 8 passes, route B_c [2]. The specimen surface was prepared by mechanical polishing and the affected surface layer was removed by electro-polishing. Finally, the specimen was annealed for 6 minutes at 180°C in argon atmosphere and before observation in the UHV SLEEM the surface was in-situ cleaned by the ion-beam (Ar^+).

Figure 1 shows the reflectance curves acquired by measuring average image signals over three grains with orientation (100), (111) and (101) with respect to the surface normal, together with the grain orientation map obtained from EBSD. Reflectivity of very slow electrons (below ~ 20eV) from surfaces was found inversely proportional to the local density of states coupled to incident electron waves [3]. Differently oriented crystals have specific energy dependences of the density of states, which could become source of a microscopic contrast varying with energy. Figure 2 shows this contrast evolution between the grains with orientations (001) and (101).

UHV SLEEM [4] is an excellent method for observing grains in polycrystals thanks to much faster acquisition of data relative to EBSD. Specific energy dependences of the electron reflectance can be used for identifying the grain orientations.

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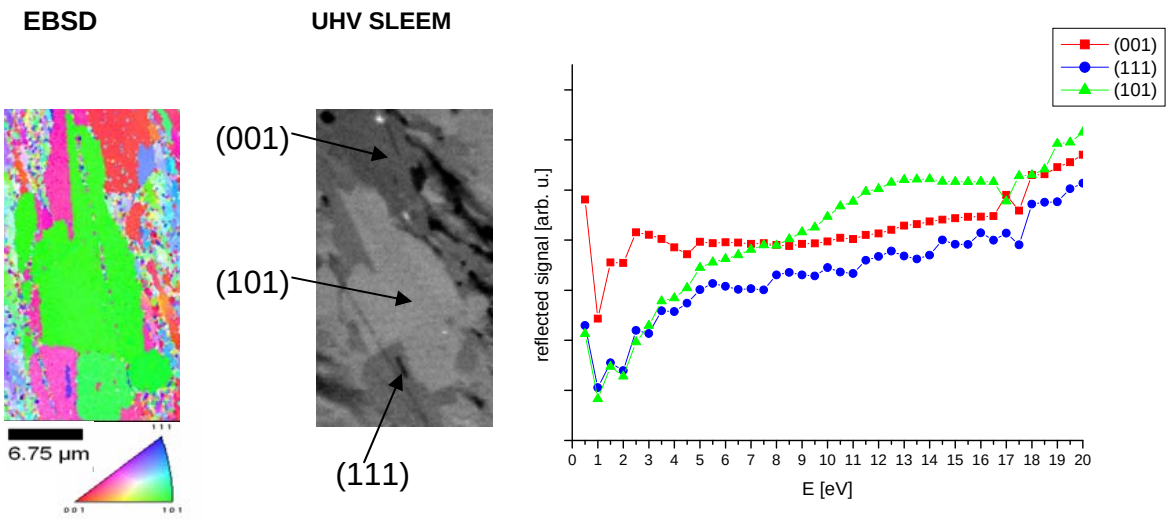

Figure 1. EBSD map of the microstructure, together with the UHV SLEEM image of the same area obtained at 11 eV and the reflectance curves for three copper grains with orientation (100), (111) and (101).

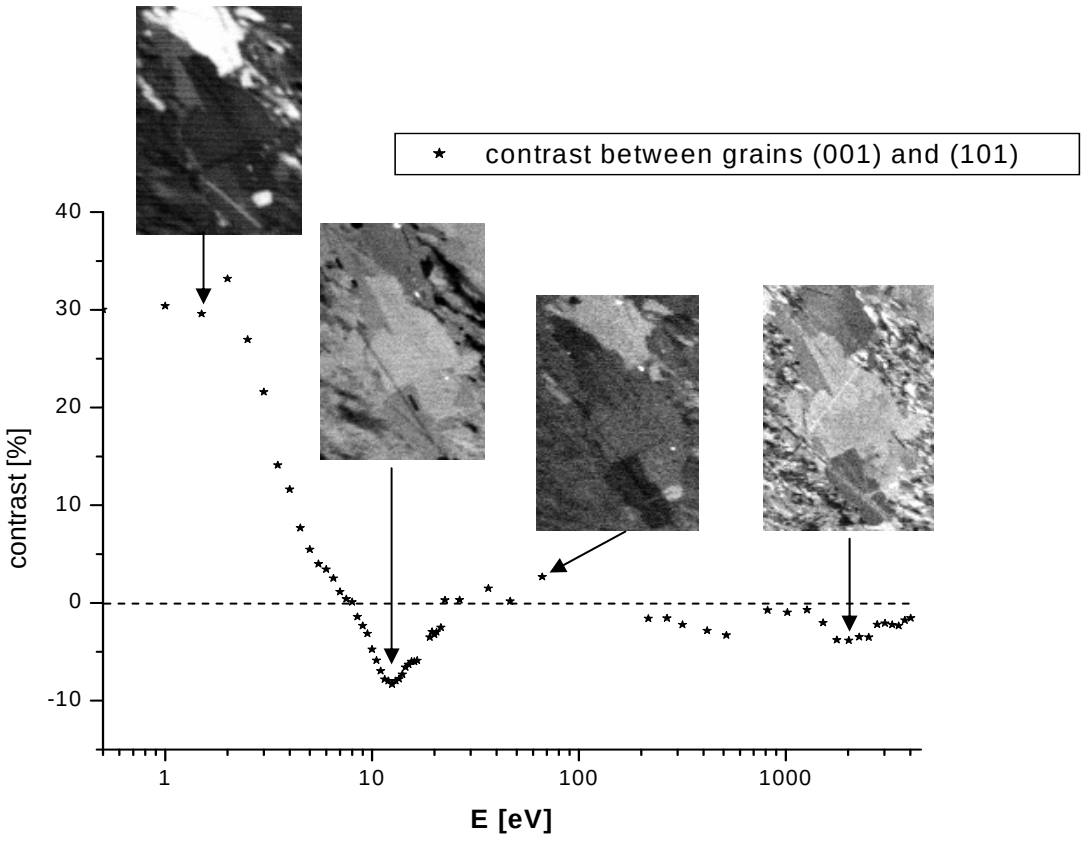

Figure 2. Contrast between grains with orientation (001) and (101) as a function of the landing energy E of the primary beam. Notice the high grain orientation contrast at units of eV.

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