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IT-9-P-2459 EBSD sample preparation: high energy Ar ion milling

Kalácska S.[1], Baris A.[1], Varga G.[1], Radi Z.[2], Lendvai A.[2], Dankházi Z.[1], Havancsák K.[1]

[1]Eötvös Loránd University, [2]Technoorg Linda Ltd.

Email of the presenting author: kalacska@metal.elte.hu

EBSD is a versatile tool providing grain size determination, orientation mapping, phase identification and 3D mapping. Since the EBSD information comes from a few tens of nanometers of the specimen surface regions the most critical issue of the EBSD measurement is the surface quality. The surface should be perfectly clean, free of amorphous or deformed surface layer and moreover it should be flat because of the shadowing effect. Lack of these factors can result either no or faded diffraction pattern.
As it is known, the usual mechanical grinding and polishing create an amorphous layer of (1-100) nm thickness on the surface. The commonly suggested colloidal silica polishment continues for hours and can embed residual polishing material in the surface grains. Electropolishing of the surface can also be tried, but this is a difficult and complex procedure, nevertheless in some cases it cannot lead to the desired result.
In the last decades a new surface milling method is spreading. This is based on energetic ion beam milling; the underlying physical process is the sputtering. One direction of this method is the focused ion beam technique (FIB) with ion energies up to 30 keV. The other direction uses near parallel inert gas (usually Ar) ion beams with energy up to 10 keV.
In this poster we present a newly developed Ar ion sample milling apparatus and show how advantageously it can be utilized to produce high quality sample surface. Surface quality development on series of metal samples was investigated using Technoorg Linda's SC-1000 SEMPrep Ar ion milling apparatus. The surface quality of samples was characterized by the image quality (IQ) parameter of the electron backscatter diffraction (EBSD) measurement. Ar ion polishing recipes have provided to prepare a surface appropriate for high quality EBSD mapping. The initial surfaces of samples were roughly grinded and polished. High quality surface smoothness could be achieved during the subsequent Ar ion polishing treatment. The optimal angles of Ar ion incidence and the polishing times were determined for several materials using a FEI Quanta 3D FEG SEM.