Surface polishing and slope cutting by parallel Ar ion beam for high-resolution EBSD measurements

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Abstract: For the large majority of materials science studies a good quality surface is extremely important. Especially, it is of great importance in case of electron backscatter diffraction (EBSD) where the information depth is as shallow as some tens of nanometres and this is the reason why this analytical method requires a damage- and oxide-free sample surface. The SC-1000 SEMPrep dual Ar ion beam workstation developed by Technoorg Linda Ltd., Hungary, is suitable for both surface polishing and slope cutting of solid state samples, preparing high-quality surfaces. These surfaces allow several types of SEM investigations including the surface sensitive EBSD analysis. The present paper demonstrates the operating principle of the SC-1000 SEMPrep apparatus and its outstanding abilities. Here we present Ar ion polishing of different conductive and non-conductive materials followed by high-resolution EBSD measurements. The average image quality (IQ) number of the Kikuchi patterns has been studied as a function of polishing angle and time in order to find the optimal polishing conditions. It has also been shown that using the optimal operation parameters high-quality surfaces can be obtained on different metallic and non-metallic materials.

Introduction: Scanning electron microscopy (SEM) is an important tool for experimental works in materials science and solid state physics. One of the available analytical methods of SEM is the electron backscatter diffraction (EBSD), which can be used to determine grain orientation, grain size and phase distribution in polycrystalline materials. In combination with focused ion beam (FIB) slice and view, 3D mapping of the grain structure can be achieved. The main difficulty of this method, however, is the appropriate sample surface preparation. The usual surface treatment techniques (mechanical grinding, multiple steps of polishing) either consume a large amount of energy and time or the process is rather complicated (e.g., electropolishing), and satisfying result can rarely be achieved.

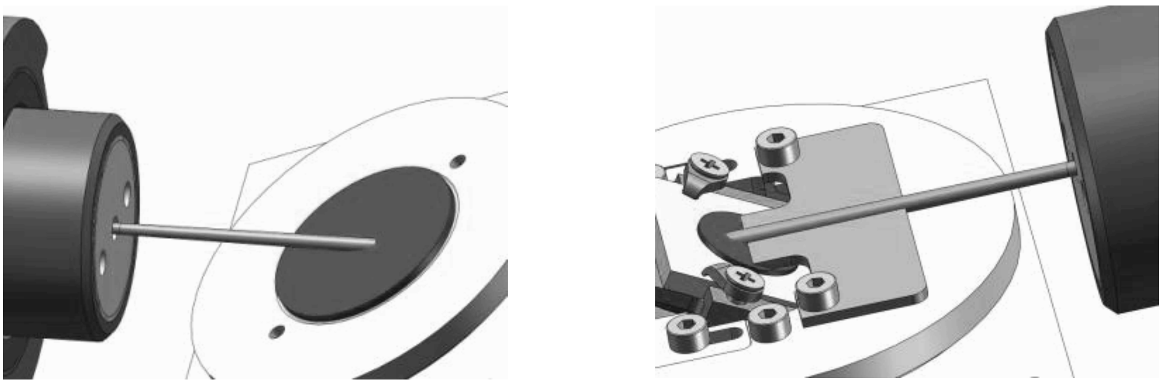
Most of the time the sample preparation begins with mechanical grinding and polishing, because the surface to be studied needs to be flat and smooth for EBSD measurements. This mechanical treatment generates a 1-100 nm thick amorphous layer (the so called Beilby layer [1]) on the sample surface which significantly blurs the diffraction patterns collected by the EBSD detector, since the backscattered electrons come from a shallow subsurface region (the spatial resolution of EBSD is about 50 nm). This is why the EBSD measurements are very dependent of the surface quality and also the morphology. On the other hand, this feature makes the EBSD a perfect tool for surface quality characterization and evaluation.

In order to remove the amorphous layer one can apply chemical etching or electropolishing, but these methods tend to coarsen the surface and therefore produce less evaluable results. Because of this a new promising technique has been developed in the last decade. This new method is the ion polishing and milling, the underlying physical phenomenon of which is the atom/cluster sputtering. This technique uses either low-energy (0.1 - 2 keV) or relatively high-energy (2 - 10 keV) ions of inert gases (e.g., Ar^+, Kr^+, Xe^+) in a near parallel beam, or some ions of metallic origin, as for instance Ga^+ ones (2 - 30 keV) in the focused ion beam (FIB) systems.

The main benefit of the ion milling techniques is that they are less sensitive to the microstructure and chemical composition of the samples. Other advantages of the inert gas parallel beam technique are the size of the processed area and the low costs of the treatment. While using FIB the size of the achievable area is in the order of 100 μm × 100 μm, with parallel noble gas beams one can easily produce a 1000-2000 μm wide and 100-300 μm deep cut/polished area, not to mention that not every scanning electron microscope has a built-in FIB system.

The new SC-1000 SEMPrep device [2] developed by Technoorg Linda Ltd. is capable of both surface polishing and slope cutting (Fig.1). It was designed specifically for SEM sample preparation. Its stage can be tilted in the range of 0 - 30° towards the active ion source with 0.1° precision, and the surface polishing sample holder can do full in-plane rotation or oscillation while using the high-energy (2 - 10 keV) or the low-energy (0.1 - 2 keV) ion gun. The size of the ion polished area is set by the stage tilt. The slope cutting head units are offered with fixed 30°, 45° and 90° microstage pretilt, but the most suitable for EBSD sample preparation appears to be the 30° version. For slope forming, a titanium (Ti) plate/mask blocks the lower half of the incoming Ar ion beam. The shaded sample part remains untreated, but the rest of the sample surface is sputtered by the upper half of the beam, therefore near the Ti plate, parallel to the beam, a smooth EBSD-ready surface is created. While slope cutting, the head units can oscillate in a ±10 - 40° angular range, and the tilt of the stage can be varied. To obtain a good surface it is recommended to use first the high-energy ion gun for fast milling and slope cutting, and afterwards the low-energy one to remove the backsputtered atoms/clusters from the freshly cut surface. In case of both guns the near parallel Ar ion beam treats an area of about 20 - 100 mm^2.The sample is located in a vacuum chamber which holds ~10^{-6} mbar base pressure and ~10^{-4} mbar dynamic pressure during the ion milling.

The aim of the present paper is to demonstrate the advantages of Ar ion polishing and the unique capabilities of the SC-1000 SEMPrep apparatus. The authors will show how the optimal parameters of the EBSD-quality surface treatment can be found.

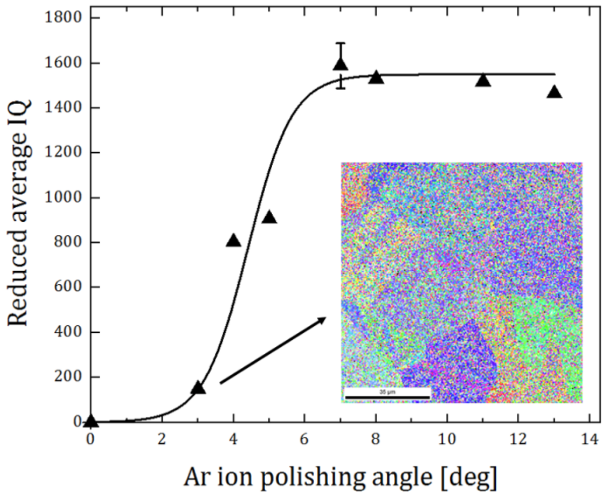


a. b.

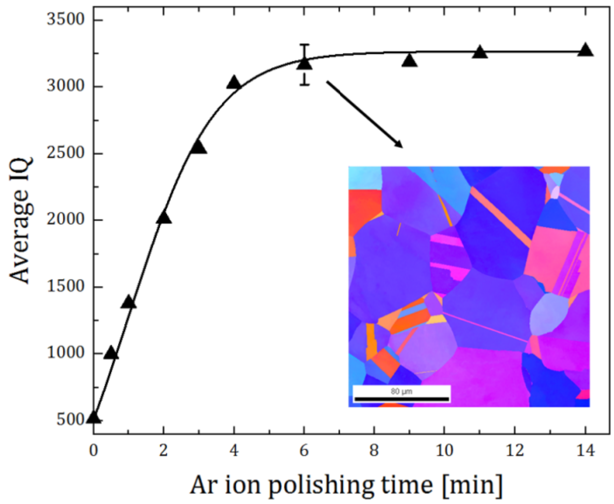
Fig. 1. Scheme of the two operating modes of SEMPrep: low-angle surface polishing (a); ion beam slope cutting (b).

Results and discussion: In the framework of cooperation between Eötvös University and Technoorg Linda Ltd. sample preparation protocols were specified in the university SEM laboratory [3]. A FEI “Quanta 3D FEG” SEM and its EDAX EBSD device were used to study the surface quality effects of ion milling. The first step of the surface treatment consisted of conventional mechanical grinding and polishing in order to remove the rough bumpiness, and ensure a rather clean, uniform surface to start the Ar ion milling. The steps of the mechanical treatment were as follows: grinding with abrasive paper of 600-, 1200-, 2500- and 4000-grit and polishing with alumina paste of 1 μm average particle size.

The EBSD measurements were begun on these samples to determine the initial average of the IQ value. In order to find the optimal ion milling/polishing angle, the angular dependence of IQ was measured. Measurements were accomplished on samples from several different materials (Cu, Ni, Fe, steel) from which the results on annealed Ni can be seen in Fig. 2.a.



a.



b.

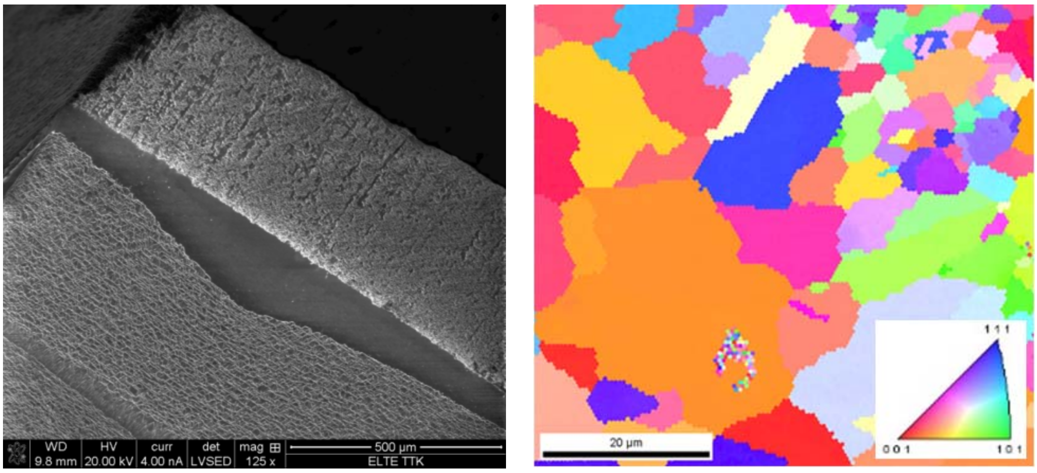
Fig. 2. Dependence of the average IQ value on the ion beam incidence angle (a) and on the total time of ion polishing (b) measured on Ni.

The horizontal axis indicates the incidence angle of Ar ion beam measured from the sample surface, while the vertical axis shows the reduced average IQ value characterizing the whole area of the EBSD image. The points were measured on different samples, hence in order to decrease the uncertainty due to distinct initial IQ, these were subtracted from the respective IQ values measured after Ar ion polishing. The milling time for all points of this curve was 6 minutes. The curve in Fig. 2.a is depressed at small angles (< 3^o), then it follows a nearly linear rise after which it saturates. It can be assumed that the rising part is in connection with the reduction of the deformed/amorphous Beilby layer. The saturation probably indicates the full removal of this layer by the Ar ion milling. After a relatively long saturation stage such curves usually start to decrease, but this is not shown in the figure. This decrease is the consequence of the inhomogeneity of the Ar ion milling/polishing due to the initial surface irregularities (i.e., shadowing phenomena) and the orientation dependence of the sputtering rate. The optimal Ar ion milling angle determined from the curve ranges from 6° to 8°. This angular range proved to be optimal in case of other materials as well [4].

The next step was the determination of the optimum duration of the Ar ion polishing treatment. The time dependence of the IQ curve is shown in Fig. 2.b. Each point of this curve was measured on the same sample and at the same area with the aim to reduce the measurement uncertainty. In the initial part of the curve there is an IQ growth with the polishing time after which saturation occurs. The explanation of the saturation phenomenon is the same as in case of the angular dependence. In order to avoid overmilling the optimal polishing time should be chosen from the initial part of the plateau. In case of Ni this optimum value falls in the range of 6 - 8 minutes. The optimum value depends on various factors; first of all on the material of the sample and its history of treatment. If one has a sample of a new material, the optimum Ar ion polishing time should be determined by similar time dependence measurements. An EBSD inverse pole figure obtained after 6 minutes of ion polishing (inserted to Fig. 2.b) shows the convincing effect of the Ar ion surface treatment.

In some cases, especially for porous and geological samples the removal of the Beilby layer is difficult due to backsputtering or high-energy ion beam induced amorphization. In such cases slope cutting proved to be an effective approach without preliminary mechanical sample treatment. Fig. 3 shows an example on a sample of limestone. A slope cut was carried out at 0° stage tilt (i.e., ion beam parallel to the Ti plate) and 30^{o} microstage pretilt using a 10 keV Ar ion beam resulting in about 1.6 µm/min milling rate. The sample was cut and oscillated in the cut plane by ±30° for 120 minutes. The shape of the slope cut zone shown in Fig. 3.a follows the intensity distribution of the Ar ion beam.

In case of limestone the result of direct slope cutting by Ar ions did not meet the requirements of a successful EBSD measurement due to the backsputtered atoms/clusters accumulated on the cut plane. In order to solve this contamination problem a 1 keV Ar ion beam generated by the low-energy ion gun was used to clean the freshly cut surface. The cleaning procedure was carried out at 5° stage tilt, ±30° in-plane oscillation and lasted for 60 minutes. The high-quality EBSD grain orientation map recorded after this extra sample cleaning step is shown in Fig. 3.b.



a. b.

Fig. 3. 10 keV slope cut of a limestone sample (a) and an EBSD grain orientation map of the slope cut zone properly cleaned by Ar ion beam of 1 keV energy (b).

Conclusions: The broad Ar ion beam surface polishing and slope cutting methods perfectly complement the traditional mechanical surface treatment and yield high-quality EBSD samples. Prior to EBSD measurements the optimum treatment parameters should be determined, some examples of which are given in this paper. The Beilby layer generated by mechanical sample preparation is quickly removed on a large (> 1 cm^2) area by Ar ion surface polishing. 1000-2000 µm wide and 100-300 µm deep slope cuts suitable for EBSD analysis are routinely produced on geological and porous samples, where the removal of the deformed/amorphous surface layers is difficult by this kind of surface polishing. Low-energy Ar ion cleaning can further improve the surface quality of these slope cuts and eliminate the problems caused by ion beam induced amorphization or backsputtering. The SC-1000 SEMPrep ion beam workstation is capable to prepare high-quality sample surfaces with both methods and also offers a low-energy cleaning option.

References

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