EBSD sample preparation: high energy Ar ion milling

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Abstract: Surface quality development on series of metal samples was investigated using a new 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**.

Introduction

EBSD is commonly used for orientation determination (Figure 1.). The information comes from a few tens of nanometers of the specimen surface regions, so the surface should be perfectly clean, free of amorphous or deformed layer [1] and it should be flat because of the shadowing effect. 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.

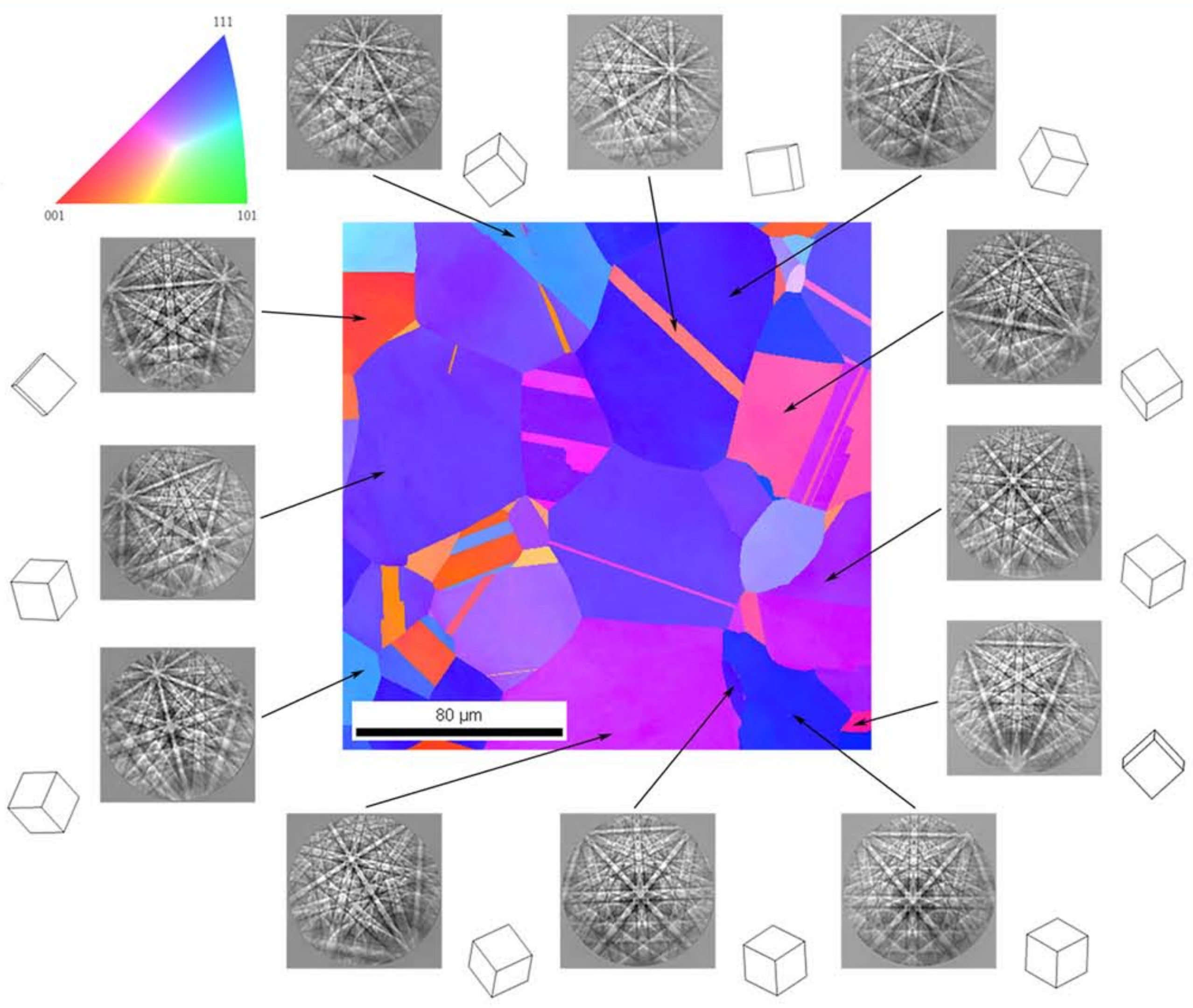


Figure 1. Orientation mapping: inverse pole figure of a Ni polycrystalline sample (middle) with the color coding triangle (upper left corner) and the corresponding diffraction patterns (grayscale images) with the unit cell positions.

Slope cutting

The new apparatus is the **SC-1000 SEMPrep** sample preparation device [2] designed by Technoorg Linda Ltd. Co. The milling apparatus has two Ar ion guns, a high-energy one (E_{max} = 10 keV) for rapid milling, as well as a low-energy (E_{max} = 2 keV) ion gun for gentle surface polishing and cleaning. Moreover, this device is capable for cross-sectional sample preparation by slope cutting for traditional SEM and EBSD measurements (Figure 2a.). In this apparatus the sample can be rotated or oscillated using different tilting angles.

Preparation of a non-conductive sample is also possible, and a good quality, EBSD-ready surface can be achieved (Figure 2b-d.).

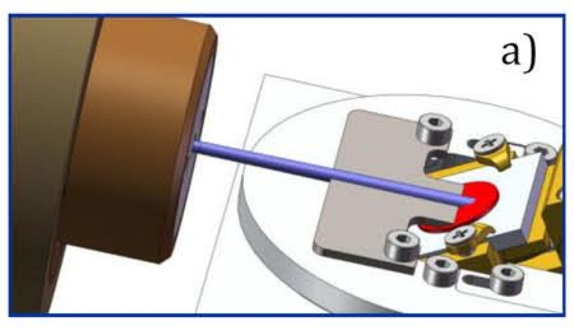
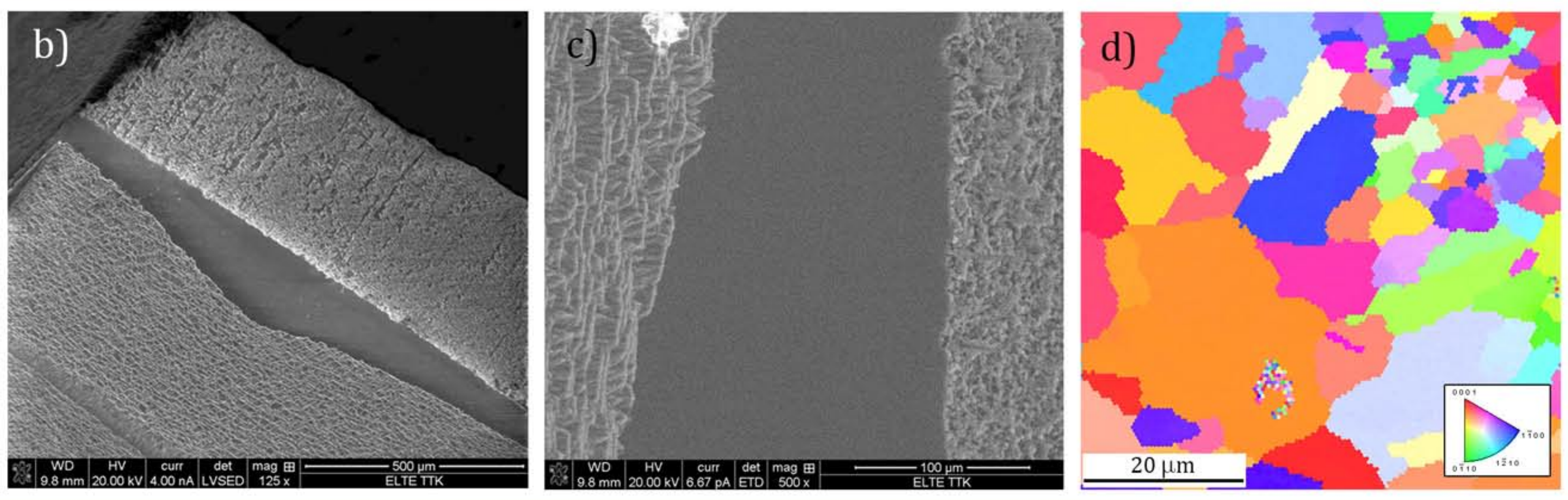


Figure 2. a) SEMPrep slope cut operation mode, b) limestone SEM image of the whole milled area, c) part of the central region and d) inverse pole figure after 2 keV Ar ion final polishing.



Surface polishing

We used the average image quatlity (IQ) parameter corresponding to the whole image for the classification of the surface quality (Figure 4-5.). The IQ value is the intensity sum of the detected and indexed diffraction bands [3]. To be careful, we kept all possible conditions constant during measurements, so the IQ would give us information on the quality of the surface.

a)

sample	Al	Cu	Ni	Fe	steel
angle (deg)	7	8	8	6	8

b)

sample	Al	Cu	Ni	Fe	steel
time (min)	6	7	6	7	26

Table 1. Ideal angle (a) and time (b) values to prepare high quality EBSD surface for various materials.

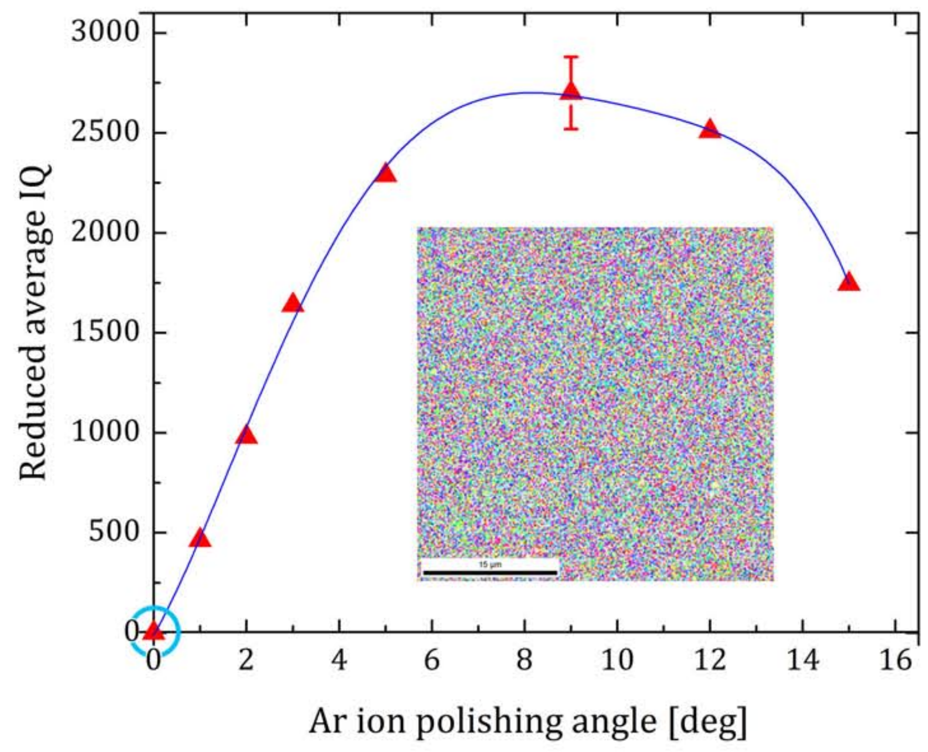


Figure 4. The reduced average IQ value can be seen as a function of angle of Ar ion beam incidence measured on a Cu sample. Since the points were measured on different samples, to decrease the deviation due to distinct initial IQ, these initial values were subtracted from the respective IQ calculated after polishing.

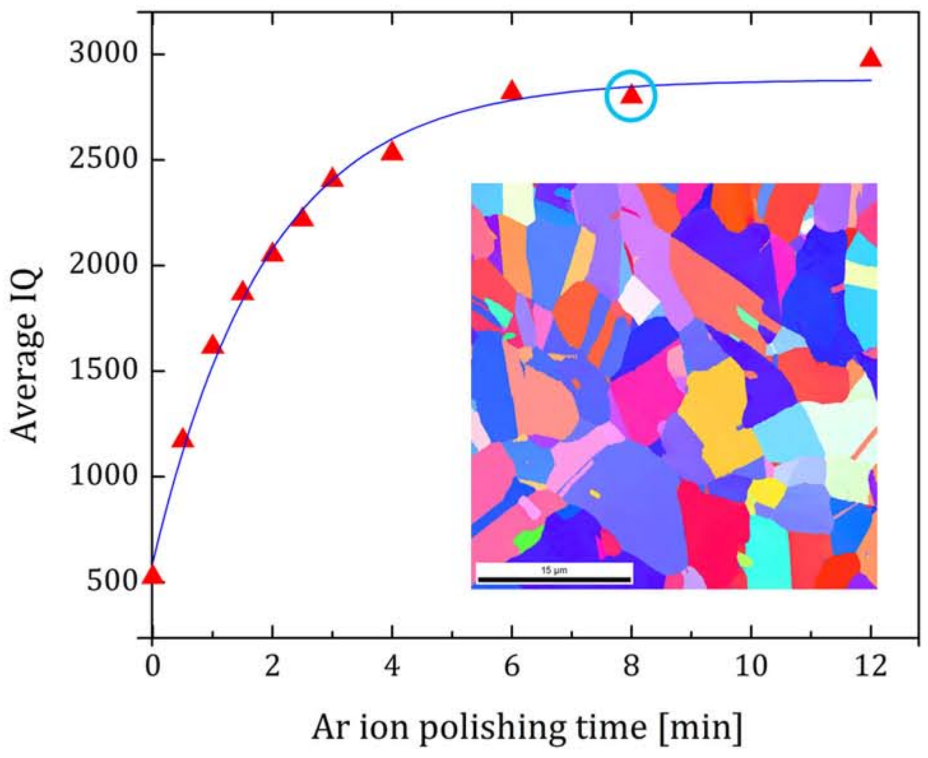
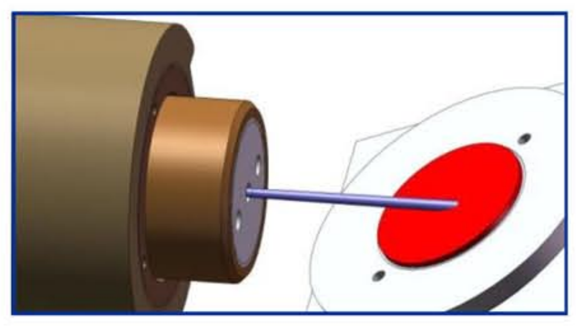


Figure 5. Effect of ion milling duration on the average IQ with the corresponding inverse pole figure (IPF) maps measured on a Cu sample. The blue circe indicates the optimal polishment based on IQ value, preparation time and surface morphology.

Figure 6. SEMPrep surface polishing mode. During ion milling the sample is being rotated and tilted.



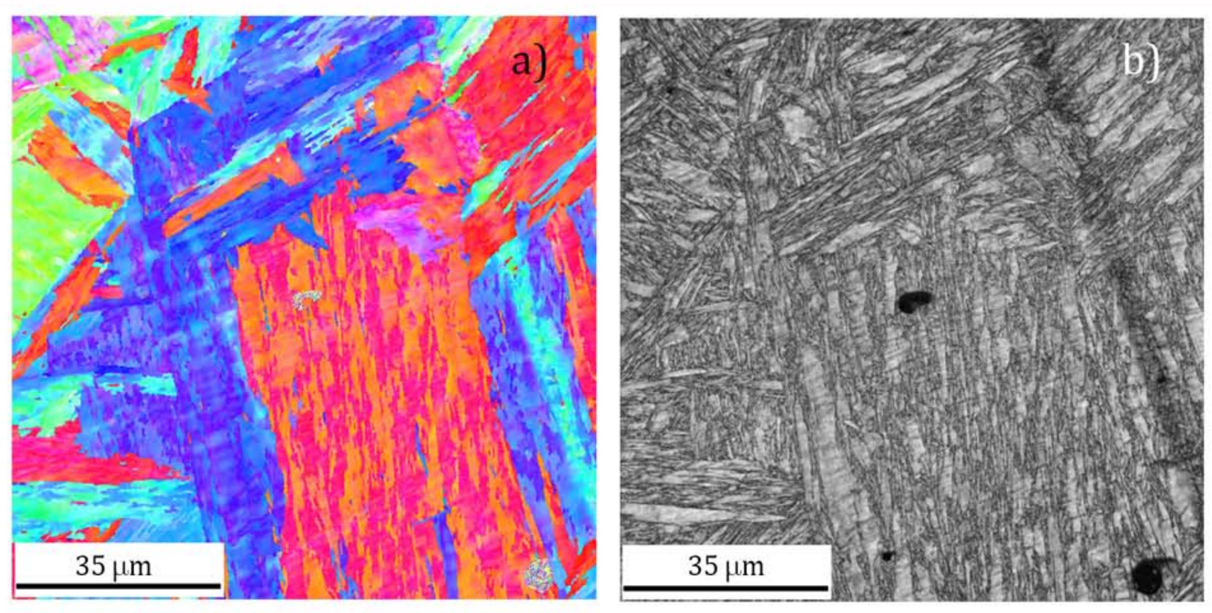


Figure 7. Inverse pole figure (a) and image quality (IQ) map (b) of a martensitic steel. This sample could only be prepared using Ar ion polishing.

Summary

It has been shown that starting with roughly polished initial surfaces the SC-1000 SEMPrep apparatus is capable to prepare a high quality surface appropriate for high resolution EBSD mapping. The preparation time is much less and the Kikuchi pattern quality of the sample is higher comparing to traditional polishing methods. The optimal parameters for several materials are determined and summarized in Table 1.

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[3] J. Goldstein, D. Newbury, D. Joy, Ch. Lyman, P. Echlin, E. Lifshin, L. Sawer and J. Michael, Scanning Electron Microscopy and X-Ray Microanalysis, Springer (2007).

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