Combined ion beam preparation procedures for simultaneous investigation of surface and bulk properties by SEM

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Ion beam processing of samples for SEM investigations offers essential information gain. The general processes together with standard applications have been described earlier[1]. However, with combined procedures special tasks can be solved concerning correlation between surface and bulk information. **Figure 1** shows three essential broad ion beam processes for 3D investigation of microstructures. With ion beam direction IB1 slope cutting is performed. The ion beam slope cutting (IBSC) technique[1] does not require a smooth initial surface. Rough samples can be cut and the IBSC technique is able to show the surface simultaneously with the cut area. Surface properties can be directly correlated with the cut through the bulk material. The surface profile can be measured along the cutting line and the inhomogeneous bulk is visible by material contrast. To avoid misinterpretations one has to consider that depending on the sputtering process and the geometry redeposition of sputtered material on topographical surface features is possible. This can modify the real profile along the cutting line depending on the bombardment time. With a short bombardment in direction IB2 the slope area as well as the initial surface and the sputtered region can be etched selectively. The internal grain and phase structure will be visible in direct correlation to the surface properties in the same microregion. Additionally also redeposition effects can be separated from the microstructures of interest by selective etching. In many cases the third process of sputter coating in **Figure 1** is useful for various purposes. Sputter-deposition can be applied before cutting to choose the cutting position on insulating material and after cutting for inspection of the cut region. However, also on conducting material this coating step can be used to protect the original surface profile and clearly to detect the upper surface contour by SEM with high resolution and not modified during long-time cutting. Exact depth and thickness measurements can be performed. The 3D information is not limited to small distances from the surface - it can be achieved up to hundreds of micrometers in depth and millimetres laterally and for complicate surface profiles. Also in this cases selective etching in direction IB2 can follow the cutting step.

All these processes have been carried out in the modified ion beam equipment Gatan PECS [2]. The apparatus allows various ion etching, cutting, and sputter-deposition processes as well as cleaning and polishing with typically 5 to 10 keV inert gas or reactive ions. **Figure 2a** shows micro-stamped structures on polycrystalline copper (Sample: Fraunhofer IWU Chemnitz/ Germany) cut by ions through the region of interest up to 150 µm depth. To show the influence of the stamp procedure on the material structure selective etching has been carried out. **Figure 2b** shows a detail with the grain structure deformed by the stamp process.

Figure 3 shows an ion beam cut through microtopographical structures on Si covered by photoresist on a metallisation layer. To protect the original surface contour of the resist the sample was coated with a tungsten layer by sputter-deposition before ion beam cutting.

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2. W. Hauffe, D. Glöß, R.J. Mitro, Proc. ICEM 15, Durban/South Africa 2002, Vol. 1, p. 273

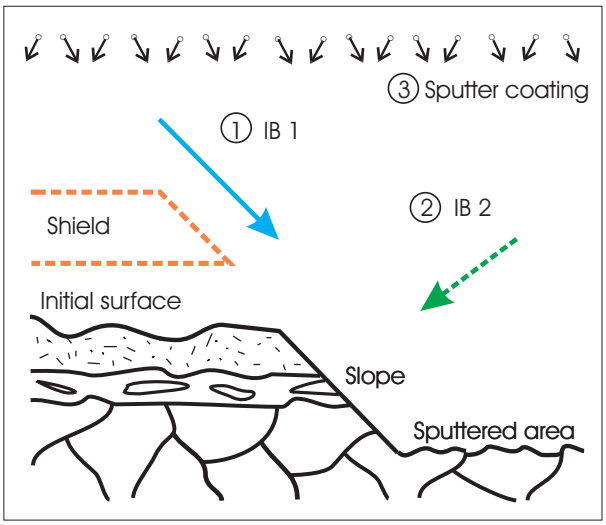


Figure 1. Ion beam processing for simultaneous investigation of surface and bulk properties of heterogeneous samples with SEM observation of the initial surface, slope cut area, and sputtered area

1 Ion beam direction for cutting (IB1)
2 Ion beam for selective etching (IB2)
3 Sputter coating for protecting the initial surface profile and/or conductive coating for SEM observation

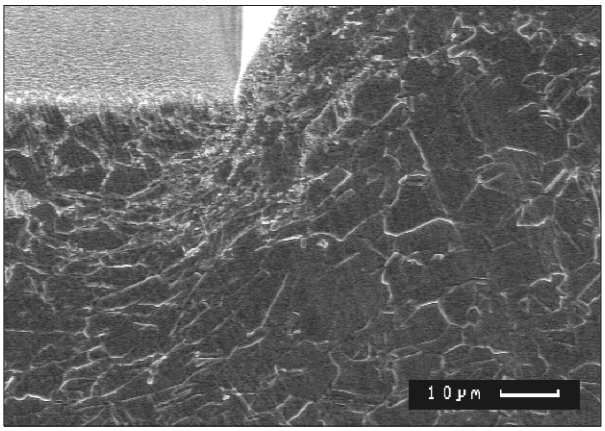


Figure 2b. Detail of the **Figure 2a** (see the marked frame) with the deformed grain structure near the concave edge of the stamped microstructure in polycrystalline Cu

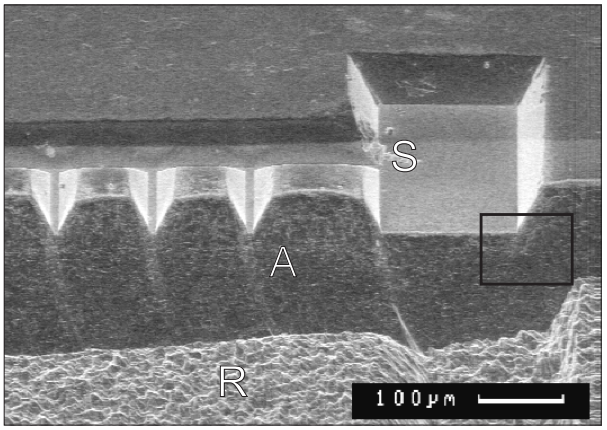
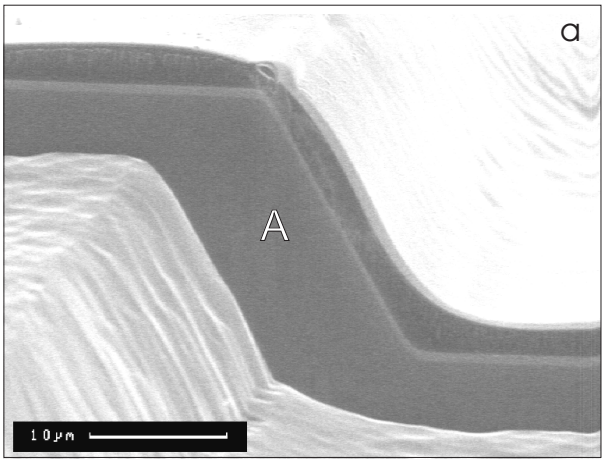


Figure 2a. Microstructures on micro-stamped polycrystalline copper after ion beam slope cutting and selective Ar ion etching to determine the internal deformed grain structure with direct correlation to the micro-stamped initial surface profile

S Initial surface structure
A Ion beam cut area
R Sputtered region
☐ Detail shown in **Figure 2b**



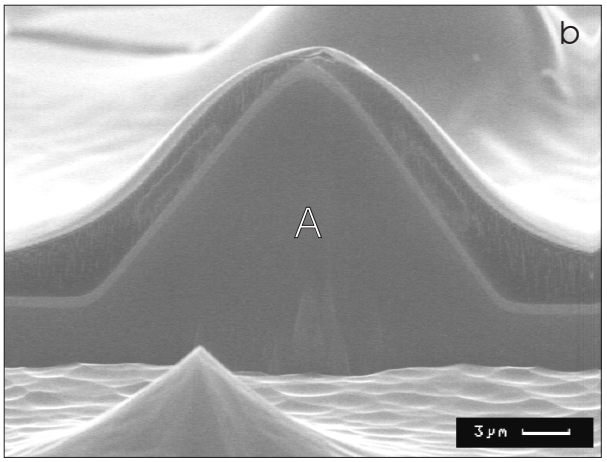


Figure 3. Ion beam slope cut (A) through microtopographical structures on Si covered with a thin metallisation layer and photoresist. The photoresist layer was coated with tungsten by sputter-deposition before ion beam cutting. The resist thickness distribution was measured along the cutting line. a) convexe and concave edge b) hillock with convex top and concave surrounding