3D Microscopy and Microanalysis of Heterogeneous SEM Samples by Broad Ion Beam Processing: Cutting - Etching - Coating

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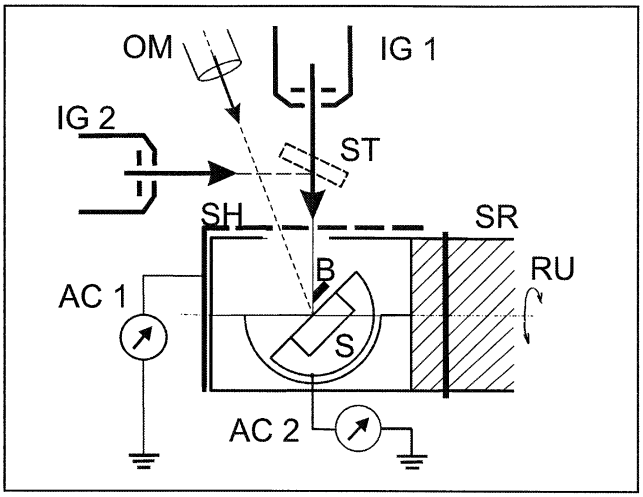
As a versatile tool for investigation of heterogeneous SEM samples ion beam sputtering can be used for various tasks, e.g. for cleaning, smoothing, thinning, etching, and coating. For these applications defined ion beam parameters and a special beam-sample-geometry (including variations during bombardment) have to be chosen to get the wanted result. Most ion beam preparation apparatus were designed for several of these applications mentioned above. However, ion bombardment allows additionally higher sophisticated methods to get information on heterogeneous samples. Ion beam slope cutting (IBSC) [1] can reveal the whole three-dimensional microstructure. Together with the cutting capability new methodical possibilities appear due to combination of cutting with other ion beam processing steps. In most applications cutting will be combined with a short-time selective etching step and in the cases of insulators with sputter coating. For many questions in materials sciences the broad ion beam (BIB) applications have essential advantages in comparison with the focused ion beam (FIB) technology concerning sample material, achievable cutting area size and combination possibilities with other ion beam processing steps as e.g. selective etching and coating. The broad ion beam apparatus Gatan Precision Etching Coating System (PECS) has been modified to fulfill the conditions for new BIB processing steps. Fig. 1 shows schematically the ion guns for cutting, etching, and coating with ion energies up to 10 keV and ion beam densities over 20 $\mu A/mm^2$ with more than 10 mm beam diameter. Sample mounting and blind positioning accuracy can be adjusted by optical microscopy and rocking/rotation is possible program-controlled. The modified PECS has been applied for new applications on 3D investigations of microstructures. Fig. 2 shows a very large ion beam cut through an embossed microstructure in polycrystalline copper (Sample IWU Chemnitz/Germany). The cut area produced with 7 keV Ar^+ ions is 10^4 times larger than a FIB cut. Compared with mechanical grinding and polishing no deformation takes place and no embedding is required. The near-surface region modified by the stamp process is etched selectively with the same ion gun for less than one minute. Figs. 3 and 4 show details of the internal grain structure in the modified region. Fig. 5 shows an example of Low Temperature Co-fired Ceramics (LTCC) technology (Sample TU Wroclaw/ Poland) after the three-step CEC process (Cutting-Etching-Coating) with cutting by the ion beam, selective ion etching of grains, phases and defects followed by AuPd coating. The information gain in all these applications depends on the selective ion etching process and its correct interpretation. The method has an extended field of applications also in microelectronics [2] and its resolution is determined by the SEM and not limited by the ion beam processing steps.

References

[1] W. Hauffe, Production of Microstructures by Ion Beam Sputtering, Chapter 6 in *Sputtering by Particle Bombardment III* (Eds. R.Behrisch and K.Wittmaack), Springer Ser. Topics in Applied Physics, Vol. 64 (Springer-Verlag Heidelberg New York (1991) 305
[2] R. Alani, W. Hauffe , and R.J. Mitro, Microsc. Microanalysis 6, Suppl.2 (2000) 496

Figure 1. Ion guns, sample arrangement and control attachments in the modified broad ion beam apparatus Gatan PECS
IG 1 ion gun for cutting and etching;
IG 2 ion gun for coating;
S sample; B blind; SR sample rod;
RU rocking/rotation unit;
SH shutter; OM optical microscope;
AC 1 absorbed shutter ion beam current;
AC 2 absorbed sample ion beam current;
ST sputter target



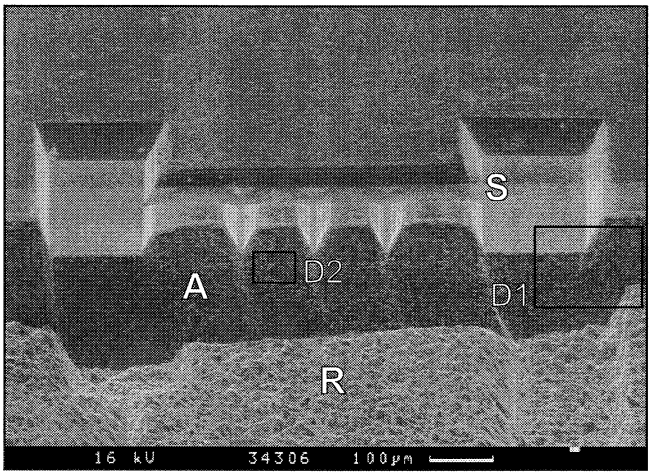


FIG. 1.Ion beam cut through micro-stamped Cu structures. S initial surface structure, R sputtered region, A cut area, D1 and D2 details shown in FIG. 2 and FIG. 3.

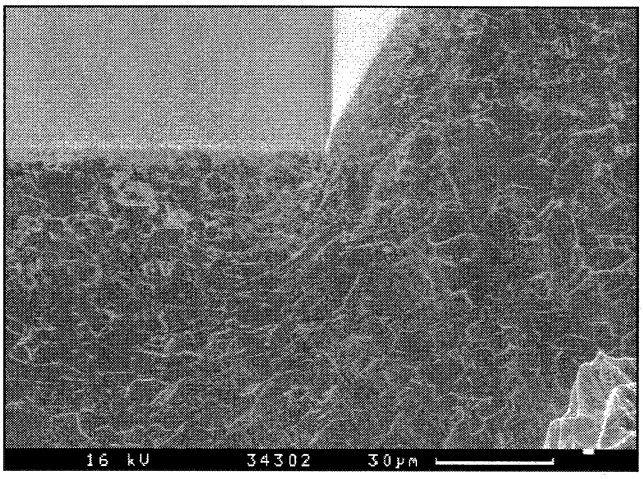


FIG. 3. Detail D1 of the cut area in FIG. 1. Deformed microstructure revealed by selective Ar ion etching.

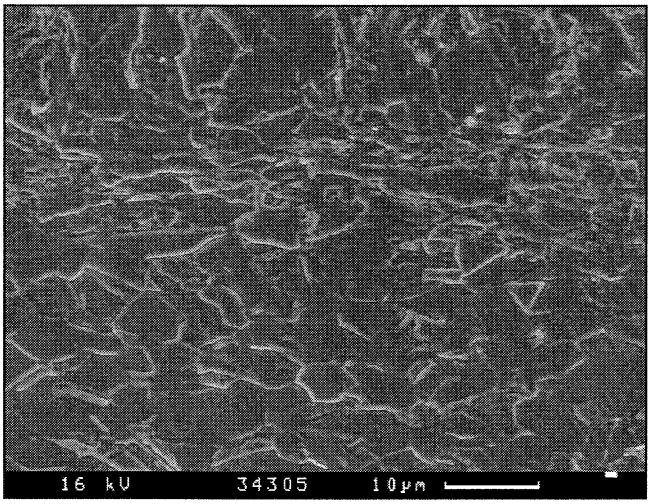


FIG. 4. Detail D2 of the ion beam cut in FIG. 1. Heavily deformed region of the Cu grain structure revealed by the selective ion etching step.

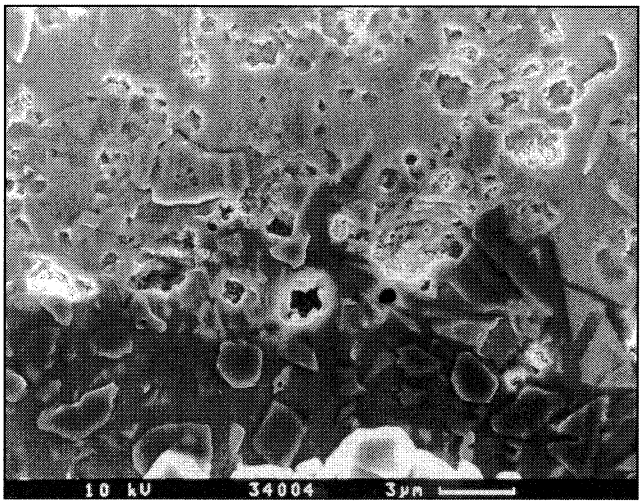


FIG. 5. Ion beam cut through a LTCC system after selective etching and AuPd coating (CEC process).