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A METHOD OF FIM-FEEM SPECIMEN PREPARATION OF SUPERCONDUCTING AND OTHER OXIDES

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Abstract

A new method is described for preparation of oxide specimens for field-ion and field-electron emission microscopy.

The recent worldwide scientific interest in understanding the structure and properties of superconducting oxides has motivated the field-ion microscopy (FIM) and field-electron-emission microscopy (FEEM) research community to investigate these new materials with its special techniques. The preparation of specimens for these studies, from ceramic wafers, has presented a challenge which, in principle, can be met in various ways (1). We wish to report a new method of FIM-FEEM specimen preparation which has proven to be routinely usable for samples of all of the known classes of superconducting oxides to date, and also for a test case of an ordinarily-conducting oxide. We will describe the technique and then comment on its potential benefits and deficiencies in comparison with other methods of specimen preparation.

The starting material is usually a wafer of oxide that has been compacted and heat-treated from a powder mixture (2). A small piece, typically about 2-3 mm on a side, is broken away from the wafer, put into a ceramic or glass bowl and crushed to fragments, using a ceramic or glass pestle (see fig. 1a). The fragments are next sprinkled onto a glass slide which is then clamped in an optical microscope (fig. 1b). Ordinarily, we use x150 magnification. The slide is scanned laterally and searched for sharply-pointed fragments. When one is found, a pointed tungsten "poker", handled with a micromanipulator, is used to do the following: 1) clear away nearby fragments, 2) prod the selected fragment in order to expose it to view from other directions and, if it appears to be acceptably sharp in all directions viewed, 3) prod it into a favorable orientation for subsequent pick-up. Alternatively, the fragment may simply be oriented for pick-up with no prodding, by rotating the glass slide appropriately. After pick-up, in any case, it should be examined in several directions to ascertain sharpness symmetry or lack of symmetry. A small (about 1 mm diameter) drop of freshly prepared electrically conducting adhesive is put onto a rod, about 1 cm long, and placed in an optical microscope with the edge of the drop in focus. This second optical microscope, which we operate at x75 magnification, is then used (see figure 1c) with the micro-manipulator to apply a small amount of the wet electrically conducting adhesive (we are using silver epoxy) to a pre-polished, not-too-sharp (-500-1000 nm diameter, for example) metal point which is attached or can be attached to the FIM-FEEM specimen holder. The adhesive-laden point is then guided into position just above the selected oxide fragment, in the first optical microscope, using the micro-manipulator, as in fig. 1d, lowered carefully to contact the fragment with the adhesive, and then raised. Next, the fragment-specimen is clamped in an optical microscope and examined. If it is mis-aligned relative to its support point, an
L-shaped pointed wire is introduced in the microscope, using the micro-manipulator, configured so that its point is directed upward. When this tool is adjusted, it appears as a dark dot in the microscope, that is, only some cross-section of the tapered point is in focus (figure le). The tool is then used to prod the fragment so that can also be assessed during this procedure. If acceptable, the specimen is set aside, preferably in a dry, dust-free environment, to allow the adhesive to harden. We usually allow about 10-12 hours for this, although shorter times may suffice. For additional bonding, it is possible to add more adhesive after the first has hardened, using a micro-manipulator and an adhesive-laden point. An example of an array of fragments is shown in Fig. 2 and a mounted specimen is shown in Fig. 3.

No chemical, electrochemical or other treatment of the specimen is needed. The size range of fragments which we have used usually was about 0.01-0.10 mm, usually with apex radii of curvature less than 50 nm, and sometimes less than 20 nm. We have successfully imaged in the FIM, using this method of specimen preparation, superconductor specimens of the La-Sr-Cu-O type (3), the R-Ba-Cu-O ("1,2,3") type (with R=Y, Yb, Sm, Gd, Dy, Er, Pr, Eu, Ho or La) (3), the Bi-Ca-Sr-Cu-O type (4), the Tl-Ba-Ca-Cu-O type (4) and the (not-superconducting oxide La-Cr-O. Micrographs showing an array of YBa$_2$Cu$_3$O$_{7-x}$ fragments on a glass slide and an epoxy-mounted fragment on a tungsten support are displayed on figure 2.

An obvious weakness of this method of specimen preparation is the fact that its success depends upon the fracture mechanics of the initial oxide, and indeed we have occasionally encountered samples which did not produce usable specimens. Also, the orientations of the specimens depend on fracture mechanics. The potential benefits of the technique, however, make it very useful. Only a small amount of sample, typically about 10 cubic mm, suffices to provide many FIM specimens. Specimen preparation is rapid; for example, we have imaged material about 15 hours after receiving the wafer sample. The average time for doing the procedures illustrated in figure 1 is about 10-20 min., and the adhesive setting time is then the rate-limiting step. This time, in principle, can be reduced significantly. The most important benefit of using this technique may well be the fact that no extraneous chemicals are introduced, for example by grain-boundary diffusion, and that no selective loss of elements occurs due to chemical or electrochemical interactions.

A preliminary description of this method of specimen preparation and further examples of its use have been reported (5,6).

References
Figure 1: A method of specimen preparation.

(a) Producing fragments.

(b) Finding sharply-pointed fragments, clearing away other fragments and testing for symmetry, using poker (P).
(c) Applying electrically conducting adhesive to a bluntish point (Y).

(d) Picking-up a fragment.
Adjusting the position of a fragment, if necessary (also, testing for symmetry, adding more adhesive, etc.).

Figure 2: Optical micrograph of $Y_{123}\text{Cu}_3\text{O}_{7.5-}\text{X}$ fragments on a glass slide. The scale marker line represents 0.1 mm.

Figure 3: Transmission electron micrograph of a $\text{GdBa}_2\text{Cu}_3\text{O}_{7-\text{X}}$ fragment attached to a tungsten support point (blunt) by conducting silver epoxy. The fragment length is about 0.019 mm. (Micrograph courtesy H. B. Elswijk.)