Sample Preparation of YBa$_2$Cu$_3$O$_{7-δ}$ for High-Resolution Electron Microscopy

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An overview is given of three techniques for preparing thin specimens of YBa$_2$Cu$_3$O$_{7-δ}$ for high-resolution electron microscopy: grinding, cleaving, and ion milling. Advantages and disadvantages are described, with particular attention to the artefacts that may be introduced by the different techniques. It is concluded that the most serious problem encountered during high-resolution electron microscopy studies is a surface-initiated decomposition leading to the ultimate degradation of the structure. This problem was found to be most pronounced in ion-milled specimens.

KEY WORDS: YBa$_2$Cu$_3$O$_{7-δ}$; high-resolution electron microscopy; sample preparation.

1. INTRODUCTION

Specimens for high-resolution electron microscopy must be clean, thin, and flat in order to guarantee that phase contrast images can be properly interpreted. Cleanliness is needed to avoid background signals from surface overlayers or oxide films. Sufficiently strong background, even from amorphous layers, can completely disrupt a phase-contrast image [1]. Specimen thickness should be controlled to maintain the image formation process within the linear regime [2] for accurate interpretation. This requires thicknesses no greater than one extinction distance for primary scattering by the lowest-order Bragg reflection (e.g., copper must be less than 24 nm thick, while aluminum could be 55 nm thick). Finally, flatness, is essential if proper orientation is to be preserved over large image areas.

Unfortunately this combination of characteristics induces competition in the sample preparation process. Thin samples are less likely to remain...
flat due to bending (or fracture) under the beam, and they are much more susceptible to image artefacts from contamination overlayers that might be introduced by careless handling or exposure to reactive atmospheres. Surface cleaning, often performed by ion milling, can introduce other artefacts due to local damage or heating. Finally, the actual observation of the specimen might also be a source of electron irradiation damage which the microscopist must recognize. Since the objective of any preparation activity is the production of a representative sample, all phases of the process are critical.

These considerations place stringent requirements on the preparation of the new oxide superconductors for examination by high-resolution electron microscopy. The materials as a class are brittle, often examined in powder form, notorious for variations in oxygen stoichiometry, and complex in structure. In this paper an overview and comparison of specimen preparation techniques is presented for the specific case of YBa$_2$Cu$_3$O$_{7-\delta}$.

2. DESCRIPTION OF TECHNIQUES

2.1. Mechanical Grinding

This procedure is both rapid and simple, and a large number of variations are possible. Beginning with a pellet fragment or coarse powder of YBa$_2$Cu$_3$O$_{7-\delta}$ compound, the material is ground to a fine powder using a hard (e.g., agate) mortar and pestle. Porcelain vessels are not recommended because any surface abrasion can introduce impurities into the powdered sample. After grinding, the powder is suspended in an inert liquid, where particle dispersion can be facilitated by various agitation methods (e.g., ultrasonic). A few droplets of the suspension are then put on a carbon-coated thin film and transferred immediately into the microscope. An example of a dispersion of crystals on a holey carbon film is given in Fig. 1.

Variations in this basic procedure are used to give control over the final specimen morphology. For example, if the 1-2-3 compound is partially ground, rapidly cooled in liquid nitrogen, then further ground in the suspension medium, the powders are more flakelike, due to preferential fracture along c planes. This causes them to settle onto the carbon film with a strong c-axis texture. Similar results are obtained in $\beta$-alumina type materials [3]. By changing the type of suspension fluid and/or the settlement time after agitation, the size range of the selected powders can be varied. After settling, the suspension contains more fine particles at the top of the fluid, and coarse particles at the bottom.

The support on which the suspension is deposited can be either a continuous film or a holey carbon-coated film covering a metal grid. In the latter case, adjustment of the size of the holes leads to size selectivity; larger