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Current Developments in TEM and SEM Sample Preparation

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Abstract Materials characterization by TEM or SEM requires preparation of cross sectional samples that are representative of the bulk state, atomically clean and free of physical and chemical artifacts. Rapid and reproducible results are achieved with TEM specimens when mechanical sectioning and grinding are combined with argon ion milling, and followed by argon - oxygen plasma cleaning. The same treatments can be applied to SEM samples and may further include reactive ion etching and ion beam sputter coating. Analytical results obtained using FETEM or FESEM for various materials will be discussed.

Introduction The initial process in producing a specimen for TEM is sectioning the bulk material by sawing or cleaving. TEM specimens are thinned mechanically until the region of interest is ideally less than 10 mm thick. Argon ion etching at low incident angles, low ion energies and liquid nitrogen temperature is used to further thin the region of interest to electron transparency. Material is removed from the specimen by means of momentum transfer from the impinging incident argon ions. Plasma cleaning with Ar - 25%O2 removes residual organic contamination from the specimens. Potential sources of contamination are mounting compounds, cutting and grinding media, and specimen handling. The hydrocarbons are removed by chemical reduction to CO, CO2, H2O. [1] A plasma cleaned specimen may be imaged or analyzed (EDX, PEELS) for an extended time in a FETEM without recontamination.

Plasma cleaning is also an important tool for the final processing of SEM samples. Whether mechanically sawed or cleaved, the sectioned material may contaminate during imaging if not plasma cleaned beforehand. Current practice is first to plasma clean, then to argon ion etch in order to produce a planar surface free of physical damage. [2] This surface is then etched with reactive ions to enhance the topography in selected areas within the microstructure. An ionized gas consisting of fluorine, chlorine, bromine or oxygen may be used depending on the particular materials contained in the microstructure. The result is increased image contrast. The image quality may be further improved by ion beam sputter coating. Amorphous, electrically conductive and thin (< 2 nm) layers of carbon or certain metals (Cr, Ir, Pt, Ta, W) are preferred.

Results A cross sectional TEM specimen of LaTiO3/SrTiO3 was prepared by mechanical sectioning and grinding, followed by ion milling (Ar), and plasma (Ar - 25%O2) cleaning, Fig. 1 [3].

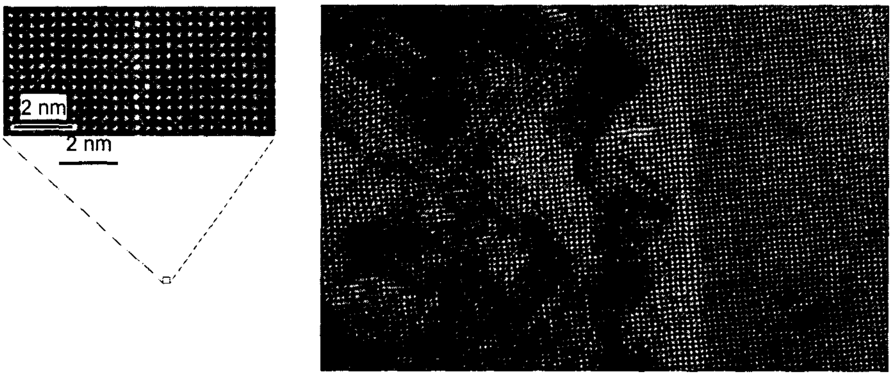

Fig. 1: High angle ADF STEM images of LaTiO3/SrTiO3 after ion etching (Ar) and plasma cleaning (Ar - 25%O2) at lattice (left) and atomic (right) resolution

This material was cooled to liquid nitrogen temperature during argon ion milling to retain the complex microstructure. The interfacial area was studied using a FETEM equipped with a high angle, annular dark field (ADF) STEM detector. Atomic resolution images were obtained. The image contrast scales with atomic number (Z) such that the La atomic columns appear brighter and Sr appears darker. The spacing between the columns of Sr and Ti was ~ 1.9 Angstroms.

A copper (Cu) based microelectronic material was mechanically cross sectioned to produce 4 mm by 4 mm by 750 mm samples for SEM. One edge of the cross section was ground to a 0.05 mm surface finish. This surface was first plasma (Ar- 25%O2) cleaned, then ion etched (Ar) at a low (10o) incident angle. This planarized surface was reactive ion etched with CF4-10%O2 to produce topography between the Si and SiO2, then coated with 1 nm of Pt. The total process time following sectioning and grinding was 17 min. The cross section was imaged in a FESEM at 2 kV before and after the combined treatment, Fig. 2. A mixed signal of secondary and backscattered electrons was used for the imaging.

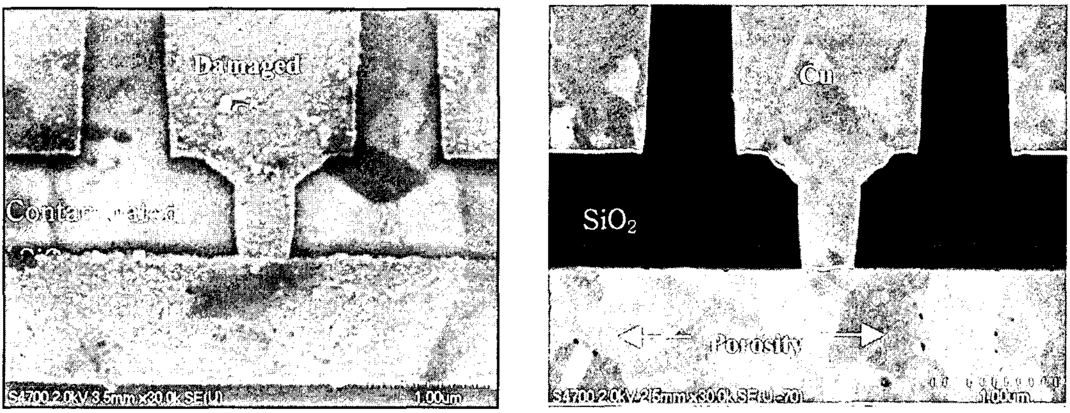

Fig. 2: SEM cross section of a Cu based microelectronic material before (left) and after (right) the combined treatment of plasma cleaning (Ar - 25%O2), ion milling (Ar), reactive ion etching (CF4 -10%O2) and ion beam sputter coating (Pt, 1 nm)

High resolution imaging of the copper areas revealed grain boundaries and residual porosity from the interconnect fabrication process. This porosity was associated with boundary triple points and twin boundaries. Interlayers were identified between the Cu and SiO2.

References

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