Specimen Preparation For Electron Microscopy Of Materials On The Nano And Atomic Scales

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The initial process in producing a specimen for FETEM is sectioning the bulk material by sawing or cleaving. Such specimens are thinned mechanically until the region of interest is ideally less than 10 µm 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 handling. The hydrocarbons are removed by chemical reduction to CO, CO2, H2O. [1] A plasma cleaned specimen may be imaged or analyzed for an extended time without recontamination.

A cross sectional FETEM specimen of LaTiO3/SrTiO3 was prepared by mechanical sectioning and grinding, followed by ion milling (Ar), and plasma (Ar . 25%O2) cleaning, Fig. 1 [2]. 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.

Plasma cleaning is also an important tool for the final processing of FESEM specimens. 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. [3] 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, or oxygen may be used depending on the particular materials contained in the microstructure. A second plasma cleaning often follows. 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.

A copper (Cu) based microelectronic material was mechanically cross sectioned to produce 4 mm by 4 mm by 750 μm specimens for FESEM. One edge of the cross section was ground to a < 0.1 μm 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 substrate, fine grained (or "Poly") Si, and SiO2. The sample was 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 after the combined treatment, Fig. 2. A mixed signal of secondary and backscattered electrons was used for the imaging. High resolution imaging of the Cu and tungsten (W) phases revealed grain boundaries, and residual porosity in the case of the Cu.

Differential contrast was evident for all of the nonmetallic phases, including the thin (~ 1.5 nm) gate oxide detected at the transistor level.

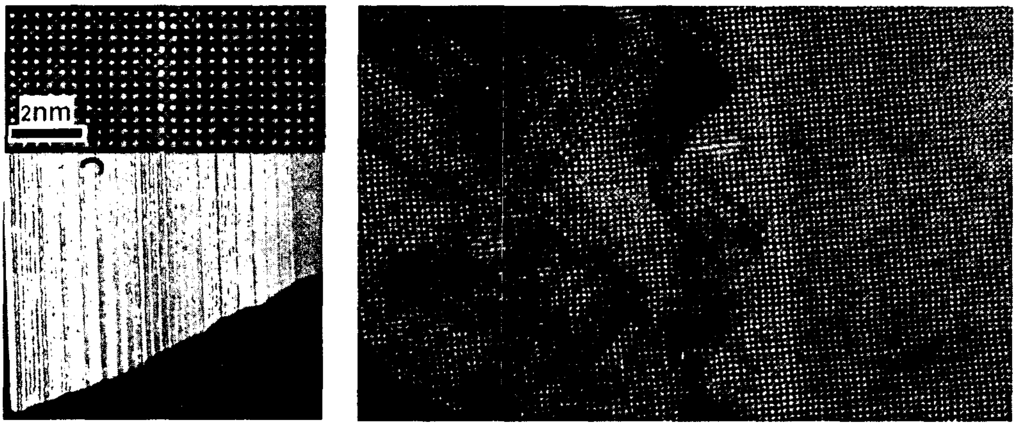

Fig. 1. High angle ADF STEM images of LaTiO3/SrTiO3 after ion etching (Ar) and plasma cleaning (Ar . 25%O2) at nano (left) and atomic (right) resolution

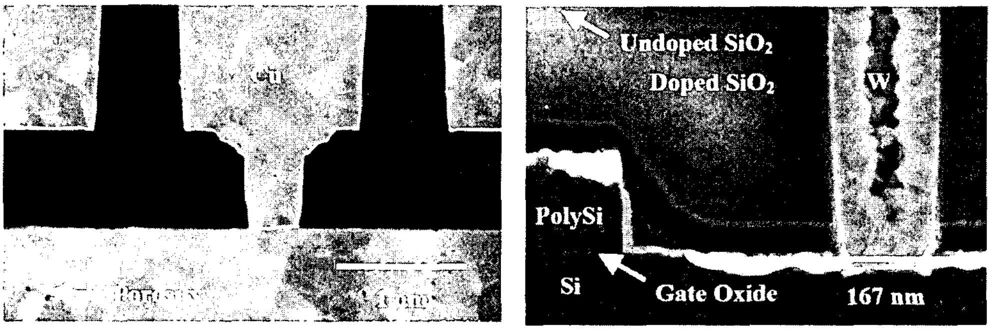

Fig. 2. FESEM of nanoscale features at the interconnect (left) and transistor levels after a combined treatment of plasma cleaning (Ar . 25%O2), ion beam etching (Ar), reactive ion etching (CF4 - 10%O2) and ion beam sputter coating (Pt, 1 nm)

1. T. C. Isabell et al., “Plasma Cleaning and Its Applications for Electron Microscopy”, Microsc. Microanal., 5, 126-135 (1999).

2. D. A. Muller, A. Ohtomo, J. Grazul, H. Y. Hwang, “Artificial Charge Modulations in La - Doped SrTiO3 Superlattices”, Microsc. Microanal. 8 (Suppl. 2), 2002, 576CD.

3. R. R. Cerchiara, P. E. Fischione, J. J. Gronsky, W. F. Hein, D. D. Martin, J. M. Matesa, A. C. Robins and D. W. Smith, “Recent Developments in Automated Sample Preparation for FESEM ”, Proceedings of the 29th International Symposium for Testing and Failure Analysis, Nov. 2-6, 2003, Santa Clara, CA, pp. 288 . 300.