FINAL REPORT

Structure of Ferroelectric Superconductor Interfaces

by

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Subcontract Number : 9-XU3-7595J-1

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OBJECTIVE

To carry out detailed Transmission Electron Microscopy (TEM) analysis of a multilayered film whose desired configuration is shown schematically in Figure 1.

Figure 1. Schematic of desired multilayered film (as provided by Dr. Ross Muenchausen).

RESULTS

TEM Sample Preparation

The Si wafer was sliced into two pieces and glued into a sandwich such that the film surfaces were back to back (separated by a glueline). Cross sectional TEM samples were then sliced from the sandwich and mounted on Cu grids. The samples were polished to a thickness of 50 μm and then mechanically dimpled such that the remaining thickness of the sample in the region of the coating was of the order of 10 μm . The samples were then ion milled to perforation in a GATAN Duomill, using 5.5 KV ions at 8 degree incidence angle.

The samples were then examined by a JEOL 200CX TEM. High resolution lattice imaging was carried out using an Akashi 002B High Resolution Electron Microscope (HREM) while chemical analysis was carried out using a VG603 dedicated Scanning Transmission Electron Microscope (STEM).

TEM Observations

Figure 2 shows a low magnification TEM micrograph of the cross sectional sample. The multilayered film was separated from the silica substrate by a thick (~500 nm SiO_2 (silica) layer. Figure 2 shows the position of the two silica layers and the back to back films separated by a thin glue line (as marked by arrows).

Figure 2. Low magnification TEM micrograph of sample.

Figure 3 shows a digitized image of one side of the sandwiched sample at a slightly higher magnification. The white area at the bottom is the silica layer, while that at the top is the glue line. The multilayered coating itself shows regions of three distinct contrasts, a light gray bottom layer, a black central layer and a dark gray top layer. Figure 4 shows a high resolution image of the Si- SiO_2 interface.

Figure 3. TEM micrograph of coating showing positions from which EDX spectra were collected.

Figure 4. HREM micrograph of the Si/SiO₂ interface.

Figures 5a to 5d show EDX (energy dispersive x-ray spectroscopy) spectra from regions marked as 1, 2, 3 and 4 by arrows in Figure 3. The spectra were collected in the STEM using a 1 nm diameter electron probe.

1

Figure 5a. EDX spectrum from region 1 in Figure 3.

2

Figure 5b. EDX spectrum from region 2 in Figure 3.

3

Figure 5c. EDX spectrum from region 3 in Figure 3.

4

Figure 5d. EDX spectrum from region 4 in Figure 3.

Figure 5a shows that the light gray bottom layer in Figure 3 is essentially metallic Ti, not TiO_2 , due to the lack of an O peak in the EDX spectrum. This is further confirmed by the HREM micrograph of the silica/Ti interface, as shown in Figure 6. The figure shows the Ti layer to be a randomly oriented and polycrystalline. The enlarged inset micrograph in Figure 6 shows the c-axis orientation of the HCP Ti grain.

Figure 6. HREM micrograph of the silica/Ti interface.

Figure 5b shows that the central black layer is Pt with a small concentration of Ti. The Si and Cu peaks are due to scattering from the Si substrate and Cu grid respectively. The presence of Ti in the Pt layer was further confirmed by elemental distribution maps of the film constituents shown in Figure 7. The Ti map shows the presence of approximately 25 nm sized Ti islands in the Pt layer.

Figure 5c is an EDX spectra at the interface of the Pt and PZT layer. The figure shows that apart from scattering by the Pt and PZT layers as well as from the Si substrate and Cu grid, there are peaks of Co and La. The lack of a substantial O peak indicates that this layer is not an oxide. This was confirmed by the elemental dot maps in Figure 7, that clearly shows a layer of La and Co between the Pt and PZT layers. The thickness of this La-Co layer is approximately 25nm.

Figure 7. Elemental distribution map of multilayered film.

Figure 5d is an EDX spectrum from the dark gray top layer in Figure 3 (area 4), identifying it to be PZT. Again, the Si and Cu peaks are due to scatter from the substrate and grid respectively. Figure 8 is a brightfield TEM micrograph of the multilayered film, showing the various layers. The 3000Å PZT film clearly has two distinct layers. The bottom half of the PZT layer is clearly crystalline. Selected area diffraction (SAD) showed this to be randomly oriented polycrystalline in nature. The figure also shows the presence of a few larger columnar grains, one of which is marked by an arrow. The upper portion of the PZT film is amorphous as confirmed by the SAD diffraction pattern from this region. EDX spectra from various regions of the film did not show any significant composition differences within and between the crystalline and amorphous PZT regions.

Figure 8. TEM brightfield micrograph of multilayered film, showing the crystalline and amorphous regions in PZT.

Figure 9 is a HREM micrograph of a region in the crystalline portion of the PZT film. The presence of a twin can be clearly seen in the figure and is marked as T in Figure 9.

Figure 9. HREM micrograph of PZT film.

In conclusion, the observed microstructure of the multilayered film was somewhat different than anticipated and is summarized in a schematic in Figure 10.

Figure 10. Schematic of the observed multilayered film microstructure.