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# APPLICATION OF FIB AND TEM FOR THE CHARACTERIZATION OF DEWETTING BEHAVIOR ON CERAMICS

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The Focused Ion Beam (FIB) tool is proving to be invaluable for specimen preparation for transmission electron microscopy (TEM) observations[1]. Systems with both an electron source and ion source (dual beam) are ideal for preparing specimens from specific sites. While site-specific sample preparation is possible with an ion source alone, the use of an electron source enables the viewing of the sample without simultaneously milling it. In the present study, a dual-beam FIB has been utilized to prepare samples of the interface between glass and alumina. Impurities/additives, which form glassy phases at high temperature, are often incorporated in ceramics and aid in the sintering process (liquid-phase sintering). The manner in which the glass wets the crystalline ceramic dictates the microstructure and therefore the properties of the material. The wetting behavior of anorthite glass, $CaAl_2Si_2O_8$ (CAS), on $(10\bar{1}0)$ (m-plane) $Al_2O_3$ surfaces is a model system being studied to understand the complexities involved in the wetting/dewetting process[2-4]. The m-plane surface is unstable and reconstructs into a hill and valley structure during heat treatment.

Thin films (~100 nm) of CAS glass are deposited by pulsed-laser deposition on $(10\bar{1}0)$ single crystals of alumina. The thin film/alumina system is then annealed at high temperature to form a liquid phase. On cooling the liquid dewets the faceted alumina surface. Samples are characterized by low-voltage field-emission SEM (Hitachi S-900 operated at 5 kV) and TEM (Philips CM30 and Tecnai F30-ST operated at 300 kV). TEM specimens were prepared by a dual beam FIB (FEI DB235). Figure 1 is an SEM image of a dewet glass droplet on the m-plane of alumina. The glass preferentially dewets the reconstructed surface. The darker facets in figure 1 correspond to facets that are wet by the liquid. Figure 2 is an SEM image taken in the FIB, in which a specific region has been selected. This region includes a single dewet droplet and adjacent facets. The strip of protective Pt indicates the region from which the FIB was cut. Figure 3 is a BF TEM image and corresponding SAD pattern of a specimen prepared in such a manner. This image, taken with the electron beam parallel to the zone, shows the interface between the alumina surface and the dewet droplet. The serrated structure in Figure 3 is a result of the $(10\bar{1}0)$ surface reconstructing into $(10\bar{1}\bar{2})$ and $(10\bar{1}1)$ facets during the heat treatment. Figure 4 is a BF TEM image of a region of facets near a droplet. Glass can be seen in this image preferentially wetting the $(10\bar{1}1)$ facet.

The FIB is a powerful tool in the preparation of TEM specimens with distinct surface morphologies. In the case of investigating the dewetting behavior of glass on ceramic surfaces, the difference in hardness between the glass and alumina makes conventional polishing methods for sample preparation difficult. The thin film of glass (~ 2 nm in thickness) apparent in figure 4 would be nearly impossible to see if conventional preparation methods had been employed. Also, the thickness of these samples is uniform across the entire region of interest, avoiding the limitations associated with traditional wedge-shaped specimens.

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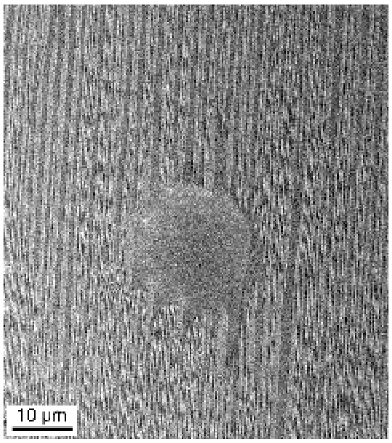


Fig. 1 SE image of CAS glass that has dewet the reconstructed m-plane of alumina.

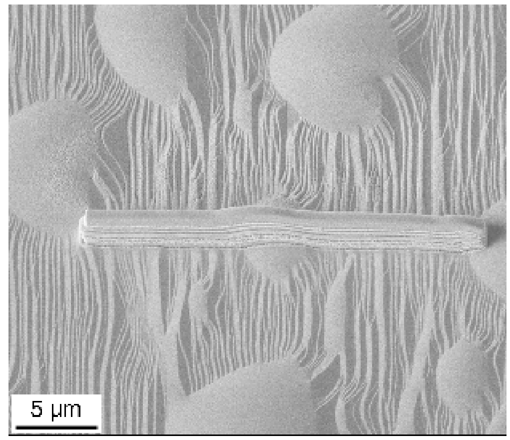


Fig 2. SE image taken in the FIB. The protective Pt overlayer can be scene on the area of interest. This area includes a dewet glass droplet and adjacent facets.

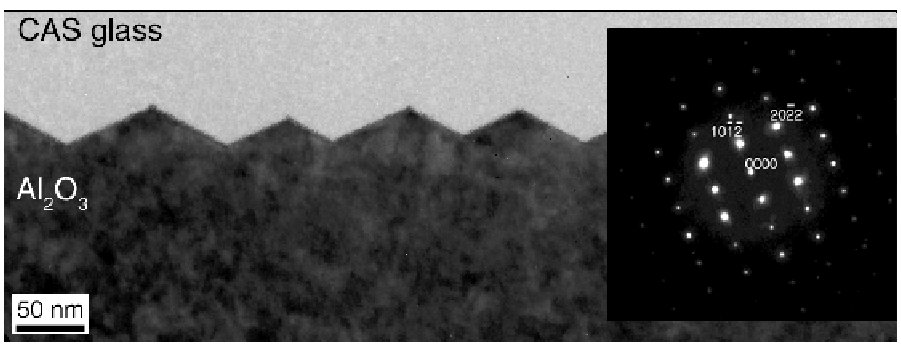


Fig. 3 BF TEM image and corresponding SAD pattern of glass/alumina in terface within a droplet. The serrated facets correspond to the (10$\bar{1}\bar{2}$) and (10$\bar{1}$1) planes.

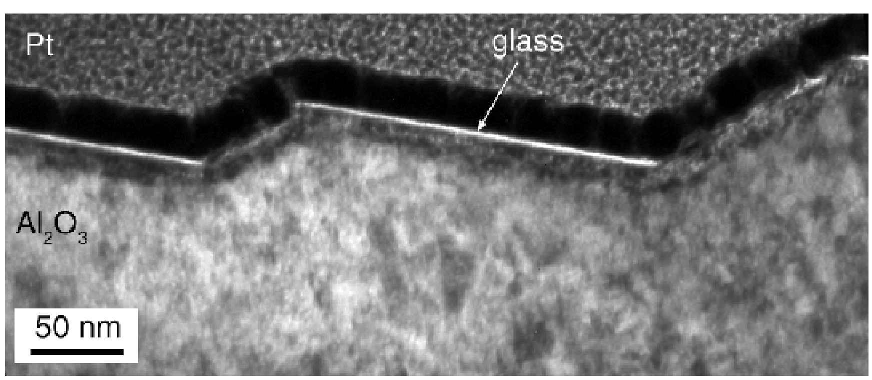


Fig. 4 BF TEM image of facets near a droplet. The glass can be seen wetting only the (10$\bar{1}$1) facet.

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