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Ultrahigh Temperature *In Situ* TEM Based Small-scale Mechanical Characterization

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The thermal, mechanical, and microstructural stability of materials in extreme environments often depend strongly on interfacial transport processes occurring in response to stresses. The complex nature of polycrystalline microstructures presents challenges for characterizing interfacial transport mechanisms in bulk materials, particularly at grain boundaries and solid-solid phase boundaries. Small-scale mechanical testing at room temperature has provided a wealth of information enabling improved understanding of plasticity in materials by isolating microstructural features of interest [1]. Application of localized laser heating extends similar capabilities to the high and ultrahigh temperature regimes, where the diffusional response of materials may be characterized readily [2]. For example, bicrystal tensile creep experiments were recently employed to characterize the temperature dependence of grain boundary diffusivity, the surface energy, the activation volume for grain boundary mediated creep, and the grain boundary diffusion mediating point defect volume [3, 4]. The stress coupling in these experiments provides a valuable way to probe various aspects of the thermodynamics and kinetics of the material at the scale of individual boundaries. Similar methods have recently been extended to characterize the diffusivity at solid-solid phase boundaries [5].

Prior work assumed that the electron beam did not significantly influence the measured kinetics and thermodynamics based on reported thresholds for electron beam damage in the systems of interest. The question as to the role of the electron beam in affecting diffusional kinetics has been raised when discussing this work. To address this concern, experiments like those in reference [4] were implemented at $T \approx 1900$ ^{o}C, wherein tensile bicrystal Coble creep was performed under conditions of constant displacement rate, 10 nm s^{-1}. In these experiments, however, the electron beam was turned on and off during the duration of the experiment using electrostatic beam blanking in the I^3TEM at the Center for Integrated Nanotechnologies at Sandia National Laboratories, which is a highly modified JEOL 2100 LaB_6. Figure 1 shows an example of Sc_2O_3-doped ZrO_2 bicrystal tensile creep that results in the formation of two nanowire structures that result from two initial contact points/grain boundaries. This structure forms as surface diffusion drives a flux into the grain boundaries loaded under tension. The width of the nanowire is determined by the grain boundary diffusivity normalized to the applied stress driving force. Since the grain boundary plane orientation, i.e. grain boundary anisotropy, surface anisotropy, and instantaneous stress state will vary during the experiment, there is some variation in the specimen geometry with time. The width of the nanowires, nevertheless, are reasonably consistent. Furthermore, the presence of the electron beam flux at 200 keV does not appear to influence the overall evolution of the structure. This is consistent with the expectation that 200 keV electrons fall below the damage threshold of 1000 keV measured previously [6]. Performing experiments at low magnifications, such as those shown here and in prior work, could also help avoid electron beam induced damage. Ionization and electrostatic charging might play a more important role at higher magnifications, especially at higher temperatures where ions can diffuse in response to electrostatic gradients.

The results in Figure 1 suggest a negligible effect of the electron beam on kinetics observed *in situ* at high temperatures in systems where the accelerating voltage is below the threshold and where the samples do not experience significant electrostatic charging. In this talk, these experiments are extended to understanding the mechanisms for interfacial transport. Of particular interest is the question as to how capillary process can overcome large activation barriers during processes governing microstructural evolution. The *in situ* measurements provide new insights into answering this question [7].

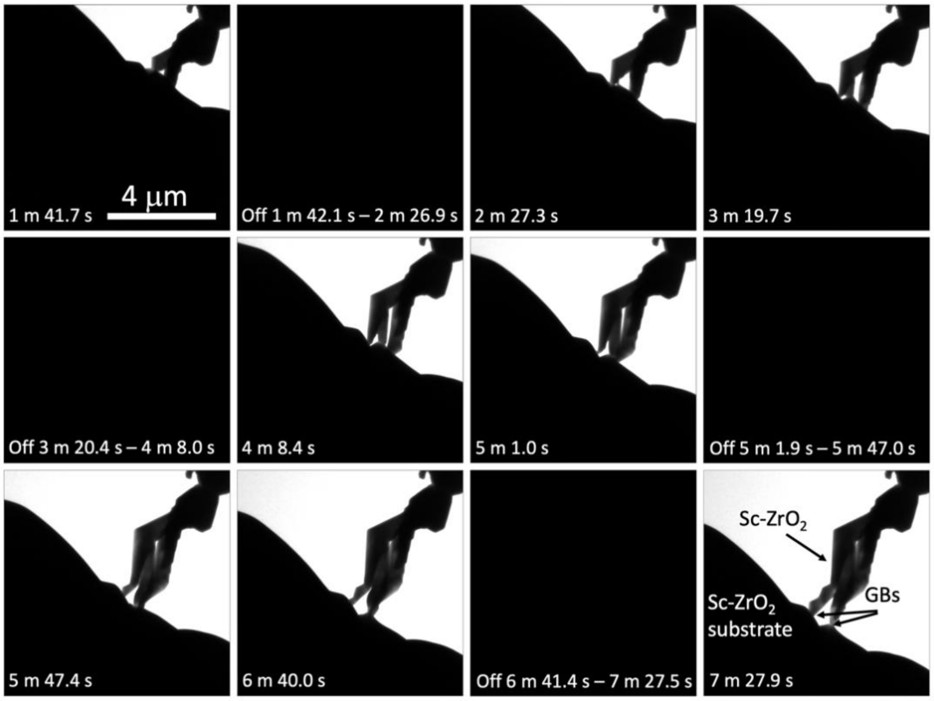

Figure 1. Time-lapse *in situ* TEM images of grain boundary GB) tensile creep performed on Sc_2O_3-doped ZrO_2 at 1900 ^{o}C at a rate of 10 nm s^{-1}. The electron beam was blanked for the periods of time indicated, but the creep response persists while the electron beam is not impinging the sample.

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