

ROOM TEMPERATURE PLASTICITY IN SUB-MICROMETER THERMALLY GROWN OXIDE SCALES

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Thermally grown oxides (TGOs) are generally considered to be brittle, capable of sustaining very limited plastic deformation before fracture. As they are prone to exhibit different forms of defects, the fracture toughness, typically measured to be some 1–2 MPa m^{1/2} [1], is typically reached well before sufficiently high stresses to induce plasticity can be applied [2]. This is particularly true at room temperature, where possible low-stress thermally activated creep mechanisms are suppressed. However, the occurrence of plasticity in e.g. Al₂O₃ single crystals at room temperature can occur for samples in the micrometer range [3]. Most measurements of the deformation of TGOs have been made on relatively thick scales, (>1 micrometer), which are limited by the fracture originating from inherent defects. Furthermore, the studies have been limited in resolution and sensitivity, as the scales were adherent to the substrates and tested as a composite. Recently, micro-mechanical testing has been introduced as a method to evaluate mechanical behavior of TGOs on a ferritic/martensitic steel [4], where micro-cantilever bending was used to test specimen extracted from different layers in a 5–10 micrometers thick oxide. Still, the cantilever cross-section was typically several micrometers, and the very similar fracture stresses for notched and un-notched cantilevers seems to indicate that the deformation is still limited by inherent defects.

Here we present new experimental methods to assess the mechanical behavior of thin TGO scales, with thicknesses in the order of a few hundred nm, grown on a commercial Ni-base superalloys. We present several experimental approaches, all based on micro-cantilever bending in a scanning electron microscope, and discuss the advantages and disadvantages with each method. Finally, we demonstrate how specimens with a nominal thickness in the order of 200 nm, which are manufactured to be detached from the superalloy substrate and free of residual stresses, can be subjected to significant plastic deformation at room temperature, without fracturing. The possibility of accurately measuring properties such as true fracture strain of TGO scales of all thicknesses is essential as input to more sophisticated mechanistic based predictive methods for oxide failure [5].

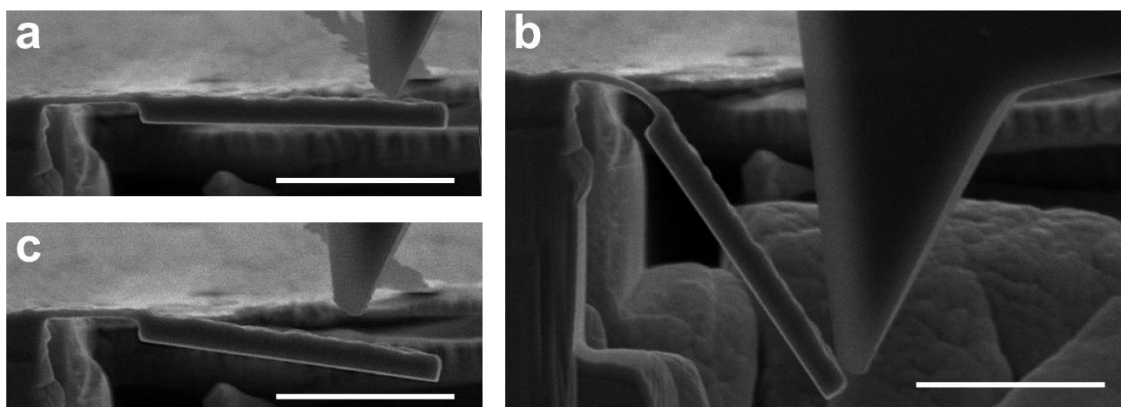


Figure 1 – Bending of a cantilever with a ~200 nm thick TGO. (a) Before test; (b) at maximum deflection; and (c) after unloading, showing permanent deformation. All scale bars are 5 micrometers.

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