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Advances in TEM *in Situ* Mechanical Testing for Nuclear Alloys

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The objective of this work is to present recent advancements in transmission electron microscopy (TEM) *in situ* depth-sensing mechanical testing of nuclear and irradiated materials. This work specifically emphasizes those advancements enabled by the US Department of Energy (DOE) Nuclear Science User Facilities (NSUF). TEM *in situ* depth-sensing mechanical testing offers the potential for gaining unprecedented insight into mechanical behaviors of materials because it uniquely couples quantitative mechanical measurements with qualitative microstructure-scale observations of plastic phenomena. The nuclear and irradiated materials research community has a growing interest in TEM *in situ* mechanical testing because material volumes less than a cubic micrometer can be tested. Such a configuration could open unprecedented opportunities for accelerating material qualification by ion irradiation, or reducing the cost of in-pile neutron irradiations.

Initial TEM *in situ* mechanical testing work on irradiated materials has focused on simple specimen geometries, such as compression pillars [1,2]. Compression pillars are easy to fabricate by focused ion beam (FIB) milling, and can provide meaningful measurement of yield stress [1,2], elastic modulus when coupled with finite element method (FEM) models [2], and strain hardening coefficient [3]. Size effects arise when reducing the mechanical testing volume, but the high number density of irradiation-induced defects sufficiently confines the plastic zone and enables meaningful quantitative mechanical properties to be measured using TEM *in situ* methods [4].

More recently, however, novel TEM *in situ* mechanical testing configurations have been demonstrated on irradiated materials. This study will describe these recent advancements in four-point bend fracture testing, tensile testing, and mechanical testing intermitted with TEM phase mapping.

Fracture properties can be probed using a four-point bend configuration (Figure 1a), and has been demonstrated on a model Fe-9Cr oxide dispersion strengthened (ODS) alloy in the as-received, proton irradiated, and self-ion irradiated conditions. The irradiated specimens exhibit a more abrupt and brittle fracture behavior, with flat fracture surfaces, whereas the as-received specimens exhibit more ductile fracture surfaces. Because traditional fracture mechanics breaks down at nanoscopic length scales, we utilize extended finite element method (XFEM) modeling to calculate J-integral values from notch length propagation observed from the TEM *in situ* videos. These J-integral values provide an assessment of the relative fracture toughness values of the specimens, and reflect the more brittle fracture behavior of the irradiated ODS compared to the as-received ODS.

Tensile testing is also conducted on electron-transparent tensile bars (Figure 1b). The TEM *in situ* tensile testing is demonstrated on a commercial FeCrAl alloy C37M (nominally Fe-13Cr-7Al), in both as-received and self-ion irradiated condition. Tensile bars are single crystalline and have orientations of [100], [110], and [111]. Dislocation-mediated plasticity is observed in all of the [110] and [111] specimens, but an inhibition of dislocation activity is observed in the [100] specimen. These results are consistent with molecular dynamics models, which suggest that numerous slip systems are active in [110] and [111] oriented single crystals, but twinning is the dominant deformation mechanism in [100] oriented crystals.

Finally, TEM *in situ* lamella indentation [5] has been intermitted with precession electron diffraction (PED) to investigate precipitate and phase evolution. This work is demonstrated on Cu-10Ta nanocrystalline alloy in the as-received and a proton irradiated condition (Figure 1c-d). The intermitted PED reveals dynamic precipitation and growth of Ta-rich phases in the irradiated specimen during TEM *in situ* indentation loading, but the as-received specimen does not exhibit dynamic precipitation. The cause of this difference remains to be understood, but this coupling of TEM *in situ* mechanical testing with intermitted PED presents tremendous potential for providing unparalleled new insight into the dynamic nucleation process.

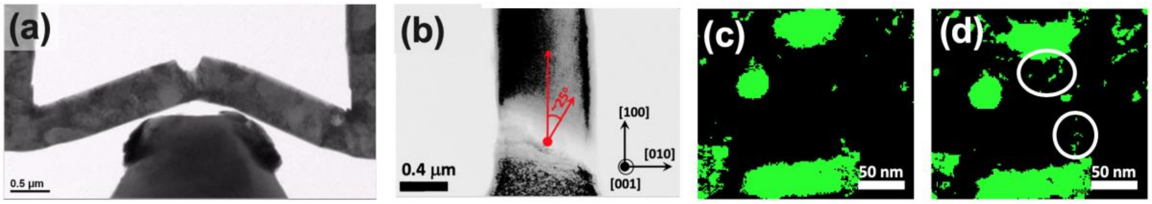

Figure 1. (a) TEM in situ four-point bend fracture testing of self-ion irradiated Fe-9Cr ODS; (b) TEM in situ tensile testing of as-received FeCrAl alloy C37M; (c) initial Ta phase (green) map in proton-irradiated nanocrystalline Cu-10Ta; and (d) Ta phase map after TEM in situ indentation, showing dynamic nucleation of Ta nanophases (circled).

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