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In-Situ Characterization Techniques for Investigating Nuclear Materials

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Nuclear material research is an exciting field of material science since it addresses the core of material science in multiple facets while being truly multiscale on time and length scales.[1,2] Radiation damage occurring at the picosecond and nanometer length scale is the cause of material degradation over decades on meter-sized components.[3] Nuclear components reach an operation age of 60 and possibly 80 years and beyond and are therefore among the longest serving engineering components in modern applications along with civil engineering structures such as the Golden Gate Bridge (finished in 1937 with the 83rd anniversary in 2020). Nuclear components are simultaneously exposed to temperature, radiation, corrosion, and stress, making for unique operation conditions. An unfolding tetrahedral representation of the combination of environments is shown in Fig. 1.

It is the combination of these environments that poses the true challenge to current and future materials. The fundamental understanding of the effects as they take place must be fostered to make long-term predictions for and assessments of the materials in service. Recently, scientists have been addressing the combination of environments on materials coupled with in situ diagnostics, be it stress under radiation,[5,6] corrosion under radiation,[7–9] or multiple irradiation sources simultaneously.[10–13] While the multiple conditions can have synergistic effects on the material, to truly understand the effects taking place at a fundamental level, in situ material testing is needed. It is only with in situ material examination that a direct observation of the effects of multiple conditions taking place simultaneously in real time can be examined. This special topic is dedicated to the conventional and new in situ techniques enabling new research and science in the area of nuclear materials.

The article by C. Taylor et al. investigates "Using In-situ TEM Helium Implantation and Annealing to Study Cavity Nucleation and Growth." Noble gases are implanted in palladium during direct TEM observation to collect real-time cavity evolution dynamics. For the first time, a dependence of cavity nucleation on temperature was observed. This is an excellent example of in situ observations that can lead to improved understanding of defect formation mechanisms.

W.J. Williams et al. investigate "Structure Refinement of U-10 wt%Zr by Neutron Diffraction with In-situ Annealing." Here, high-pressure-preferred orientation time-of-flight neutron diffractometry with in situ heating was used to observe phase transitions, lattice parameter ratios, and linear coefficients of thermal expansion. This work adds real-time data to clarify areas of a contested phase diagram.

Work performed by D. Frazer et al. entitled "Cryogenic Stress-driven Grain Growth Observed via Micro Compression with In-situ Electron Backscatter Diffraction" uses in situ mechanical testing and EBSD while the samples are cooled to cryogenic temperatures. This is the first time experimental evidence has been provided for the fact that nanograined materials can experience grain growth at mechanical deformation at cryo-temperatures. This extremely difficult to conduct experiment marks a milestone for in situ material testing combining two characterization methods (SEM and EBSD) while two external conditions are applied (temperature and stress).

The article by P.H. Warren et al. investigates a "Method for Fabricating Depth-specific TEM In-situ Tensile Bars" using a focused ion beam (FIB). While

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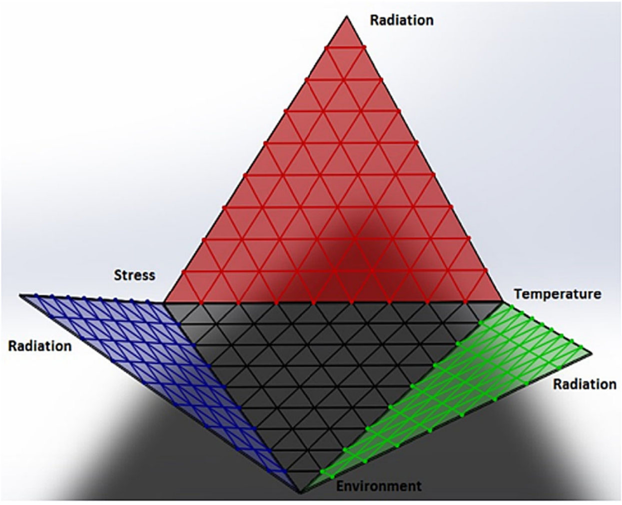

Fig. 1. Tetrahedral representation of four effects on materials (stress, temperature, environment, and radiation)[4]

FIB techniques have been used to shape pre-prepared samples for tension, this article presents a method to directly create samples from the irradiated layer of a sample in in situ tensile specimens.

The “Method for Evaluating Irradiation Effects on Flow Stress in Fe-9%Cr ODS Using TEM In-situ Cantilevers” is presented by K.H. Yano et al. to assess a newer method of in situ testing. Defect size plays an important role in the ability to use the technique in metals to measure mechanical properties while observing how the material accommodates stress. Materials with complex defect structures compared with pure materials can be investigated using this in situ small-scale testing while still measuring properties that compare with those measured in the bulk material. This is a promising technique to assess small irradiation zones.

L. Feng et al. investigate “Grain Boundary and Lattice Fracture Toughness of UO_2 Measured Using Small-scale Mechanics.” This method also uses cantilevers to isolate the behaviour of individual grain boundaries and observe crack paths in oxides, which are known to fracture in an intergranular manner. The observations show that for oxides microstructural toughening mechanisms, such as irradiation damage, may promote crack deflection and change the crack path and toughen the material. Thus, this methodology could be a fruitful approach to examining brittle materials used in nuclear applications.

J. Qiu’s work features a chemical in situ experiment entitled “An Electrochemical Impedance Spectroscopic Study of Oxide Films in Liquid Metal” performing electrical impedance spectroscopy at elevated temperature in liquid metal. The study found that this powerful in situ technique can be performed on pre-oxidized samples and the liquid metal acts just like an electrolyte.

This special topic highlights the interest in and need for in situ measurements of materials to enhance the understanding of materials in their natural engineering environment. A wide range of techniques, such as electrochemical impedance spectroscopy, transmission electron microscopy, scanning electron microscopy, electron backscatter diffraction, and neutron diffraction, while loading, heating/cooling, or irradiating materials, enhances the toolkit available to researchers as well as knowledge that can bridge the gap between science and engineering. We therefore hope that these papers will be of interest to the readers and spark new innovative ideas and concepts advancing our knowledge and understanding of material science.

To download any of these papers, follow the url https://link.springer.com/journal/11837/72/5/page/1 to the table of contents page for the May 2020 issue (vol. 72, no.5).

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