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Microstructural and Nanomechanical Characterization of In-Situ He Implanted and Irradiated fcc Materials

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Nuclear Materials research focusing on the degradation of materials utilized in nuclear environments has been studied for decades. This research aims to assess the microstructural changes and therefore mechanical property degradation as a function of radiation dose at representative temperatures and environments. Traditionally specimens are exposed to reactor or ion beam irradiation and subsequently examined using transmission electron microscopy, atom probe tomography, X-ray techniques and mechanical testing. However, all these techniques are mostly conducted ex-situ and one only observes the resulting changes but not on the same sample or same location making interpretations sometimes challenging. Following the defect development has been achieved utilizing tools like the Intermediate Voltage Electron Microscopy (IVEM) facility or other similar in-situ irradiation facilities [1]. Modern He ion beam microscopes also allow direct implantation of He in samples on a very localized level allowing localized He implantation on specific regions of interest on the same TEM foil [2]. While these are a very useful tools the damage evolution has only been followed on TEM foils and no mechanical data has been extracted from the irradiated samples. Ideally one would be able to extract a change in mechanical property associated with the microstructural changes. Recent advantages in nanomechanical testing and in-situ ion beam irradiation have the potential to obtain both microstructural data as well as mechanical property data on the same sample. This work attempts to perform the entire study in-situ spanning from He implantation to displacement damage and subsequent mechanical property evaluation to follow the defect development from the beginning to its implications for mechanical properties. It is anticipated that this procedure developed here will allow to establish a process producing data for model benchmarking bridging the gap between modeling and experiments on the same length-scale.

Ion beam irradiation capabilities are available today either ex-situ or in-situ. Ex-situ irradiations do not allow the observation of the microstructural changes as a function of dose whereas in-situ irradiations in the transmission electron microscope do lead to more insight. Of course this calls for thin foils being irradiated. In addition, in situ mechanical testing in the TEM allows also for direct observation of the mechanical test and plastic deformation processes. In this work we present an experiment where the irradiation was carried out in-situ as well as the mechanical testing. The IVEM was utilized to perform an ion beam irradiation on 304SS on a pre-FIBed foil as it can be seen in Figure 1. The foil was pre-implanted using He ions utilizing the Orion Nanofab He ion beam microscope. The subsequent dose accumulation was conducted using the IVEM up to 3dpa at 300 ºC using Ar ions. The subsequent mechanical testing on the He implanted and ion beam irradiated pillars was also conducted in an electron microscope and a stress strain curve was recorded quantifying the mechanical properties of the material.

It was found that the He implantation and subsequent heavy ion beam irradiation causes the formation of small spherical cavities in the material which likely are He bubbles in the range of 10^{17}-10^{18} He/cm^2 implantation condition and 3dpa at 300 ºC heavy ion irradiation. The subsequent mechanical testing showed high yield strength in the material. The yield strength is comparable of what has been found in 10dpa proton irradiated materials tested in the SEM on the same material [3]. It was also found that the mounting of the FIB foil held up in the IVEM in-situ irradiation tool opens the door to many similar studies.

Detailed analysis of the data is still ongoing but this approach presents a new way of investigation radiation damage in materials being able to follow all developments of radiation induced defects in the microstructure while also being able to quantify the resulting changes in the mechanical properties. We anticipate that this work will be matched by ongoing modeling efforts and will become a useful method to merge modeling and experiments [4].

References:

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[4] The Authors want to than the IVEM microscope at Argonne National Laboratory as well as the Molecular Foundry which is supported by the Office of Science, Office of Basic Energy Sciences, of the U.S. Department of Energy under Contract No. DE-AC02-05CH11231.

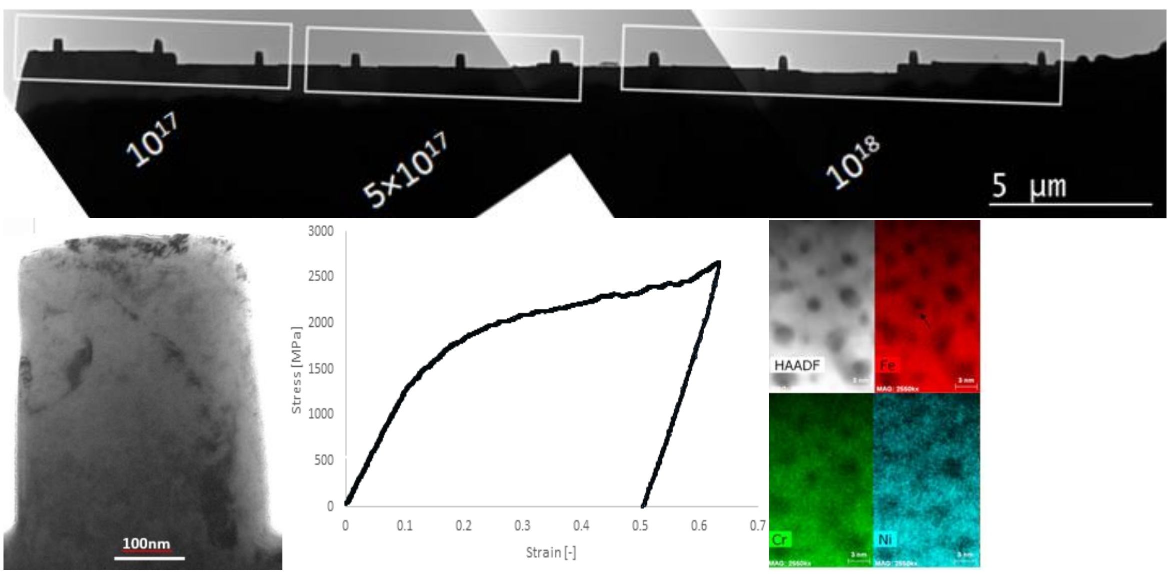

Figure 1. Array of nanopillars implanted to different He doses (flux labeled in the image). TEM of a pillar before testing b) and resulting stress strain curve c). ChemiSTEM of the as implanted and irradiated sample showing the development of He bubbles after 3dpa irradiation.

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