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Multimodal characterization of samples from Spallation Neutron Source components exposed to extreme radiation and transmutation-inducing environments

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Simultaneous high-energy proton and neutron irradiation induce microstructural and mechanical responses in structural materials that are unique from irradiation with fission/fusion neutrons or accelerator-based ion beams. The target module and proton beam window (PBW) at the Spallation Neutron Source (SNS) at Oak Ridge National Laboratory (ORNL) are irradiated in these unique environments during operation. In addition to recoil damage up to about 10-15 displacements per atom (dpa) at moderately low component temperatures (100-120℃), transmutation reactions produce large levels of helium (up to ~190 appm/dpa) and hydrogen (up to ~740 appm/dpa). To maintain neutron production and extend component lifetime, accurate understanding of the radiation-induced changes to the microstructure and mechanical properties is essential, with particular focus here on the interactions with transmutation produced hydrogen and helium. Tensile testing with digital image correlation has shown that the materials in these components have interesting and potentially unique behaviors not found in other radiation environments. Samples of 316L stainless steel target module material deformed via deformation waves with little martensite transformation, and material from a solution-annealed 718 PBW displayed a partial recovery in ductility with increasing radiation dose. To investigate these interesting phenomena further, specimens from these materials were characterized using a multitude of microstructural and microanalytical characterization techniques including scanning transmission electron microscopy (STEM) with energy dispersive x-ray spectroscopy (EDS) and electron energy loss spectroscopy (EELS), thermal desorption mass spectrometry (TDS), differential scanning calorimetry (DSC), and in-situ scanning electron microscopy (SEM) and TEM heating and tensile/compression testing.

In the as-irradiated condition, both the 316L and 718 materials exhibit a high density of nanometer-size cavities. These cavities were assumed to contain both He and H, similar to the cavities found in metals and alloys that have a high He/H concentration after triple-beam ion irradiation or neutron irradiation and transmutation. However, the results show that the cavities did not necessarily contain both gasses after the low temperature irradiation, as only H was observed by STEM-EELS. Post-irradiation annealing above 500 ℃ resulted in the colocation of H and He in the same cavities, though with a core-shell structure of H surrounding He, coinciding with the growth in cavity size and drop in cavity number density. To understand why this is the case and to examine the stability of the transmutation gas/defect complexes and their effect on mechanical properties, the materials were characterized using multiple different methods before and after post-irradiation annealing. TDS and DSC experiments were used to gain insights into the types of defects that are present and how the gasses are stored in the materials. DSC experiments have previously shown that a large amount of stored energy from irradiation is present in defects that are too small to be seen by TEM. Here, the radiation-induced stored energy –difference between energy released during first scan and subsequent annealed scans –were strongly correlated with the radiation dose and H and He release levels and recrystallization of amorphized phases. This correlation may help explain why there is an increase in radiation ductility with increasing dose in the PBW 718 material. In-situ SEM and TEM deformation testing will be presented to highlight the mechanisms for the ductility recovery.

With the DSC and TDS results in mind, post-irradiation annealing microstructural characterization and mechanical testing revealed that 1) the nanocavities retain relatively stability to high temperatures and are highly pressurized –enhancing dislocation emission during straining and thus work hardening and 2) annealing transforms the grain structure and dislocation structure enabling further recovery of ductility while maintaining strength greater than the reference condition. The microstructural process(es) responsible for the increased ductility with radiation dose could provide insights into deformation mechanisms that might be exploited in future materials.

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