The Role of Precipitate Coherency on Helium Trapping in Additively Manufactured Alloy 718 (NSUF-RTE#4272)

Stephen Taller (PI), Timothy Lach (collaborator), Oak Ridge National Laboratory
Kai Sun (collaborator), University of Michigan

Ni-based superalloys are a candidate alloy class for advanced reactor applications because of their intrinsic resistance to creep, adequate corrosion resistance and the ability to tailor the microstructure for high strength possibly through additive manufacturing. These high strength Ni-based alloys gain their strength mostly through secondary precipitating phases in the lattice, such as the intermetallic phases δ, γ' or γ''. The absorption of transmutation-produced helium at grain boundaries at high temperature becomes a key factor in the propagation of cracks, possibly leading to subcritical crack growth. The precipitate-lattice interfaces can act as benign locations in the microstructure to trap helium – possibly controlled by the degree of coherency – reducing the detrimental accumulation of helium and formation of cavities at boundaries.

Two heats of Ni-superalloy Inconel 718, one wrought designated as ASTM and one additively manufactured designated HT2, with large pre-existing precipitate densities with varying coherency were evaluated using the in-situ dual ion irradiation capability within the 300 kV FEI Tecnai transmission electron microscope at the Michigan Ion Beam Laboratory. Irradiations were conducted nominally up to 10 dpa at 0.6-1.1 × 10^{-3} dpa/s using 1.17 MeV Kr ions with ~400 appm He/dpa co-injected from 23 keV He ions at temperatures from 500-700 °C. Detailed information about the irradiations performed is included in Table 1. High angle annular dark field (HAADF) images were continuously collected during each irradiation to capture the time-dependent evolution of the microstructure. A representative set of images is included in Figure 1.

Table 1. Summary of the irradiations and information gathered as part of this proposal, RTE 4272.

Irradiation #	Sample ID	Irradiation Date	Temperature (°C)	End DPA	Time avg. DPA/s	Time avg. Kr Flux (ions/cm^2)	In-situ Video Capture	PIE on final microstructure
1	ASTM#1	12/13/21	500	9.9	9.7×10^{-4}	3.55×10^{11}	√	√
2	ASTM#4	12/14/21	600	10.0	9.4×10^{-4}	3.42×10^{11}	√	√
3	ASTM#3	12/14/21	700	9.6	8.3×10^{-4}	3.04×10^{11}	√	√
4	HT2-9B#4	12/15/21	500	10.5	1.0×10^{-3}	3.67×10^{11}	√	√
5	HT2-9B#3	12/15/21	600	10.3	9.9×10^{-4}	3.62×10^{11}	√	√
6	HT2-9B#2	12/16/21	700	7.9	1.01×10^{-3}	3.69×10^{11}	√	√

Dissolution of pre-existing γ'' precipitates occurred early (<1 dpa) for all conditions. At 500 °C, small cavities nucleated by 1 dpa, and a phase with similar contrast to γ'' emerged at about 5 dpa. At 600 °C and 700 °C, both cavity nucleation and precipitate dissolution and reemergence were accelerated. Further analysis will be conducted to identify the emerged phase or phases and quantify the irradiated microstructure using a dynamic segmentation convolutional neural network on each image frame of each video. The utilization of a neural network will allow for time dependent evolution of nearly every microstructure feature visible in the HAADF images. The experiments and data from this RTE are expected to result in one journal article on the use of neural networks to process in-situ microstructural evolution data and one journal article on precipitate and cavity evolution in Inconel 718 under irradiation focusing on temperature and damage level dependences.

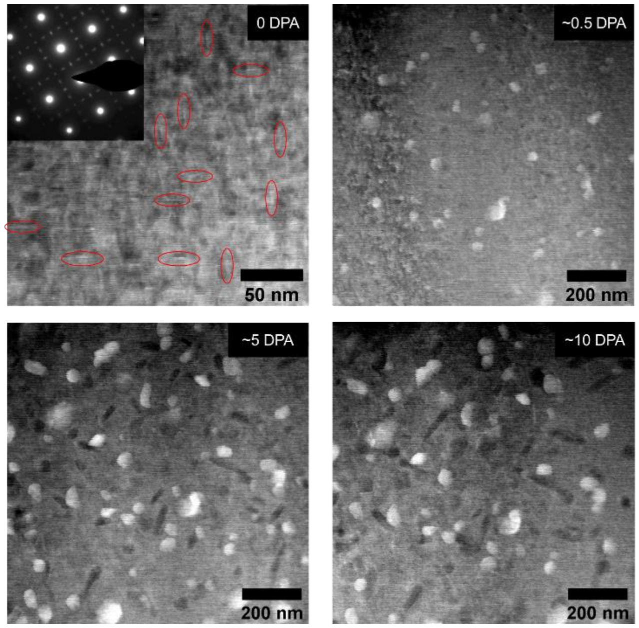

Figure 1. STEM HAADF images of the microstructure from dual ion irradiation of one heat of additively manufactured Inconel 718 with 1.17 MeV Kr and 23 keV He ions at 600°C. The bright features are Nb-rich precipitates and cavities can be observed as dark features. At 0 dpa, the γ″ precipitates (highlighted with red ovals) form a superlattice and dissolved early. Features believed to be cavities nucleated by 0.5 dpa and elongated along preferred planes with continuing irradiation up to 10 dpa.

Several key lessons were learned as part of this RTE. The first is that clear communication between the investigators and the Michigan Ion Beam Laboratory (MIBL) prior to Focused Ion Beam (FIB) specimen preparation resulted in minimal setup time and downtime during the week of scheduled experiments. Prior to the experiment, the need to use specialized FIB liftout procedures for the high temperature TEM nanochip stage was communicated and MIBL provided written instructions that were executed by the investigators. This resulted in TEM transparent lamellas optimized to have a low order zone axis visible during the ion bombardment and minimize the time needed to collect images at high temperatures. Related to this, however, is the second lesson of flexibility in that after the experiments, the liftouts were too fragile to remove from the specialized nanochips. The planned PIE in LAMDA was no longer feasible and PIE was completed at the University of Michigan on the specialized holder. Shifting characterization to the Michigan Center for Materials Characterization, an additional NSUF partner facility, aided in finishing the work scope awarded.

This work was supported by the U.S. Department of Energy, Office of Nuclear Energy under DOE Idaho Operations Office Contract DE-AC07-051D14517 as part of a Nuclear Science User Facilities experiment. Additional research efforts sponsored in part by the Laboratory Directed Research and Development Program of Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the US Department of Energy.