Heavy Ion Irradiation and Characterization of Light-Refractory, Body-Centered Cubic, High-Entropy Alloys

Michael Moorehead - Idaho National Laboratory - michael.moorehead@inl.gov

While body-centered cubic (BCC) steels are known to incur less void swelling than their face-centered cubic counterparts under similar irradiation conditions, few studies have examined the performance of BCC high-entropy alloys (HEAs) under irradiation at temperatures relevant for promoting void swelling (i.e., approximately half the melting point).The purpose of this research project is to experimentally examine the role of chemical complexity on the irradiation response of single-phase, BCC, light-refractory HEAs, which are relevant for advanced nuclear reactor applications.

Experimental or Technical Approach

Four increasingly complex light refractory HEAs were examined, $Cr_{33}Mn_{33}V_{33}$, $Cr_{31}Mn_{31}Ti_7V_{31}$, $Al_{15}Cr_{20}Mn_{20}Ti_{10}V_{35}$, and $Al_{15}Co_4Cr_{20}Mn_{20}Ti_6V_{35}$, and are all in atomic percent. Alloy composition selection was informed by calculation of phase diagrams (CALPHAD) modeling, via PanDat and the PanHEA database, which were used to search for HEAs that exhibited a single-phase microstructure at elevated temperatures and contained large amounts of Al, Cr, and Ti to form passivating oxides and remove interstitial impurities from solid solution. These impurities are known to embrittle refractory metals. Alloys were produced via arc melting and subjected to a 1200°C homogenization heat treatment to ensure each started with a single-phase, BCC microstructure as confirmed by X-ray diffraction. Samples of each alloy were irradiated at the Wisconsin Ion Beam Laboratory at 500°C using 4-MeV, defocused V^{2+} ions to a maximum-dose of 100 dpa in the damage plateau region. PIE was performed at the University of Wisconsin-Madison Characterization Laboratory for Irradiated Materials facility, where a focused ion beam was used to prepare sample lamellae for TEM using an FEI Technai TF-30 TEM instrument. TEM analysis was used for assessing the presence of radiation-induced precipitation and void swelling and for examining the dislocation loops and networks generated under irradiation.

Results

After 100 dpa at 500°C, TEM analysis revealed radiation-induced precipitation in the damage region in three out of four studied alloys. Based on CALPHAD predictions, the radiation-induced precipitates in $Cr_{33}Mn_{33}V_{33}$ are likely a Mn-rich

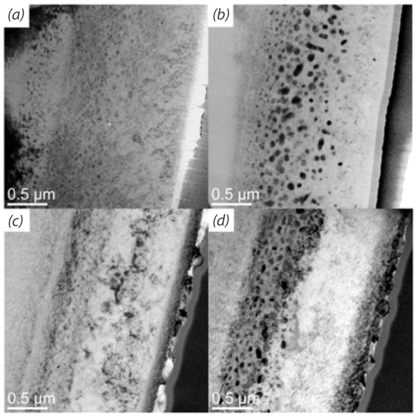

Figure 1: Bright-field TEM micrographs of (a) $Cr_{33}Mn_{33}V_{33}$, (b) $Cr_{31}Mn_{31}Ti7V_{31}$, (c) $Al_{15}Cr_{20}Mn_{20}Ti_{10}V_{35}$, and (d) $Al_{15}Co_{4}Cr_{20}Mn_{20}Ti_{6}V_{35}$ after irradiation with 4-MeV V2+ ions at 500°C to 100 dpa in the damage plateau region.

sigma phase near the damage peak, while $Cr_{31}Mn_{31}Ti_{7}V_{31}$ appears to have formed a Ti-rich Laves phase, which is pervasive over the entire damage region. $Al_{15}Co_{4}Cr_{20}Mn_{20}Ti_{6}V_{35}$, with both Co and Ti additions, likely formed a combination of Co-rich, ordered, B2 precipitates and formed Ti-rich Laves phase precipitates. Of the light refractory HEAs irradiated to 100 dpa, only the least complex alloy ($Cr_{33}Mn_{33}V_{33}$) showed evidence of minor void swelling, which lends credence to the idea that increased chemical complexity leads to improved tolerance to irradiation damage. However, further irradiations at different temperatures and higher doses would be needed to compare the swelling curves and the incubation doses for the more-complex alloys before a causal determination can be made.

Discussion/Conclusion

While sparce voids were observed in one of the light-refractory HEAs studied, most alloys formed radiation-induced precipitates after irradiation. Being that V is only a minor constituent in the radiation-induced precipitate compositions, it is suspected that the formation of these precipitates is caused by the irradiation-enhanced diffusion kinetics rather than changes in chemistry caused directly by the injection of V ions. This suggests that only $Al_{15}Cr_{20}Mn_{20}Ti_{10}V_{35}$ could be expected to remain microstructurally stable after prolonged exposure in a reactor environment.

Distributed Partnership at a Glance

NSUF Institution	Facilities and Capabilities
University of Wisconsin-Madison	Characterization Laboratory for Irradiated Materials
Collaborators	
University of Wisconsin-Madison	Adrien Couet (Co-Principal Investigator), Hongliang Zhang (Team Member)
Fermi National Accelerator Laboratory	Frederique Pellemoine (Team Member), Kavin Ammigan (Team Member)