Clarifying phase instabilities induced by irradiation in a FCC-based FeNiCrMn HEA

A. Dartois[1], M. Nastar[1], B. Décamps[2], A. Fraczkiewicz[3], M. Loyer-Prost[1], E. Meslin[1]

[1]Université Paris Saclay, CEA, Service de recherche en Corrosion et Comportement des Matériaux,
SRMP, 91191 Gif Sur Yvette, France
[2]Laboratoire de Physique des 2 infinis Irène Joliot-Curie (IJCLab), CNRS/IN2P3, (UMR9012),
Université Paris-Saclay, 91405 Orsay, France
[3]MINES St-Etienne, CNRS (UMR 5307), Laboratoire Georges Friedel, LGF, 42100 Saint-Etienne, France

Recent studies have highlighted the promising behavior of High Entropy Alloys (HEAs) under irradiation, exhibiting improved swelling resistance [1] and reduced radiation-induced segregation (RIS) [2]. For evident safety concern, they need to maintain high single-phase stability to ensure stable mechanical properties under operating conditions.

In this context, our study focuses on the quaternary $Fe_{37}Ni_{34}Cr_{16}Mn_{13}$. Before irradiation, this high-purity alloy, annealed at 1473 K for 4 hours, is a single FCC phase. However, after heavy-ion irradiation within the JANNuS Saclay facility at 823 K up to 2 dpa, phase instability was detected by means of CTEM (Conventional transmission electron microscopy) and STEM/EDX (Scanning TEM/Energy dispersive X- rays) within a double-corrected Jeol Neo-ARM TEM.

It is well known that irradiation generates point defects – vacancies and self-interstitials – that subsequently agglomerate into dislocation loops, voids or precipitates [3]. Besides, net fluxes of point defects toward sinks lead to RIS potentially leading to radiation-induced precipitation (RIP) [4].

In the current study, phase instability is characterized by the precipitation of nanometer-sized Cr-Mn enriched precipitates. These intragranular precipitates have a spherical shape with an approximate diameter of 10 nm. Their crystallographic structure needs to be examined thoroughly. Considering previous analyses on similar alloys [5], a BCC phase could be expected. The matter balance between self-interstitial atoms (SIAs) and vacancies respectively stored into dislocation loops and precipitates tends to confirm the less dense BCC structure of the precipitates. We hence may expect these precipitates to result from a RIP mechanism explained by the elimination of irradiation-generated vacancies accommodating the volume mismatch between less dense precipitates and the matrix [3].

This phase instability is also correlated with a microstructural evolution. Indeed, both faulted and perfect dislocation loops are formed. According to atom probe tomography (APT) and STEM/EDX analysis, RIS occurs at these radiation-induced defects. Indeed, a strong Ni enrichment and Fe, Mn and Cr depletions, have been detected. An inverse Kirkendall model of atomic fluxes, mediated by vacancies and fitted on tracer diffusion coefficients of the well-known FeCoCrMnNi Cantor alloy, corroborates the identified RIS trend. We highlight a reduced Cr depletion, compared to conventional austenitic steels [6], potentially reducing irradiation-assisted stress corrosion cracking (IASCC) phenomena.

In order to elucidate the precipitation mechanisms, microstructures exposed to several radiation doses have been compared. First, samples have been irradiated up to 0.2 dpa. Secondly, an analysis of the microstructure at increasing thicknesses has enabled us to compare various stages of irradiation progress. Indeed, point defects elimination on surfaces is stronger in thinner areas, where effective damage is therefore reduced, whereas this phenomenon is negligible in thicker areas, experiencing a stronger effective damage. On-zone axis STEM imaging shows the formation of a dislocation network, resulting from the interaction of perfect loops as well as a decrease of the density of both Frank loops and precipitates with increasing depth.
To further investigate the precipitation mechanisms, in-situ irradiations are essential. These will be conducted inside a TEM in November 2023 within the JANNuS-Orsay facilities, to track both the microstructural evolution as well as precipitation using electron diffraction spots signal produced by the formation of Cr-Mn precipitates.

References

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