

Advanced Characterization of Phase Stability Under Ion Irradiation of Ultrafine-grained and Nanocrystalline SS304L

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SS304L is a Fe-18Cr-8Ni austenitic steel and a common core-internal structural material for light-water reactors (LWRs) due to its advantageous mechanical properties and corrosion resistance. Previously it was observed that during irradiation austenitic steels like 304L can undergo a phase transformation under irradiation from face centered cubic (FCC) to base centered cubic (BCC) phase [1]. It is thought that the mechanism for this transformation may be due to both Ni depletion in the matrix due to grain boundary segregation and precipitation of Ni enriched phases as well as strain induced martensite transformation. This work attempts to characterize the enhanced resistance to phase transformation of nanostructured SS304L by utilizing grain boundaries (GBs) that act as sinks for radiation-induced defects [2]. The reduction of radiation induced defects in the material helps to reduce both segregation and precipitation effects as well as strain due to accumulated dislocations in the material.

SS304 commercial bar stock was manufactured into UFG and NC samples using two forms of SPD: equal-channel angular pressing (ECAP) and high-pressure torsion (HPT). ECAP 304L had an average grain size of 500 nm and HPT 304L had an average grain size of 85 nm. XRD showed that during SPD no deformation induced martensite had occurred at both HPT and ECAP 304L were fully austenitic. After SPD, samples were irradiated using 3.7 MeV Fe²⁺ ions up to 50 displacements per atom (dpa) at 500 °C.

Grazing incidence XRD was performed in order to compare the phase change during irradiation. Qualitatively, the nanostructured steels showed less phase transformation from FCC to BCC under irradiation. Because of SPD and irradiation induced textures XRD proved difficult to give quantitative information on phase, and therefore precession electron diffraction was used in order to determine a more qualitative phase fraction (see Figure 1). From PED the BCC phase fraction of CG, ECAP and HPT 304L was 33%, 6.5%, and 2% respectively. Energy dispersive x-ray spectroscopy was performed on all samples showing that in CG 304L after irradiation many of the BCC grains were enriched in Cr and depleted in Ni. Additionally, in CG 304L many intragranular Ni enriched precipitates were observed. These intragranular Ni enriched precipitates were not observed in either HPT or ECAP 304L. The smaller grain size also helps to reduce the effects of radiation induced segregation. Figure 2 shows APT results of the irradiated HPT 304L showing that in the larger UFG in the sample irradiation induced segregation is much more severe than in the NC grains.

In addition to reduced segregation the CG304L showed larger dislocation loops and the formation of larger dislocation networks, as opposed to ECAP and HPT 304L which showed no dislocation network formation under irradiation, and only small dislocation loops thus reducing the strain mechanism which may cause deformation induced martensite [3].

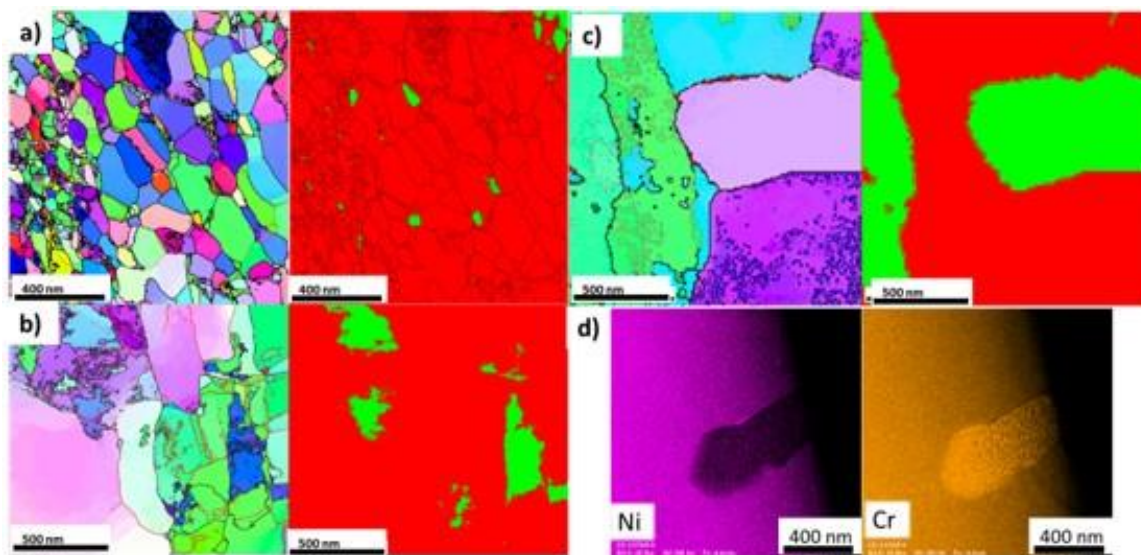


Figure 1. a)–c) Precession electron diffraction misorientation maps (left) and phase maps (right) showing BCC in red and FCC in green for a) HPT 304L b) ECAP 304L and c) CG 304L. d) EDS maps of Ni and Cr of the region in c) showing that some BCC grains are enriched in Cr and depleted in Ni.

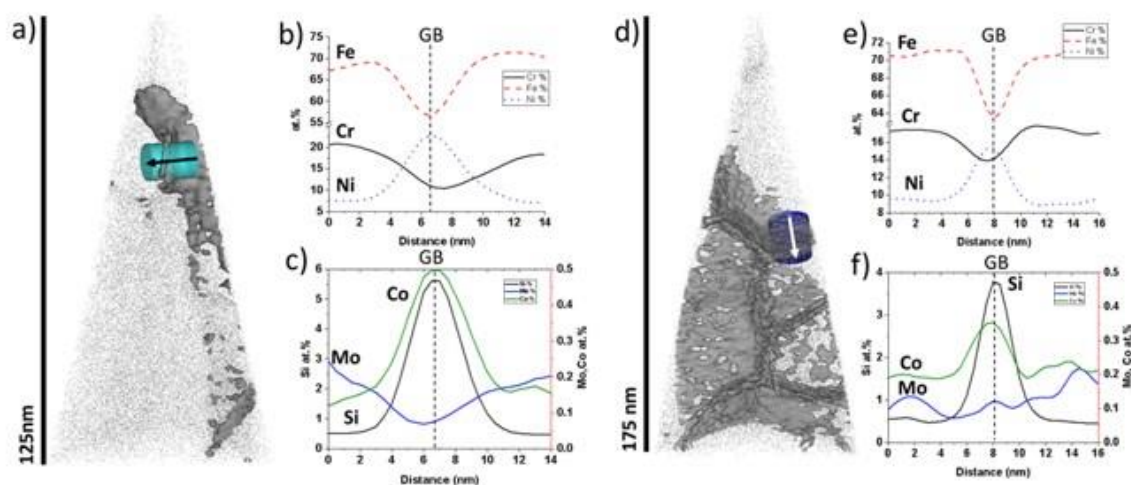


Figure 2. APT reconstruction showing Si atoms and 4 at.% Si isoconcentration surface for irradiated HPT 304L showing a) an UFG and d) a region with many NC grains. The 1-D concentration profiles of the shown cylinders across grain boundaries are shown in b) and c) for subfigure a) and e) and f) for subfigure d). The NC grains show significantly reduced segregation after irradiation.

References

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- [2] Y Chen, et al., *Nat. Commun.* 6 (2015), p. 7036.
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