

## **Bubble Formation of In situ He-Implanted 14YWT and CNA Advanced Nanostructured Ferritic Alloys**

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The objective of this work is to investigate the effects of nanoparticle density and their binding energy on the formation of helium bubbles. The synergistic effect of helium along with radiation damage can cause unwanted degradation of the mechanical performance of structural materials and impact the safety of nuclear reactors [1]. It has been proposed that these effects could be mitigated by increasing the number of He trapping sites to control the bubble size or to shield the grain boundaries from He [2]. This concept has led to the development of nanostructured alloys with engineered high sink-strength microstructures.

### **Experimental or Technical Approach**

Helium bubble formation in Fe-9/10Cr binary alloys and two dispersion strengthened nanostructured alloys (CNA3 and 14YWT containing carbide and oxide particles, respectively) was examined by scanning/transmission electron microscopy (S/TEM) after *ex situ* and *in situ* He implantation to ~10,000 appm at 500–900°C. The thin TEM foils were irradiated at the Intermediate Voltage Electron Microscopy (IVEM)-Tandem facility in Argonne National Laboratory. The TEM thin foils of Fe-10Cr, CNA3, and 14YWT materials were irradiated with a 10 keV single He ion beam at a 15 degree incident angle, at 600 and 900°C. For the 600°C *in situ* irradiated

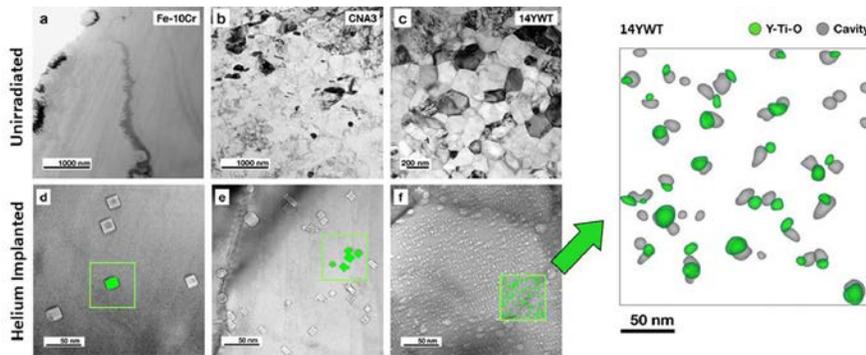


Figure 1. Under focused TEM images of bubbles in in situ He-implanted Fe-10Cr and NFA thin foils at 600°C (a–c) and 900°C (d–f). The green filled portions of the micrographs highlight the bubbles in the materials.

samples, once we achieved the target dose, we stopped the implantation and performed post irradiation annealing to 900°C. The annealing results were used to compare with the 900°C directly hot implanted experiments.

*In situ* TEM combined with post irradiation analysis using high resolution scanning transmission electron

microscopy (STEM) and electron energy loss spectroscopy (EELS) techniques were utilized to examine the microstructures. TEM magnifications of 100 kx or 200 kx were used for measuring the bubble sizes. Analysis of the STEM-EELS data was performed using Gatan GMS software. The spatial distribution and morphologies of various types of nanoparticles were evaluated from elemental maps acquired by STEM-EELS.

## Results

Cavity formation in bulk He irradiated and *in situ* He irradiated ferritic alloys containing different nanoparticle densities: Fe-9/10Cr (without nanoparticles), CNA3 (intermediate), and 14YWT (highest nanoparticle density) was examined from 500 to 900°C by TEM and STEM. At all temperatures, the cavity density in the He implanted materials was generally in the order of Fe-9/10Cr < CNA3 < 14YWT, which directly corresponds to the nanoparticle density, whereas the cavity size showed the opposite order. The observed bubble number densities for the nanostructured alloys are comparable to the nanoparticle density, suggesting that the nanoparticles in both alloys

were effective in trapping He. The combination of high resolution STEM images and EELS revealed that the MX carbides in CNA3 and Y-Ti-O oxides/TiN particles in 14YWT showed a good capability for trapping He bubbles. The Y-Ti-O nanoparticles in 14YWT were uniformly distributed and exhibited a one to one relationship for bubble attachment to the nanoclusters. In the *in situ* experiment at 900°C, grain boundary cracking was severe in the Fe-10Cr model alloy but not in the nanostructured alloys.

## Discussion/Conclusion

In agreement with previous studies, the addition of high-density nanoparticles were found to sequester the helium into finely dispersed tiny

bubbles at the particle-matrix interface (leading to a lower volume swelling compared to conventional alloys) and suppress He diffusion to the grain boundaries. The present results suggest that very high He concentrations can be managed well in nanostructured alloys with a high density of nanoparticles ( $>10^{22} \text{ m}^{-3}$ , which corresponds to a sink strength  $>10^{15} \text{ m}^{-2}$ ). Furthermore, nanoparticles in both the CNA3 and 14YWT alloys were found to exhibit high (favorable) binding energies for helium cavities up to temperatures as high as 900°C.

## References

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- [2.] Odette, G. R., M. J. Alinger, and B. D. Wirth. "Recent developments in irradiation-resistant steels." *Annu. Rev. Mater. Res.* Vol. 38 (1). 2008. pp. 471–503.

## Publications

### Journal

- [1.] Lin, Y., W. Chen, L. Tan, D. T. Hoelzer, Z. Yan, C. Hsieh, C. Huang, and S. J. Zinkle. "Bubble formation in helium-implanted nanostructured ferritic alloys at elevated temperatures." *Acta Materialia*. Vol. 217. 2021. p. 117165.

### Conference

- [2.] Lin, Y., W. Chen, L. Tan, D. T. Hoelzer, Z. Yan, C. Hsieh, C. Huang, and S. J. Zinkle. "Bubble formation in helium-implanted nanostructured 14YWT and CNA ferritic alloys at elevated temperatures." *The Nuclear Materials Conference (NuMat)*. 2018. Seattle, Washington. October 2018. (Poster).

## Distributed Partnership at a Glance

NSUF Institution	Facilities and Capabilities
Argonne National Laboratory	The Intermediate Voltage Electron Microscopy – Tandem Facility
Collaborators	
University of Tennessee, Knoxville	Steven Zinkle (co-principle investigator), Wei-Ying Chen (collaborator), Lizhen Tan (collaborator), David Hoelzer (collaborator)