A Novel Experimental Technique for Exploring the Transport and Fate of Helium in RAF/M Steels for Fusion

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Reduced Activation Ferritic/Martensitic Steels for Fusion (Fe-0.1C-9Cr-2W-0.25V-0.07Ta)

Advantages

- Well-developed technology for nuclear and other advanced technology applications.
- Fusion materials program has developed reduced activation versions with equivalent or superior properties.
- Resistant to radiation-induced swelling and helium embrittlement.
- Compatibility with aqueous, gaseous, and liquid metal coolants permits range of design options.

Issues

- Upper operating temperature limited to ~550°C by loss of creep strength.
- Potential for radiation-induced embrittlement at temperatures <400°C.
- Possible design difficulties due to ferromagnetic properties.

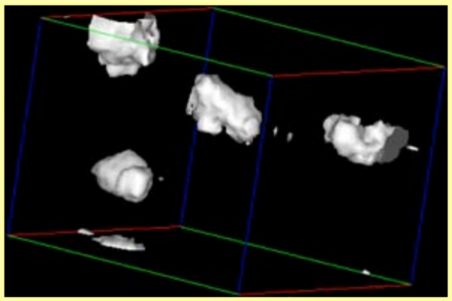
CURRENT RESEARCH

Expand Low Temperature Operating Window

- Pursue collaborative international fission reactor irradiation program (IEA activities)
    - Investigate micro-mechanics of fracture and radiation-induced reductions in fracture toughness.
    - Understand the role of helium on fracture and crack propagation.
    - Develop Master Curve to characterize effects of irradiation on temperature dependence of fracture toughness.

Expand High Temperature Operating Window

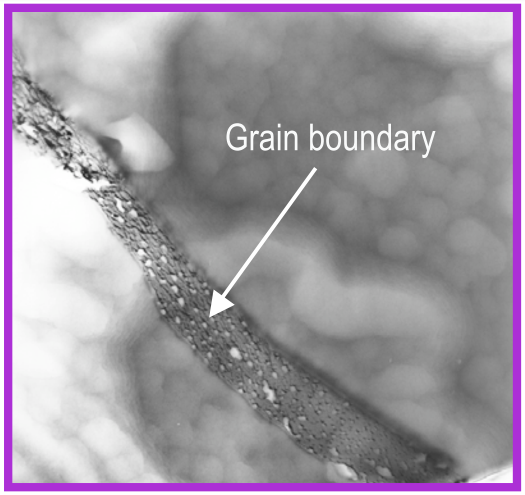
- Explore nitride dispersions and improved TMT.
- Develop nanocomposited ferritic alloys (NFA).
    - Expand upper operating temperature.
    - Radiation-stable, tough microstructures.

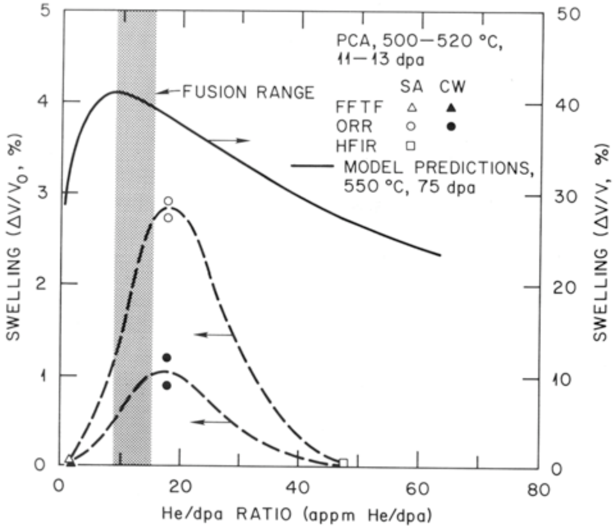


3-D atom probe image; clusters of ~100 atoms of Y, Ti, and O responsible for high strength of NFA materials

Impact of He-Rich Environment on Neutron Irradiated Materials

- A unique aspect of the DT fusion environment is substantial production of gaseous transmutants such as He and H.
- Accumulation of He can have major consequences for the integrity of fusion structures such as:
  - Loss of high-temperature creep strength.
  - Increased swelling and irradiation creep at intermediate temperatures.
  - Potential for loss of ductility and fracture toughness at low temperatures.
- Trapping at a high-density of tailored interfaces is a key strategy for management of He.

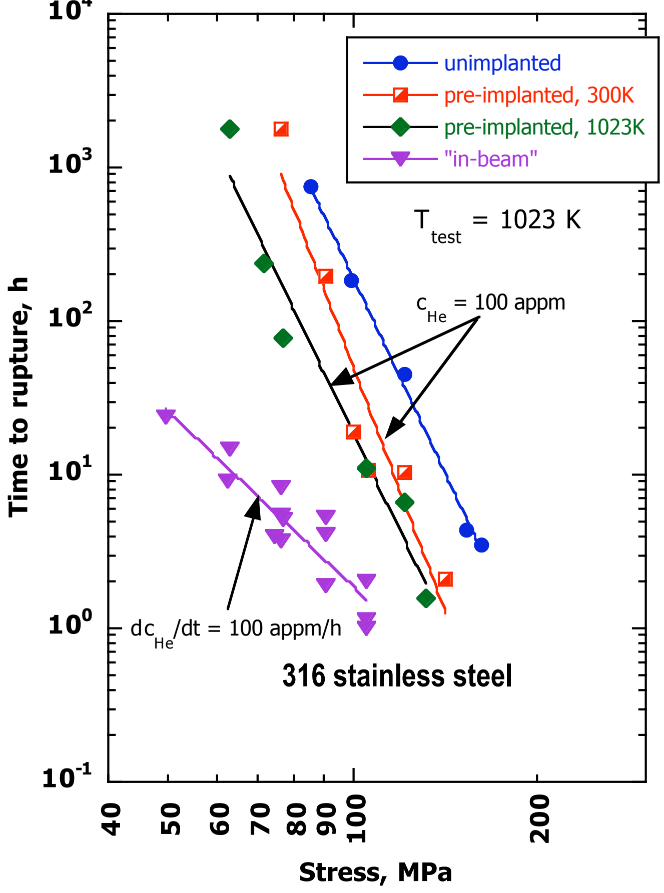




Swelling in stainless steel is maximized at fusion-relevant He/dpa values.

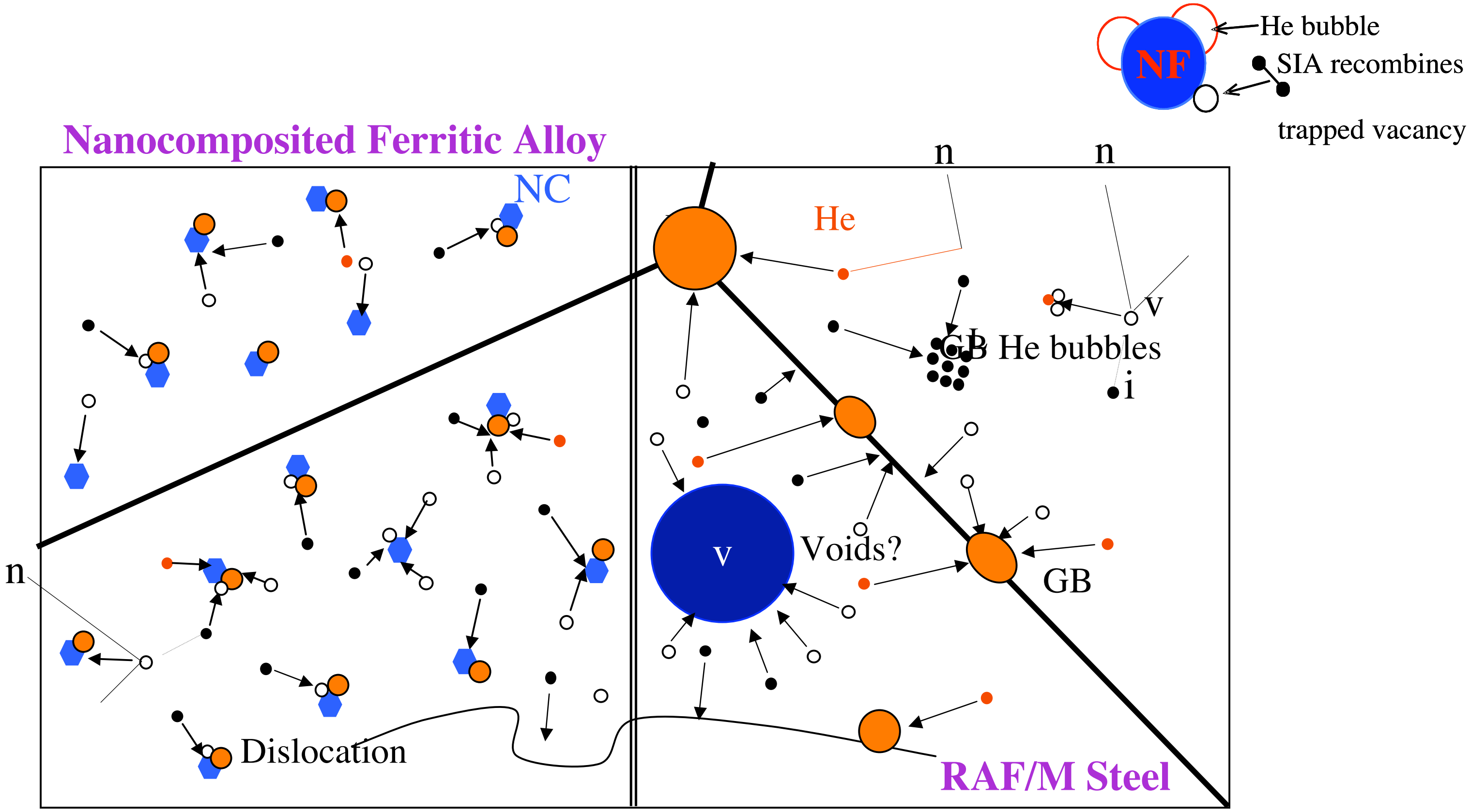
He Embrittlement: Unresolved Questions

- What is the sequence of events after He generation that controls its fate?
  - How does He diffuse?
  - How and where is He trapped?
  - What is the effect of grain boundary type on He bubble density and size?
  - How does He behave at trapping sites to form bubbles?
- Can nano features in advanced ferritic alloys stably trap He and render it innocuous in very fine bubbles?



Schroeder and Batflasky, 1983

Managing Radiation Effects in Ferritic Alloys



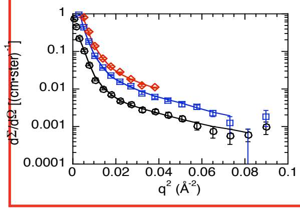
Use high sink strength of nano-features (NF) to trap (getter) both He (in fine bubbles) and vacancies (to enhance self-healing of damage by recombination with self-interstitial atoms)

Coupling of Modeling and Experiment to Determine He Transport and Fate

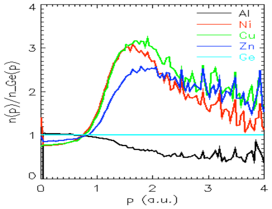
Experimental characterization

feature 'signals'

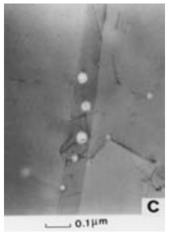
SANS/ ASAXS



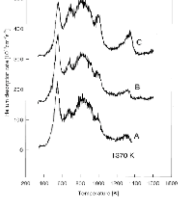
PAS



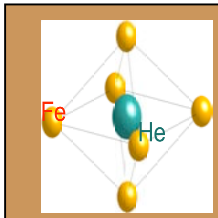
TEM

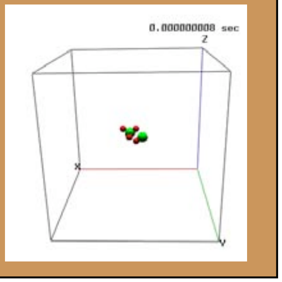


He desorption & release studies



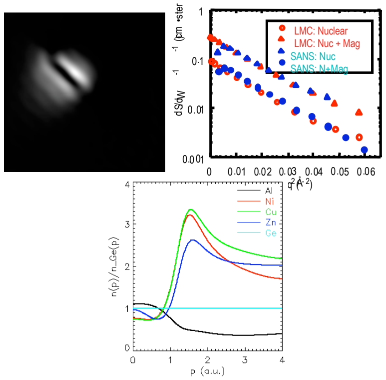
Multiscale modeling





self-consistent 'understanding' of He effects

TEM, SANS, Positron theory

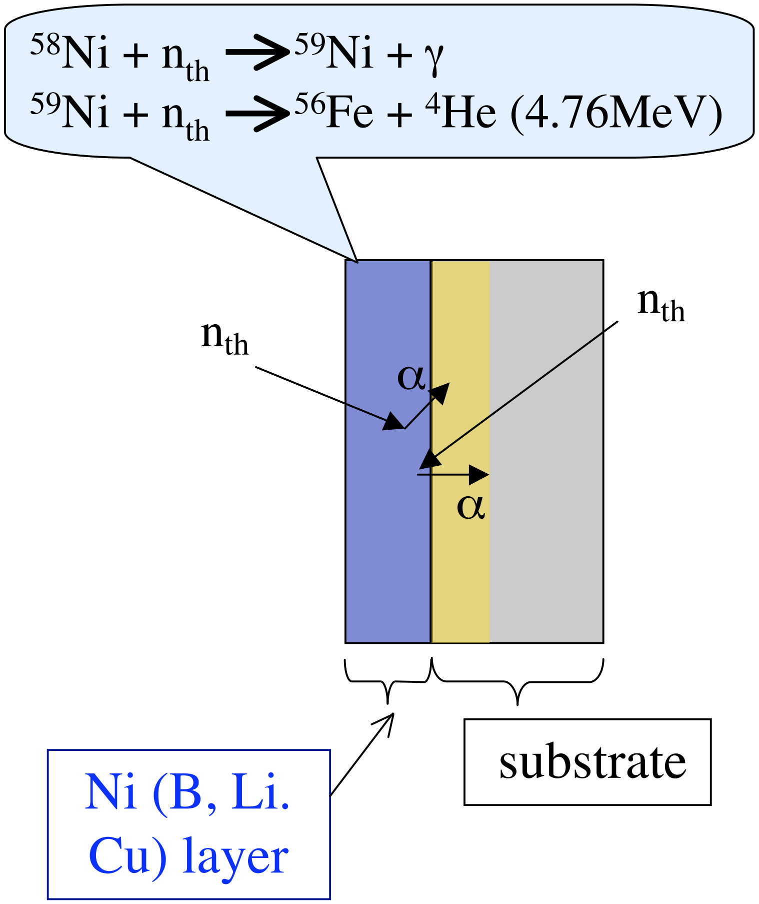


predict features

simulate observables

In Situ Helium Implanter Layer

- Use n,α reactions (various sources) in mixed spectrum reactors to produce controlled He/dpa at fusion relevant conditions.
- Avoids most confounding factors.
- Apply to any material - e.g., SiC and a variety of specimens.
- Ni injector produces up to 18 µm uniformly deposited He in Fe.
- Can not obtain bulk property information.

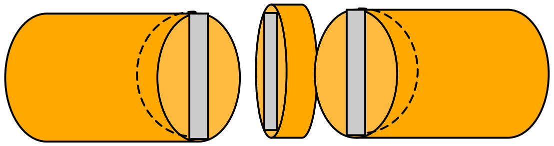


$^{58}\mathrm{Ni} + n_{th} \rightarrow {}^{59}\mathrm{Ni} + \gamma$

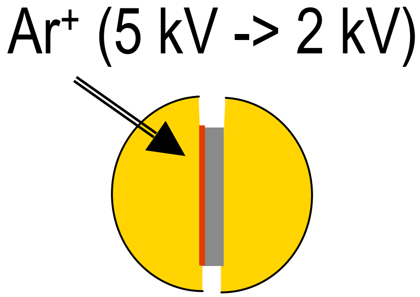
$^{59}\mathrm{Ni} + n_{th} \rightarrow {}^{56}\mathrm{Fe} + {}^{4}\mathrm{He}\ (4.76\mathrm{MeV})$

Eurofer97 Specimen Preparation Method

=> Cu/TEM/Cu - cross sectioned to $t \approx 0.5$ mm

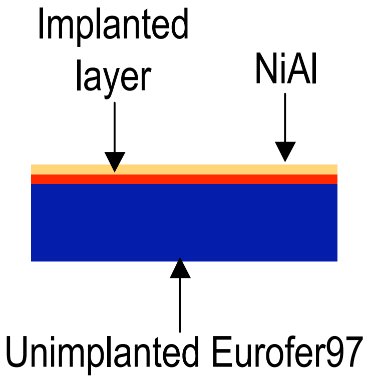


- Ar ion milling for TEM observation (V_{acc}=5 kV to 2 kV variation)
- JEOL-2010F (200kV FE)



- High precision lapping
- He desorption - isotope-dilution magnetic sector mass spectrometer

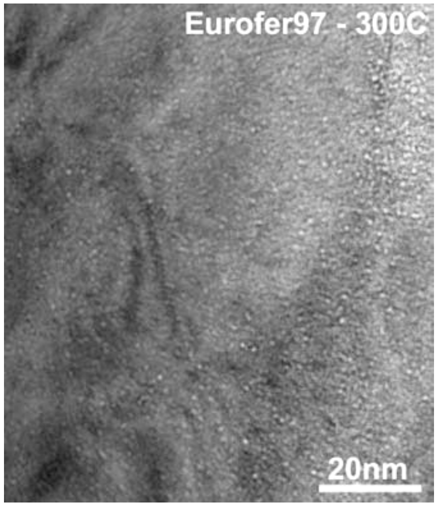
- Cross-sectional Knoop hardness
- Nano-indentation (Hysotron)

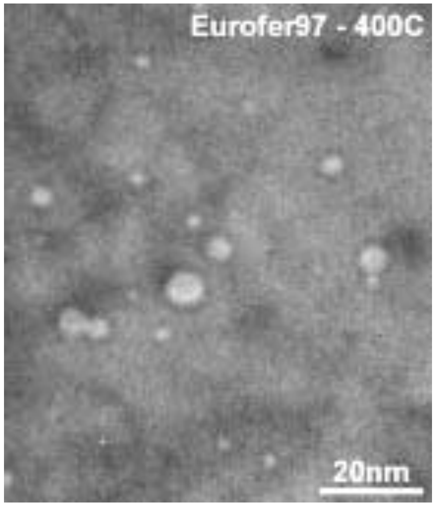


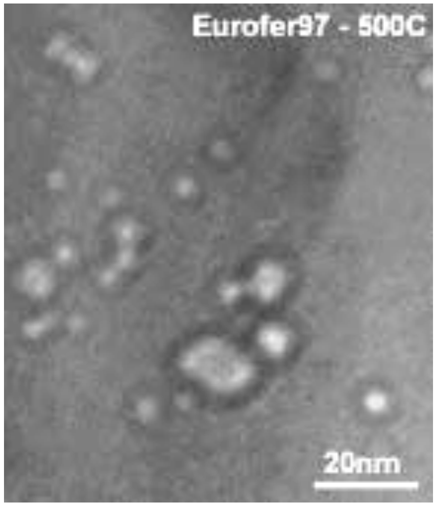
Overview of He Bubble Structure

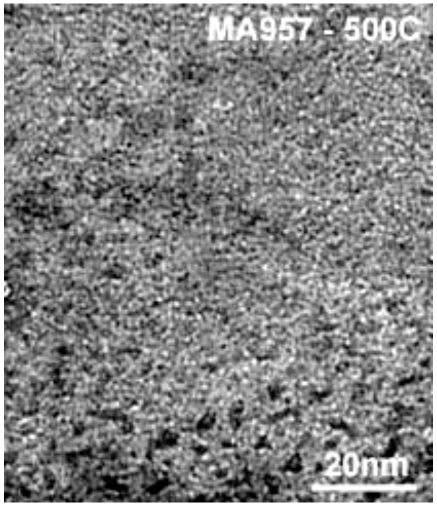
Material	T,°C	dpa - He	<2r>, nm	N, m^{-3}
Eurofer97	300	4 - 89	0.9 ± 0.2 (est.)	3.6x10^{23}
Eurofer97	400	4 - 82	3.0 ± 1.4	1.2x10^{22}
Eurofer97	500	10 - 380	4.3 ± 1.6	1.5x10^{22}
MA957	500	10 - 380	0.9 ± 0.4 (*)	3x10^{23} (*)

* preliminary

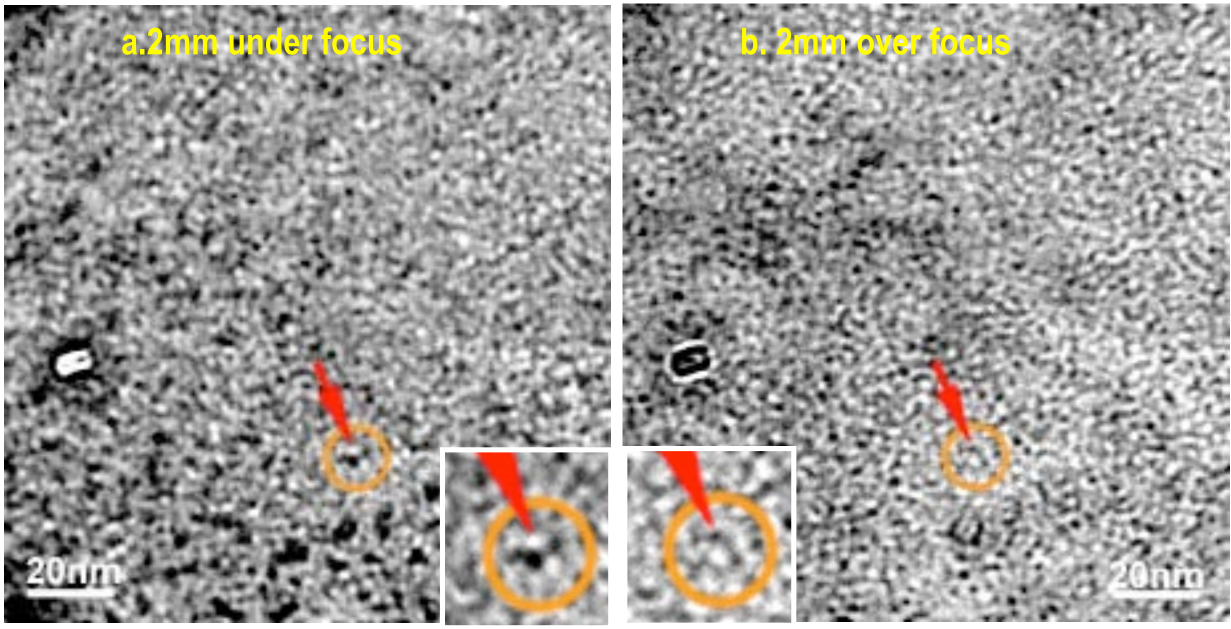




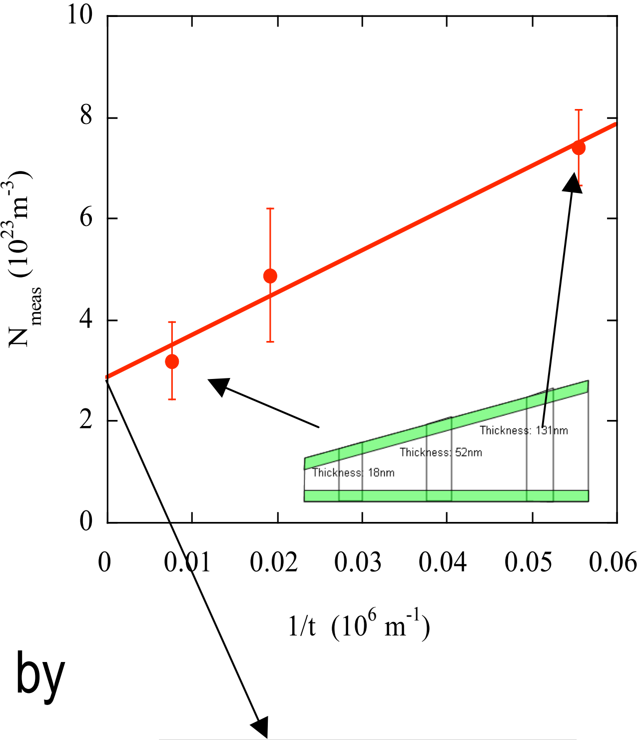




MA957 Through-Focus Series TEM (Fe-14Cr-0.3Mo-0.9Ti-0.25Y_2O_3)



N vs 1/t plot



$N_b \approx 3 \times 10^{23}$ /m^3

- Features showing bubble contrast are associated with black features which correlate with Fe depletion areas by ≈ 69%.
- N density of bubble contrast depends on location thickness =>

  Surface features (oxides?) + Bulk features bubbles

Calculated He Concentration From Measured Bubble Sizes and Number Densities

High P equation of state used to calculate the mole fraction He in bubbles of radius <r>.

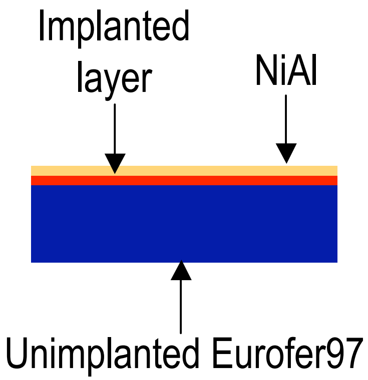
$$P \approx \frac{2\gamma}{<r>} = \frac{ZNkT}{V}$$

$$\gamma = 2J/m^2$$

Specimen	Injected He, appm	<2r>, nm	T, °C	No. of He atoms	N, m^{-3}	Bubble He, appm
Eurofer97	90	0.9	300	64	3.6×10^{23}	270
Eurofer97	90	3.0	400	909	1.2×10^{22}	128
Eurofer97	380	4.3	500	2186	1.5×10^{22}	384
MA957	380	0.9	500	35	3×10^{23}	131

Measured He Concentration in Specimen R25: 500°C, 10 dpa, 170 appm He

High-sensitivity isotope-dilution magnetic sector mass spectrometer used to determine the He concentration in two specimens with duplicate measurements. NiAl layer removed by very careful sanding.



Implanted layer

NiAl

Unimplanted Eurofer97

Thin = implanted layer only.

Thick = implanted layer only + unimplanted Eurofer97.

Specimen	m*, mg	[He], 10^{14} atoms	[He], appm
R25A (thin)	0.013	0.184	132
R25B (thin)	0.022	0.310	132
R25C (thick)	1.138	1.556	12.8
R25D (thick)	1.127	1.395	11.6

*Mass of specimen for analysis. Mass uncertainty is ±0.001 mg.

Summary

- The He implanter layer concept for producing controlled He/dpa ratios under neutron irradiation has been validated.
- Bubbles were found in the implanted region in Eurofer97 at all three irradiation temperatures.
- The minimum bubble sizes observed were near the TEM resolution limit. Additional work is needed to confirm that specimen preparation procedures or surface oxides did not influence these results.
- Loop and void formation at 300°C may have been suppressed by a high-density of small He bubbles serving as point defect recombination centers.
- At 400 and 500°C pre-existing dislocations appear to be preferred bubble nucleation sites.
- Addition of a high-density of nano-scale Ti-Y-O particles to a ferritic matrix effectively trapped He atoms and dramatically suppressed bubble growth.