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The Fuel Cycle R&D Initiative

Update on Effect of Irradiation on Microstructure and Tensile Properties of MA957

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- **SEM Exams (PNNL)**
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- **TEM Exams (PNNL)**
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- **Tensile Testing (PNNL)**
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Examination of High Dose MA957 ODS Ferritic Alloy

- **High dose (up to 121 dpa) MA957 ODS ferritic alloy creep and swelling tube specimens recovered from FFTF-MOTA**
- **Temperatures and doses**
  - (385°C, 18-43 dpa)
  - (412°C, 110 dpa)
  - (500-550°C, 18-113 dpa)
  - (600-670°C, 34-110 dpa)
  - (750°C, 33-121 dpa)

- ***Obtain creep data from tubes and extract other test specimens from the tubes:***
  - *Tensile testing (axial tensile and ring tensile specimens)*
  - *Notched tensile test*
  - *Microstructural stability under irradiation with and without applied stress*
- ***Examine tensile, swelling, in-reactor creep, and microstructural response from specimens at all irradiation temperatures***
- ***Extrapolate swelling and selected microstructural response to 500 dpa using ion irradiations***

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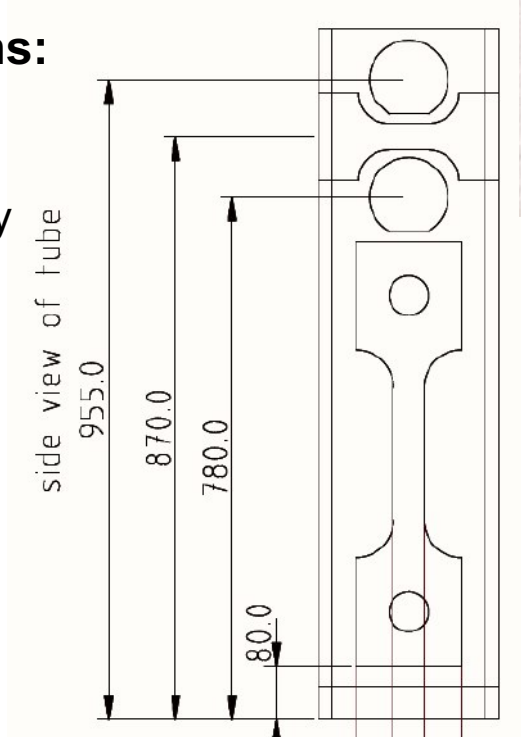
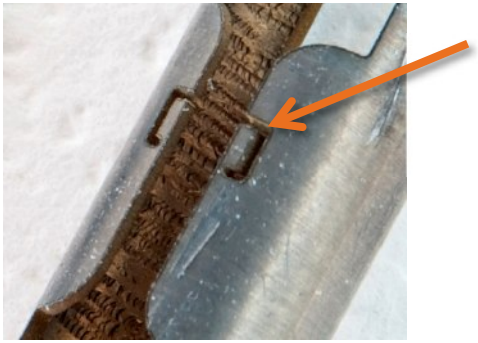
Status of Examination of High Dose MA957

- **High dose MA957 is in the form of 28 mm long creep and swelling specimens.**
- **Test specimens have been cut from these pressurized tubes.**
- **Ongoing investigations:**
  - General microstructure
  - Atom Probe Tomography
  - Swelling
  - In-reactor creep
  - Tensile properties
  - 500 dpa ion irradiations

Machined tensile, ring pull, and TEM disk specimens.



Machining plan



Material for fabricating APT specimens

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MA957 Tubing

- **Data obtained from two sets of tubes.**

	EV tubing	TX tubing
Heat	DBB0111	DBB0122
Fabrication	Drawn bar, followed by gun drilling to final dimensions	Drawn bar, followed by tube drawing to final dimensions
Dimensions	0.230" OD x 0.200" ID (0.015" wall)	0.270" OD x 0.226" ID (0.022" wall)
Final Heat Treat	760°C/30 min/AC	not reported
Irr. Temperatures	*412°C* – 750°C	*385°C* – 750°C
Dose	109-121 dpa	43-103 dpa

alloy	heat	Cr	Ni	Mn	Mo	Si	C	V	W	Ti	Y_2O_3
MA957	DBB0111	13.8	0.13	0.05	0.31	0.05	0.014	---	---	1.05	0.22
MA957	DBB0122	14.2	0.10	0.06	0.31	0.03	0.013	---	---	1.03	0.22

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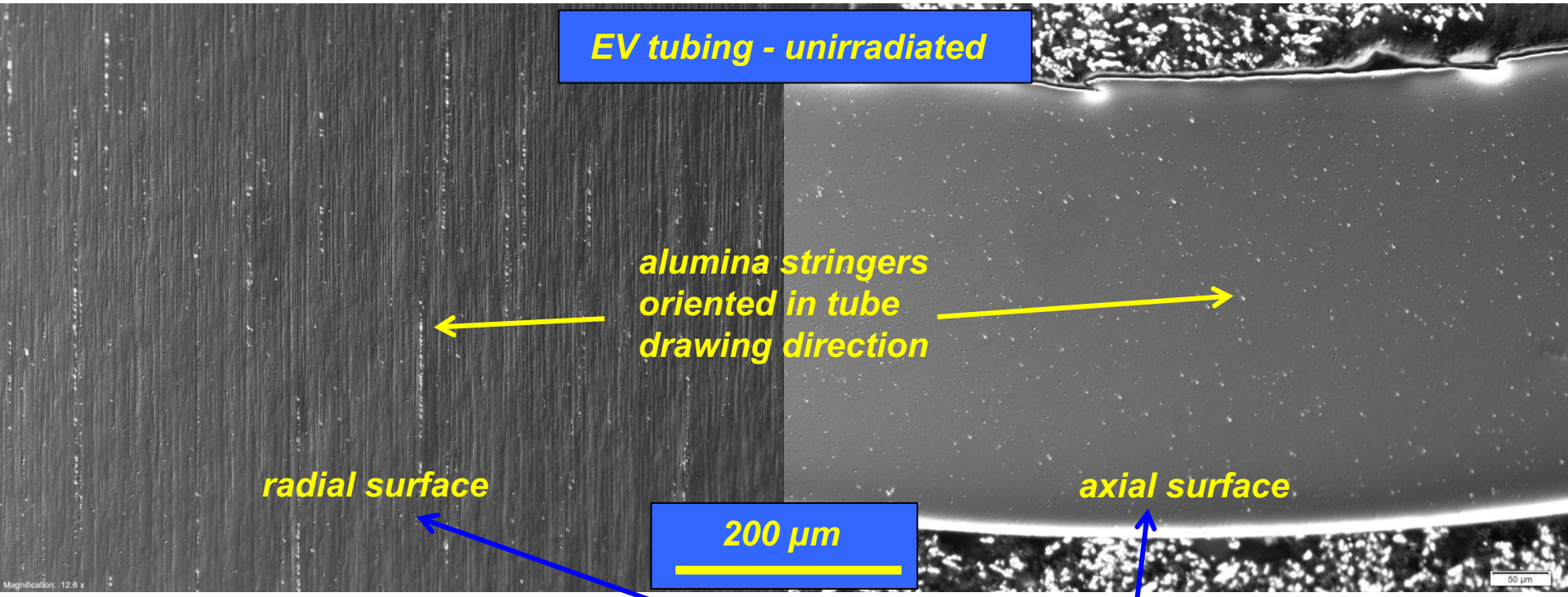
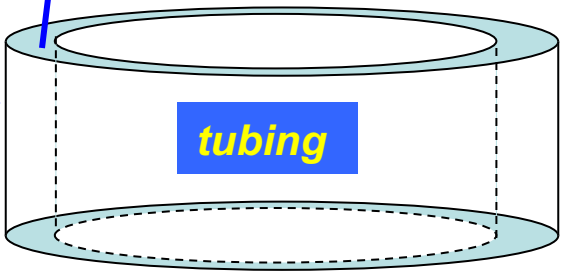
Microstructure – Optical



- Elongated grain structure is apparent.
- Alumina stringers are apparent and uniformly dispersed through the material and are oriented in drawing direction.



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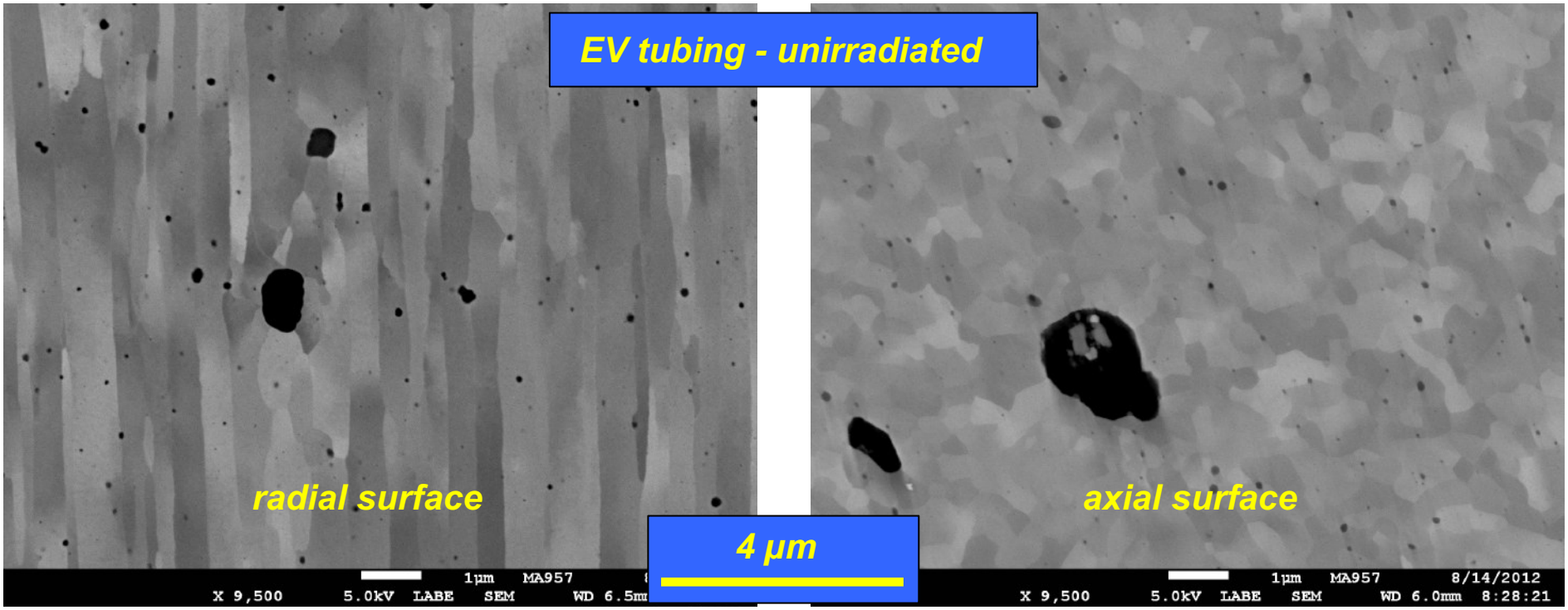
Microstructure – SEM



- Highly elongated grains
  - Grain diameter = 300-600 nm
  - Grain length = ~10 µm
  - Aspect ratio is ~20:1

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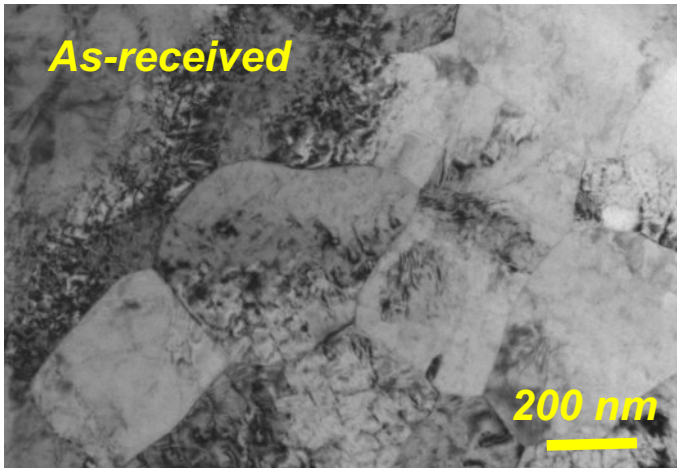
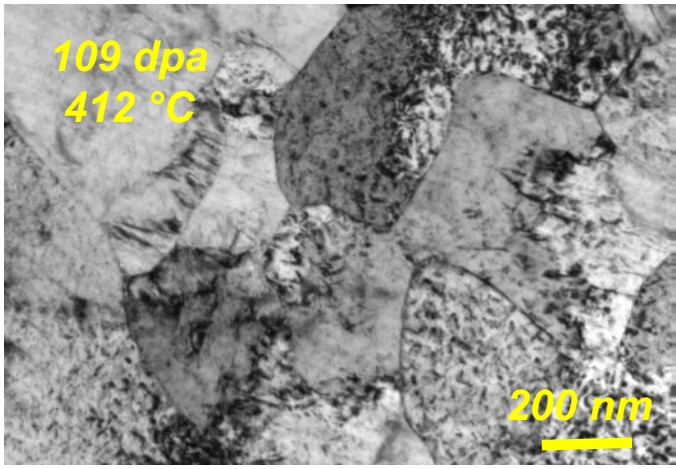
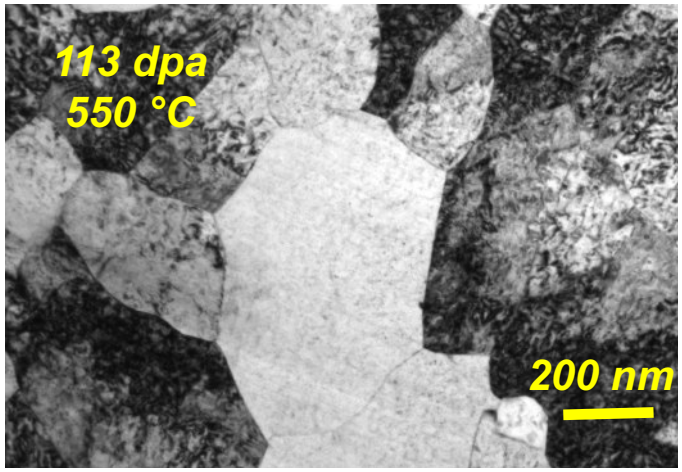
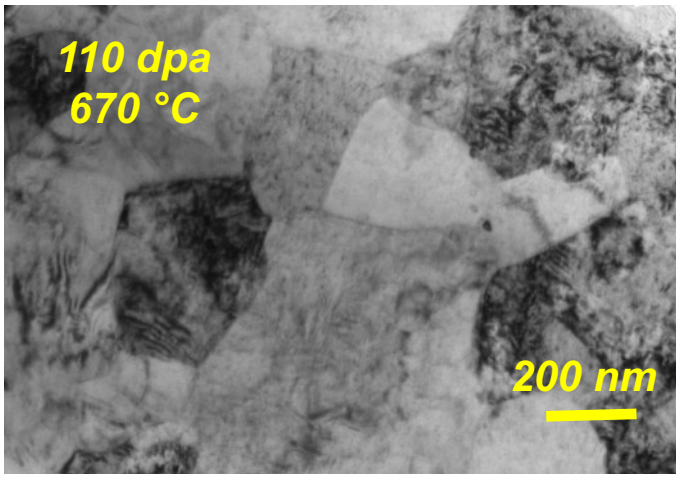
Microstructure - TEM

- Ongoing TEM observations.
- High dislocation density. No obvious change in dislocation density after irradiation.
- No swelling observed by TEM in any irradiated specimens.









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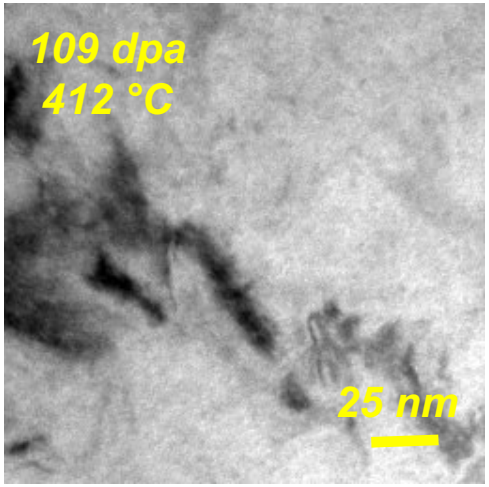
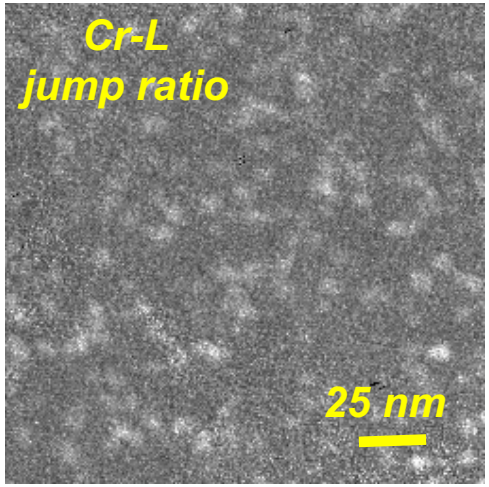
Microstructure/Chemistry Within Grains - EFTEM

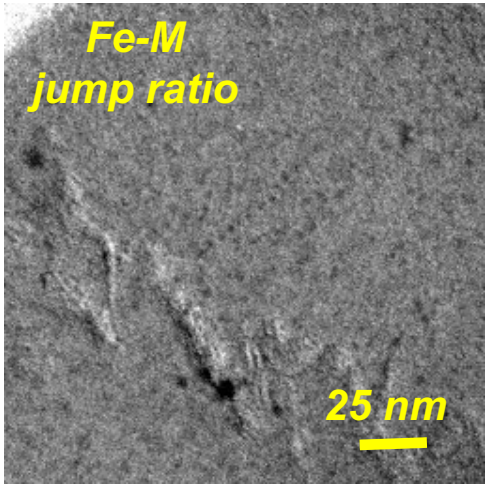
brightfield image



alpha-prime imaging



oxide particle imaging



- **Alpha-prime easily imaged by EFTEM by examining the Cr-L jump ratio. Found at 412°C, expected to be present at 385°C. Contributing to irradiation induced hardening.**
- **Constituents of oxide particles (Y, Ti, O) are not easily imaged by EFTEM in this microscope - instead look for iron-deficient regions (dark areas in Fe-M jump ratio image). Still a challenge by this method.**

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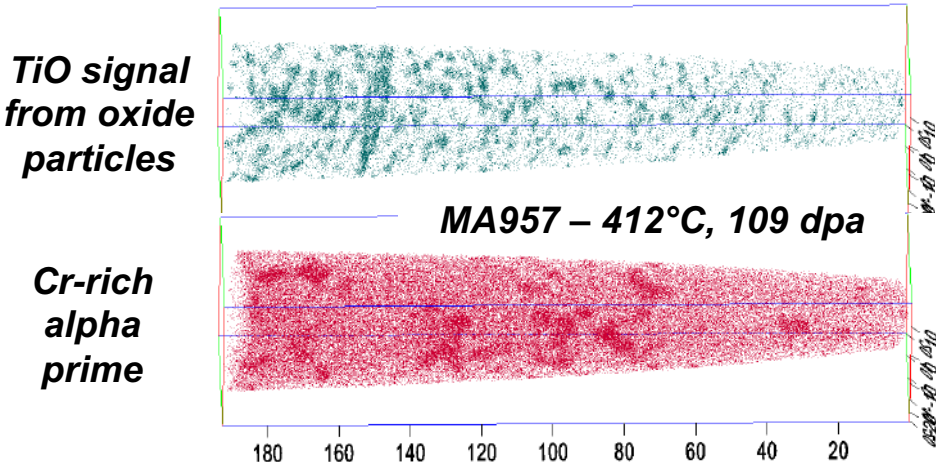
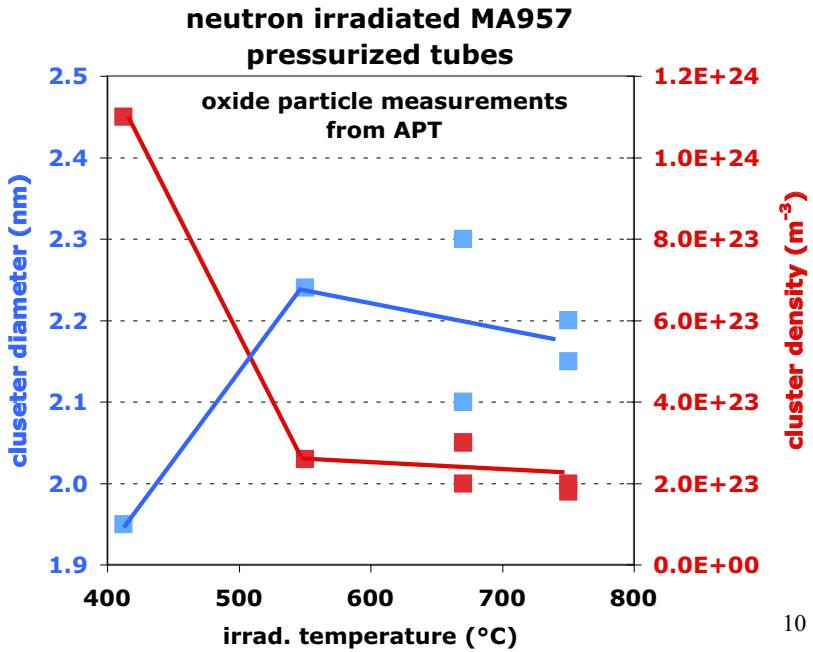
Microstructure/Chemistry Within Grains – APT Examinations

- Using 110-120 dpa neutron irradiated MA957 from in-reactor pressurized tube creep specimens.
- Preliminary APT examinations completed on specimens irradiated at 412, 550, 670, and 750°C to 109-121 dpa.

Initial Results

- MA957 pressurized tubes have a small YTiO particle size of ~2 nm similar to newer ODS ferritics such as 14YWT.
- Composition is ~0.1Y-0.45Ti-0.45O.
- No apparent ballistic dissolution at these irradiation temperatures, but possibly a small difference in oxide particle population at 412°C, perhaps due to measurement statistics.
- Cr-rich alpha-prime clusters observed at 412°C irradiation temperature.





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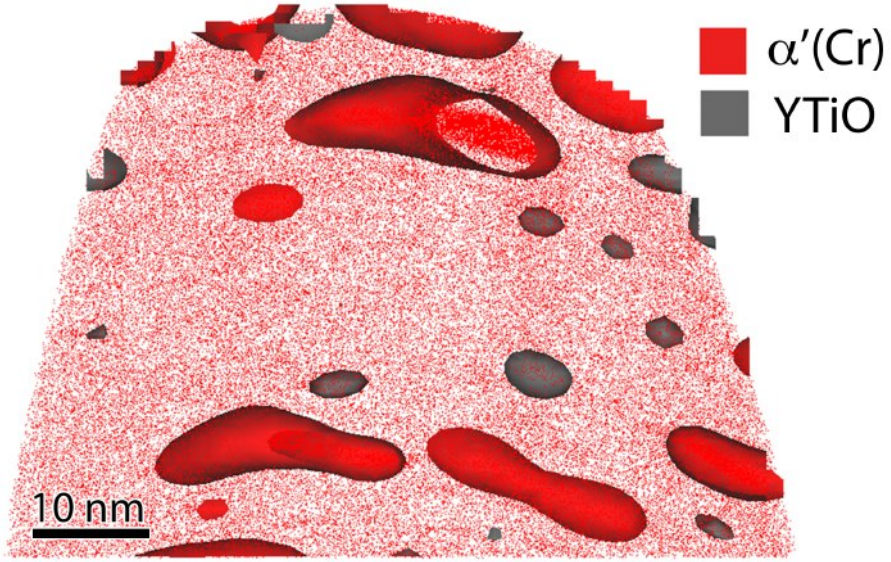
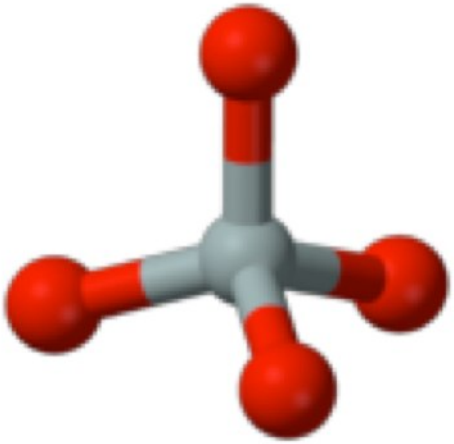
Microstructure/Chemistry Within Grains – APT Examinations

- **Details on alpha-prime after 412°C irradiation**
  - Segregation of Cr is 40-80 at%
  - Size is 5 -25 nm



- **Alpha-prime and YTiO are roughly anti-correlated, i.e., alpha prime sits between YTiO clusters rather than randomly intermixed.**



Anti-correlated Cr precipitation should maximize distance from 4 nearest YTiO neighbors

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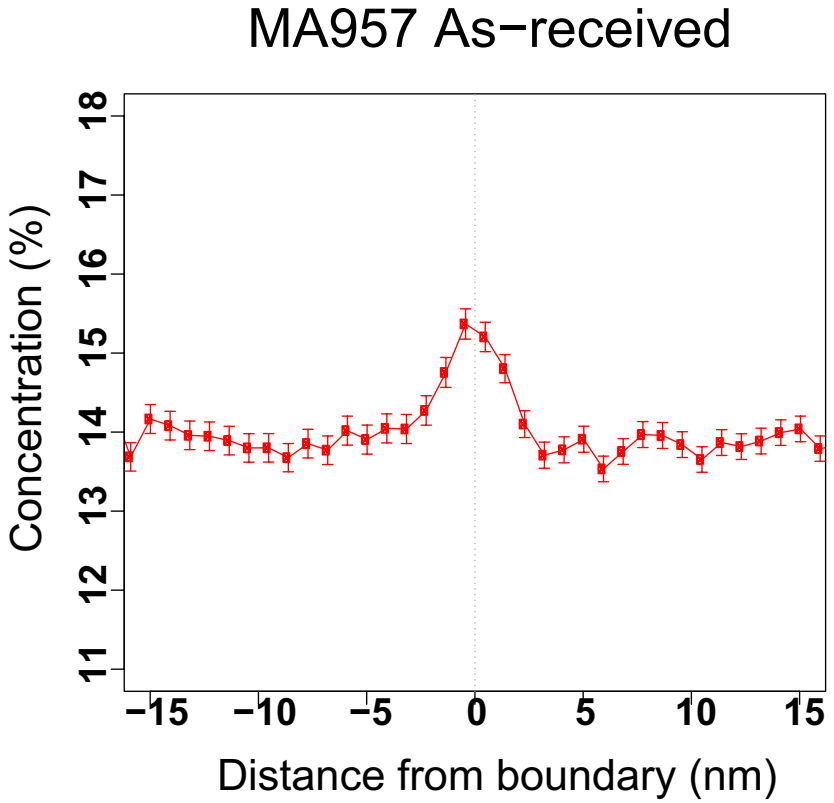
Grain Boundary Chemistry – TEM (STEM/EDS) Examinations

- Grain boundaries were aligned and positioned normal to the scan direction. 50 nm x 50 nm EDS maps were collected at a spot size of 0.75 nm and a step size of 0.9 nm.
- Assuming the boundary is straight in the vertical direction of the scan and the boundary is homogeneous, the data was binned to simulate a line scan
- This method prevents specimen destruction typical of traditional line scans



Slight enhancement of chromium at grain boundaries was found to be typical in unirradiated MA957.

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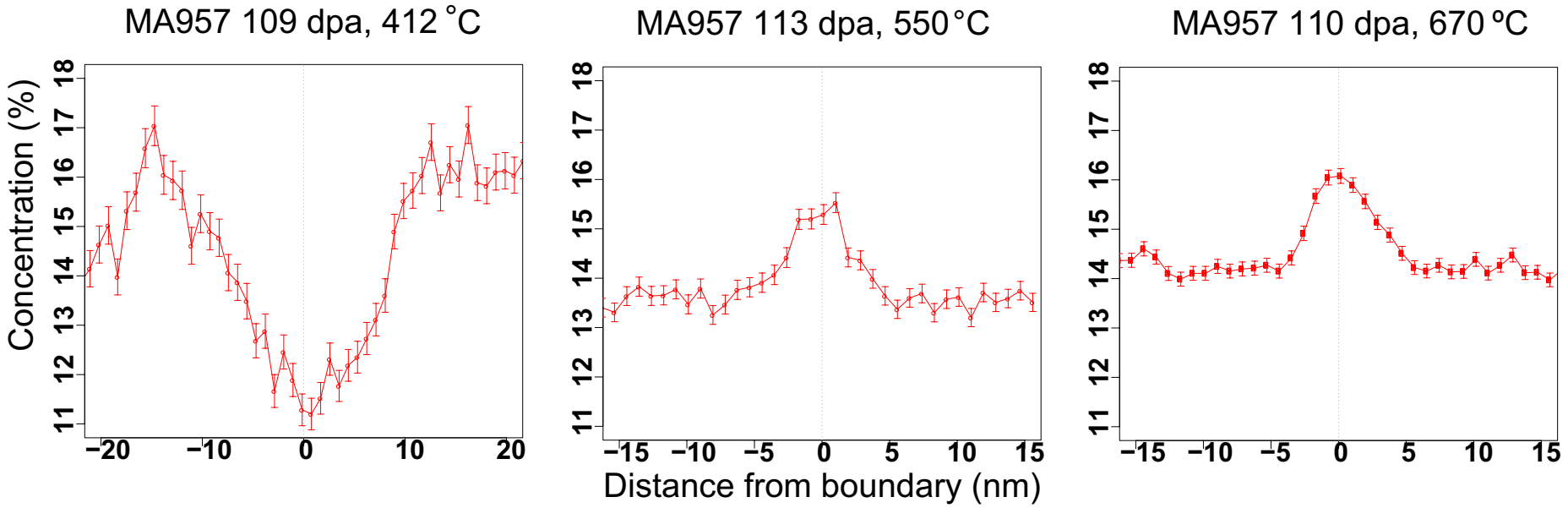
Grain Boundary Chemistry – TEM (STEM/EDS) Examinations



- **Clear depletion of Cr on grain boundaries after irradiation at 412°C. Bulk is 14%, value on grain boundary is ~11%. Depletion width is ~30 nm.**
- **At higher irradiation temperatures, grain boundary Cr profile was unaffected by irradiation.**

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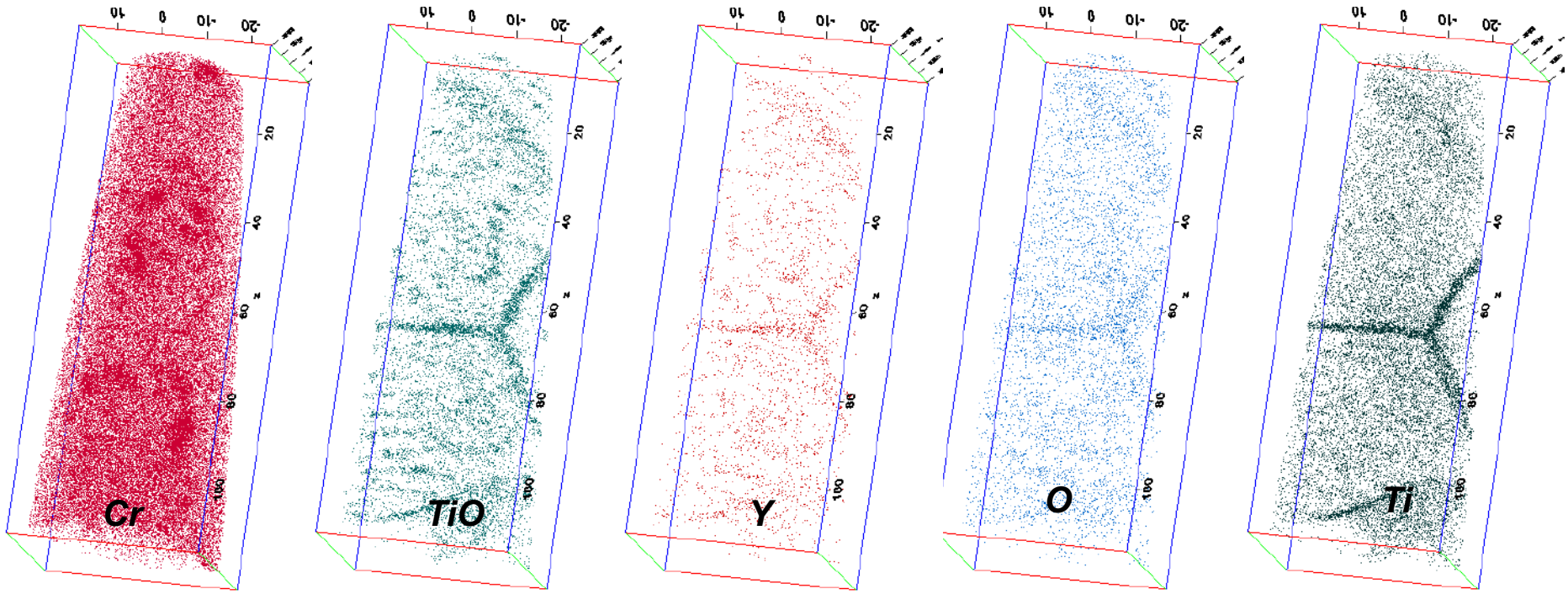
Grain Boundary Chemistry – APT Examinations

- **Grain boundary observation after irradiation to 109 dpa at 412°C**
  - Y, Ti, O, Ni, Si enrichment at intersected grain boundary.
- **Different results at different irradiation temperatures. Further observations needed to better understand.**



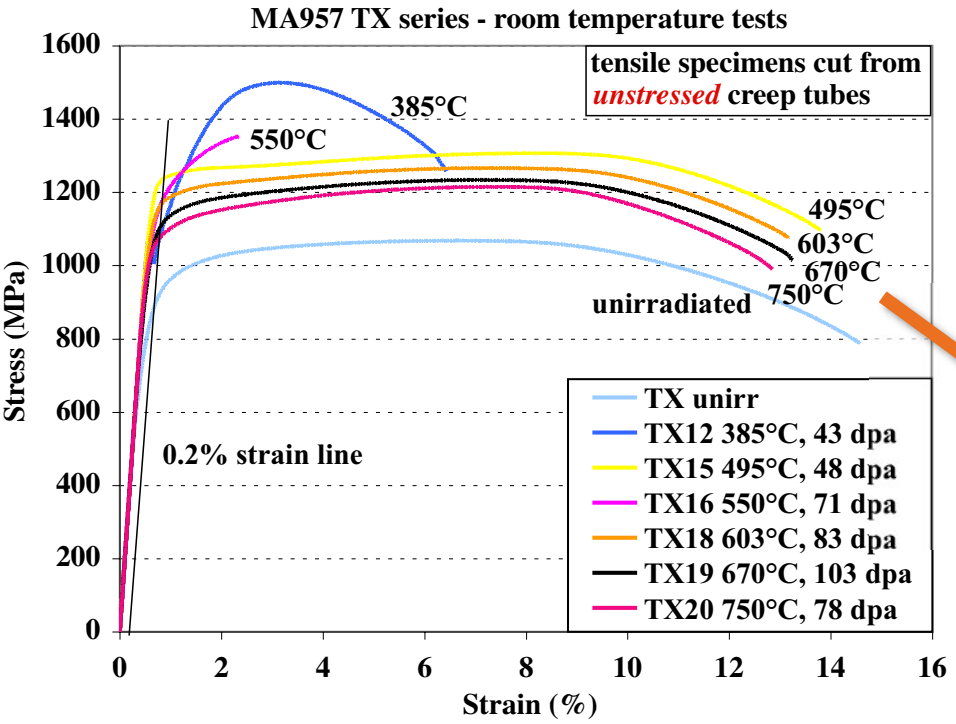
Cr-rich alpha prime

412°C, 109 dpa – map of selected elements

needle 1124

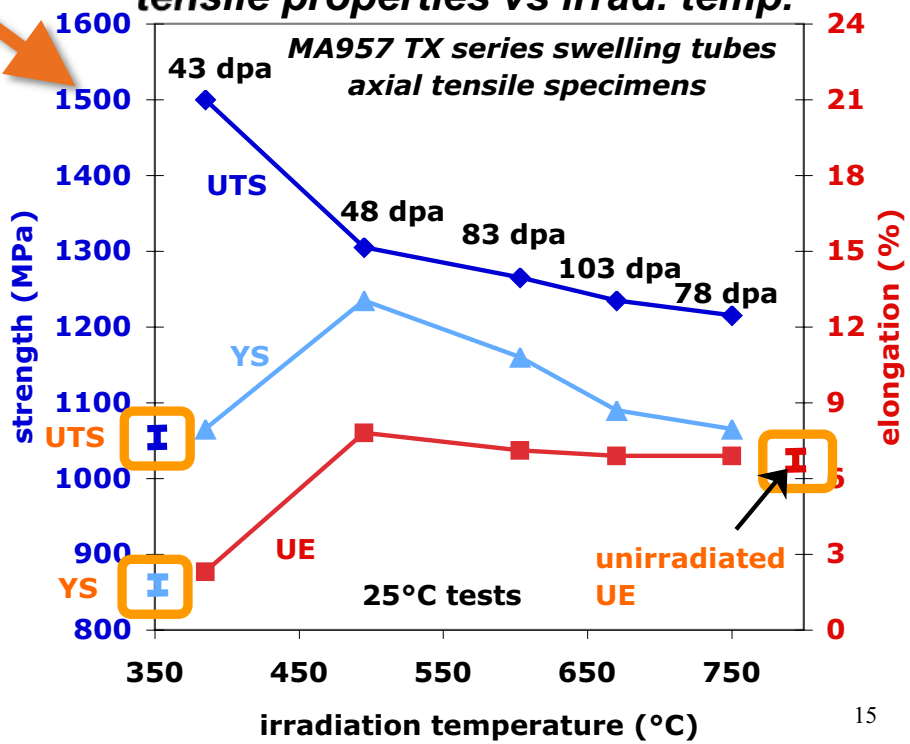
Tensile Properties – TX Swelling Tubes

tensile test data from unpressurized tubes



Strength: Irradiated yield/ultimate strength exceeds unirradiated values at all irradiation temperatures. Indicates microstructural evolution even at high temperatures where recombination and thermal point defect populations dominate evolution.

tensile properties vs irrad. temp.



Ductility: Irradiated uniform elongation equals or exceeds unirradiated value *except at 385°C irradiation temperature*.

Note: Lower YS of 385°C irradiated specimen due to early cracking.

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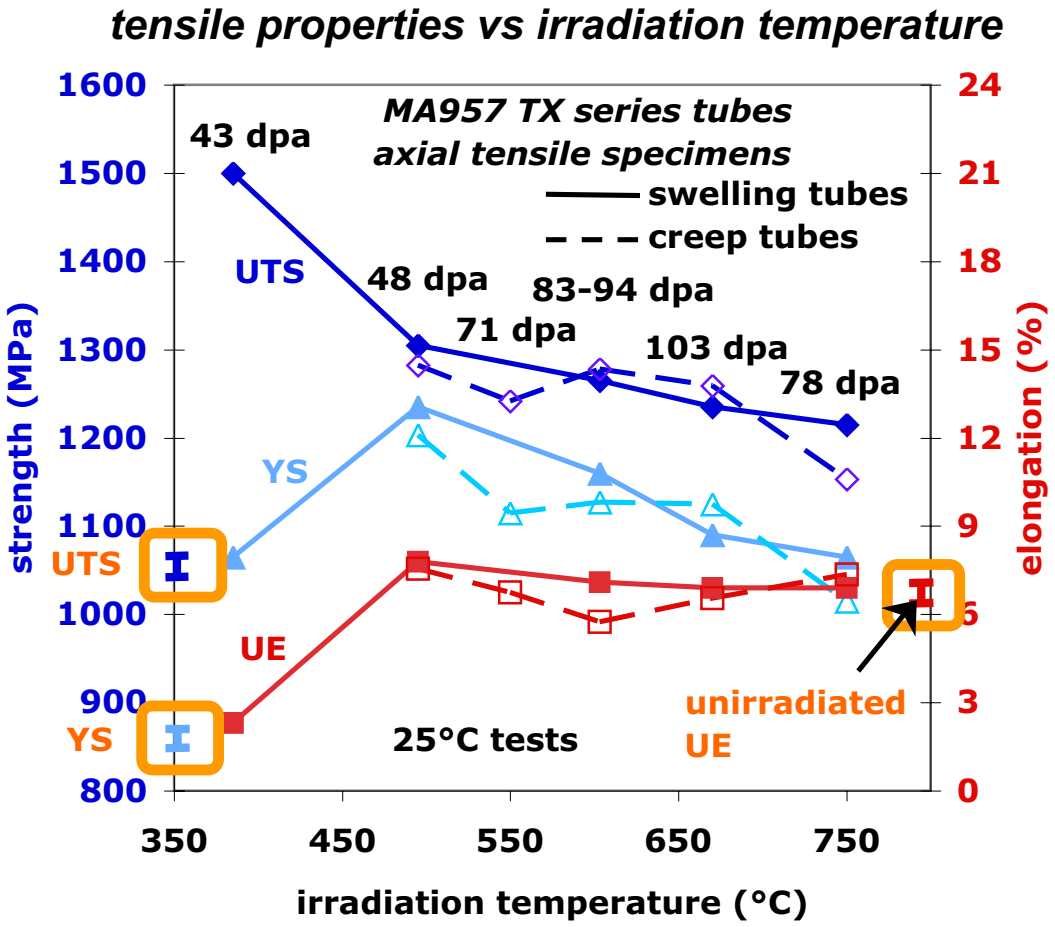
Tensile Properties – Comparison of Swelling and Creep of TX Tubing



Tensile properties of stressed tubing are similar to unstressed tubing suggesting little difference in dislocation and oxide particle microstructures as a result of creep deformation.

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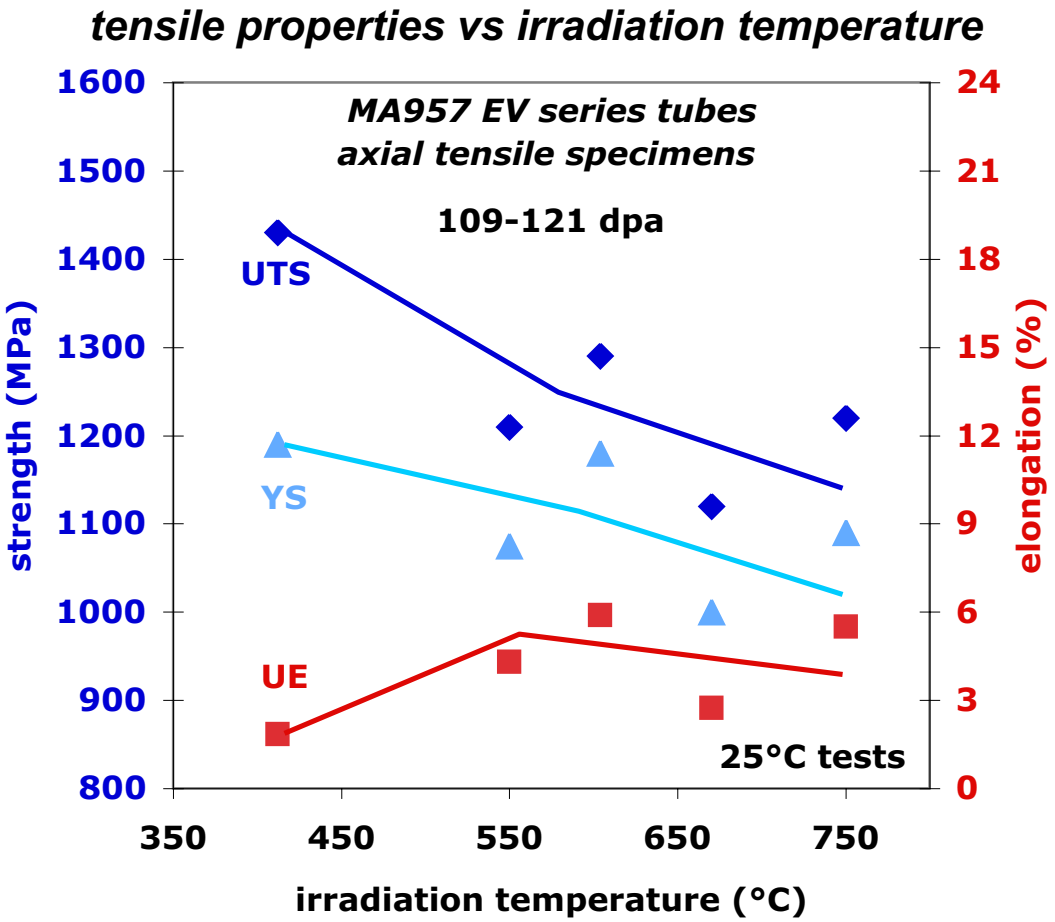
Tensile Properties – EV Swelling Tubes

- **More scatter in EV series of tubing, but tensile property trends are identical to the TX series tubing.**
  - Highest strength and lowest ductility at 412°C
  - Uniform elongation is relatively independent of irradiation temperature from 550-750°C.



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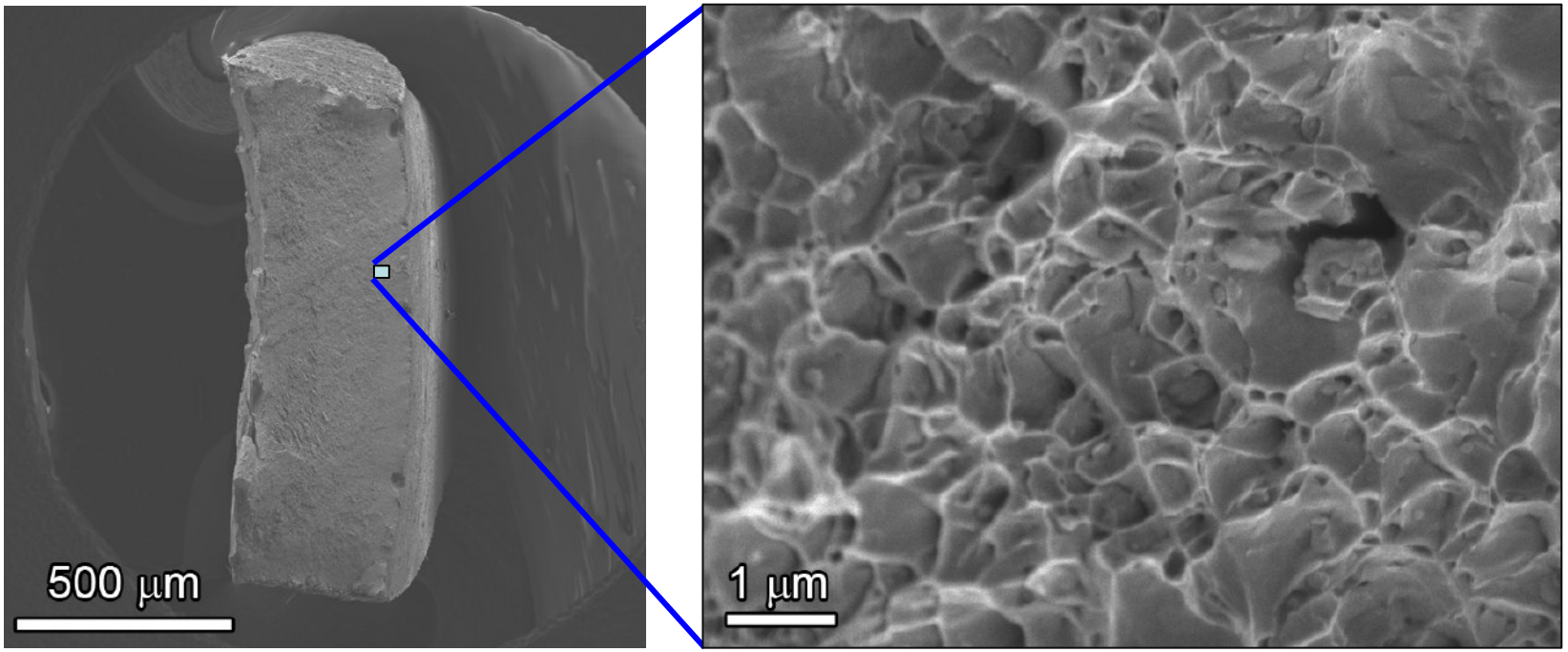
Tensile Testing – SEM of EV Series Fracture Surfaces

EV34 – 550°C, 113 dpa

tensile specimen fracture face



After irradiation at 550°C – 750°C to 110-121 dpa, classic ductile dimple fracture was observed.

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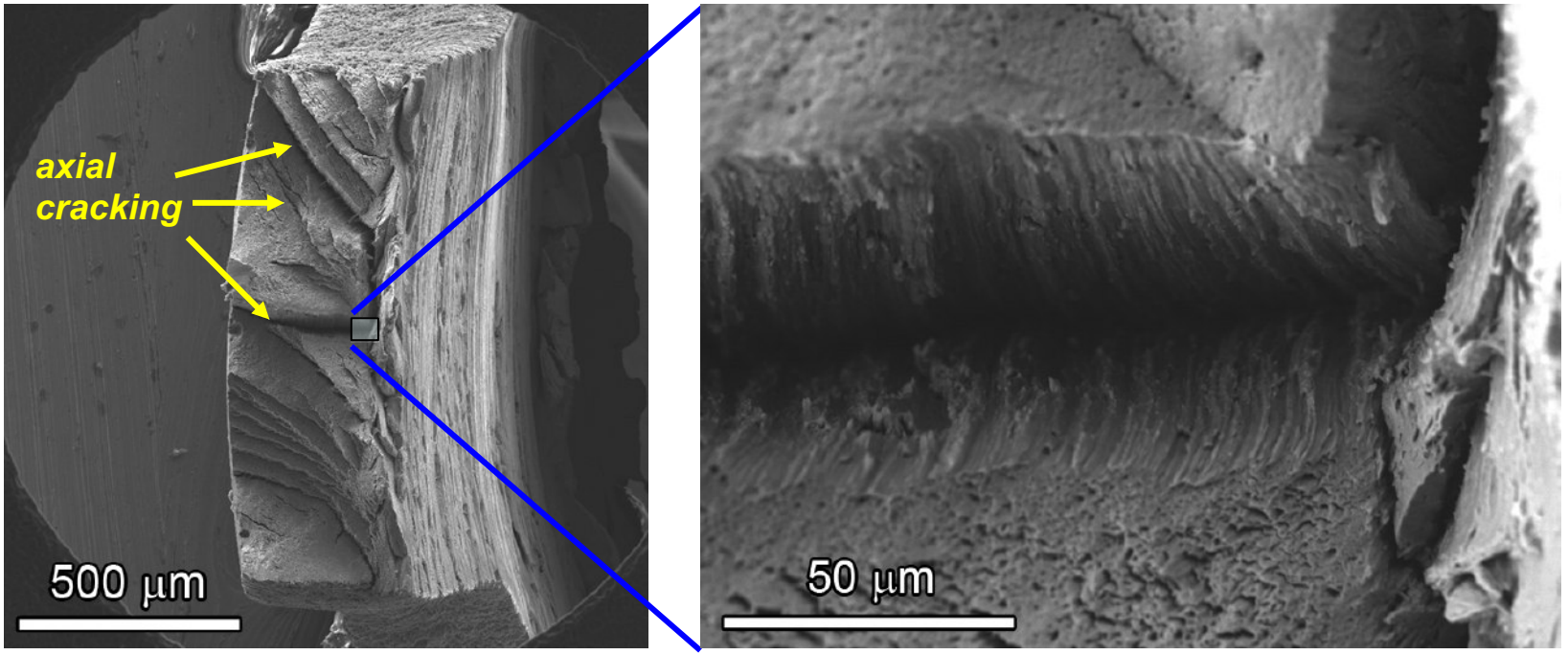
Tensile Testing – SEM of EV Series Fracture Surfaces

EV46 – 412°C, 109 dpa

tensile specimen fracture face



After irradiation at 412°C to 109 dpa, ductile-dimple fracture observed on fracture faces, but extensive axial cracking (splitting) of the specimen along the gauge length also observed.

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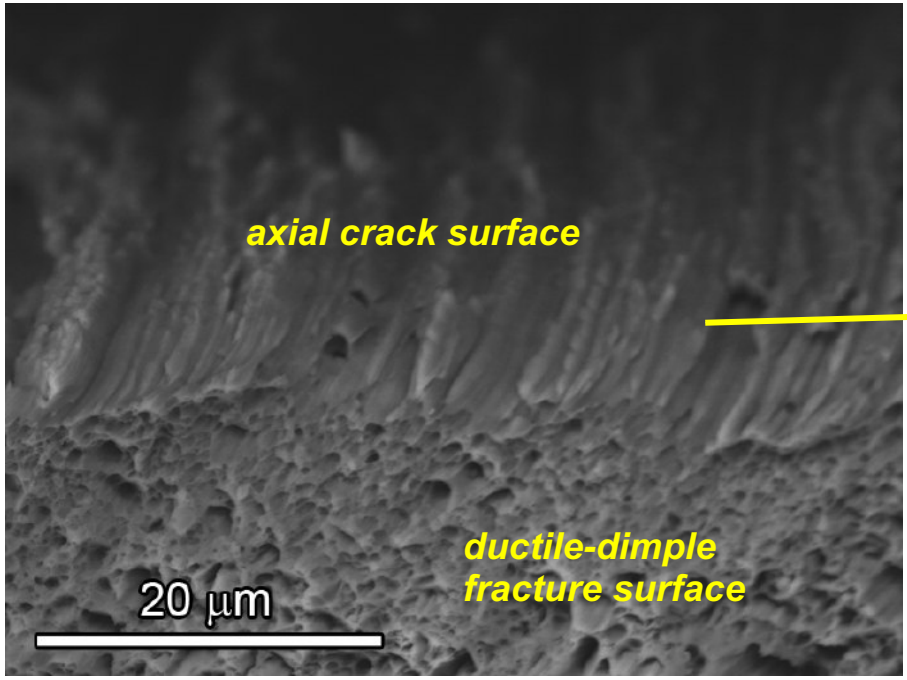
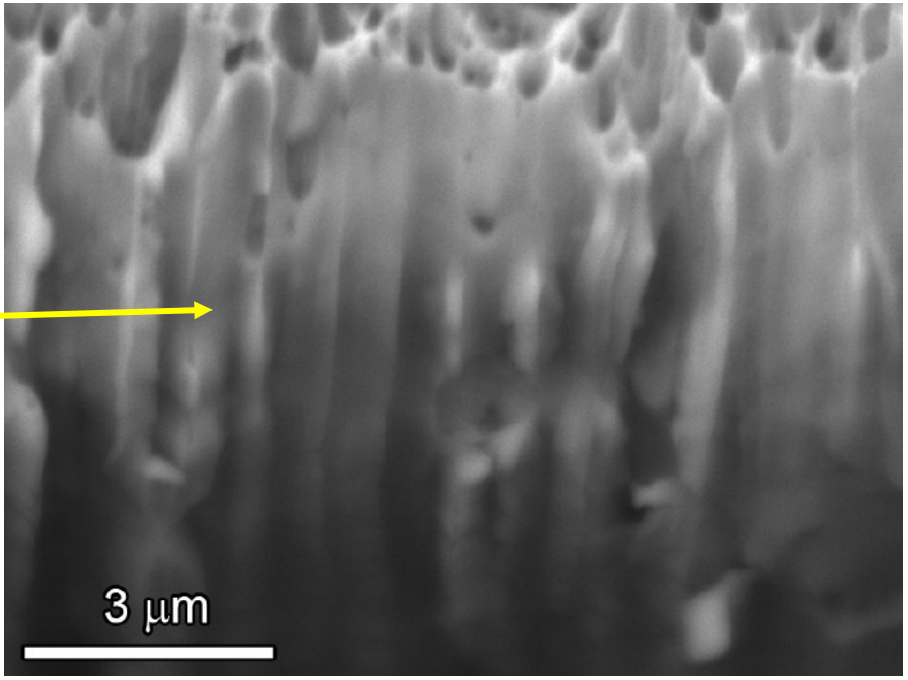
Tensile Testing – SEM of EV Series Fracture Surfaces

EV46 – 412°C, 109 dpa





Axial splitting appears to be occuring on the long sides of the grain boundaries.

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MA957 Ongoing Summary & Conclusions

- **Microstructure**
  - No dissolution of oxide particles after 110-121 dpa from 412-750°C. Possible minor refinement in oxide particle population at 412°C. Need to examine 385°C.
  - Extensive alpha-prime formation after irradiation at 412°C. Certain to be present at 385°C, probably at higher density. Plan to examine 385°C.
  - Irradiation is causing grain boundary segregation at 412°C. Just getting started, more exams planned.
- **Room Temperature Tensile Properties**
  - Significant irradiation hardening at 385-750°C, especially at 385 and 412°C.
  - Uniform elongation only suffers at 385 and 412°C.
  - Change in fracture mode at 385 and 412°C. Currently available microstructure exams suggest a combination of matrix hardening and grain boundary embrittlement.
  - Tensile properties may be better at elevated tensile test temperatures.
- **ODS ferritics have promise as a material for high temperature irradiations. Appear to need improvement to be viable for use at lower irradiation temperatures. Further analysis needed to understand causes.**