

Properties of Advanced ODS Alloys and Routes for Application

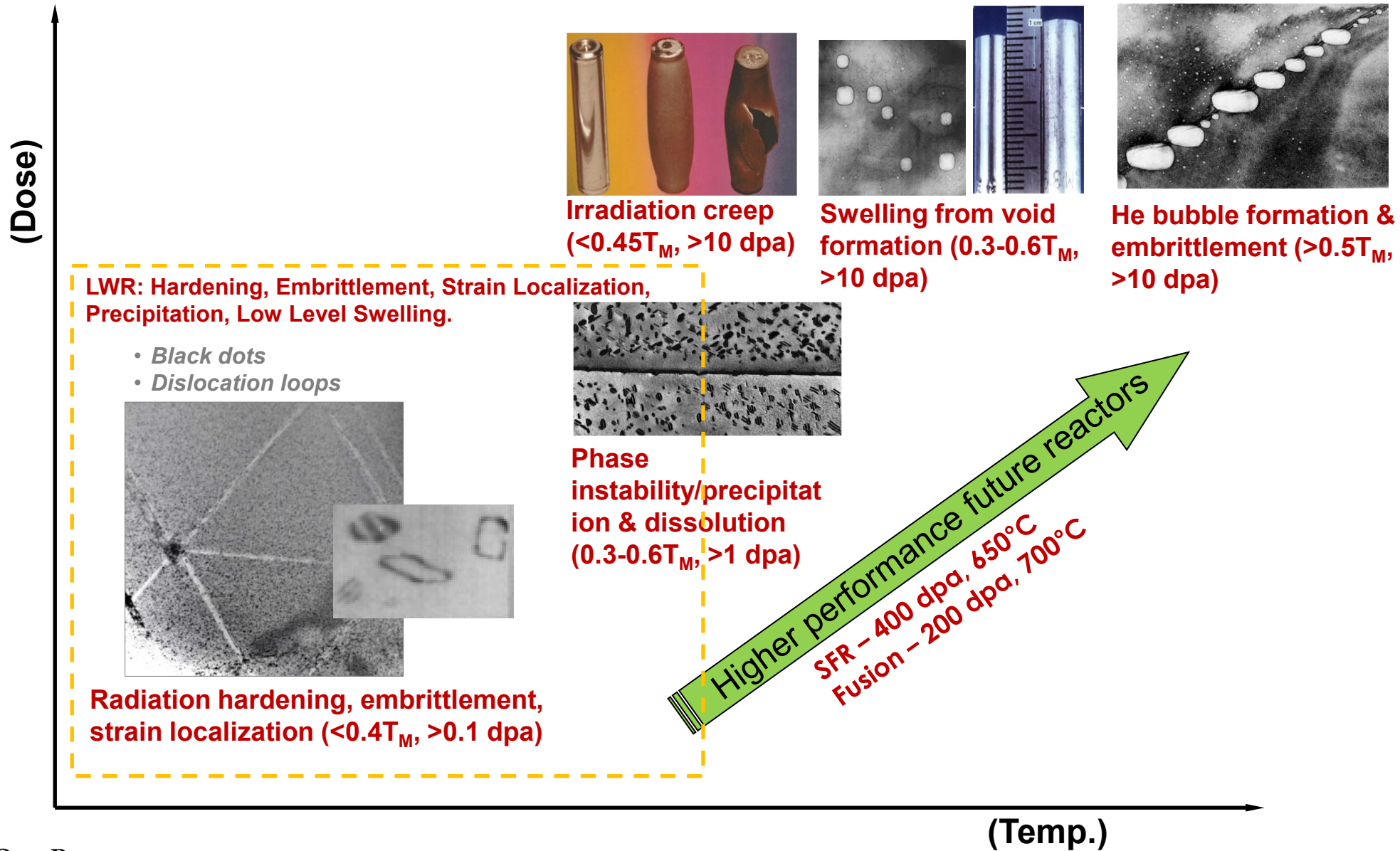
TS Byun, David T. Hoelzer, Caleb Massey (ORNL), Stuart A. Maloy (PNNL)

*GAIN Innovative Materials Research Workshop, Wednesday, June 15, 2022,
1:00 pm - 5:30 pm, ANS Annual Meeting, Hilton Anaheim, Anaheim, CA*

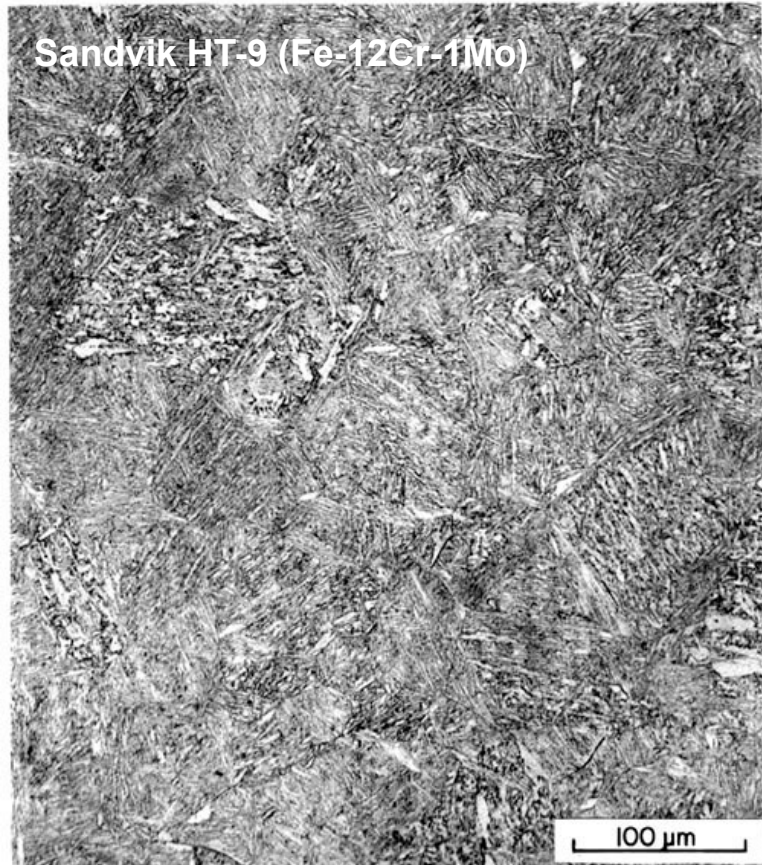
Contents

- Introduction
- Mechanical Properties & Irradiation Responses
- Latest Tube Processing
- Main Obstacle for Application
- Research Needed

Radiation Damage in Materials – Temp & Dose Dependence

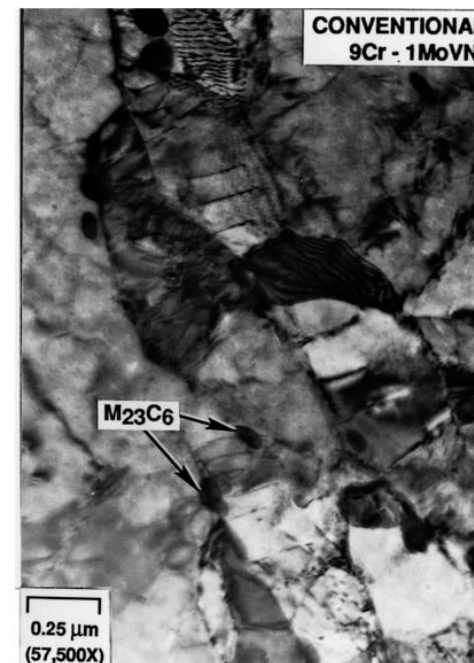
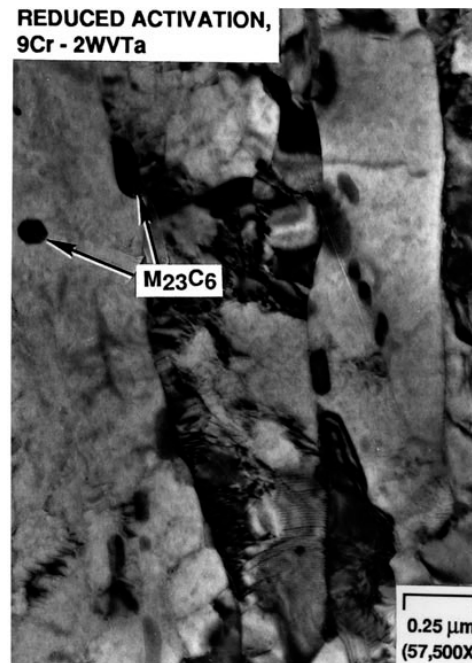


Materials for Advanced Reactors: Ferritic-Martensitic Steels for $T_{op} < 0.5T_M$



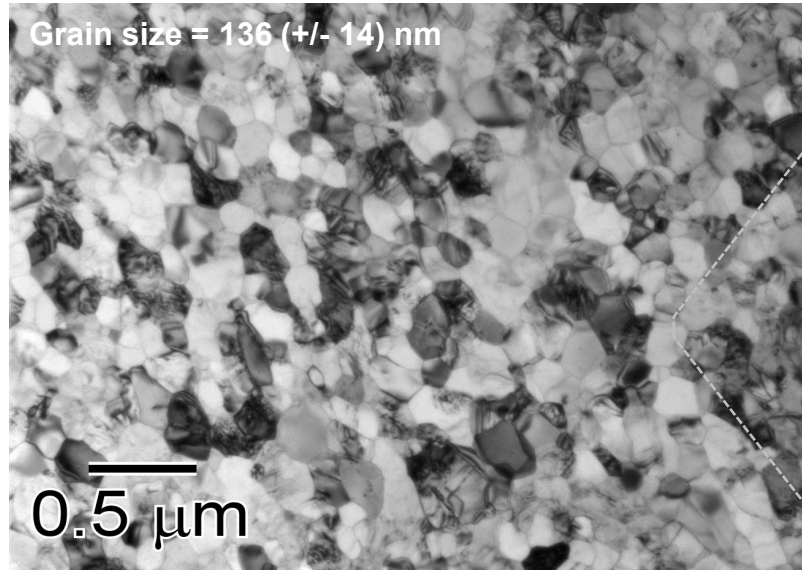
- F/M steels are used in normalized-quenched & tempered condition; Fe-(2 to 12)Cr steels.
- Microstructures of Cr-Mo and reduced-activation Cr-W steels are similar. (Fe-9Cr-2WVTa vs. Fe-9Cr-1MoVNB)
- Precipitates:
 - $M_{23}C_6$
 - Small amount of MX
 - **Nano carbides and nitrides in new materials (CNA)**

➤ **FM Steels: refined & hardened by laths and carbides**

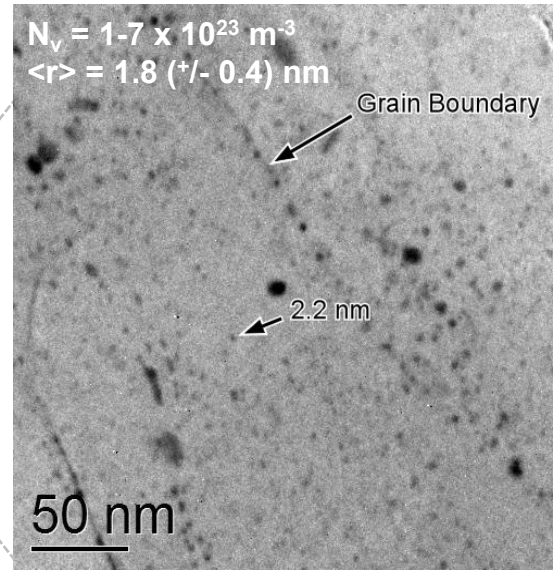


(R.L. Klueh, et al.
or J.T. Busby)

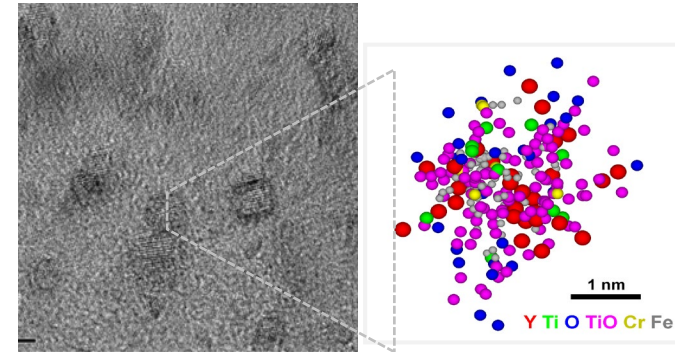
Materials for Advanced Reactors: Nanostructured Ferritic Alloys (NFAs) for $T_{op} < 0.65T_M$



BF-TEM of 14YWT SM10 Heat



Energy-filtered TEM Fe-M jump ratio map of the nanoclusters.

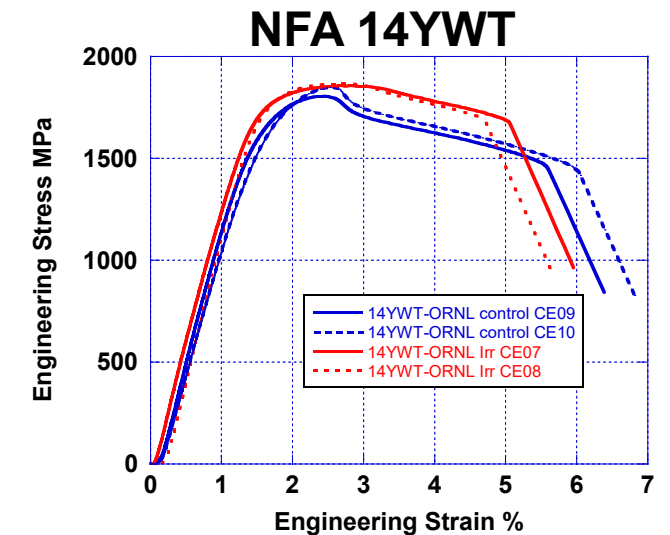
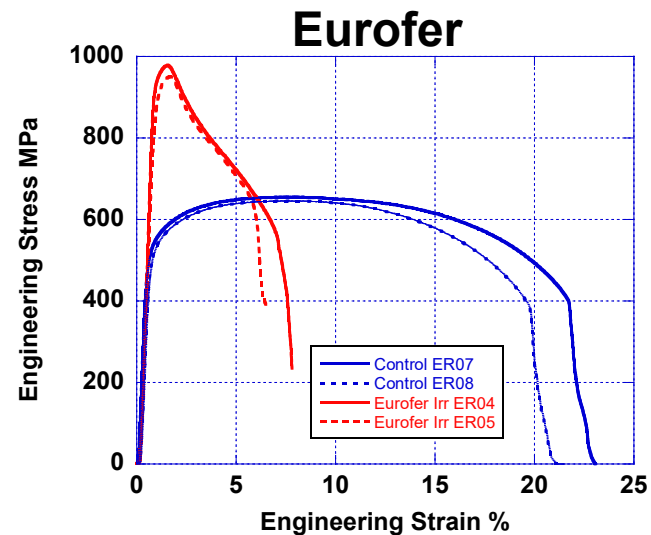
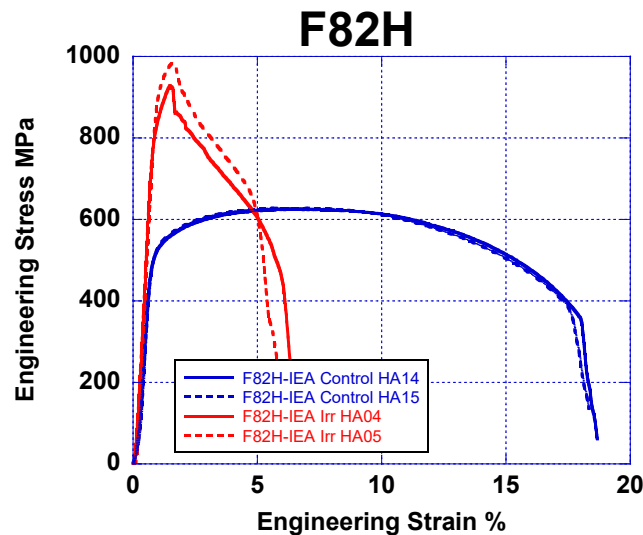
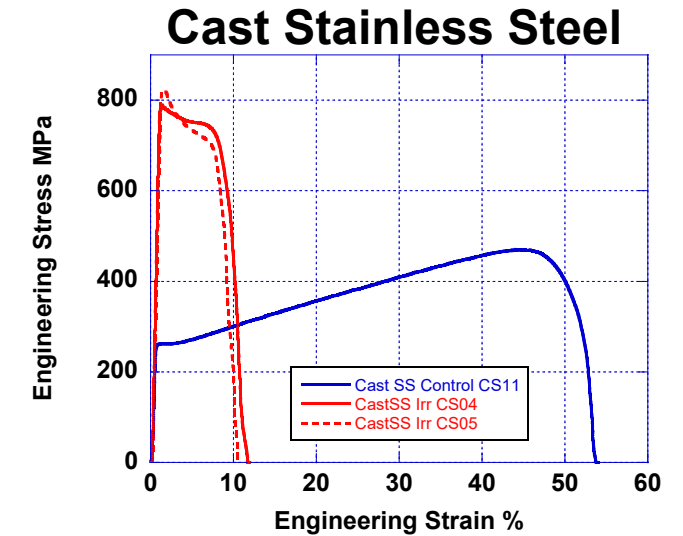
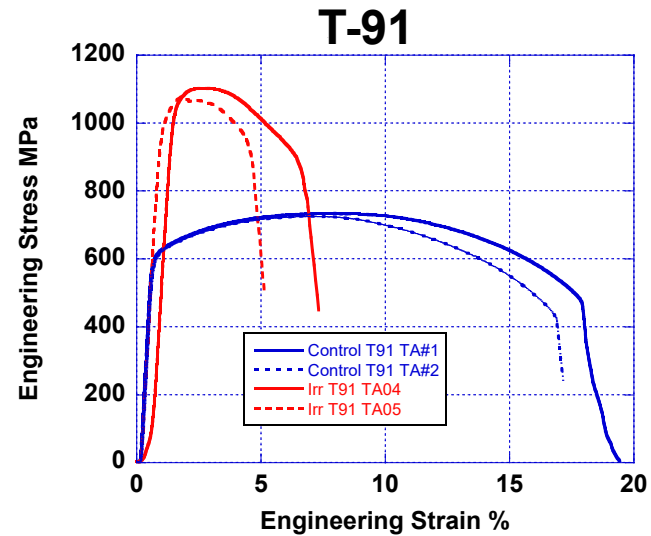
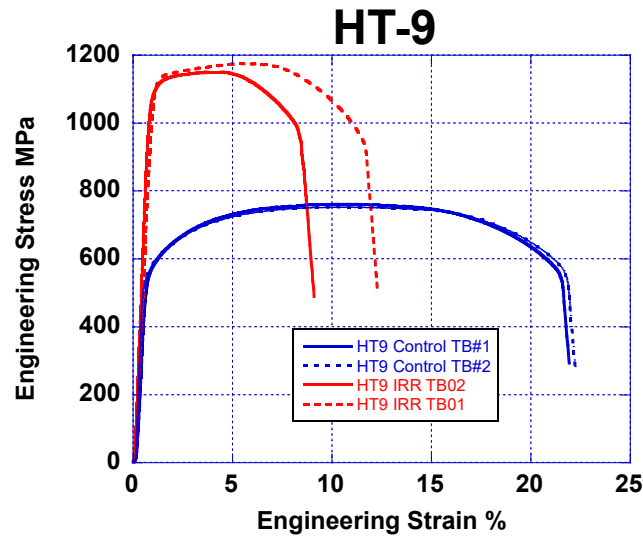


HR-TEM

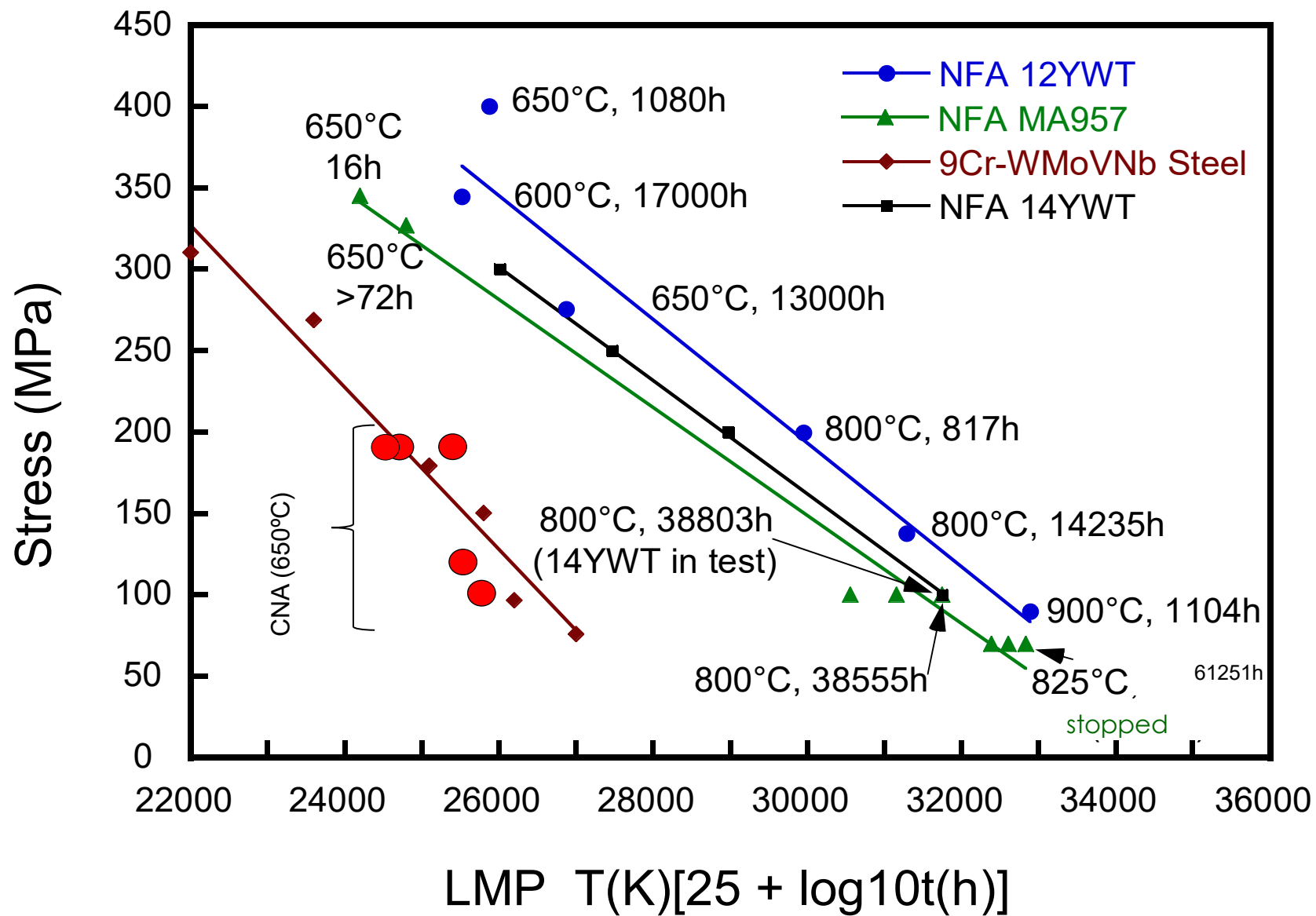
- NFAs or Advanced oxide dispersion strengthened (ODS) steels
- Nanograin structure & high density of nanoclusters.
- High energy mechanical alloying plus high-power consolidation
- Very large interfacial area (NC-matrix and grain boundaries) enhances recombination of point defects as well as accommodation of helium atoms or bubbles in irradiation.
- The ferritic nanograins & NCs in ferritic phase are known to be highly stable up to very high temperature $\sim 0.65 T_M$.
- *Considered as ideal materials for SFR cladding, fusion first wall, etc.*

Effect of Sink Strength on Radiation Hardening of Structural Alloys

All alloys/steels exhibited severe radiation hardening except for NFA 14YWT

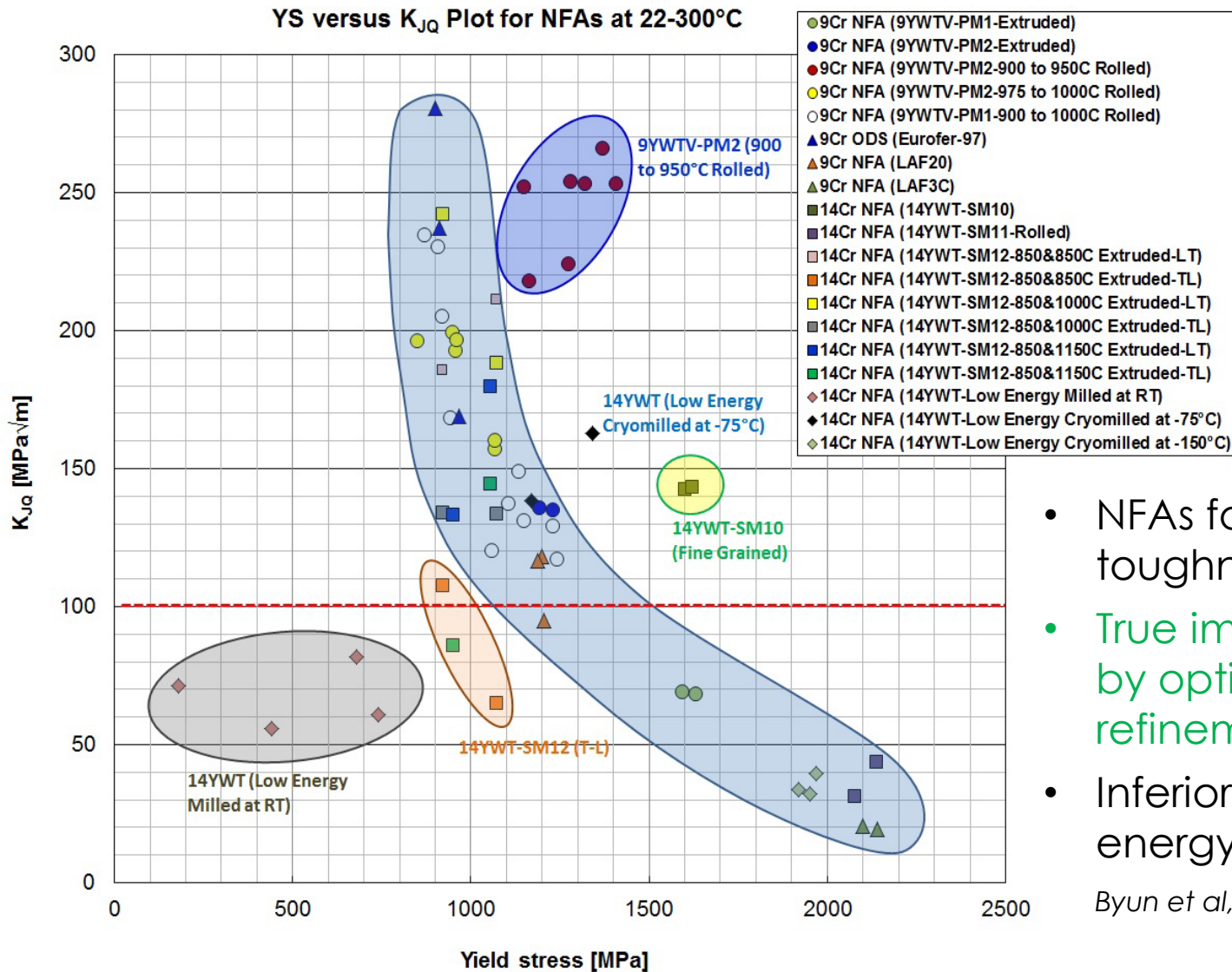


Creep Behavior (Applied Stress vs. Larson Miller Parameter)



- Klueh et al, JNM, 341 (2005) 103
- L. Tan et al., JNM 511 (2018) 598 (CNA – castable nanostructured Fe-9Cr alloy)

Fracture Toughness versus Strength Behavior of NFAs in Low-T Region

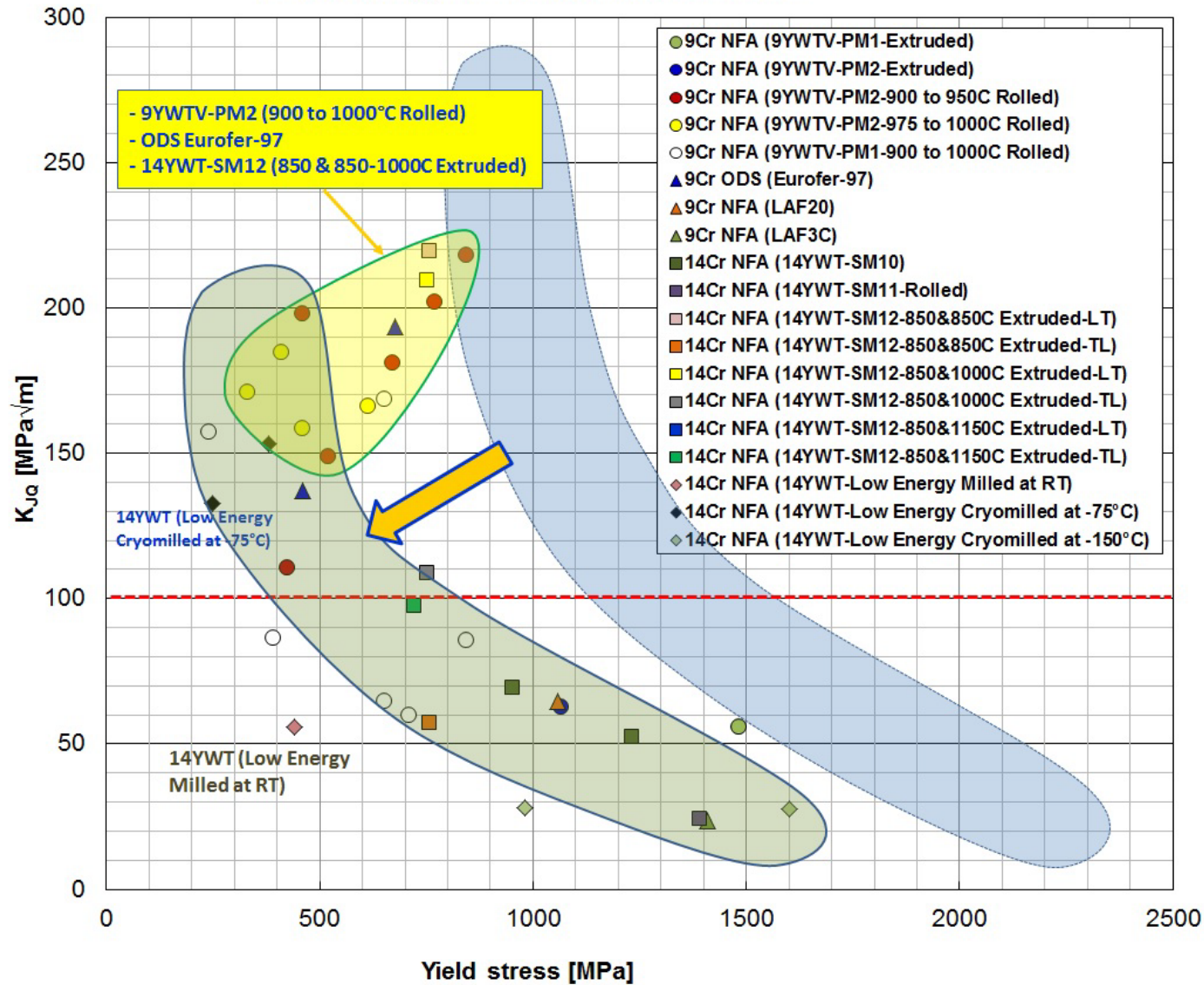


- NFAs follow general strength versus toughness behavior.
- True improvement can be achieved by optimum TMTs (9Cr) and grain refinement (14Cr).
- Inferior property is obtained by low energy milling and for T-L orientation.

Byun et al, JNM 2017

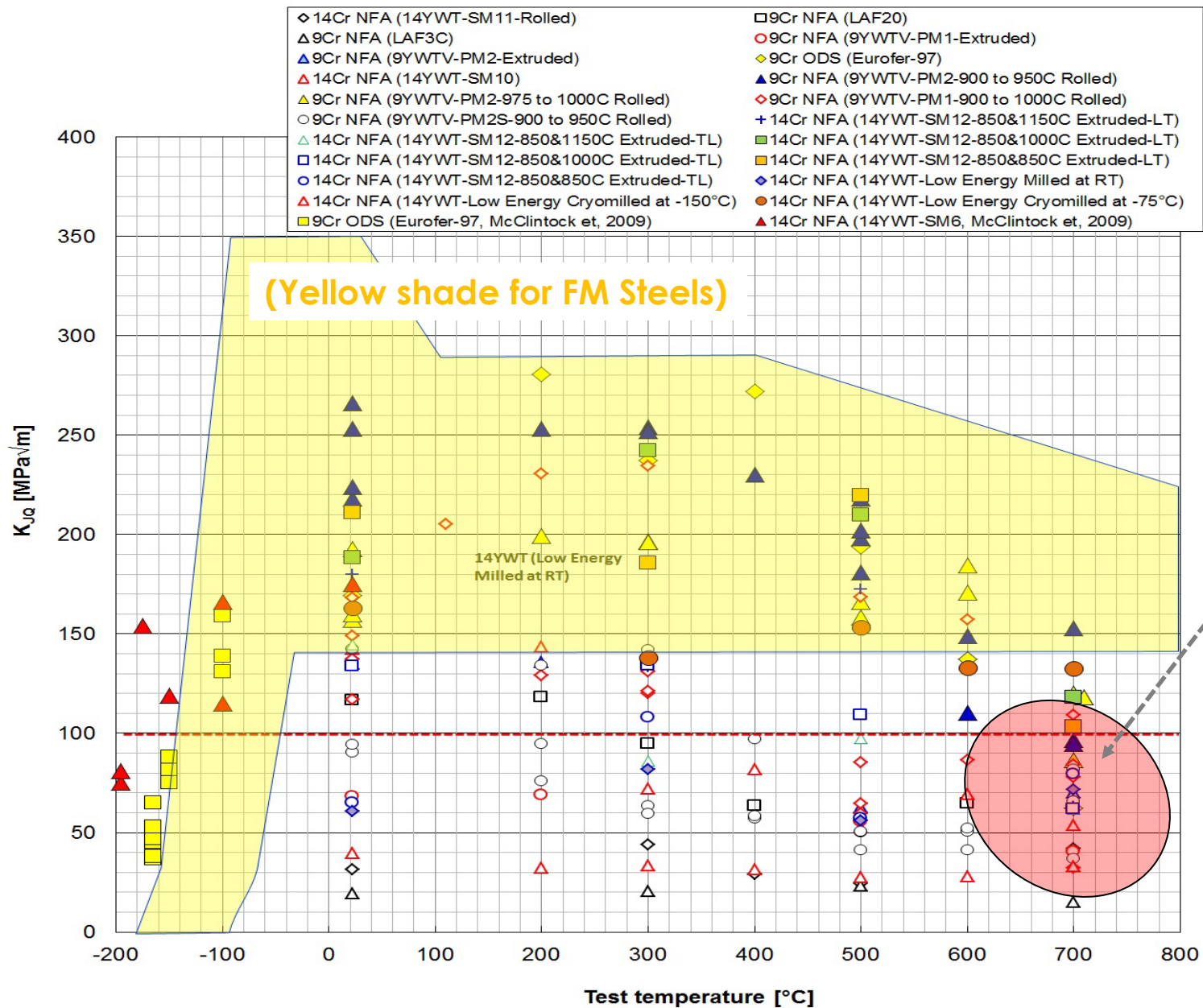
Fracture Toughness versus Strength Behavior of NFAs in High-T Region

YS versus K_{JQ} Plot for NFAs at 500-600°C



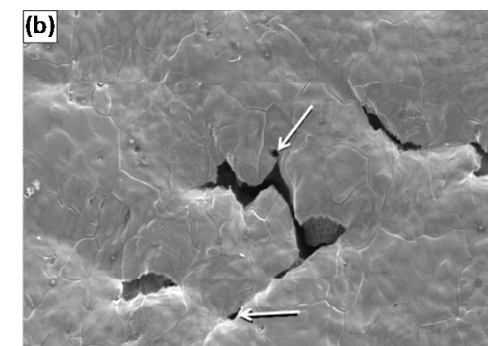
- The fracture property of NFAs generally deteriorates at high temperatures (>300°).
- High fracture toughness can be retained in the 9Cr and 14Cr NFAs with optimum TMTs.

Temperature Dependence of Fracture Toughness in NFAs

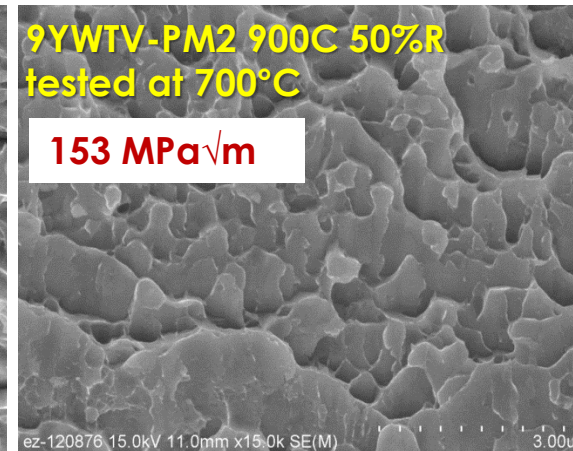
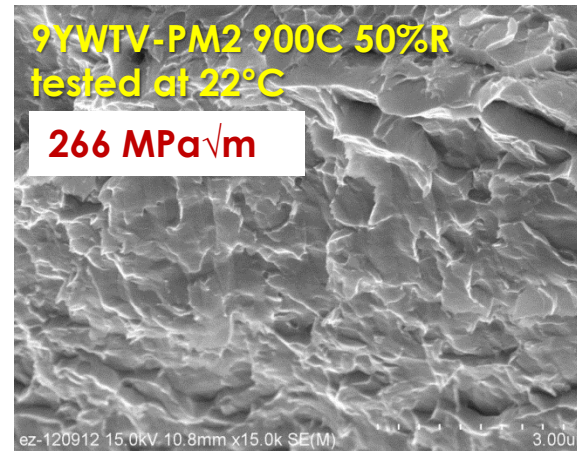
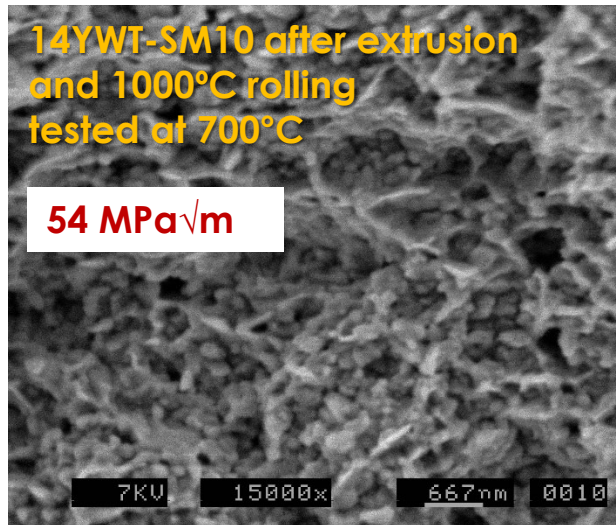
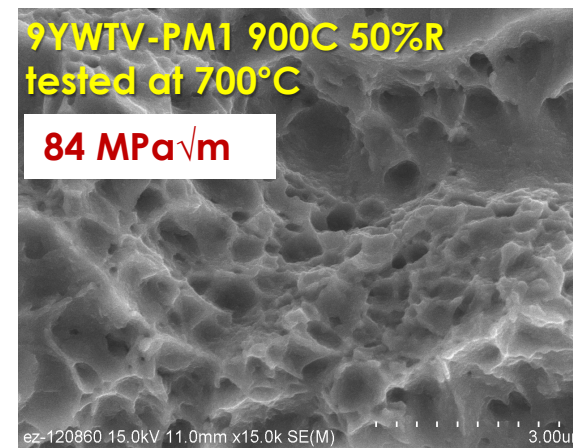
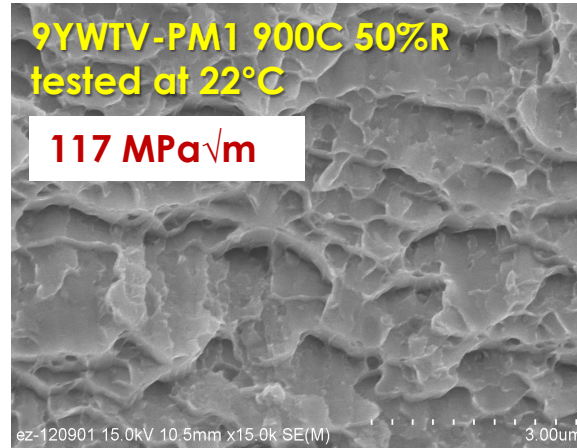
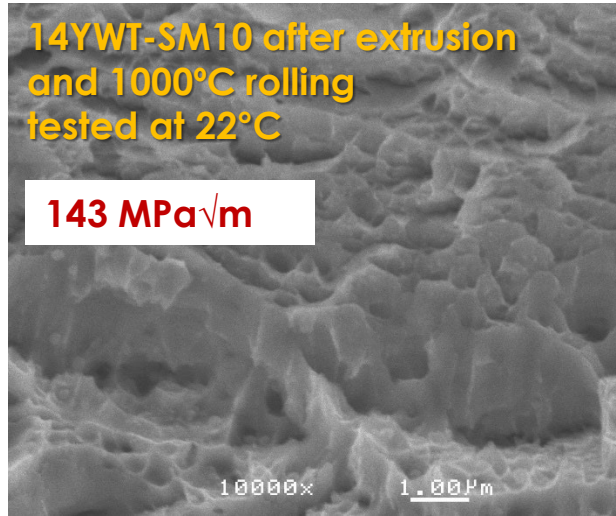


❖ **Coarse & nonuniform microstructures and excessive O and N contents lead to low fracture toughness.**

Grain (aggregate) boundary loses bond strength.



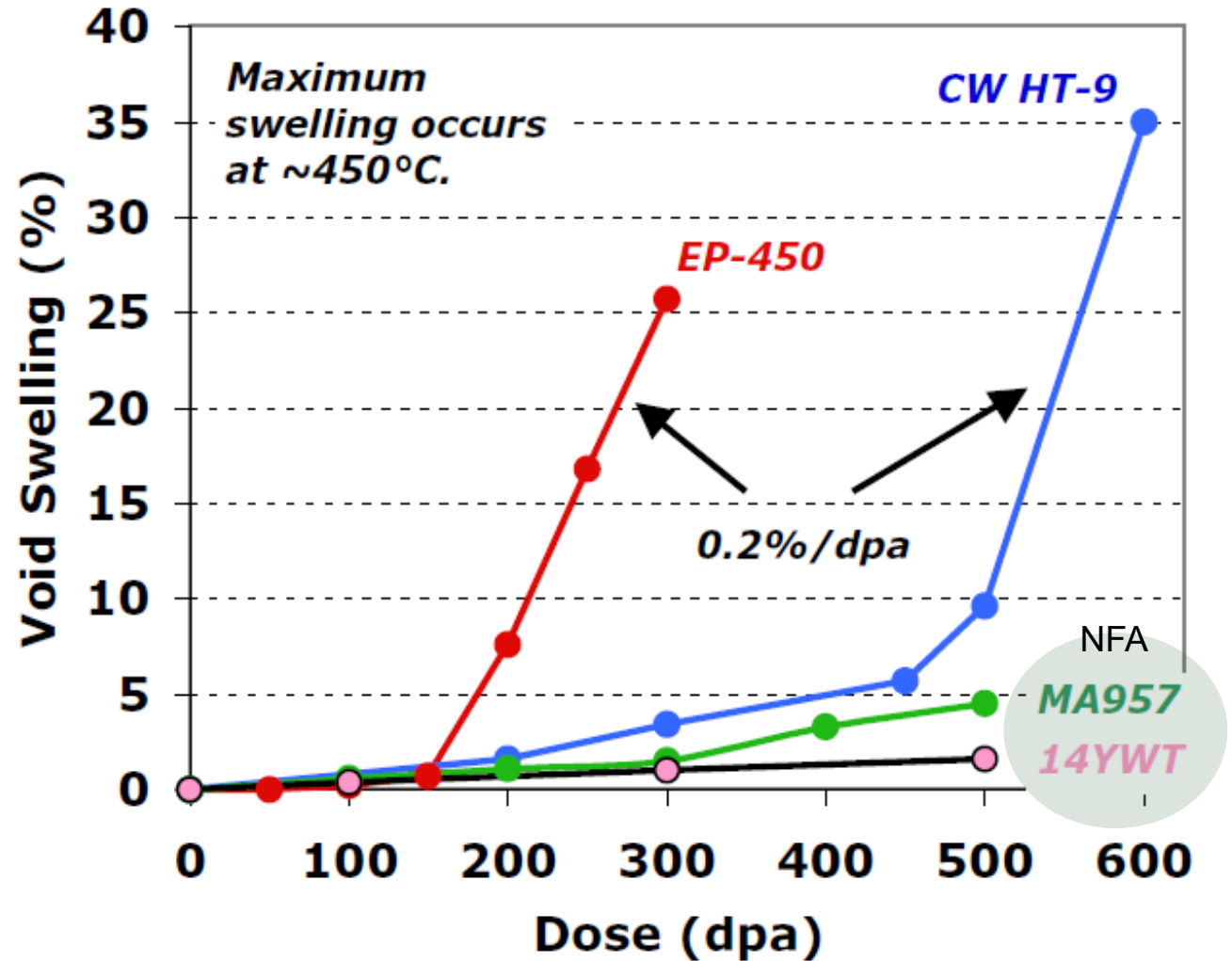
Mechanism for High Fracture Toughness



- A change in high-T fracture mechanism from boundary decohesion (and shallow dimple) formation to formation of flake-like shear tongues.

High Sink Strength & Void Swelling at High Doses

- High-dose ion irradiation at the Kharkov Institute of Physics and Technology
- ESUVI accelerator
 - 1.8 MeV Cr³⁺ ions
 - 10⁻² dpa/sec (100 dpa/hr)
 - Non-rastered beam
 - 100, 300 and 500 dpa
 - 400, 450 and 500°C
- Tempered F-M steels (EP-450/13Cr-2MoVNbB and HT-9/Fe-12Cr-1Mo-0.5W-0.5Ni-0.25V-0.2C) experience 0.2% swelling rates
- 14YWT and MA957 show extended low swelling regimes up to 500 dpa

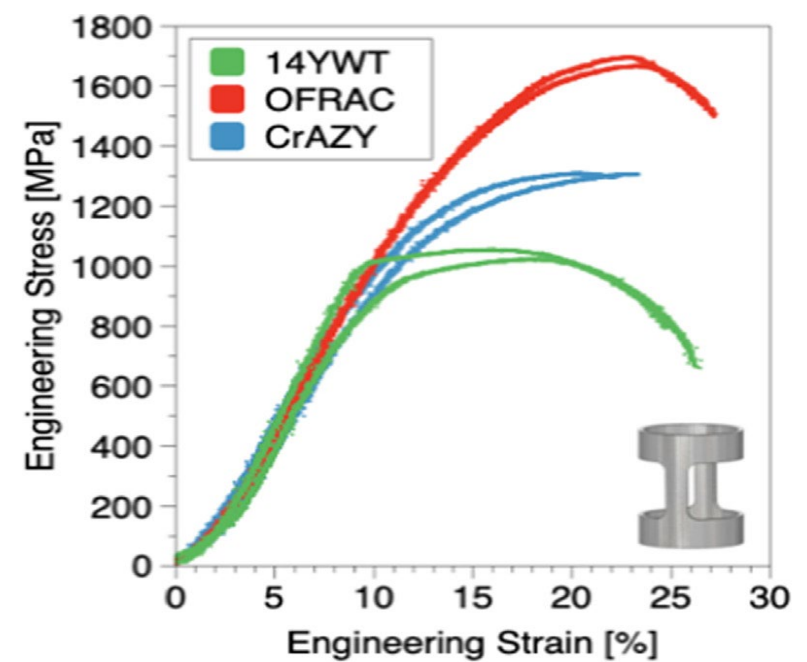


M.B. Toloczko, V.V. Bryk, F.A. Garner, D.T. Hoelzer and S.A. Maloy, FCRD Report , (2014)

Processes for Advanced ODS Cladding

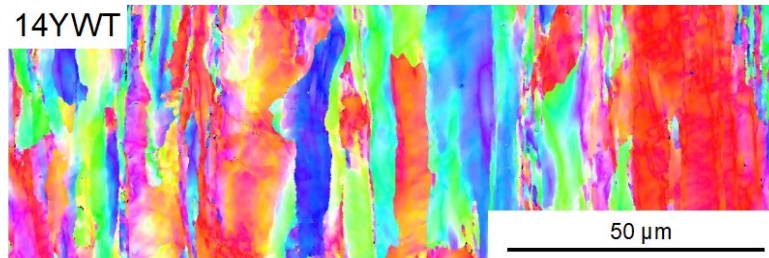
- 14YWT
- OFRAC
- CrAZY

wt.(%)	14YWT-NFA1	OFRAC-OR1	CrAZY-OR1
Fe	81.76	85.90	83.57
Cr	14.40	12.35	9.71
Al	-	-	6.03
W	3.10	-	-
Mo	-	0.95	-
Ti	0.39	0.20	-
Nb	-	0.30	-
Zr	-	-	0.27
Y	0.21	0.18	0.22
O	0.116	0.087	0.114
C	0.016	0.026	0.069
N	0.008	0.011	0.017



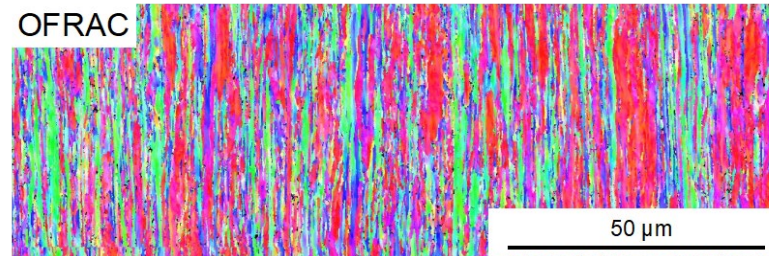
Pilger/Annealed into Thin-Walled Tube

- 40% Reduction + 1200°C/1 h
- 40% Reduction + 1200°C/1 h
- 40% Reduction + 1200°C/1 h
- 40% Reduction + 750°C/1 h



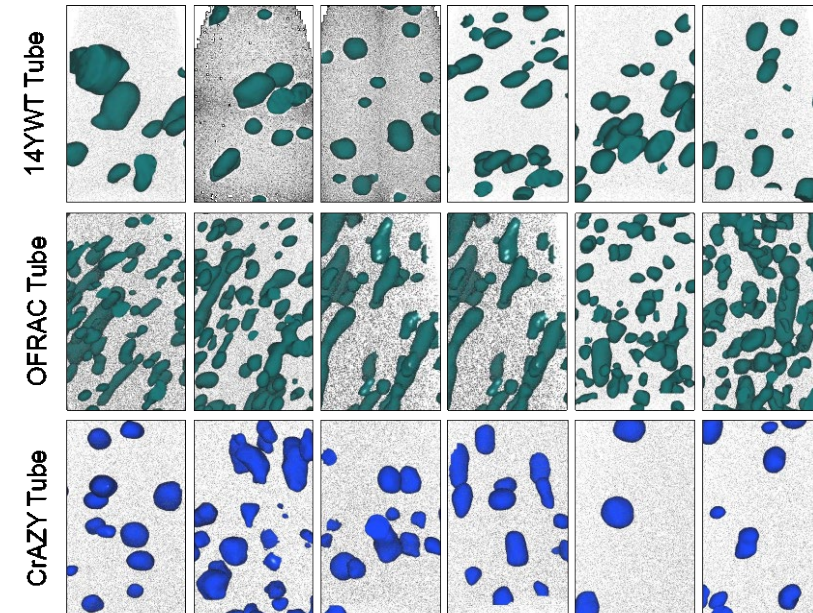
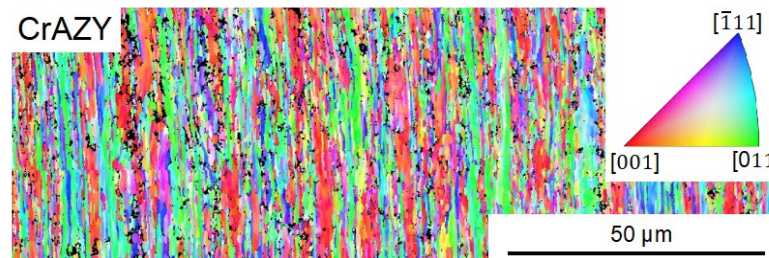
Pilger/Annealed into Thin-Walled Tube

- 40% Reduction + 950°C/30 min
- 40% Reduction + 850°C/30 min
- 40% Reduction + 850°C/30 min
- 40% Reduction



Pilger/Annealed into Thin-Walled Tube

- 40% Reduction + 950°C/30 min
- 40% Reduction + 850°C/30 min
- 40% Reduction + 850°C/30 min
- 40% Reduction

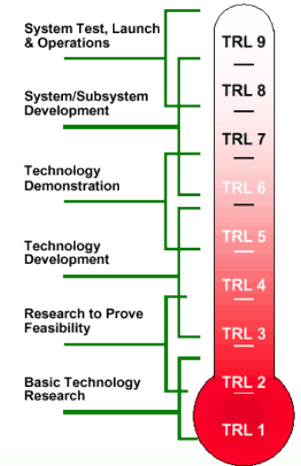


C. Massey et al, Mater. Des. 2022

Tube Fabrication with NFA OFRAC

□ Current status and critical issue

- Some of the fine tuned NFAs have demonstrated high performance, such as high temperature (creep) strength, reasonable toughness, high resistance to radiation (defect and gas) damage and oxidation/corrosion.
- **NFA development in their microstructures and properties is matured, nearing practical application for some components such as fuel cladding (TRL 5-6?)**



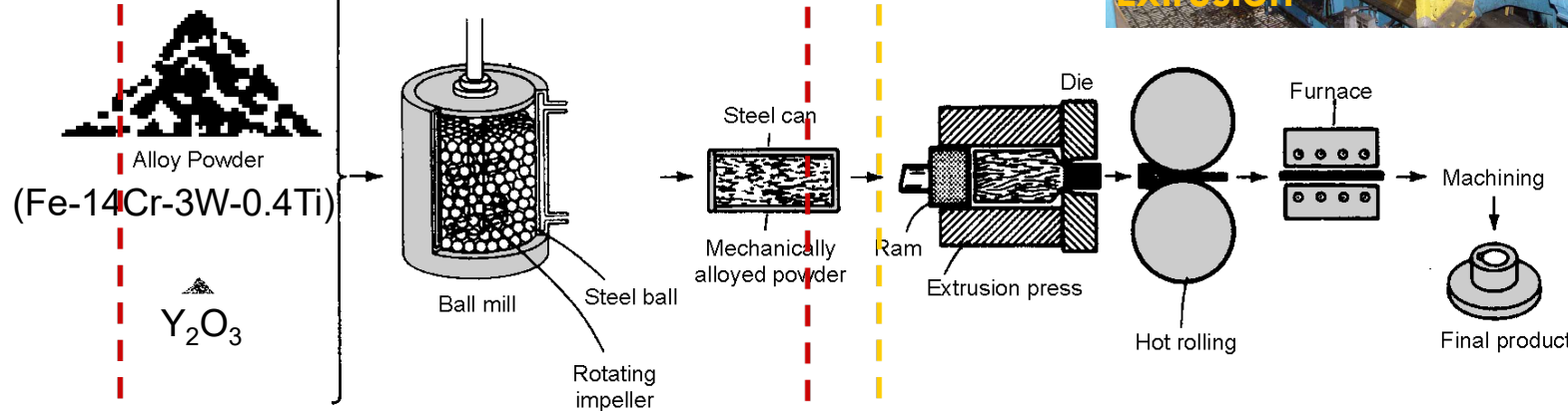
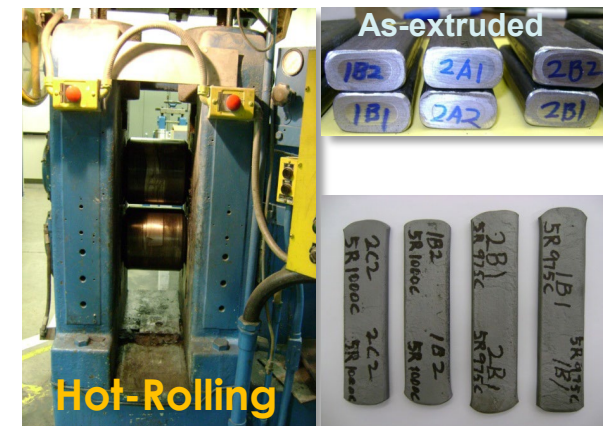
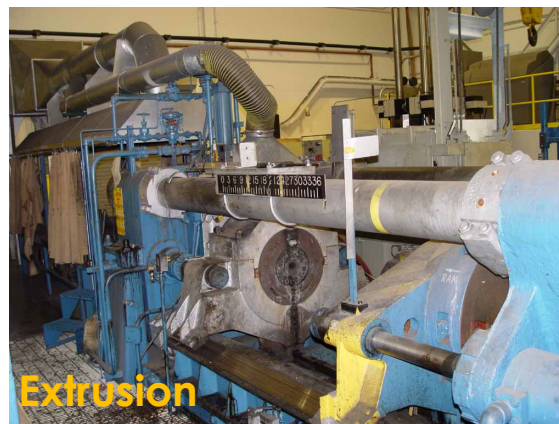
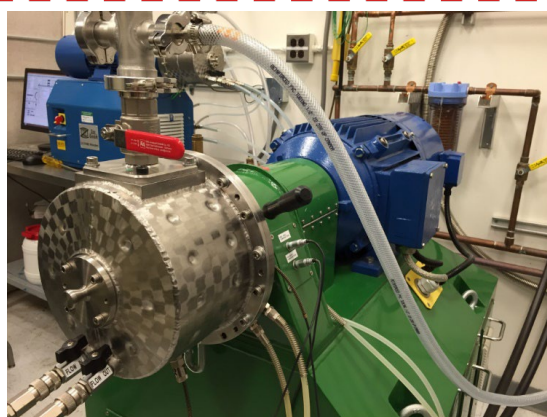
Tube Dimensions (mm)

OR8: OD=6.0; WT=0.5; L=2166
OR9: OD=6.0; WT=0.5; L=2384
OR11: OD=6.0; WT=0.5; L=2557
OR12: OD=6.0; WT=0.5; L=2380

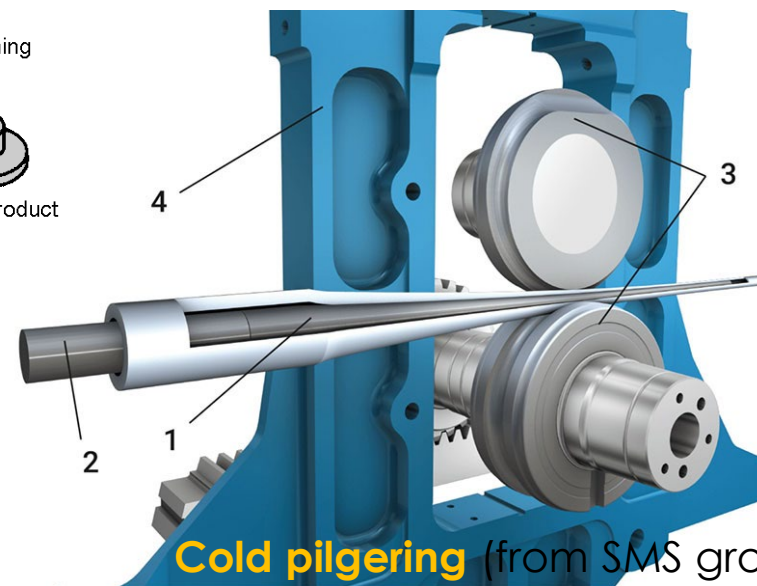


- *Despite all the advances established for 14YWT and other NFA variants, **the cost for any mass production of an NFA component through the current processing route, involving mechanical milling, may be prohibitively high, and the lengthy process route is unacceptable** for practical mass production applications.*
- *Without **a breakthrough innovation** to resolve these problems, the enormous merits of the distribution of highly stable nanoparticles in metallic materials will be missing in the future advance of fusion reactor technology*

ODS Alloy Production and Final Process



Powder Metallurgy and High-Power Mechanical Alloying (Ball Milling)



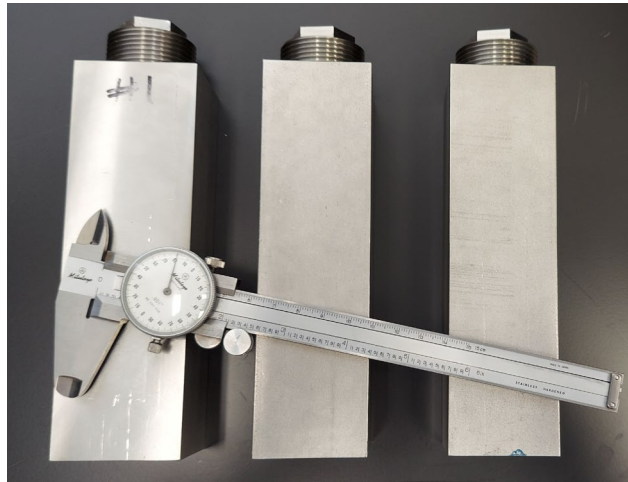
Cold pilgering (from SMS group)

: Cold pilgering is a rolling process for metal tubes that reduces diameters and wall thicknesses.

Proof of Principle Study on Melt-Based Processing

□ A research goal in FE Materials program:

- To avoid the high cost and scale-up limitation, we need to explore a manufacturing method not involving powder metallurgy.
- **This research is to design and demonstrate a new low-cost alloy processing route for nano-ODS alloys.**



- Powder is compacted into 304 SS cask
- Consolidation by multi-step hot rolling at 900°C (> 80% strain)



Nuggets from this portion were remelt in the Vacuum Ark Remelting (VAR) process



Ingots with 4.3 mm/9.5 mm diameters (left two – 14YWT alloy with high Y & O contents; right two – 14YWT plus Iron Oxide)

- Characterization underway.
- Further processing to be determined.

Research Focuses Needed for Advanced Reactor Application

❑ Processing route for cost-effective mass production

- A processing route without limitation in size and amount
- Cost effective and not time consuming
- Use or combine with traditional or existing processing routes including transformational manufacturing technologies
- Expansion to non-ferritic (austenitic stainless, hcp zirconium) alloys

❑ Proof of in-reactor performance (high dose, high temperature, neutron irradiation)

- Radiation effects on microstructures and properties under high temperature, high dose conditions
- Mechanical performance with void swelling and helium contents
- Both fundamental mechanisms and engineering properties after irradiation in application conditions

❑ Building property database for qualification

- Mechanical testing and analysis for nuclear structural alloy code case (tensile, fracture, fatigue, and creep)
- Physical and chemical (corrosion, stress corrosion) properties