U.S. DEPARTMENT OF ENERGY | Nuclear Energy

Advanced Reactor Core Materials Research

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Advanced Fuels Campaign

Contributors

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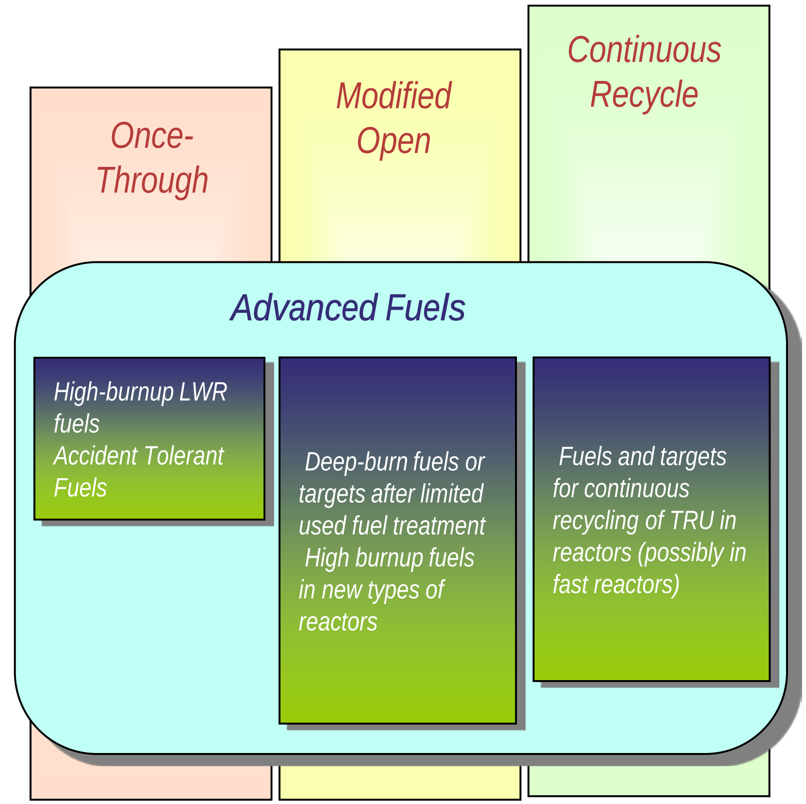
Fuel Cycle R&D's Advanced Fuels Campaign Mission & Objectives

- Mission

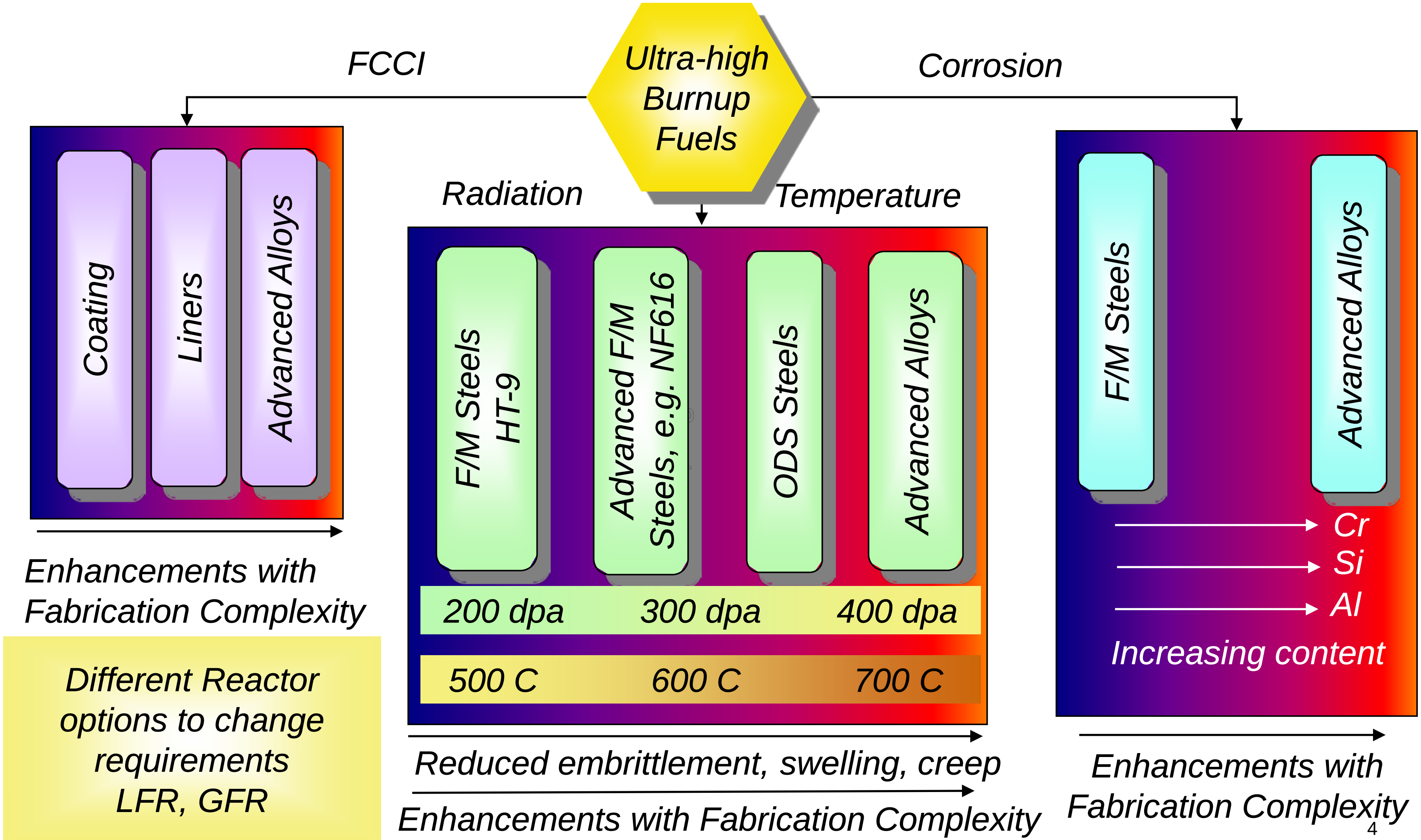
  Develop and demonstrate fabrication processes and in-pile performance of advanced fuels/targets (including the cladding) to support the different fuel cycle options defined in the NE roadmap.
- Objectives

  Development of the fuels/targets that
  - Increases the efficiency of nuclear energy production
  - Maximize the utilization of natural resources (Uranium)
  - Minimizes generation of high-level nuclear waste (spent fuel)
  - Minimize the risk of nuclear proliferation
- Grand Challenges for Closing the Fuel Cycle
  - Multi-fold increase in fuel burnup over the currently known technologies
  - Multi-fold decrease in fabrication losses with highly efficient predictable and repeatable processes



Approach to Enabling a Multi-fold Increase in Fuel Burnup over the Currently Known Technologies

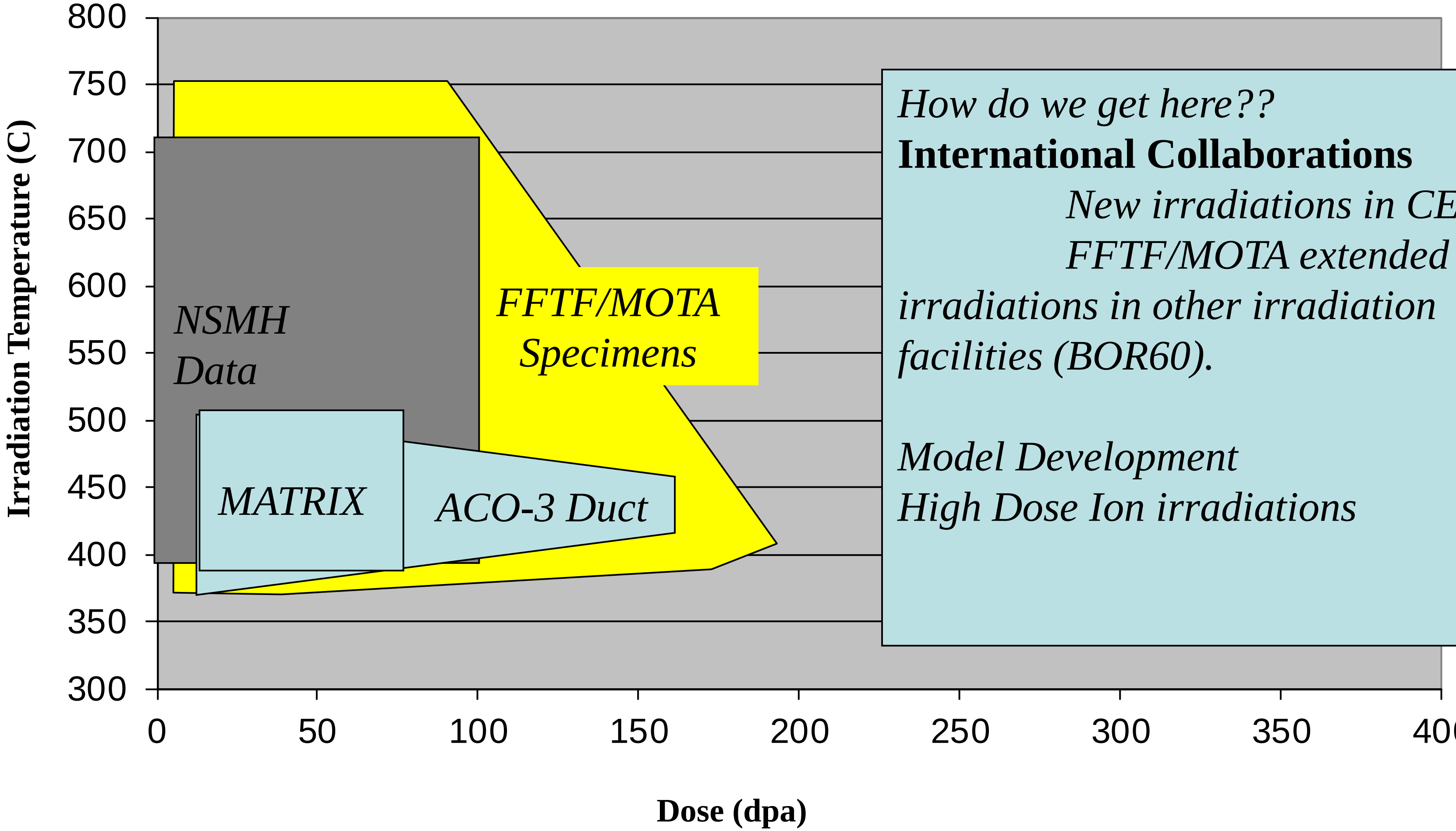
Ultimate goal: Develop advanced materials immune to fuel, neutrons and coolant interactions under specific reactor environments



Outline

- ***Qualify HT-9 to Radiation Doses >250 dpa***
  - *Obtaining high dose irradiation data*
  - *Development of new heat of HT-9*
  - *Interstitial Effects on ferritic steels*
- **Develop Advanced Radiation Tolerant Materials**
  - Production of rods, plates and tubes
  - ODS processing of new heat of 14yWT (FCRD-NFA1)- Tube Development
  - Testing of Advanced ODS alloys after low temperature irradiation
  - High Dose ion irradiation testing results

Significant data has been obtained on previously irradiated materials. How do we obtain data to dose levels out to 400 dpa?



How do we get here??

International Collaborations

New irradiations in CEFR

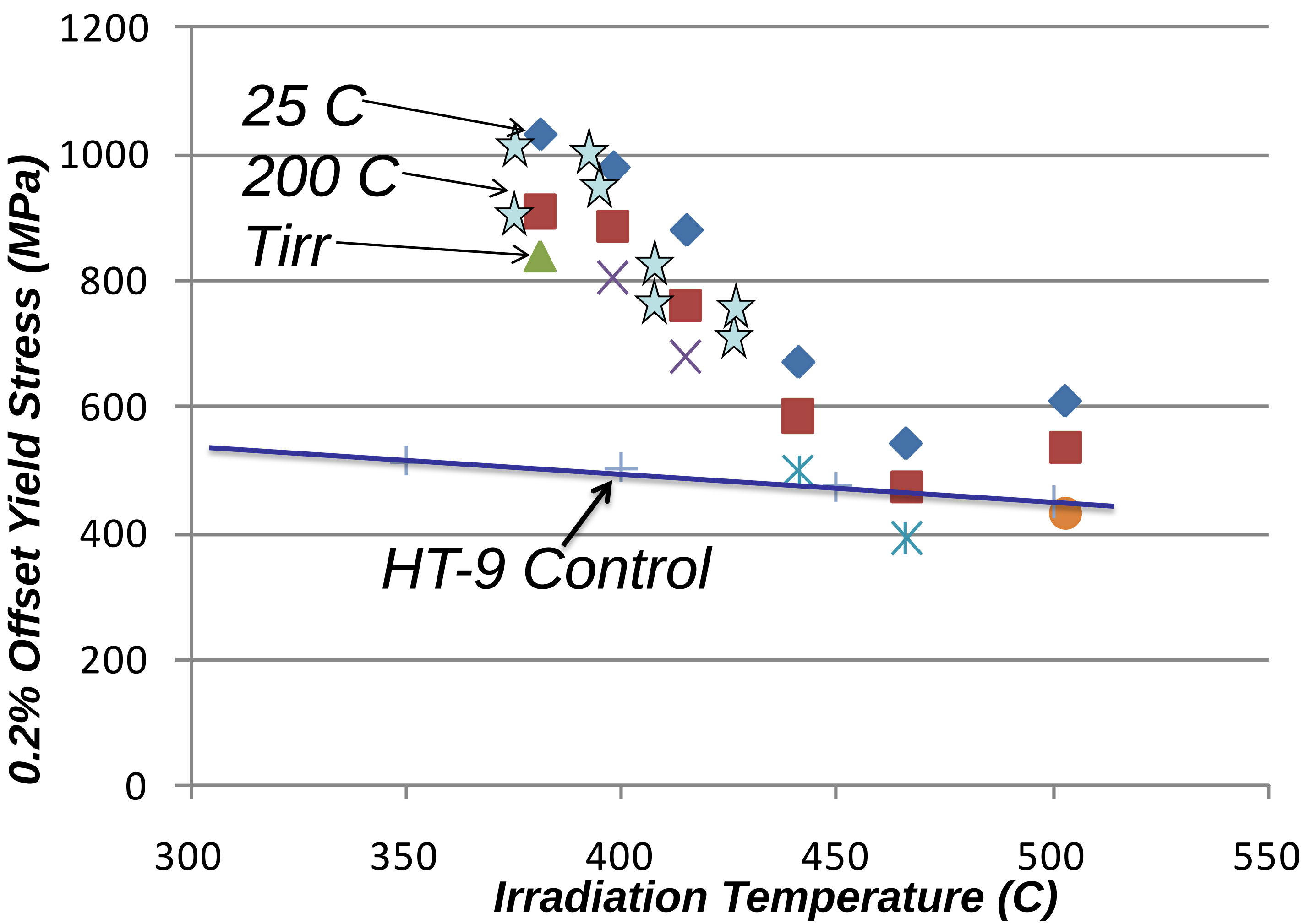
FFTF/MOTA extended irradiations in other irradiation facilities (BOR60).

Model Development

High Dose Ion irradiations

Low Temperature Hardening in Ferritic/Martensitic Steels

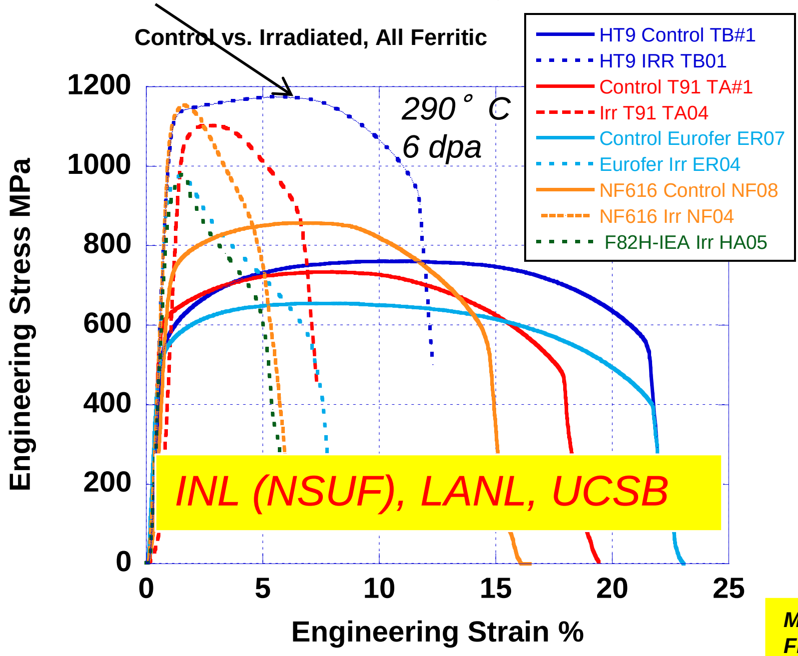
- **AC0-1 Duct and Cladding (HT-9) (total dose = ~ 88 dpa)**
  - Maximum dilatation (swelling + precipitation + creep) = 0.5 %
  - Yield stress increase ~300 MPa, Tirr =~360C, Ttest= 25C, dose = 36 dpa
- **Previous studies (stars in figure, tested at 25C) show similar dependence of yield stress on irradiation temperature**

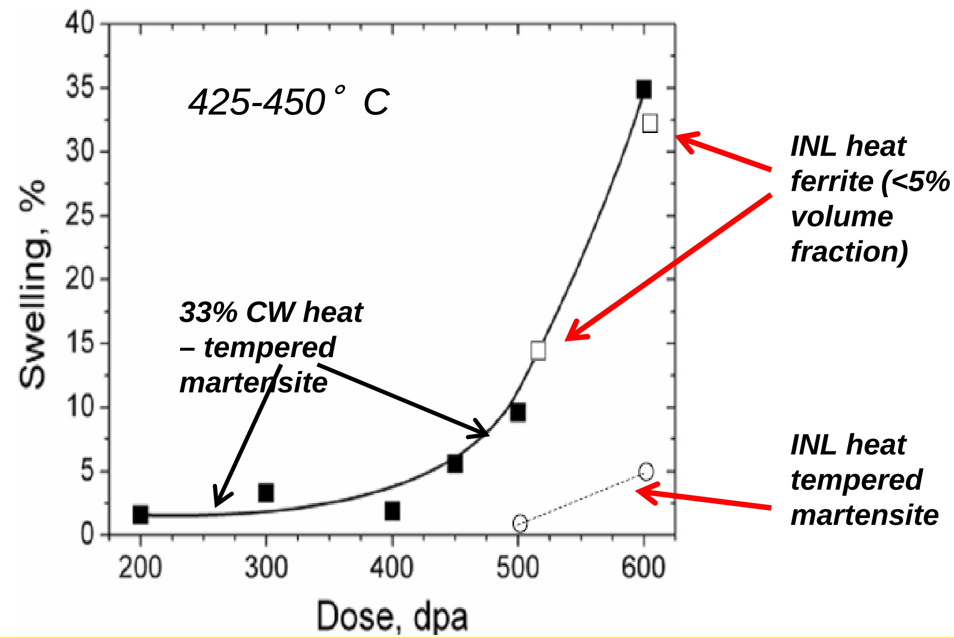


Improved Radiation Response of New NQA1 Heat of HT-9

- **300 lb heat of HT-9 produced by Metalwerks following NQA-1 quality control**
- **Tensile specimens irradiated in ATR to 6 dpa at 290° C**
  - Hardening observed but excellent ductility retained after low temperature irradiation
- **Ion irradiations performed to 600 dpa at 425° C**
  - Minimal swelling observed in tempered martensitic grains after ion irradiation to >500 dpa.
- **Four new heats of HT-9 were produced: Two by Metalwerks and two by Sophisticated Alloys with controlled interstitial content.**

INL-HT-9 Heat, best ductility





Maloy, S.A., Saleh, T.A., Anderoglu, O., Romero, T.J., Odette, G.R., Yamamoto, T., Li, S., Cole, J.I., Fielding, R., JNM, 468 (2016) 232-239.

Can annealing irradiated samples reduce nitrogen in solution?

- **Modeling studies show that nitrogen strongly couples with vacancy complexes –binding energy of 0.71-0.86 eV (**C. Domain, C.S. Becquart, J. Foct, Phys. Rev. B 69 (2004) 144112, T. Ohnuma, N. Soneda, M. Iwasawa, Acta Mater. 57 (2009) 5947–5955.)
- **Previous results on low temperature irradiated iron with nitrogen impurities show that annealing at >250C reduces the Snoek peak.**

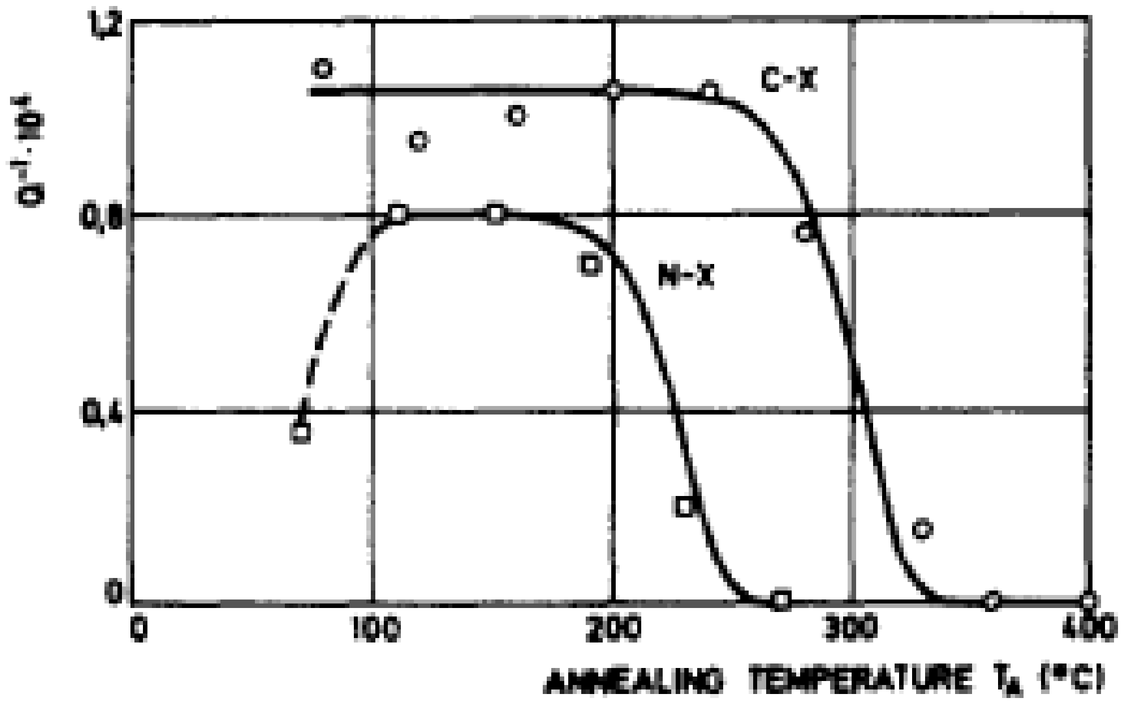
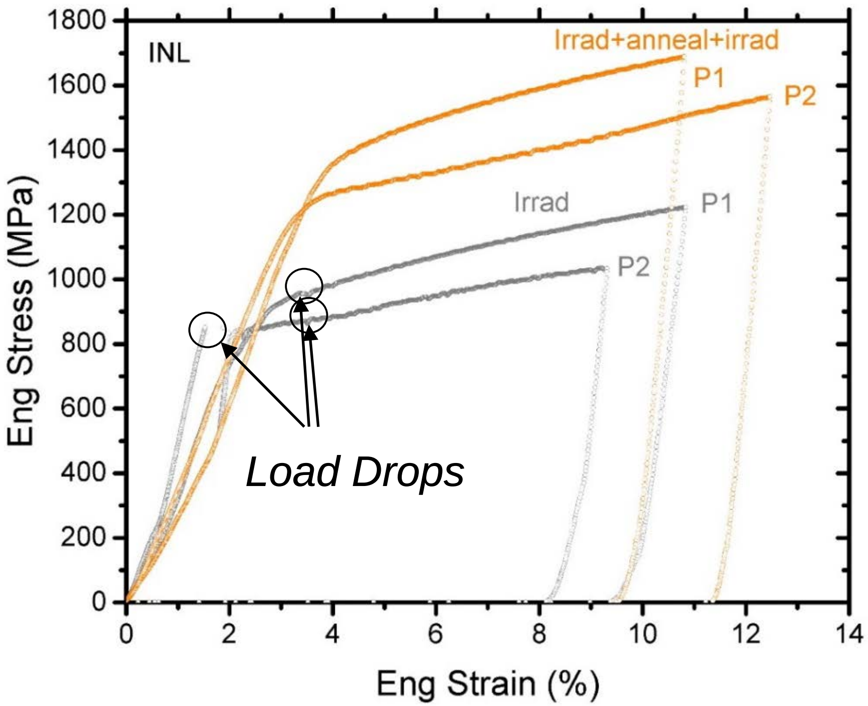


FIG. 4

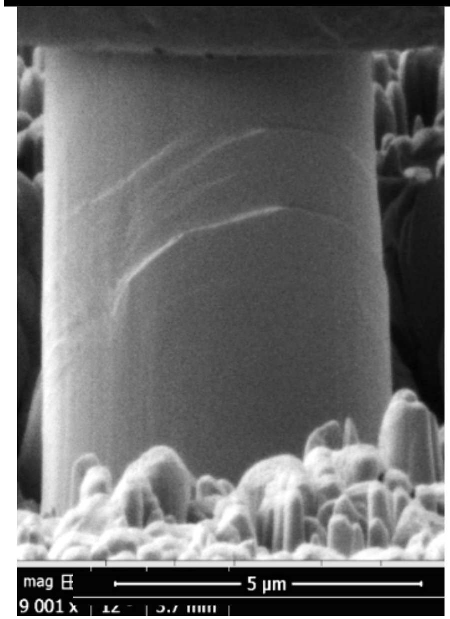
Isochronal annealing of the heights of peaks C-X and N-X.

Weller, M. and Diehl, J., "INTERNAL FRICTION STUDIES ON REACTIONS OF CARBON AND NITROGEN WITH LATTICE DEFECTS IN NEUTRON IRRADIATED IRON" Scripta Met, V. 10, pp. 101-105, 1976.

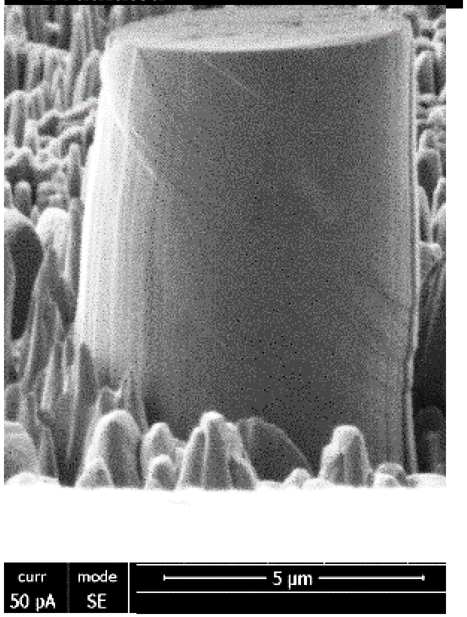
INL HT9-micropillar testing results



Irradiated ~%10 deformation



Irradiated +annealed + irradiated ~%10 deformation



- *Overall increase in σ_y after irradiation*
- *Less load drops observed in irradiated-annealed and irradiated samples*
- *More slip steps observed in irradiated-annealed and irradiated samples*
- *Re-irradiation on 550C annealed sample shows increased hardening.*

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Nanostructured Ferritic Alloys

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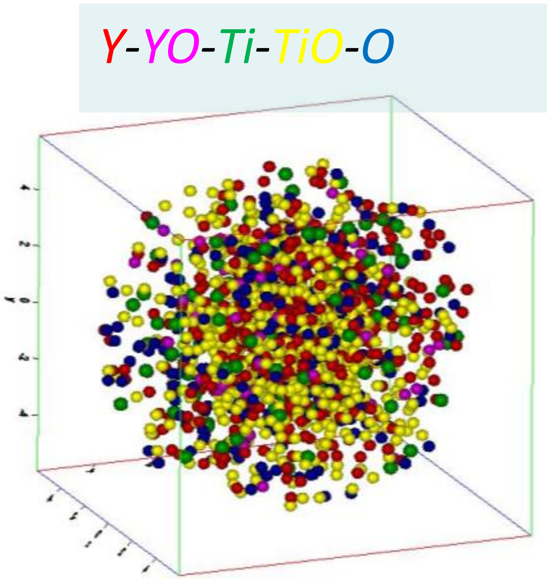
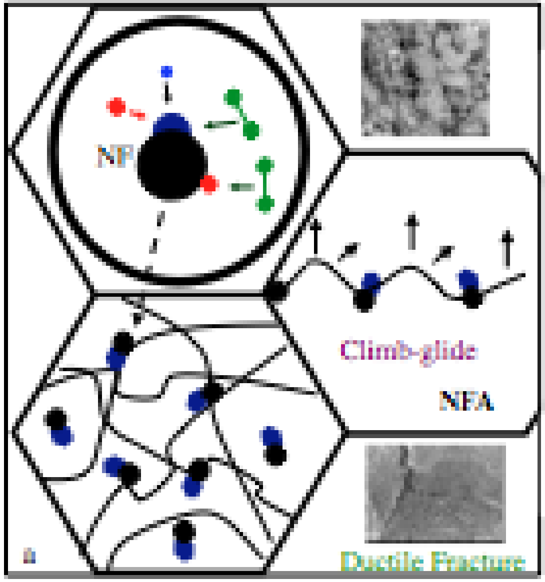
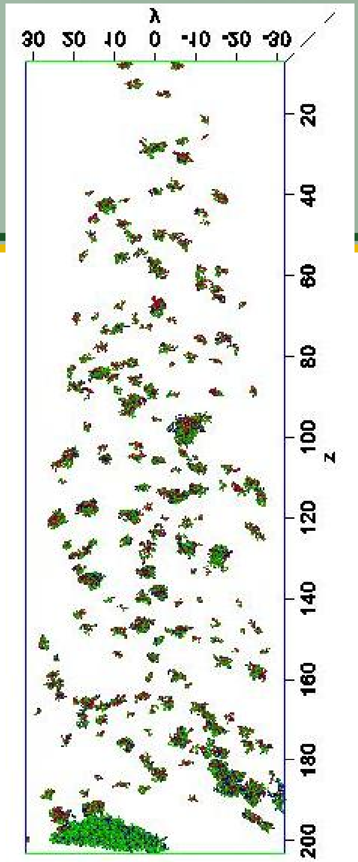
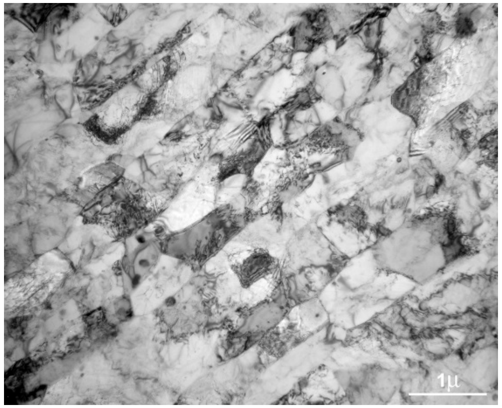
- *Strength & damage resistance derives from a high density Ti-Y-O nano-features (NFs)*
- *NFs complex oxides ($Ti_2Y_2O_7$, Y_2TiO_5) and/or their transition phase precursors with high M/O & Ti/Y ratios (APT)*
- *MA dissolves Y and O which then precipitate along with Ti during hot consolidation (HIP or extrusion)*
- *Nanostructured ferritic alloys also have fine grains and high dislocation densities*









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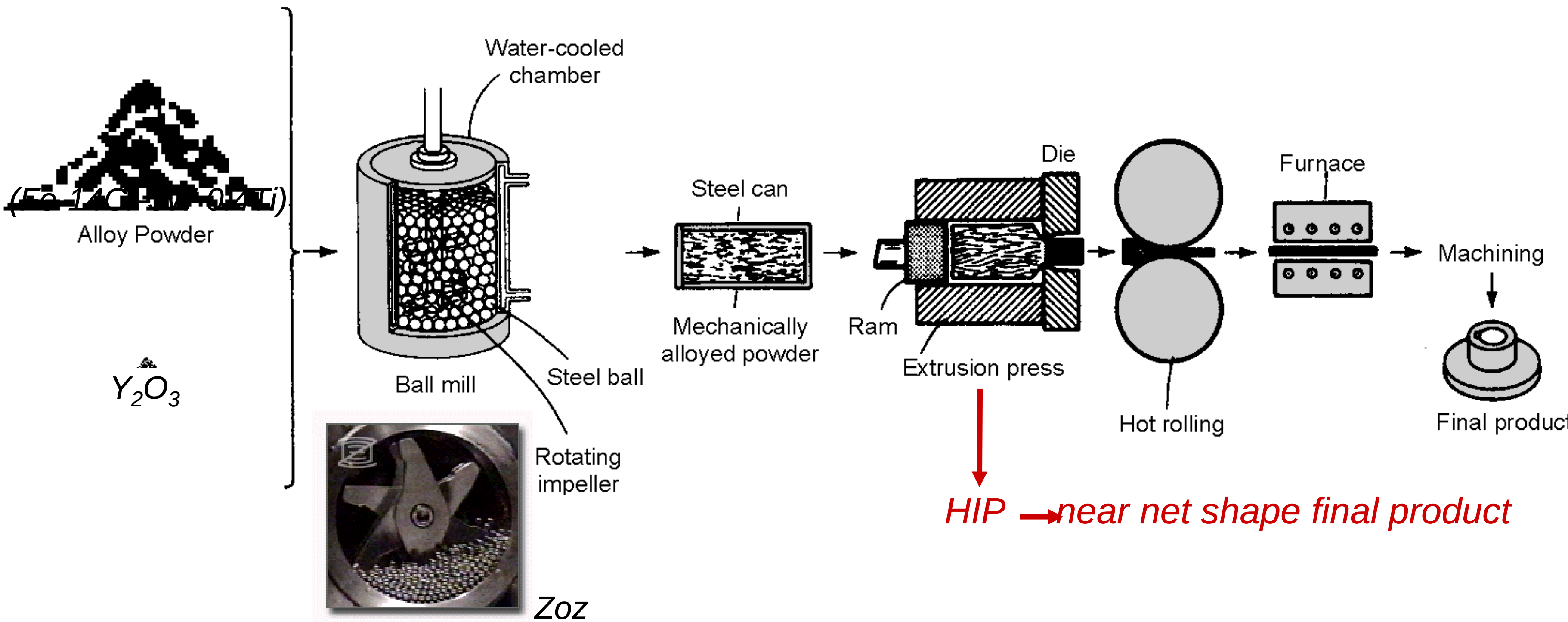
Typical Processing Route for NFA Alloys

- *Any desired combination of powders: metals, alloys, and dispersoid, such as oxides, carbides, borides, etc.*



The <u>conventional approach</u> is to ball mill alloy and Y_2O_3 powders together

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Production of rods for tube fabrication studies

- **Extruded rods of FCRD-NFA1 and 14YWT produced**
  - FCRD-NFA1: Fe-14Cr-3W-0.4Ti-0.2Y + 0.35% FeO
  - 14YWT: Fe-14Cr-3W-0.4Ti + 0.3% Y_2O_3

- *Powders were ball milled by Zoz*
- *4 inch dia. cans were extruded through 1.75 inch dia. die*

- *Extruded rods were cut to expose the ODS sections that were ~11 inches long*
- *Both rods were slightly bowed and needed to be straightened*
- *Two approaches attempted:*
  - *(1) direct decanning*
  - *(2) hot pressing prior to decanning*

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Production of plates for tube fabrication and joining studies

- *Procedures*
  - Material: FCRD-NFA1 and 14YWT (SM13) heat
  - Cans filled with ball milled powder and extruded through rectangular die
  - Bars cut and then annealed at 1000ºC in vacuum

Extruded Bars

- Bars cross-rolled at 1000ºC to 50% reduction in thickness

10 mm thick plate with can

Decanned plates

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Fabrication of thin plates of 14YWT for FSW joining studies

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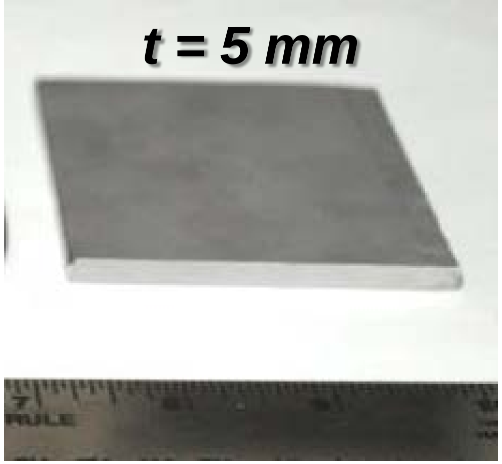
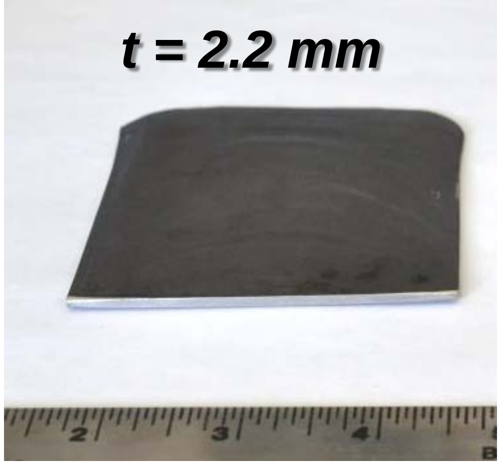
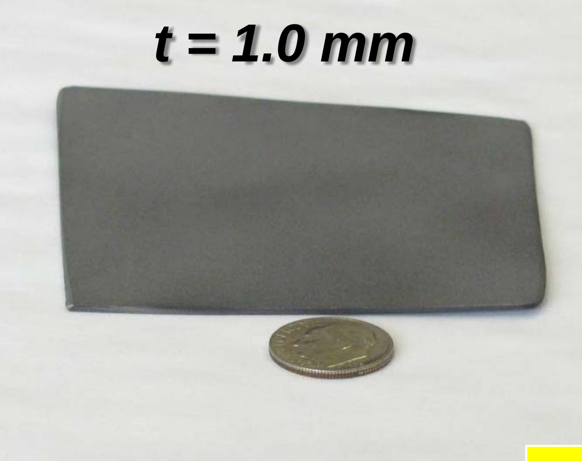
- *Starting plate thickness of 10 mm (~50% cross-rolled)*
  1) *Cross-rolled at 1000ºC to 5 mm thickness (50 % RIT) and cut in half*
  2) *One half was parallel-rolled at 1000ºC to t = 2.2 mm thickness (56% RIT) and cut in half*
  3) *One half was cross-rolled at 1000ºC to ~1 mm thickness (54.5% RIT) and cut in half*

  ➢ ***~95% RIT total with no edge cracking***







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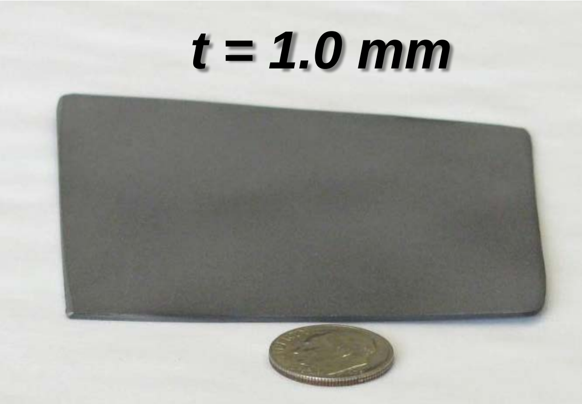
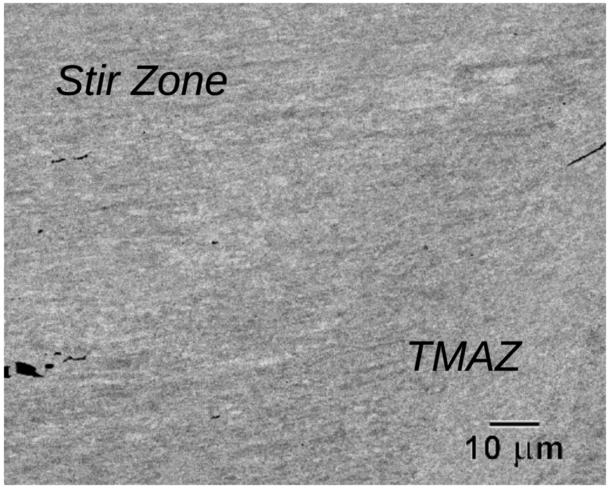
1st FSW Attempt: Bead-on-plate on 1 mm thick plate of 14YWT

- 1 mm thick plate of 14YWT (SM13) fabricated in 4 rolling sequences at 1000ºC
  - 95% total reduction in thickness
  - No edge cracking



- Bead-on-plate weld zone using a modified pin tool design

BSE Image of cross-section



ORNL

- Next: TEM, APT

Hot Extrusion at CWRU

Hot Extrusion Parameters

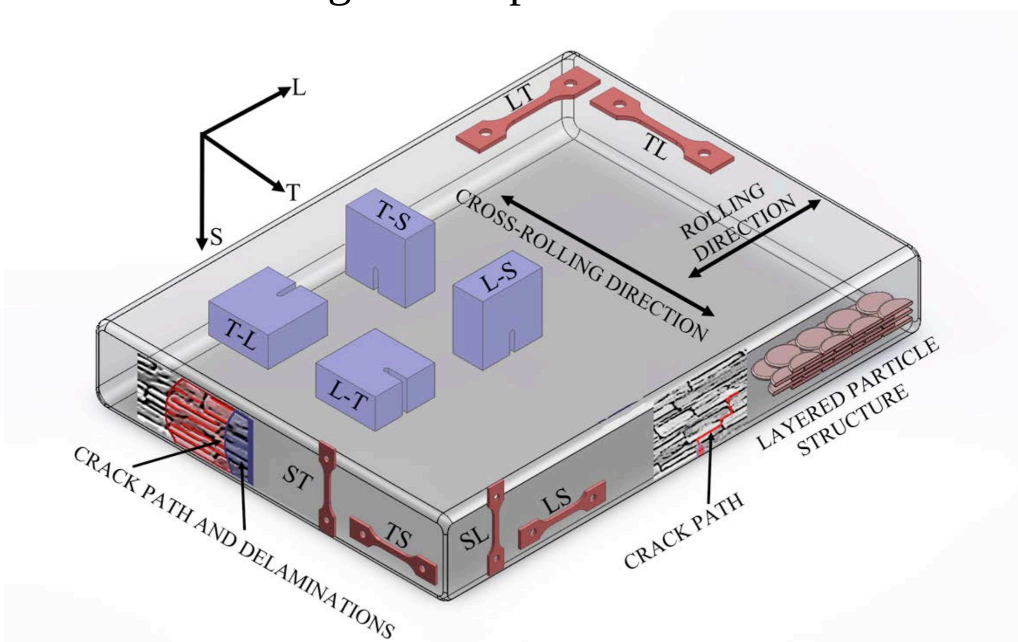
1. *Hydrostatic EXTRUSION TEMP: 1500F (815C)*
2. *RAM SPEED: 0.5 in/min, however 1st 0.5” of extrusion, speed was 0.7 in/min*
3. *SOAK TIME: 10 min*
4. *OVERALL EXTRUSION: 25 min*
5. *ER: 4:1, 45 DEG TAPER DIE (actual 0.495 diam)*

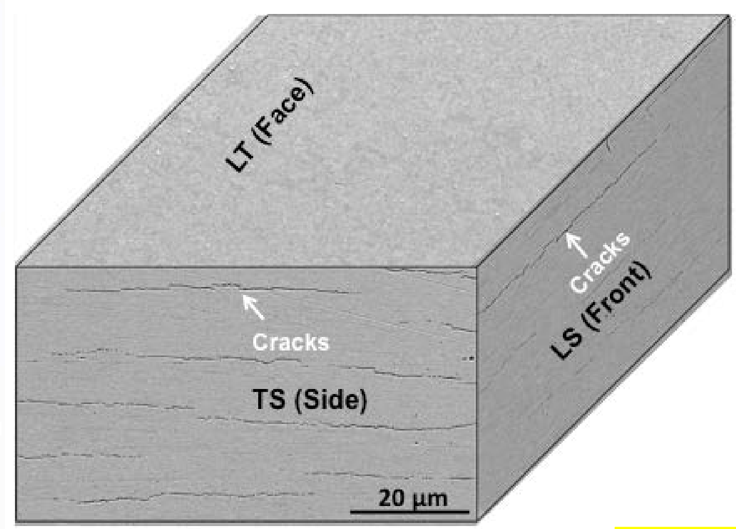
Final NFA Extruded tubes after Etching to remove liner and mandrel

CWRU

NFA-1 Deformation & Fracture Properties/Mechanisms

- The property relevant microstructures of the hot extruded and cross rolled NFA-1 plate include: a) a high density of nano oxides and dislocations; b) a bimodal distribution of highly α–<110> fiber textured, predominantly fine (< 500) nm pancake shaped grains; and, c) a very large population of pre-existing ≈ 5-30 μm microcracks laying in planes perpendicular to the broad plate faces.
- Hence, mechanical properties and the associated deformation and fracture mechanisms and processes may be highly anisotropic thus were characterized in various orientations over a wide range of temperatures.





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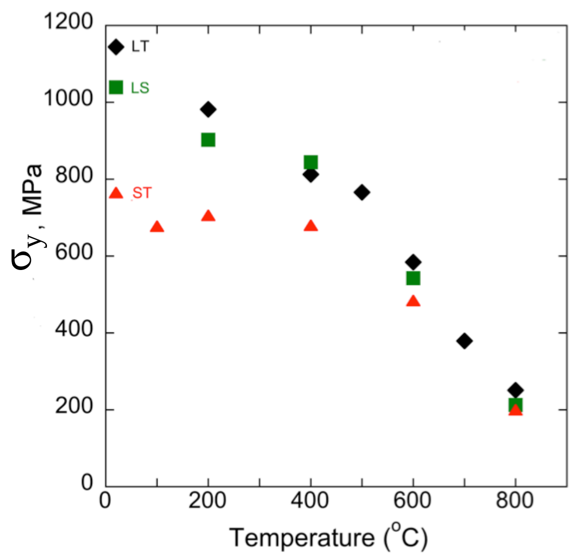
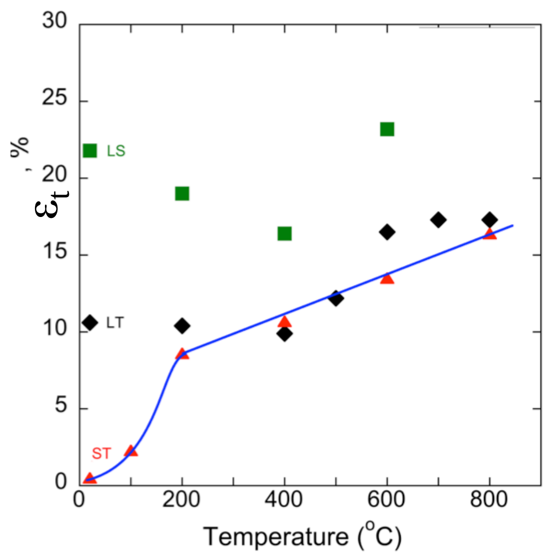
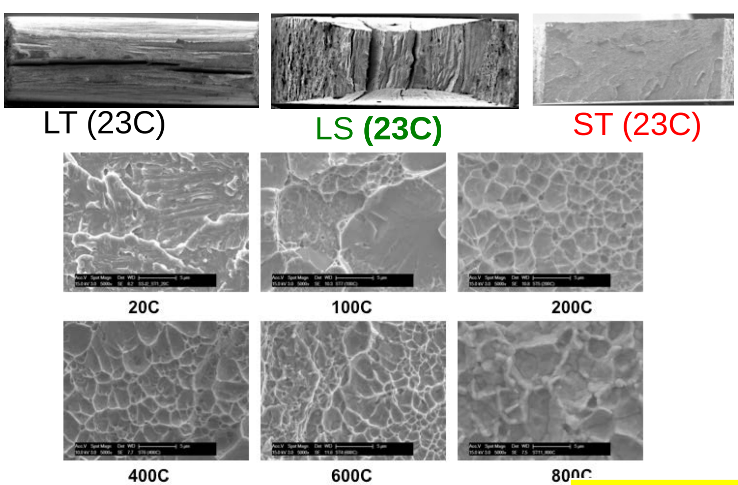
NFA-1 Tensile Deformation and Properties

- LT (from plate face) orientation 23ºC tensile results show high strength (σ_y > 1100 MPa) and with moderate ductility (ε_t: 8-11%), while LS (from plate side) shows slightly lower strength (σ_y~ 1000 MPa) with much higher ductility (ε_t: 8-22%).
- Strength decreases more rapidly at > 400° C as deformation transitions to viscoplastic creep; ductility varies, but remains significant.
- LT or LS delamination at ≤ 200ºC by propagation of preexisting microcracks.
- However, ≤ 23° C, ε_t = 0 for loading in the short (ST) plate thickness direction due to the unstable elastic propagation of brittle cleavage microcracks.
- A ST-orientation brittle-to-ductile cleavage transition occurs up to 100° C, but the effects of the pre-existing microcracks are felt up to 200° C.





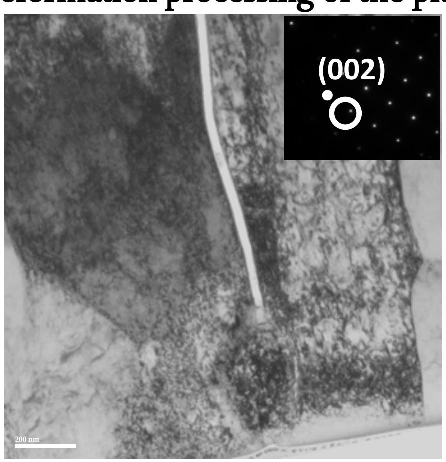


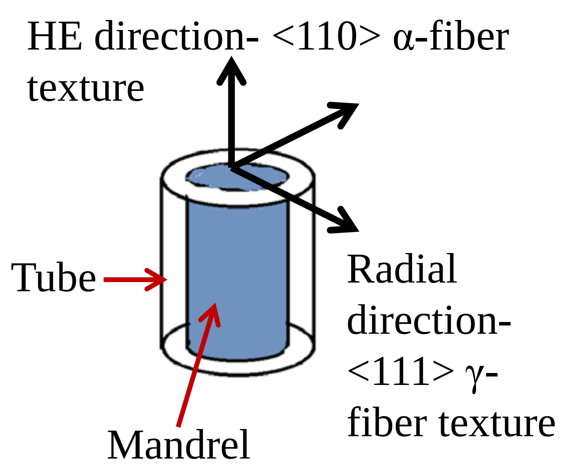
ST at different temperatures

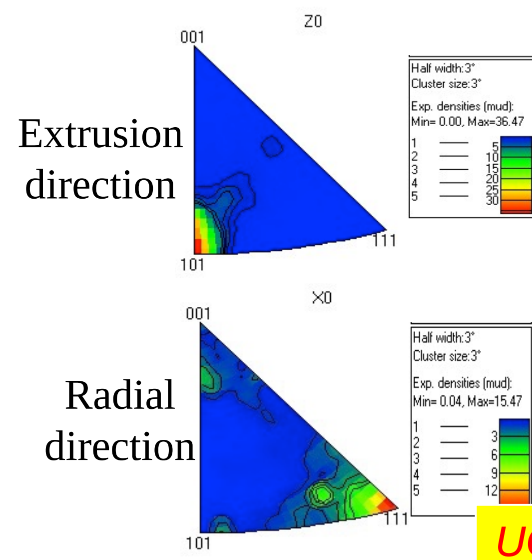
NFA-1 Tube Processing Plate and Tubing

- Extensive FIB-TEM studies show that microcrack formation during plate processing is due to formation of (002)<110> cleavage systems by sessile dislocation reactions at pile-up ends forming low angle boundaries and that microcracking is activated by local and through-thickness residual stresses.
- Formation of microcracks and texturing depends on the deformation path and elevated temperature hydrostatic extrusions of mandrel mounted mother tubes taken from the microcracked plate are crack free and have much less brittle easy slip <111> γ- fiber radial textures.
- Heat treatment paths also found to mitigate microcracking, brittle textures and anisotropic grains at the cost of modest strength reductions (not shown).

A cracked low angle tilt boundary stack up of sessile dislocations formed by glissile pile-ups during deformation processing of the plate







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Excellent Retention of Ductility observed for ODS Steels after irradiation to 6 dpa at 293C

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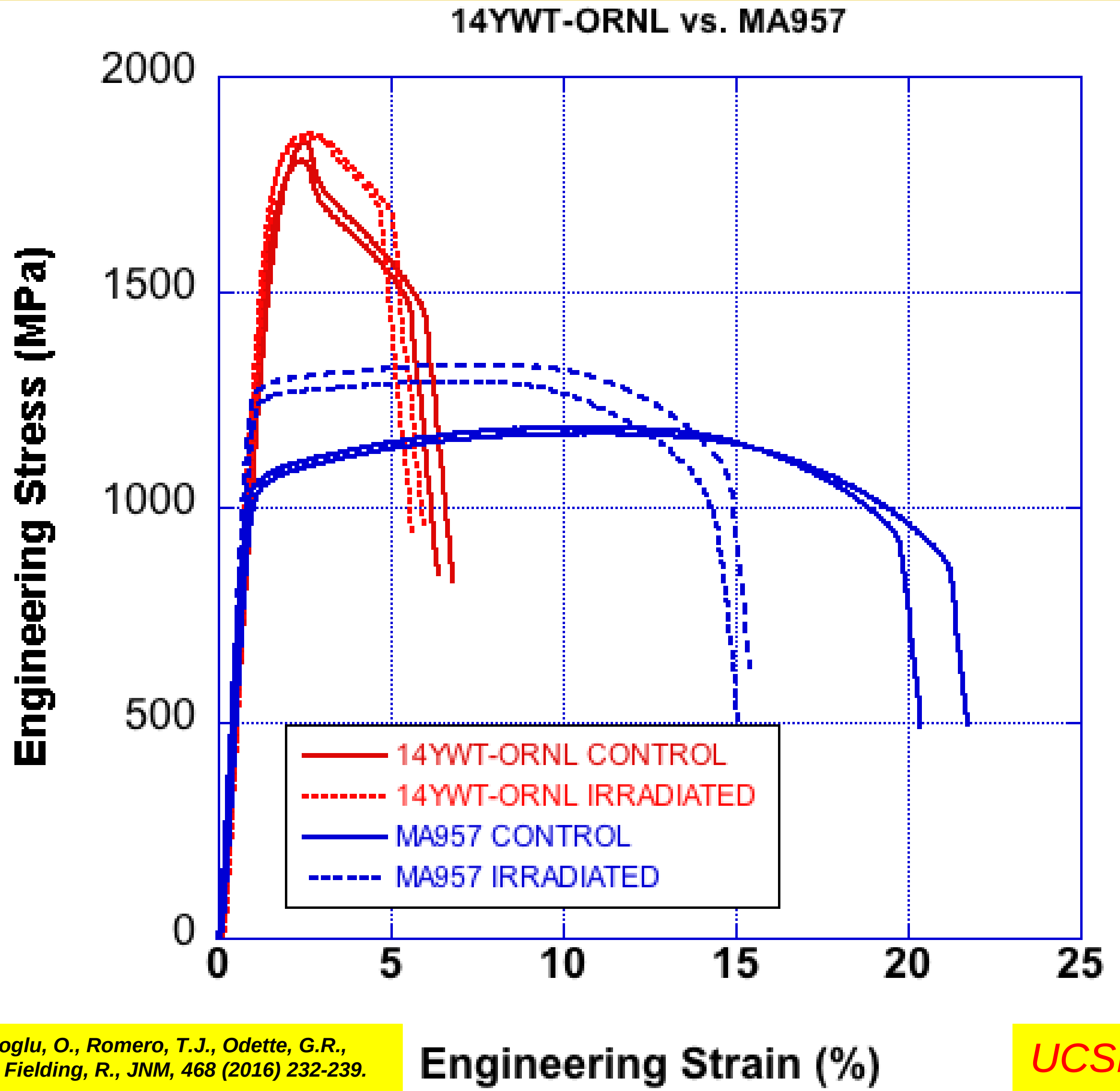

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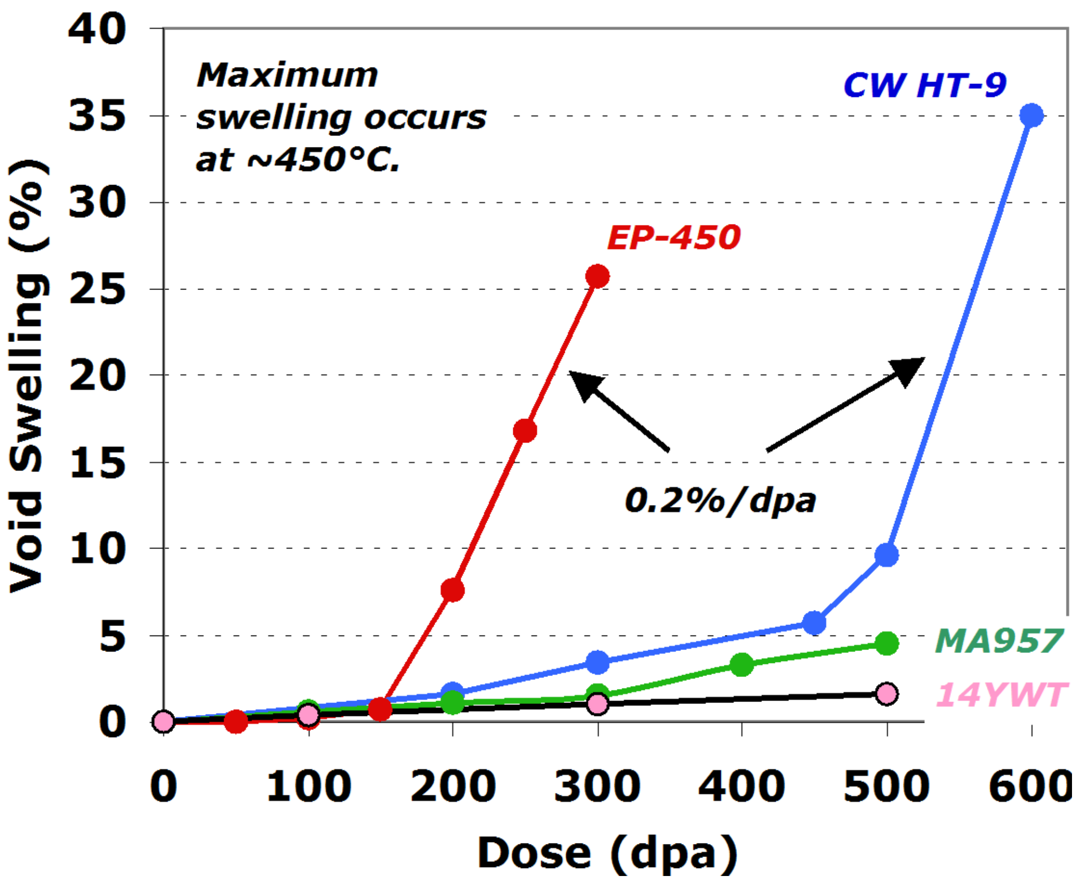
Ion Irradiation Induced Swelling Comparisons

- All materials irradiated in the same facility.
- Tempered F-M steels reached a terminal 0.2%/dpa swelling rate that has been observed ferritic alloys during neutron irradiations.



- MA957 and 14YWT are exhibiting an extended nascent low swelling period.
- Data suggests that 14YWT is exhibiting better resistance than MA957, but results are very similar.
- Will 14YWT and MA957 abruptly transition to high swelling rate?

PNNL, KIPT, LANL

Materials Integration and University and International Collaborations

- **Integrate FCRD Core Materials Activities**
  - Fuels Core Materials Work- (PNNL, LANL, ORNL)
    - *Materials teleconferences monthly*
  - University Materials Research (review quarterly progress reports)
    - *UCSB- Optimized Compositional Design and Processing-Fabrication Paths for Larger Heats of Nanostructured Ferritic Alloys (Odette)*
    - *U-Mich-High Fidelity Ion Beam Simulation of High Dose Neutron Irradiation (G. Was)*
    - *UCB- Developing ultra-small scale mechanical testing methods and microstructural investigation procedures (Hosemann)*
    - *TAMU-Development of high performance ODS alloys (L. Shao)*
    - *Northwestern U.- Electrically-Assisted Tubing Processes for Enhancing Manufacturability of Oxide Dispersion Strengthened Structural Materials for Nuclear Reactor Applications (J. Cao)*
  - ATR Reactor Irradiations (provide materials and preparing to collaborate in testing)
- **Working group meetings and Workshops**
  - NE Materials Cross-cut Meeting held through a webinar (NEET Reactor Materials Technical Lead)
- **International Collaborations**
  - DOE-CIAE Collaboration (China) – Developing CRADA for materials irradiation in CEFR
  - LANL-Terrapower CRADA – Received first specimens back from BOR-60
  - DOE-CEA collaboration (France) – receive MATRIX specimens from Phenix reactor; ODS tube development
  - CNWG (Japan) – ODS material development and high dose material data
- **New CINR FY17 Scope areas:**
  - NEET-NSUF-1.3 – ODS Steel Joining Technologies
  - NEUP-FC-2.2 – Extreme Performance Metal Alloy Cladding for Fast Reactors

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Conclusions and Future Work

- **Development of Radiation Tolerant Tempered Martensitic Steels**
  - A new heat of HT-9 shows improved retention of uniform elongation after neutron irradiation to 6 dpa at 290C.
  - Four new heats of HT-9 with controlled interstitial impurity content have been produced and processing and testing is underway.
- **Development of Radiation Tolerant Nanostructured Ferritic Alloys (NFA)**
  - A large heat (50 kg) of Nanostructured Ferritic Alloy (NFA), 14YWT was produced through a strong LANL-ORNL-UCSB collaborative effort.
  - Extrusions of rods, thick walled tubes and plates with thickness down to 1 mm were produced at ORNL.
  - Successful hydrostatic extrusions of thin walled tubes were performed at CWRU.
  - Mechanical testing at UCSB and ORNL revealed excellent tensile properties up to 800C and ductile tearing toughness down to -175C.
  - Ion irradiation testing revealed excellent void swelling resistance to doses >500 dpa
- **Future work**
  - Thin-walled tube processing
    - *Pilger processing at PNNL/Sandvik and in collaboration with CEA*
    - *Elevated temperature plug drawing at Rhenium Co.*
  - Future high dose irradiations in BOR-60 (Russia), JOYO (Japan), CEFR (China) and HFIR (ORNL)

UCSB, PNNL, LANL, ORNL, CWRU