FCRD ODS Materials Development - FCRD-NFA1

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DOE-NE Materials Crosscut Coordination Meeting - 2013

Webinar

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Collaborations

Primary Collaborators:

LANL:	Stuart Maloy, P.I.	FCRD Project: Improved processing for scale-up of 14YWT
ORNL:	Thak Sang Byun	Fracture toughness: High Temperatures INERI (KAERI): Development of ODS 9Cr
	Mikhail Sokolov	Fracture toughness: Transition Temperatures
	Kinga Unocic	Microscopy
UCSB:	Bob Odette	Long term collaboration on NFA development

Other Collaborators:

KIMS:	Jeoung Han Kim	Strengthening mechanisms
ANL:	Meimei Li	Deformation mechanism maps
M&MS:	Jim Bentley	Early collaboration on development of 14YWT Pioneering EFTEM analysis of NFA

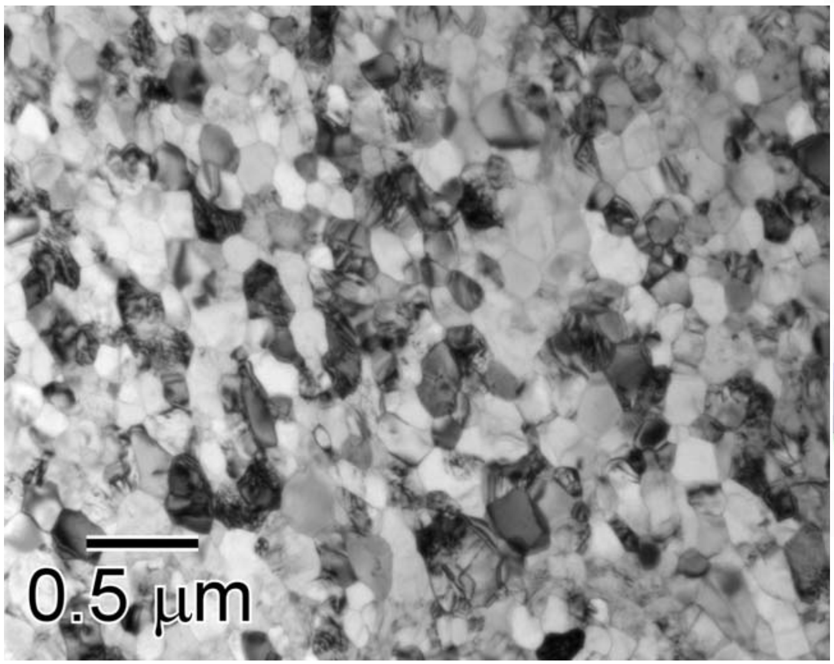
Presentation outline

- Overview of the processing, microstructure and mechanical properties of 14YWT, including concerns
- FCRD collaborative project to develop larger "best practice" heats and forms of the advanced ODS 14YWT ferritic alloy
  - Scale-up project
  - Insights obtained from studies on processing and fabrication leading up to scale-up heat
  - Processing and fabrication of the initial FCRD-NFA1 heat
- Summaries

Design strategy for developing the radiation tolerant advanced ODS 14YWT ferritic alloy

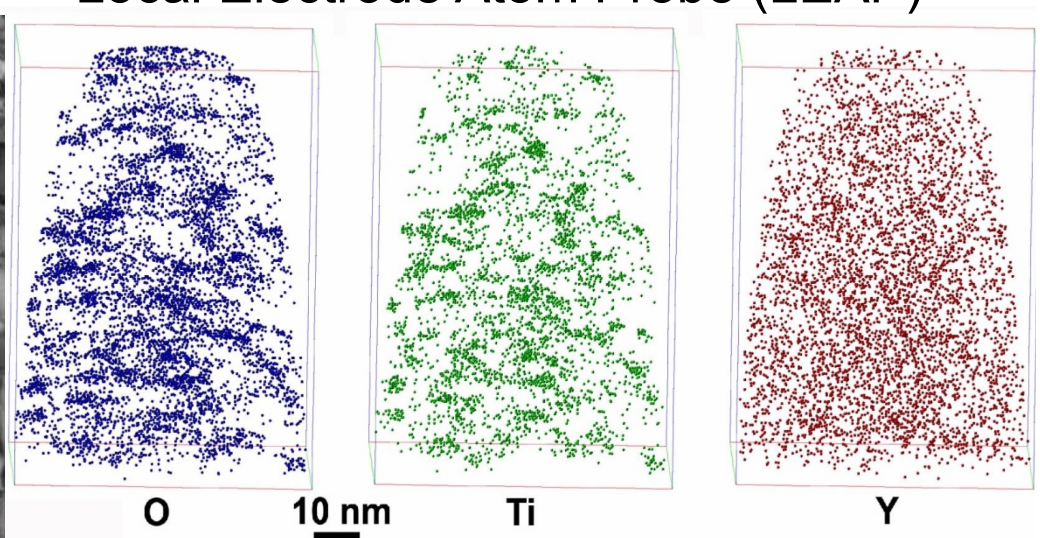
- *Create a high interfacial area (sink) to enhance trapping and recombination of point defects*
- *Combine nano-size grains with a high density of nanoclusters*

BF TEM of SM10 Heat

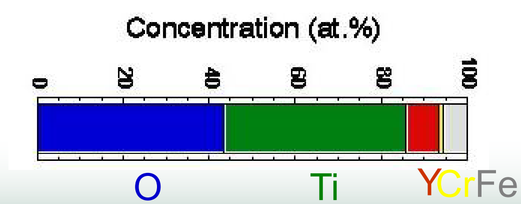


Grain size = 136 (+/- 14) nm
GAR = ~ 1.2

Local Electrode Atom Probe (LEAP)



N_v = 1-7 x 10^{23} m^{-3} <r> = 1.8 $^+$/- 0.4 nm



APT: M.K. Miller, K.F. Russell and D.T. Hoelzer, JNM, 2006

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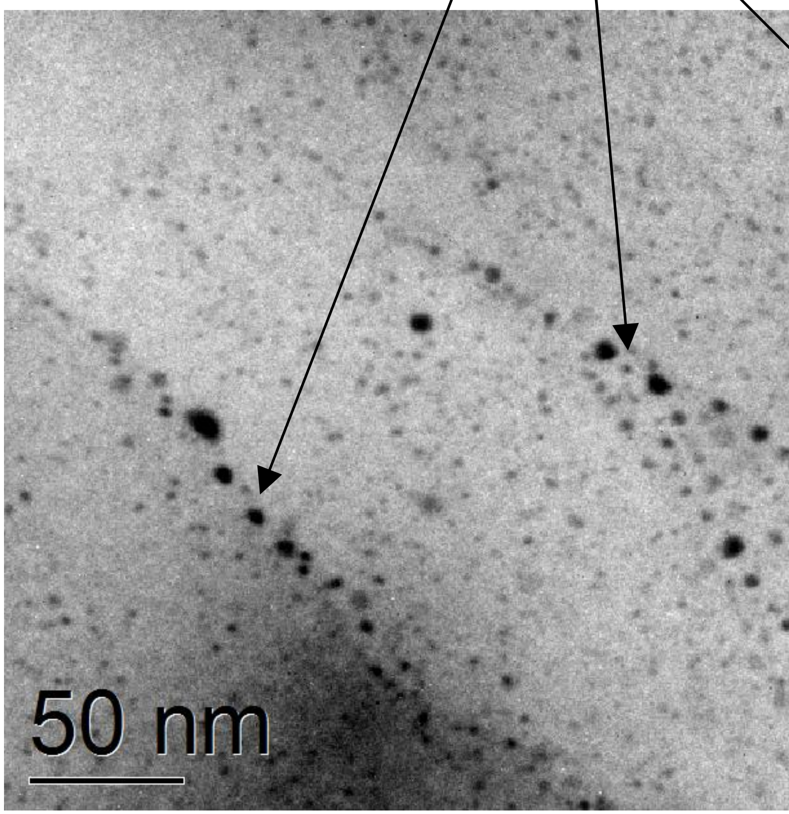
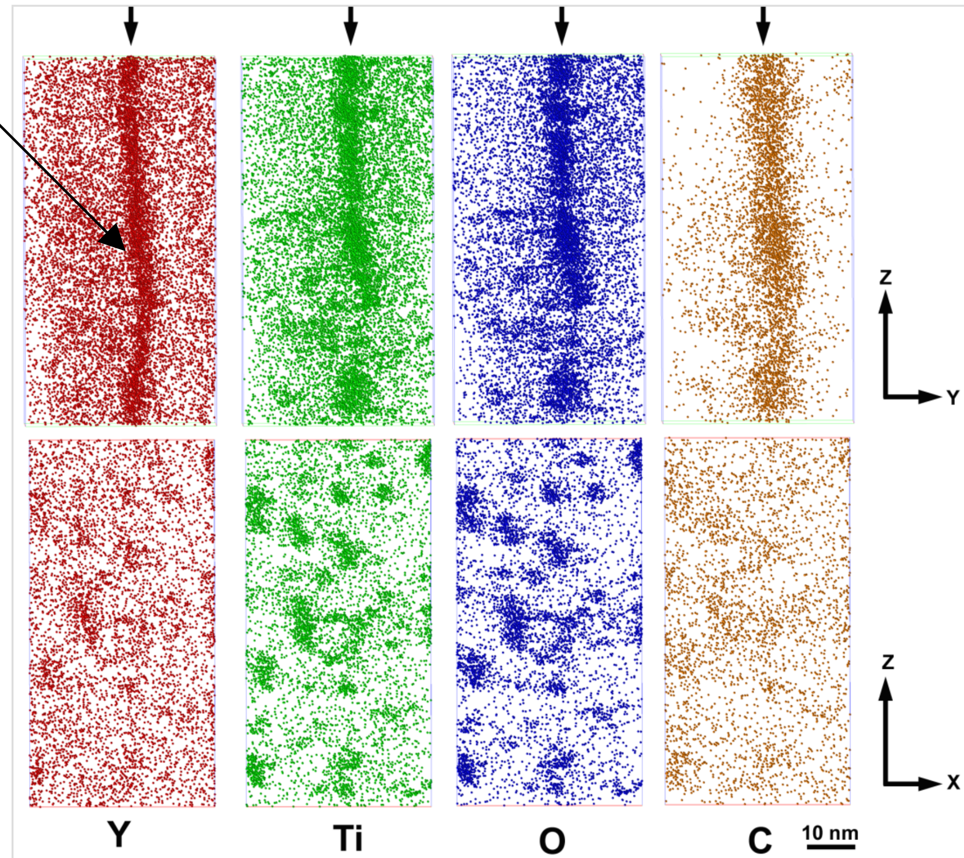
Nano-size grains achieved by nucleating NC (and larger oxide particles) on the grain boundaries

EFTEM Fe M Jump Ratio Image

Local Electrode Atom Probe (LEAP)

Grain boundaries





LEAP: M.K. Miller, D.T. Hoelzer and K.F. Russell, Materials Science Forum, V654-656, 2010

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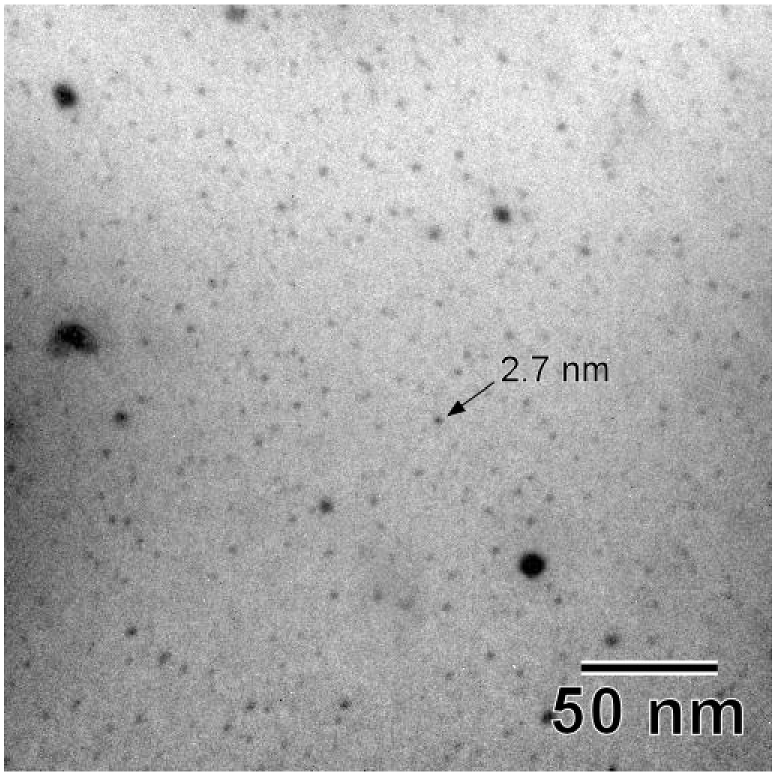
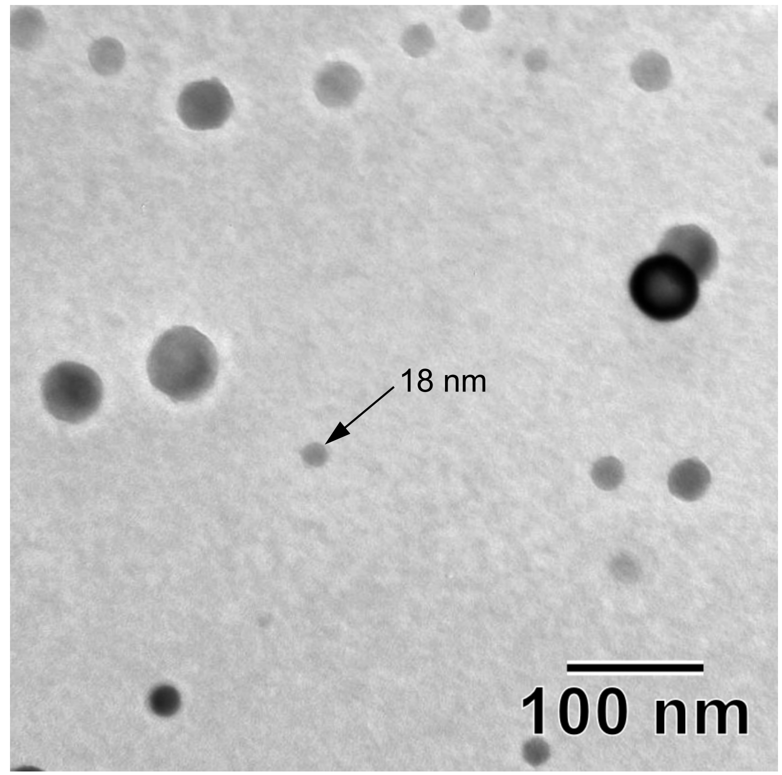
Enhanced sink strength: *High density of nanoclusters compared to typical ODS alloys*

ORNL 14YWT
EFTEM Fe M Jump Ratio Map



Plansee PM2000
TEM Bright Field Image



- 14YWT contains a significantly higher <u>number density</u> and <u>smaller size</u> of Ti-, Y-, and O-rich nanoclusters compared to the YAG oxide particles in PM2000 (and other commercial ODS alloys)

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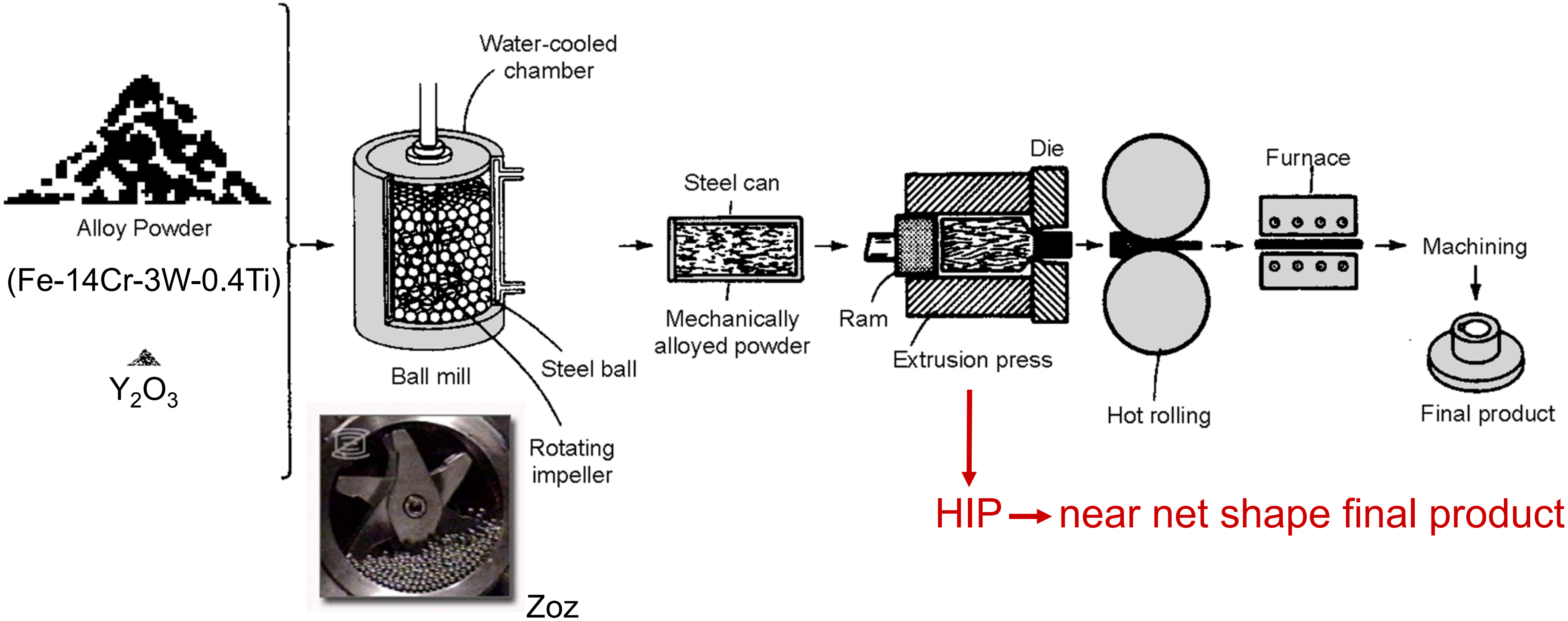
14YWT is produced by Mechanical Alloying

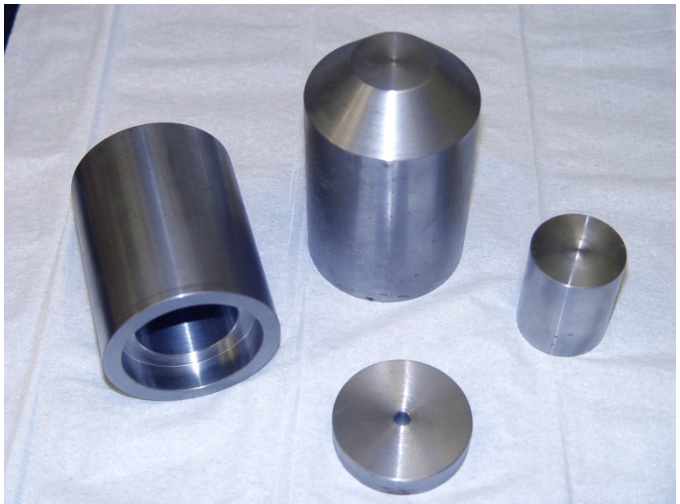
- Any desired combination of powders: metals, alloys, and dispersoid, such as oxides, carbides, borides, etc.



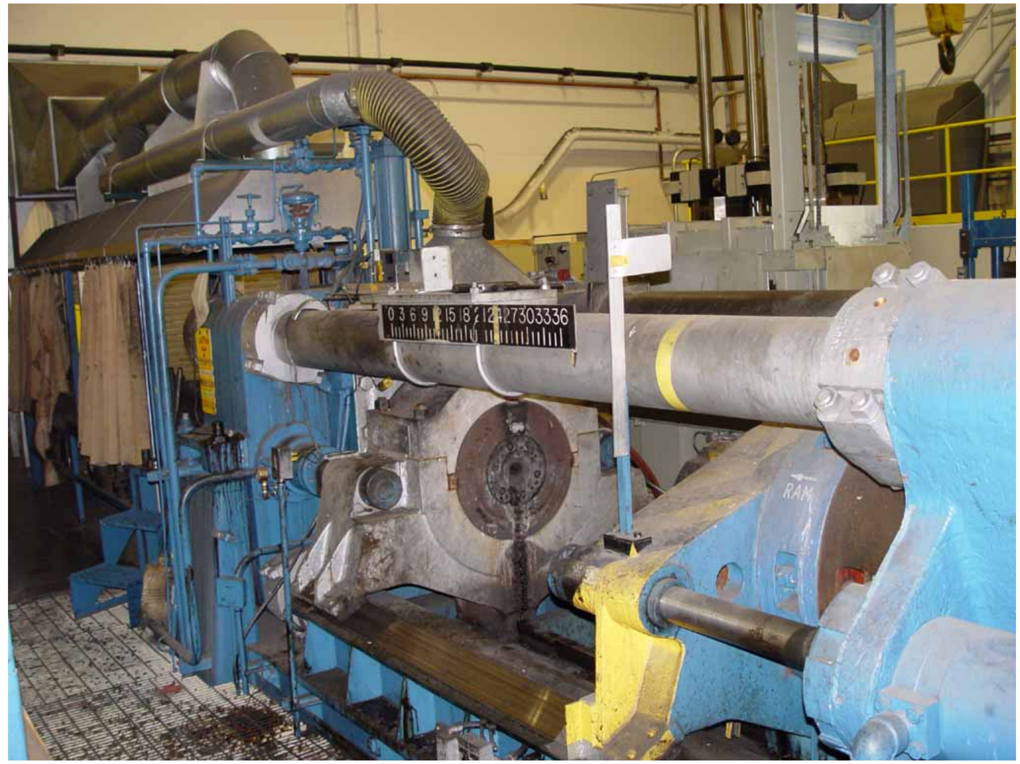
The ***conventional approach*** *is to ball mill Fe-alloy and Y_2O_3 powders together*

The ball milled powders are consolidated by extrusion

Watson-Stillman Co. (Roselle, NJ) unit with total capacity of 1,250 tons (2.5 x 10^6 lbf)



- Can made with mild steel
- Can filled with powder
- End cap welded to can
- Vacuum degassed at 400ºC
- Can hermetically sealed



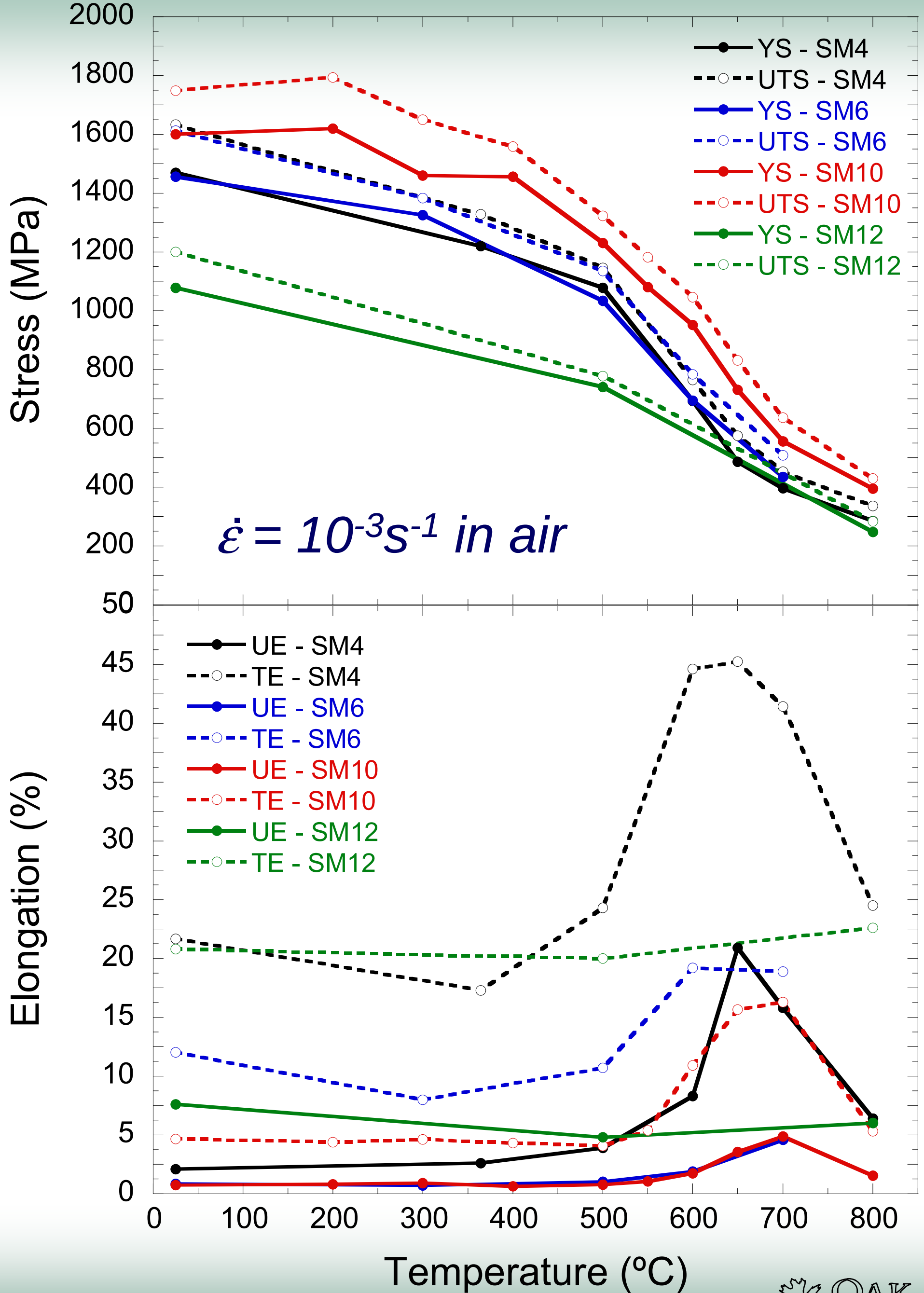
- Can heated to 850ºC for ~2 h
- Can extruded through die at ~850ºC

- **Extruded bar of 14YWT**

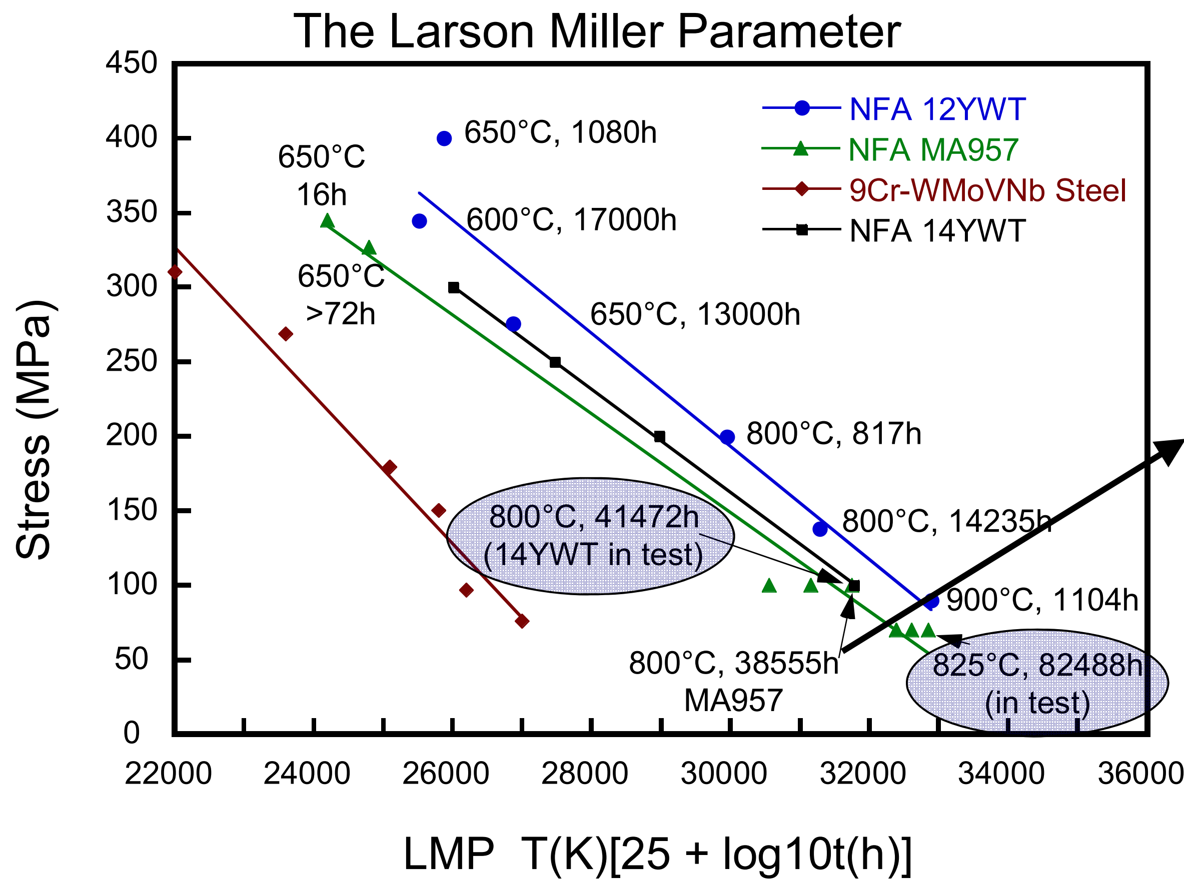
Typical tensile strength and ductility of numerous 14YWT heats from 25ºC to 800ºC

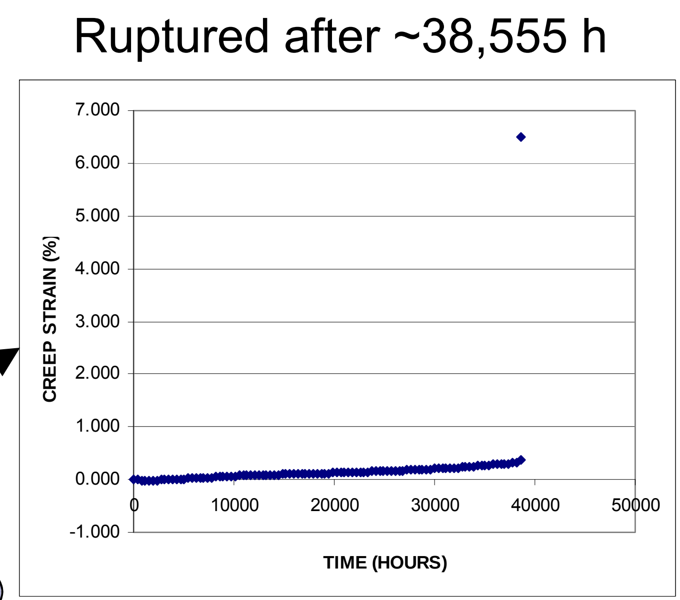
Attributes and concerns

- High strengths at low temperatures, *but sudden drop occurring above 500ºC*
- Extensive deformation after plastic instability to failure, *but low uniform elongation*
- *Variations between heats*
  - ✓ SM12 is a new “cleaner” heat containing lower O, C and N contamination levels



Creep performance of 12YWT, MA957 and 14YWT





- *Klueh et al, J. Nucl. Mat., (2005)*
- *I-NERI FY01-04*

- Creep tests still in progress:
  - *MA957: test at 825ºC and 70 MPa started in Oct., 2003 (INERI)*
  - *14YWT-SM10: test at 800ºC and 100 MPa started in April, 2008 (INERI)*

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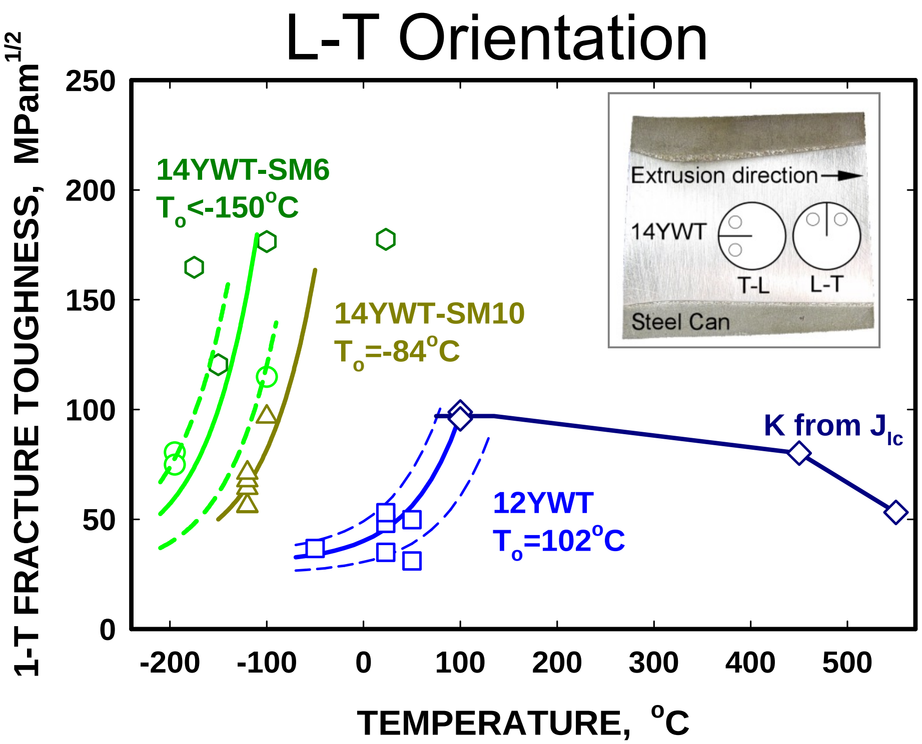
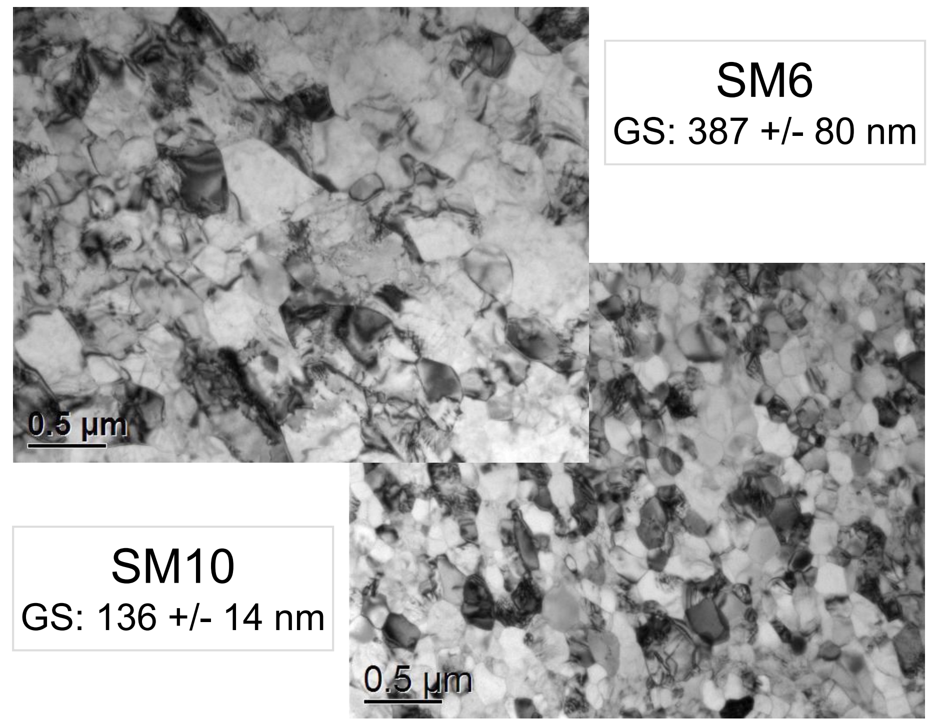
Fracture toughness properties of 14YWT



Two heats of 14YWT alloys



- Fracture toughness properties of 14YWT-SM6 were very good
  - ***T_0 = -150ºC and FT = ~175 MPa√m (25ºC)***
- Fracture toughness properties of 14YWT-SM10 were not as good
  - ***T_0 = -84ºC and FT = ~145 MPa√m (25ºC)***
  - ***Grain size may be a factor***
- FT properties for 14YWT heats still significantly better than 12YWT

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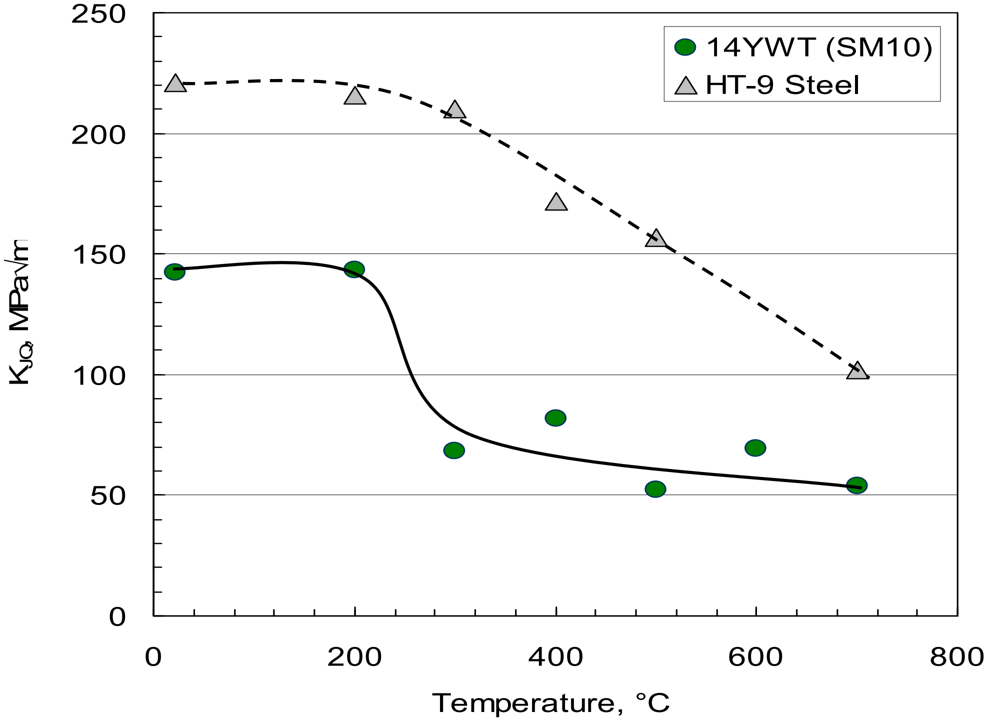
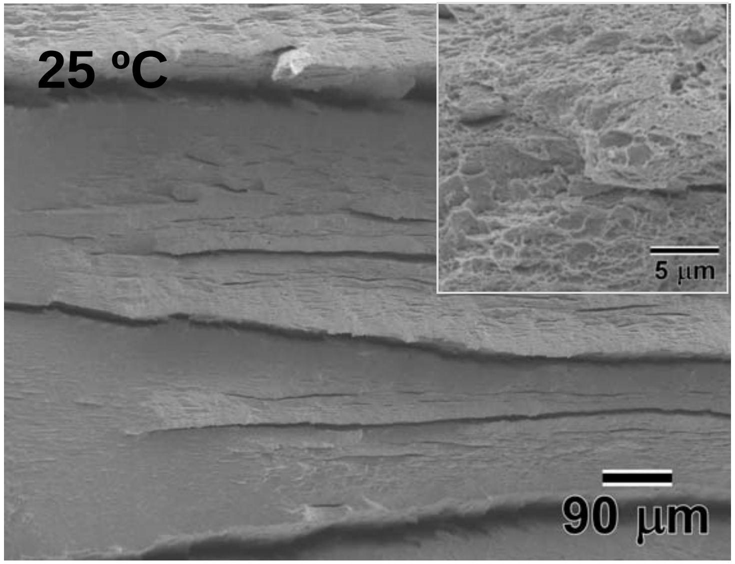
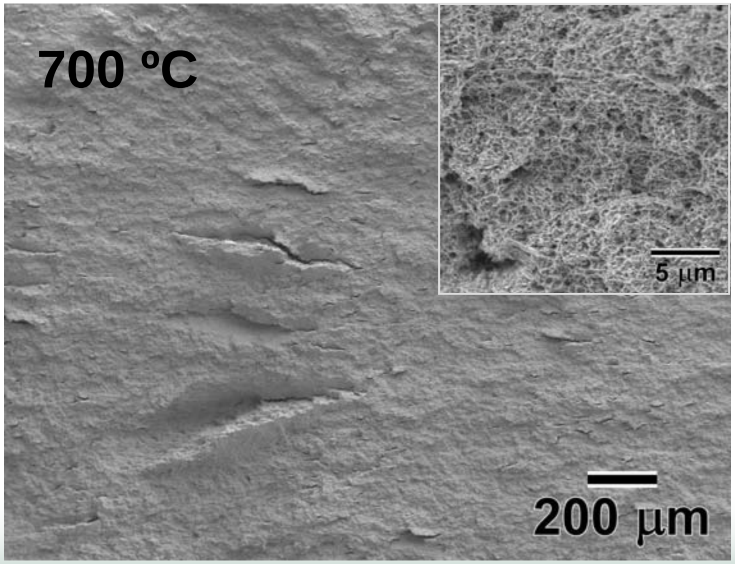
But, poor fracture toughness properties discovered for 14YWT-SM10 (and other ODS alloys) in 2010







- **At 25ºC** - Quasi-ductile fracture with nano-scale dimples and cleavage facets with shear lips plus macro-scale crevices
- **At 700ºC** - Brittle fracture with shallow nano-scale dimples possibly formed by decohesion of individual and multiple nano-size grains

*T.S. Byun et al., J. Nuclear Materials, **407**, 2010*

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US collaboration on the ODS 14YWT ferritic alloy

- Collaborative initiative to develop larger “best practice” heats and forms of nanostructured ferritic alloys (NFA)
- 14YWT ferritic stainless steel
- Collaboration led by LANL (Maloy) with:
  - ORNL (Hoelzer)
  - UCSB (Odette)
  - UC Berkeley/UT (Wirth)
  - Crucible Research/ATI Powder (Stewart)
  - South Dakota School of Mines (West)

"Best practice" processing of 14YWT: ***Powder atomization approach***

ATI Powder Metals: Y atomized with Fe alloy

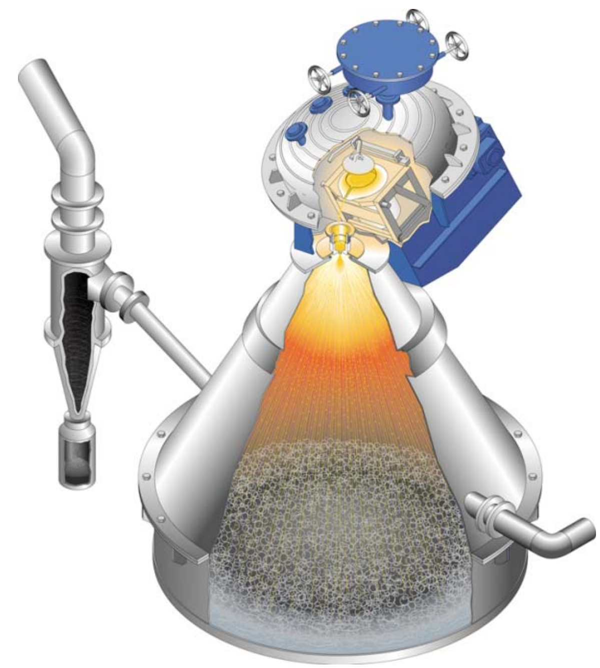
- 3 Small <20 kg heat (L2311) Ar gas: Fe-14Cr-3W-0.3Ti-0.21Y-0.014 O
  (L2312) Ar/O gas: Fe-14Cr-3W-0.3Ti-0.23Y-0.096 O
  (L2313) He gas: Fe-14Cr-3W-0.3Ti-0.21Y-0.011 O
- **Large ~55 kg heat (L2314) Ar gas: Fe-14Cr-3W-0.3Ti-0.21Y-0.012 O**

Ball Milling Studies

- UCSB - 10 h SPEX shaker mill
- UCB - 1, 5, 20 40 h CM01 attritor mill
- ORNL – 40 h CM08 attritor mill

Annealing/Consolidation Studies

- UCSB 1100 and 1150ºC ramp + anneal 3 h
  1150 ºC – 200 MPa HIP 3 h
- ORNL 1 h anneal 850ºC + extrusion



"Best Practice" processing of 14YWT: *Some Key Results*

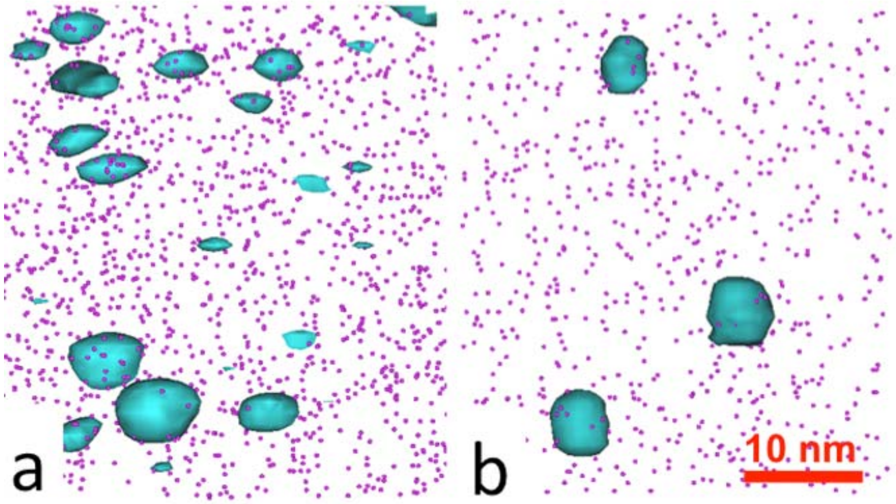
- Y phase separates in atomized powders requiring milling for mixing and NF formation
- Improved milling impurity (N, O) control showed the critical importance of O balance in forming NF and achieving excellent balance of properties
- Baseline large batch of ATI powders contained low O
  - Powder subsequently milled with FeO to optimize O level
  - Led to OW4 HIPed at 1150°C (UCSB) and 14YWT-PM2 extruded at 850°C (ORNL)
- PM2 was the final small precursor to the larger "best practice" heat (FCRD-NFA1) tentatively selected in part due to fine and uniform grain structures and balanced intermediate strength…*covered in presentation by Odette*

"Best Practice" processing of 14YWT: *Oxygen effects*

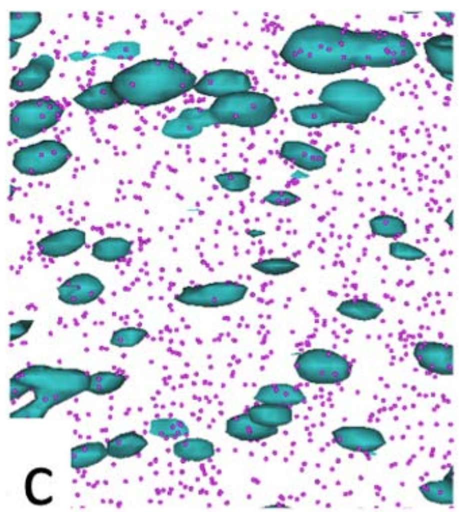
- OWH = 0.249 wt.% O
- OWI = 0.127 wt.% O
- OWL = 0.065 wt.% O

- Fine scale NF only in OWI and OWH

OWH OWL



OWI

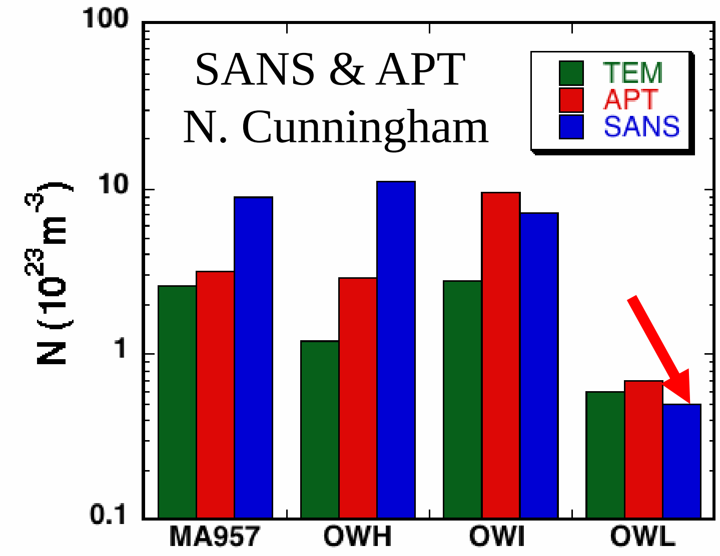


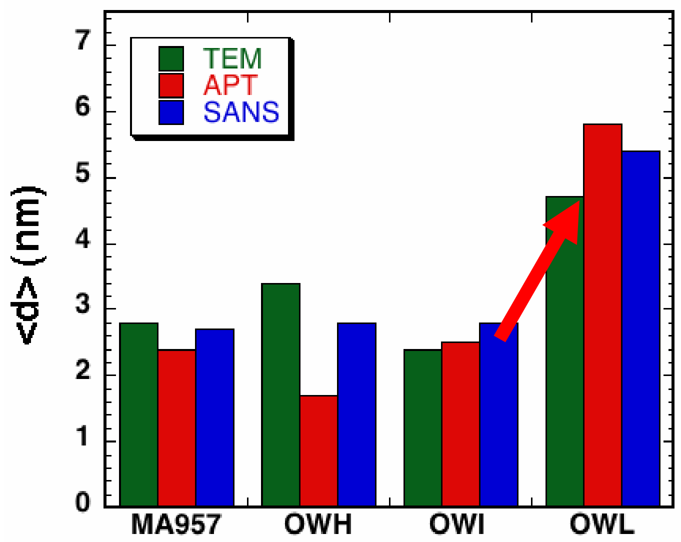
20 nm thick

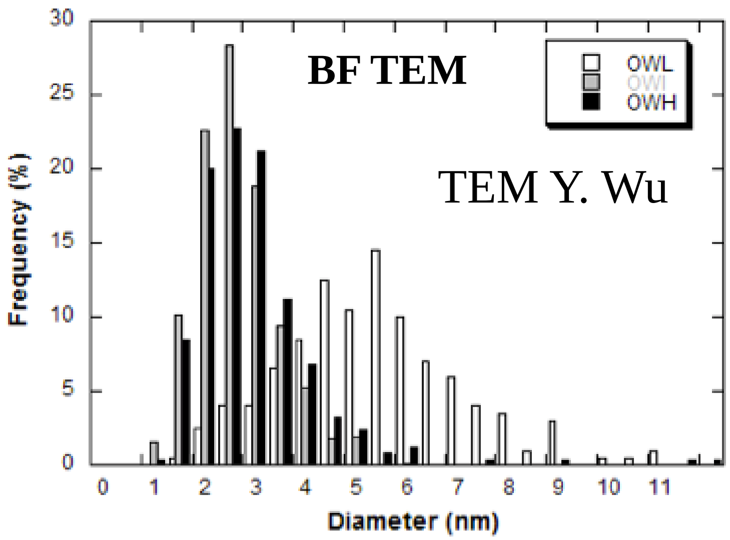
Y-Ti-O Isoconcentration Surfaces

APT: N. Cunningham

“Best Practice” processing of 14YWT: *Oxygen effects*







	μH
OWH	443±12
OWI	368±12
OWL	252±7

Partially due to grain size

- Higher Y/Ti (TEM >>1) measured in OWL

"Best Practice" processing of 14YWT:
Optimizing C, N and O levels

- Lower C and N levels achieved in developmental heats

March 2010 → July 2010 → March 2011 → August 2011

Element	14YWT-SM11 (1.6 kg) Fe-14Cr-3W-0.4Ti + 0.3Y_2O_3		14YWT-PM1 (2.4 kg) Fe-14Cr-3W-0.4Ti-0.2Y-0.014O (L2311)		14YWT-SM12 (3 kg) Fe-14Cr-3W-0.4Ti + 0.3Y_2O_3		14YWT-PM2 (1 kg) L2311 + 0.35 FeO	
(wppm)	Atomized	Milled	Atomized	Milled	Atomized	Milled	Atomized	Milled
O	150	2590	140	447	150	1184	140	1352
C	100	346	60	287	100	128	60	140
N	50	1911	25	123	50	117	25	140

- For 14YWT-SM12: O level was increased by ball milling with 0.3 wt. % Y_2O_3 powder, but C and N levels were kept low
  - *3 heats produced for HFIR JP30/31 experiment*
- For 14YWT-PM2: O level was increased by ball milling 0.35 wt.% FeO powder with L2311 powder
  - *precursor heat to the 55 kg scale-up heat (FCRD-NFA1)*

"Best practice" processing of 14YWT

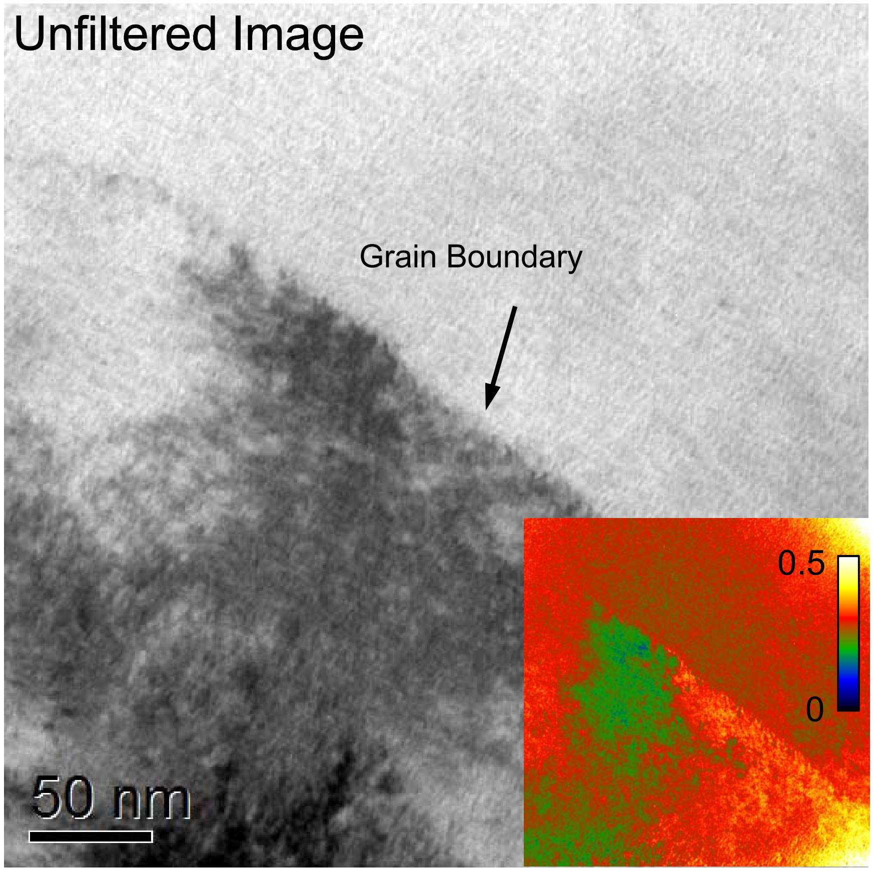
Fabrication of 14YWT-SM12 heats

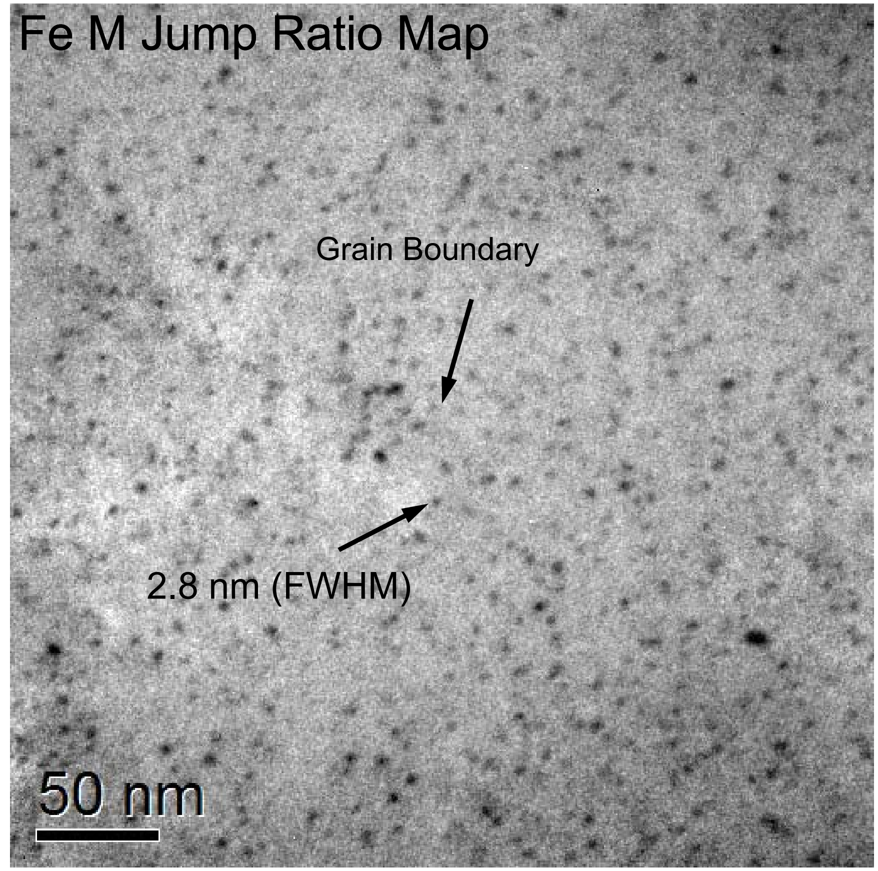
- 3 heats produced to study effect of extrusion temperature
  - SM12a: 850ºC/1h → 1150ºC/1h → extrude
  - SM12c: 850ºC/1h → 1000ºC/1h → extrude
  - SM12d: 850ºC/1h → 850ºC/1h → extrude
- Two 6 mm thick plates produced
  - Plate 1: 50% RIT at 1000ºC parallel to extrusion axis (PR)
  - Plate 2: 50% RIT at 1000ºC normal (cross) to extrusion axis (CR)

- Specimens fabricated from PR and CR plates for:
  - ***HFIR JP30/31 experiment****
  - ***Reference characterization studies****

** Funded in part by Fusion Materials Program, FY2012, ORNL*

"Best practice" processing of 14YWT
EFTEM analysis of SM12c (1000ºC)



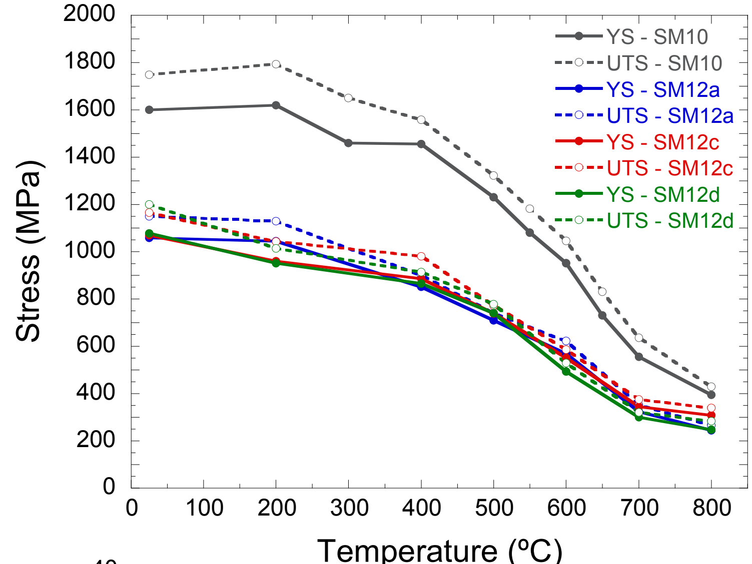


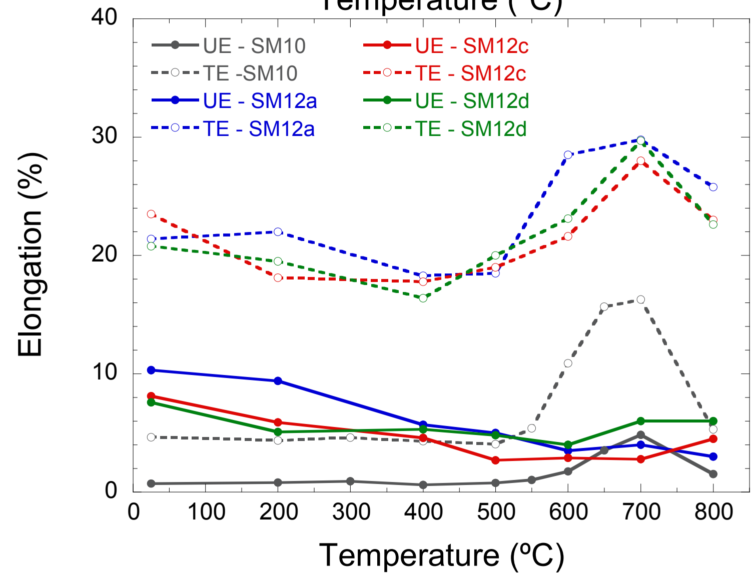
- High density of < 5 nm size NC present
- Some grain boundaries free of NC and small oxide particles
- t/l thickness map ($\lambda \cong 140$ nm; $0.5\lambda \cong 70$ nm)

"Best practice" processing of 14YWT

Tensile Properties of SM12 heats

- Strength and ductility values very similar between SM12 heats from 25ºC to 800ºC
- Strength of SM12 heats are lower, but ductility of SM12 heats are higher than 14YWT-SM10 over full temperature range
- Grain size (*D*) of the SM12 heats
  - ***SM12a****: D = 0.833 +/- 0.122 nm*
  - ***SM12c****: D = 0.714 +/- 0.079 nm*
  - ***SM12d****: D = 0.732 +/- 0.073 nm*
- *Difference in grain size between SM12 heats and SM10 accounts for much of the lower strengths*





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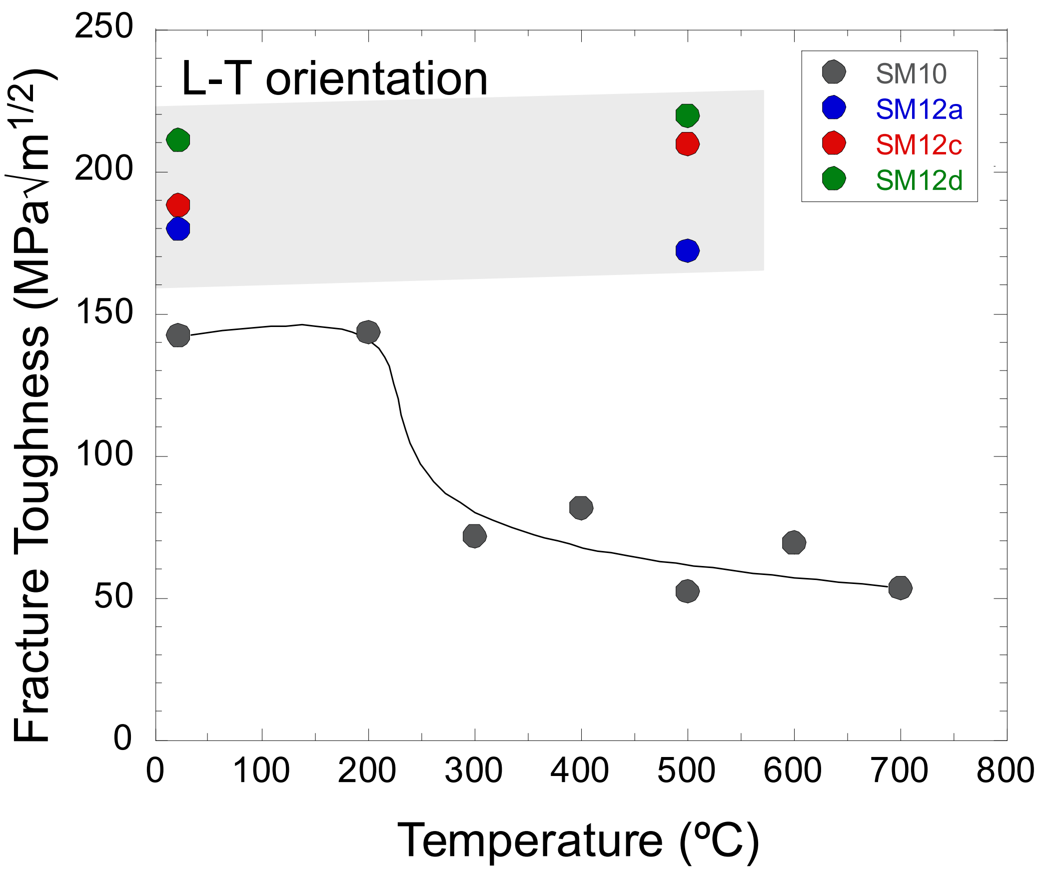
"Best practice" processing of 14YWT
Fracture toughness of SM12 heats



- HT (500ºC) fracture toughness of the three SM12 heats are up to **4x higher** than 14YWT-SM10
- Results not shown for T-L orientation, but are very good

- *These results are very promising for the scale-up FCRD-NFA1 heat currently being characterized*

"Best Practice" processing of 14YWT:

Processing history FCRD-NFA1

- ~55 kg of L2314 powder separated into 3 size ranges
  - *Coarse: 150-500 μm*
  - *Middle: 45-150 μm*
  - *Fine: <45 μm*
- Zoz, GmbH, completed 4 ball milling experiments
  - *40 h in Ar using parameters supplied by FCRD processing team*
  - Small powder samples were taken at 20 h and 40 h and analyzed
- Details of 4 ball milling experiments (in order)
  1) *Coarse size powders were ball milled*
  2) *Fine and middle size powders were mixed and ball milled*
  3) *A small quantity of ball milled coarse size particles were added to the fine and medium size powders and ball milled*
  4) ***Reference 14YWT heat:** 15 kg of 14Cr-3W-0.4Ti powder was blended with 0.3 wt.% Y_2O_3 powder and ball milled*

"Best Practice" processing of 14YWT

Zoz ball milling results

- Powders ball milled with CM100 Simoloyer

- Ball milling conditions achieved the desired goal of elevated O level and low N and C levels

Sample Identification	20hr Milling	40hr Milling
V540-01	%	%
Oxygen	.091	.111
Nitrogen	.006	.008
Carbon	.012	.019
Chromium	13.7	13.7
Tungsten	2.90	2.88
Titanium	.38	.38
Yttrium	.18	.19

"Best Practice" processing of 14YWT
EPMA of medium (45-150 μm) and fine (<45 μm) size powders

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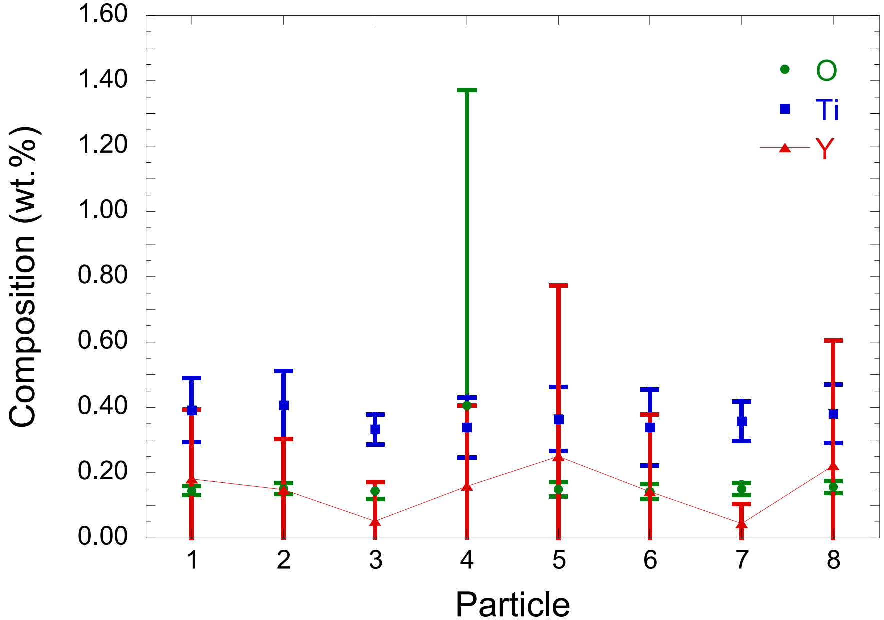
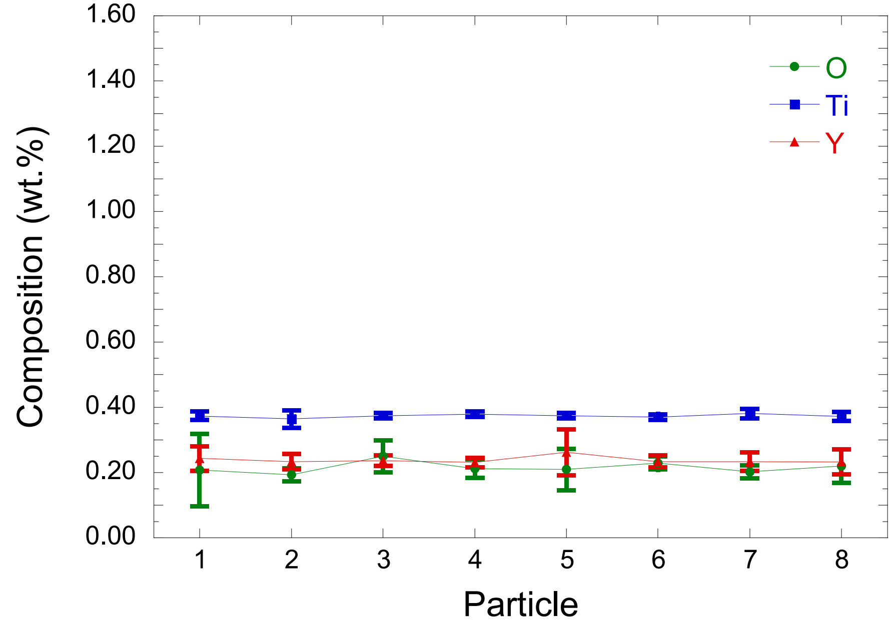
Atomized Medium Size Powder

8 Particles	Wt. %		
	O	Ti	Y
Average	0.181	0.364	0.150
SE 95%	0.096	0.061	0.168



Ball Milled Medium Size Powder

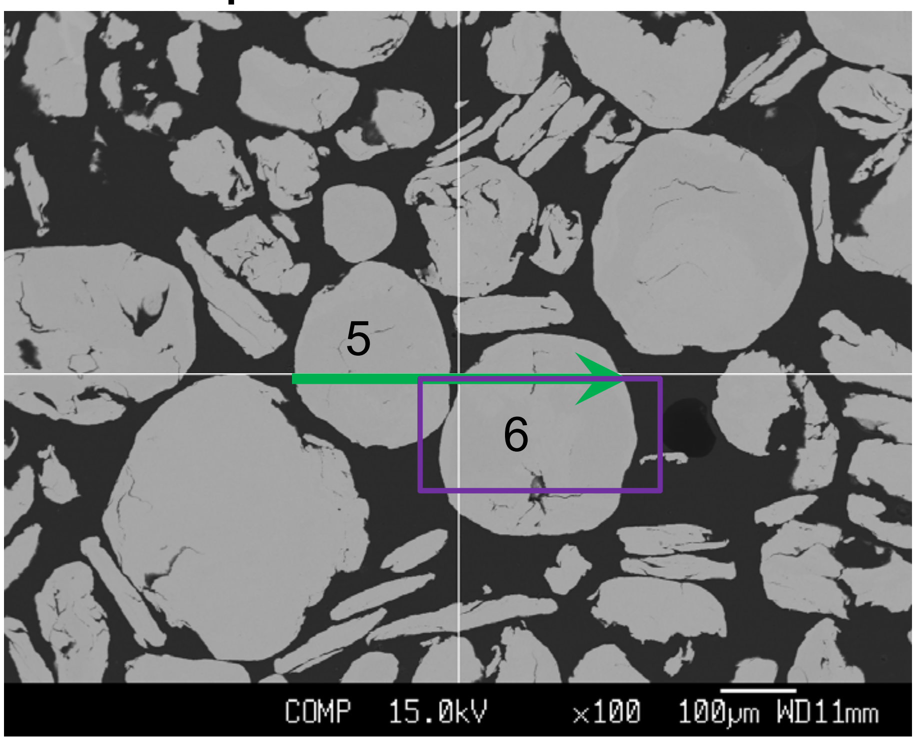
8 Particles	Wt. %		
	O	Ti	Y
Average	0.216	0.374	0.238
SE 95%	0.032	0.009	0.022

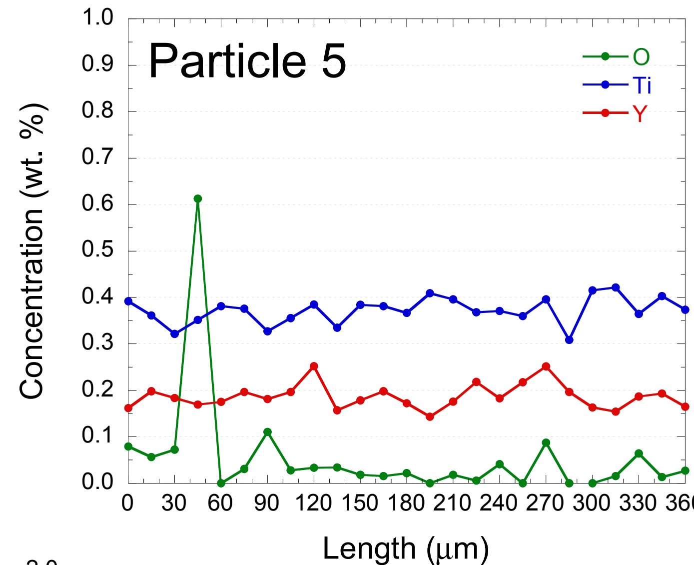


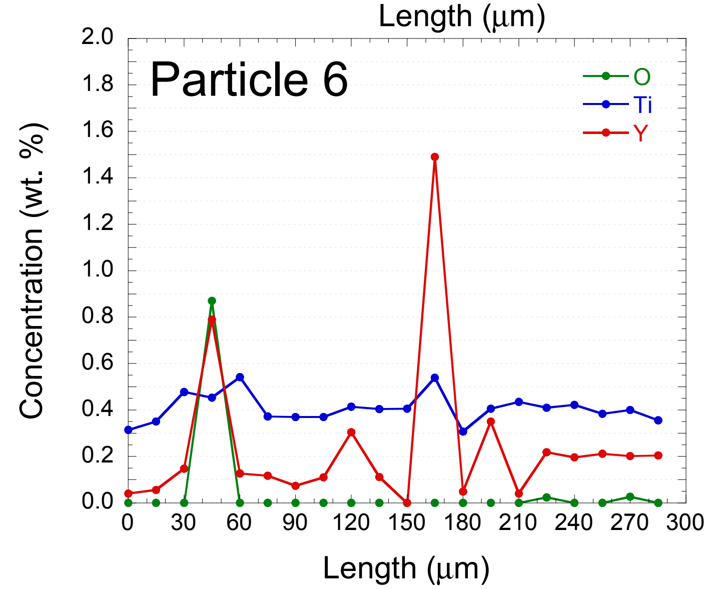
- Ball milling improved both the concentration level and distribution of Y in the medium and fine size powders

"Best Practice" processing of 14YWT *EPMA of coarse (150-500 μm) size powders*

- Y was detected, but amounts varied by location and particle size
- Conclusion: >40 h ball milling is required for uniform mixing with coarse powder







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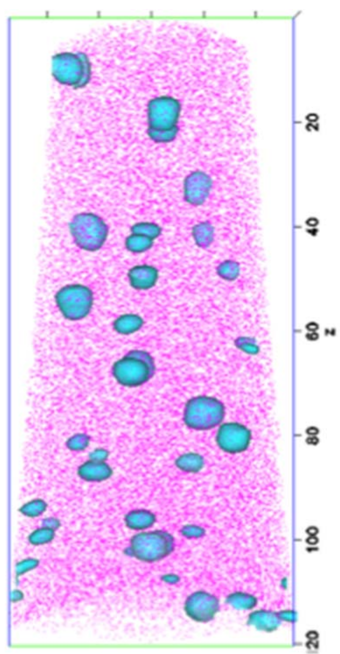
"Best Practice" processing of 14YWT
Microstructure analysis of medium (45-150 μm) size powders

- Ball milled powder was annealed for 3 h at 1150ºC and characterized by TEM and APT (UCSB)

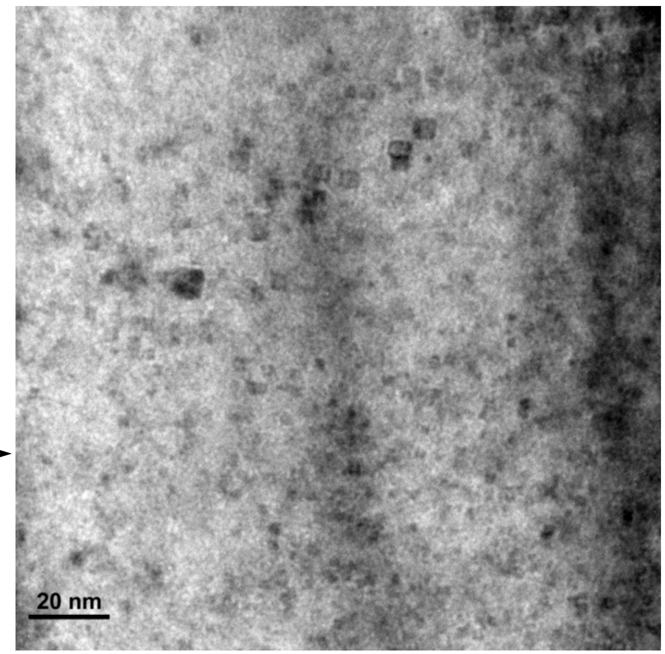
APT: N. Cunningham



$N = 1.2 \times 10^{23}/m^3$
$\langle d \rangle = 2.25$ nm

TEM: Y. Wu

$N = 6.6 \times 10^{23}/m^3$
$\langle d \rangle = 1.9$ nm

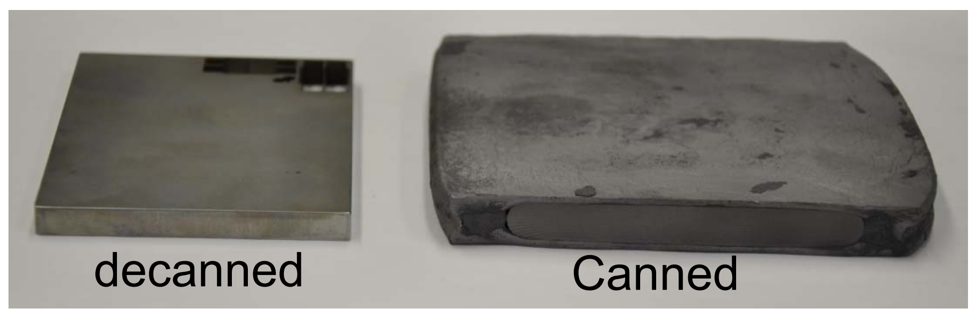


- Formation of a high concentration of nano-size oxygen-enriched particles was achieved in the annealing study

"Best Practice" processing of 14YWT: *Extrusion and plate fabrication*

- 2.8 kg of ball milled powder degassed at 400ºC in sealed 3.9 in diameter steel can
- Can extruded at 850ºC through 2.5 in x 1.2 in rectangular die
  - ODS section cut into 3 equal lengths
  - Annealed at 1000ºC/1h in vacuum

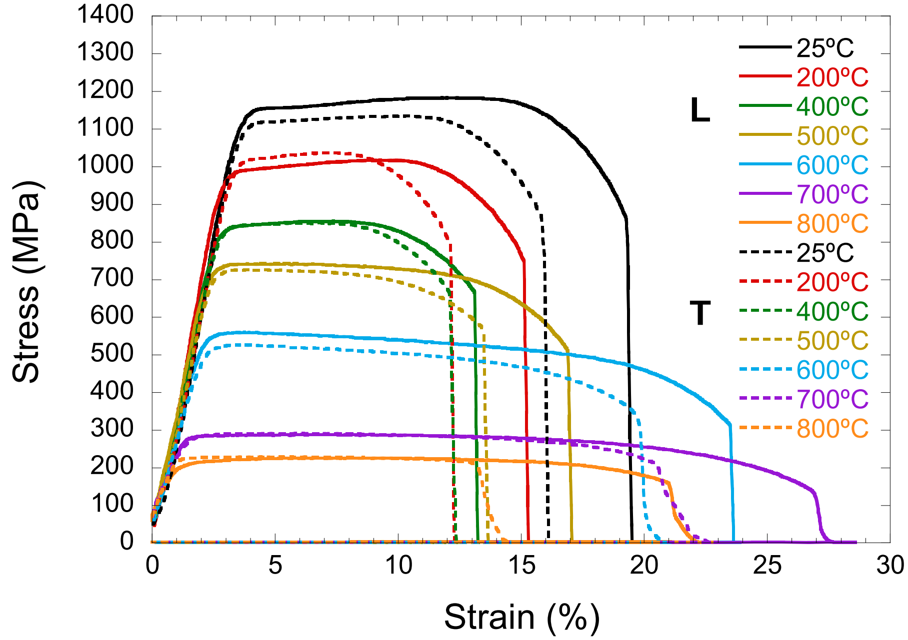
- Cross-rolled to 50% reduction in thickness at 1000ºC
  - 1 plate decanned at ORNL for characterization
    - ✓ *Tensile tests completed*
  - Canned plate sent to UCSB and LANL

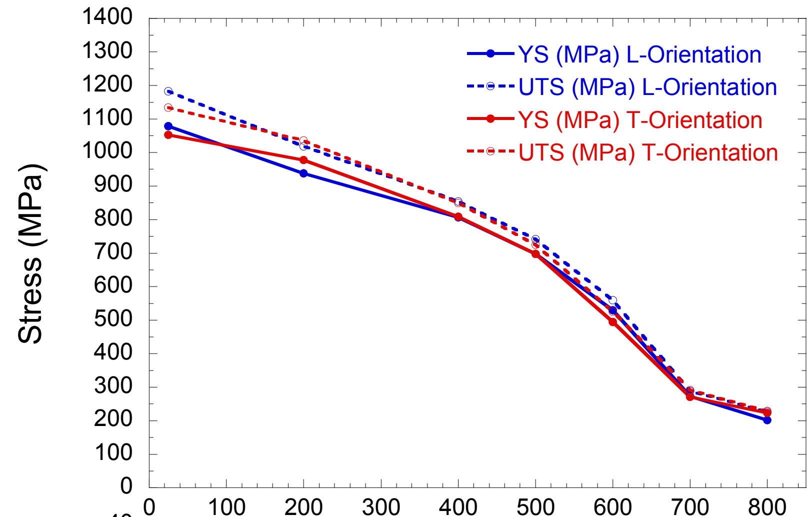


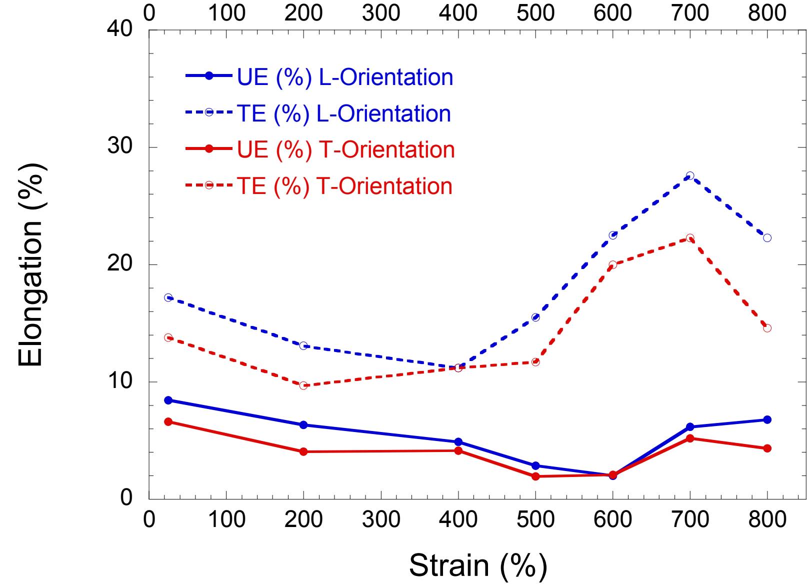
"Best Practice" processing of 14YWT:

Tensile properties of FCRD-NFA1

- Comparing L and T orientations







- Trends:
  - *Strength is lower; ductility is higher compared to previous heats*
  - *Only minor effect of anisotropy*

Summary

- Significant progress has been made in development of the larger "best practice" processing path for 14YWT ferritic alloy with optimum balance of mechanical properties, *including significant improvement in high temperature fracture toughness properties*
  - Improvements in mechanical alloying have led to lower levels and better control of C, N and O, i.e. *cleaner ODS alloys*
  - Controlled temperature-deformation conditions for rolling have been developed
- Future Work Plans
  - Continue characterization and fabrication studies on FCRD-NFA1 and reference 14YWT heat including mechanical properties, microstructure and irradiation studies
  - Demonstration of fabricating thin wall tubing, including establishing collaborations with industry and international research partners