Development and Characterization of a Larger Scale NFA Heat: FCRD-NFA1

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Secondary Sponsor DOE Office of Fusion Energy Sciences (irradiation studies)

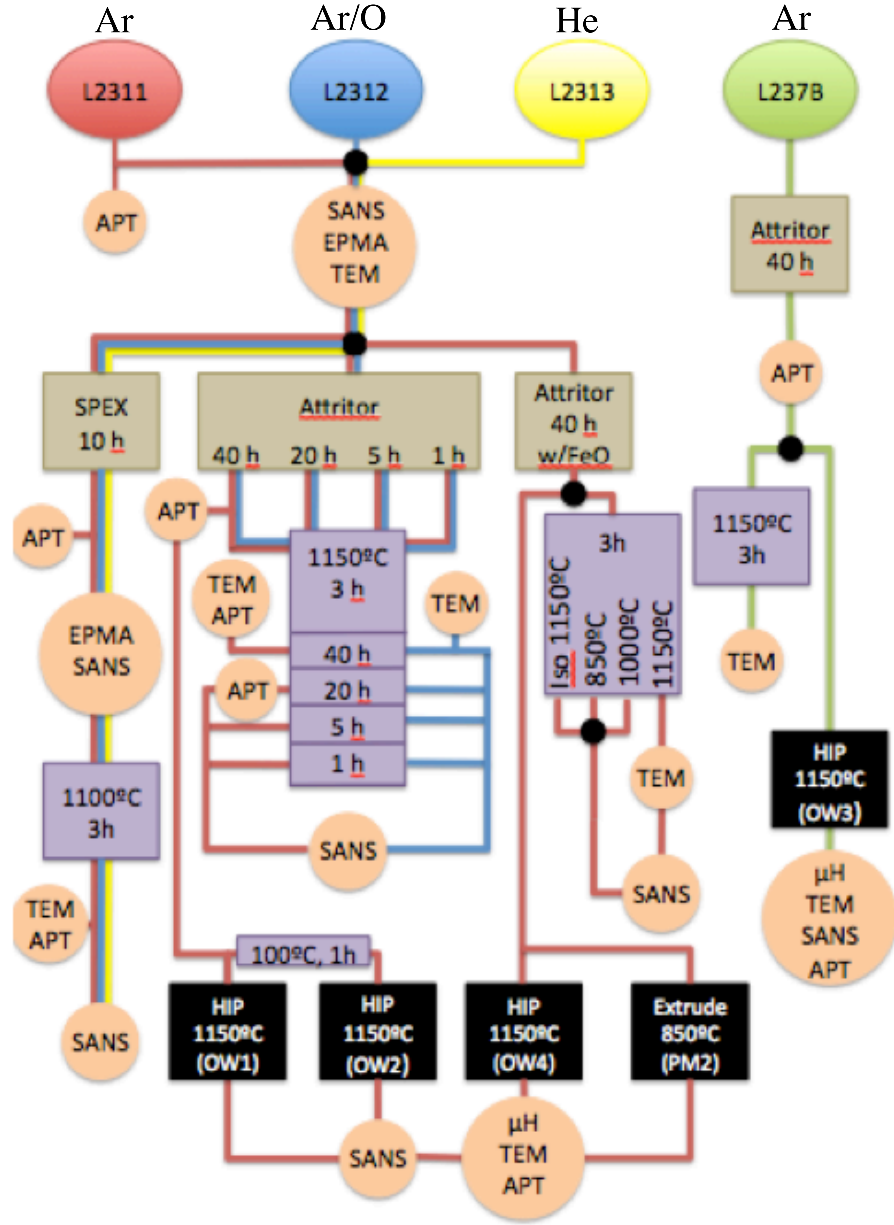
2012 Summary and Conclusions

- Systematic development of ‘best practice” alloy design processing path for larger heat of 14YWT NFA - FCRD NFA1
- Guided by step by step characterization at UCSB and collaborators using state of the art SANS, APT and TEM techniques – including NF phase and OR
- Determined critical parameters - milling time, consolidation temperature, O-level, post consolidation deformation processing
- ORNL produced PM2 as the final precursor alloy
- Industrial scale milling and powder characterization of larger heat and primary consolidation path identified
- Also showed friction stir welding improved strength and microstructure
- Initial dual ion irradiation shows PM2 stable and can manage helium at 650°C

Processing Path Research

Some Key Results

- Y phase separates in as-atomized powders
- 20-40 h milling required for full mixing
- O balance critical in forming NF
- Low O in baseline powders required milling with FeO
- 14YWT-PM2 is the final small precursor to the larger best practice heat (FCRD-NFA1)



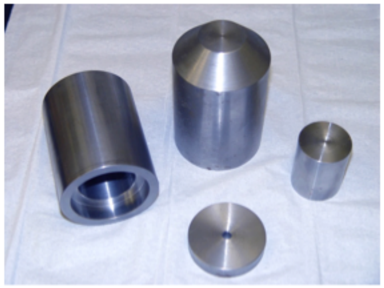
PM2 Summary

Low O base powder 40 h Zoz CM08 attritor milled with FeO and extruded at 850°C (D. Hoelzer) final precursor heat

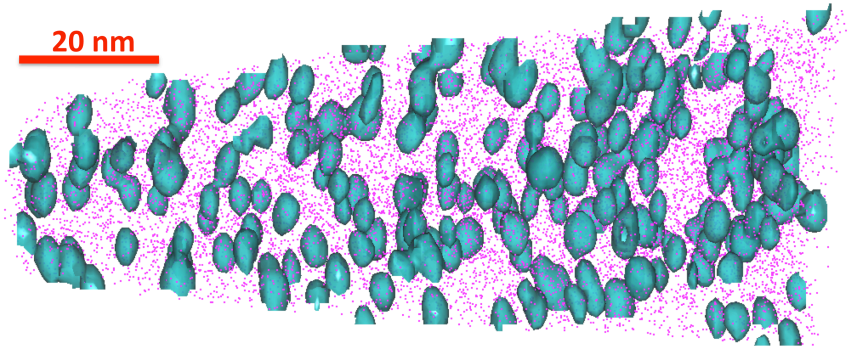
Heat	Chemical Analysis from ATI Powder Metals (wt.%)						
	Cr	W	Ti	Y	O	C	N
L2311	14.0	3.04	0.34	0.20	0.014	0.006	0.003

Element	14YWT-PM2 (1 kg)	
(wppm)	Atomized	Milled
O	140	1352
C	60	140
N	25	140

Cross rolled at 1000°C



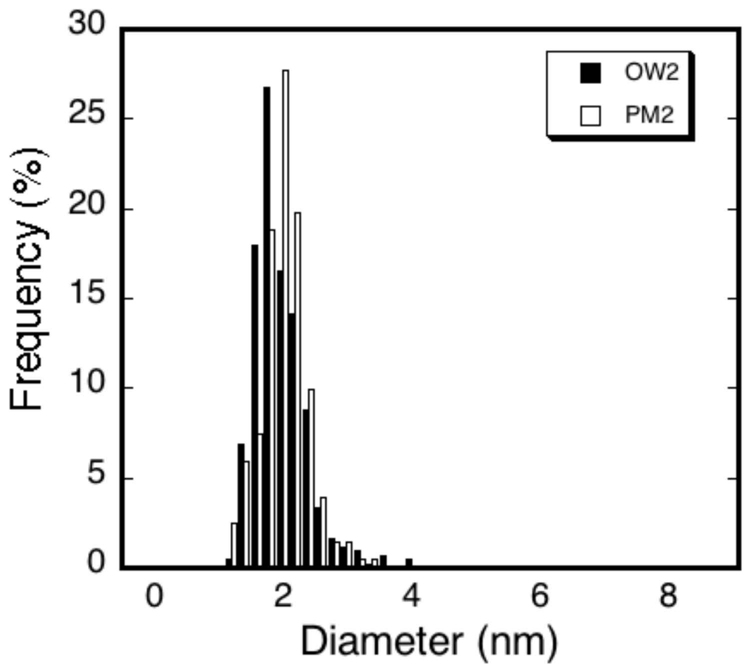
PM2 Summary Cont. - APT



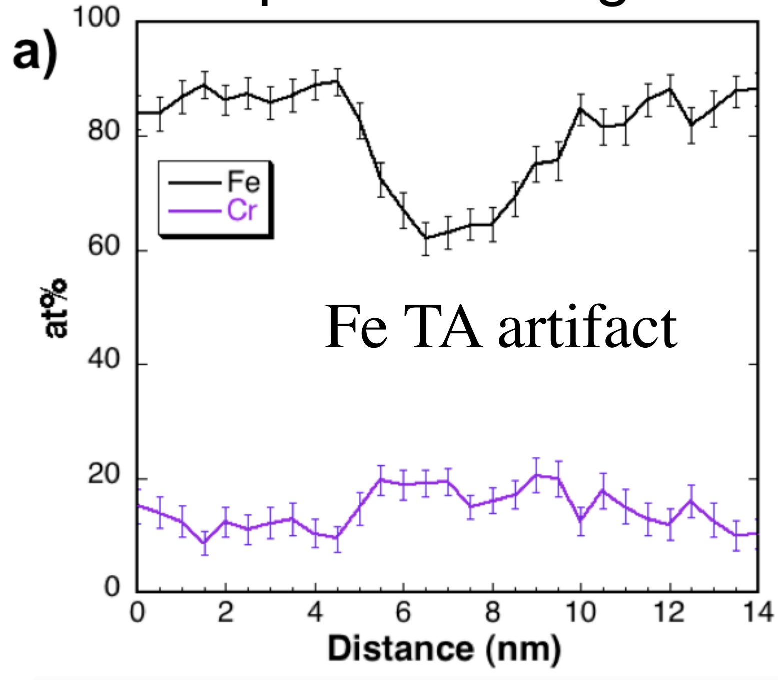
$\langle d \rangle^* \approx 2.1$ nm

$N \approx 10^{24}/m^3$

Y/Ti/O = 12/43/45



1-D profile through NF

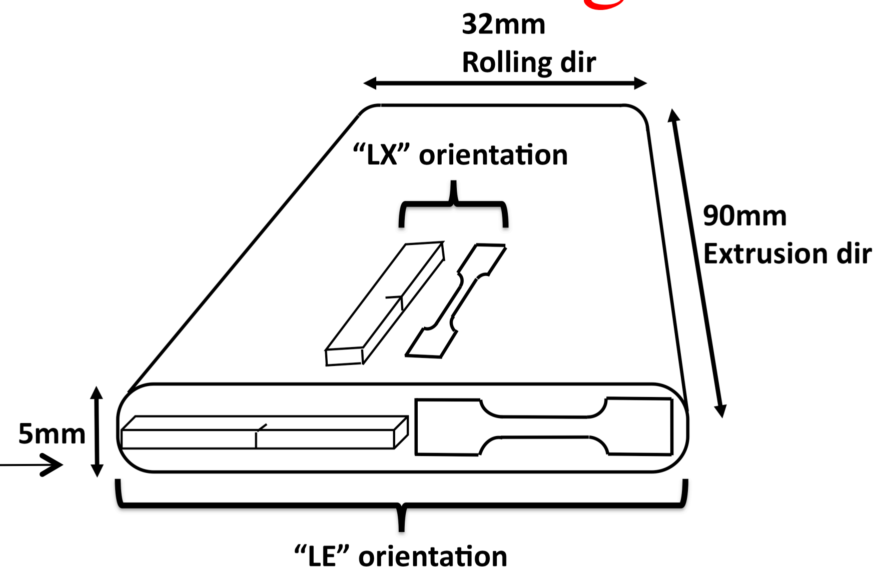


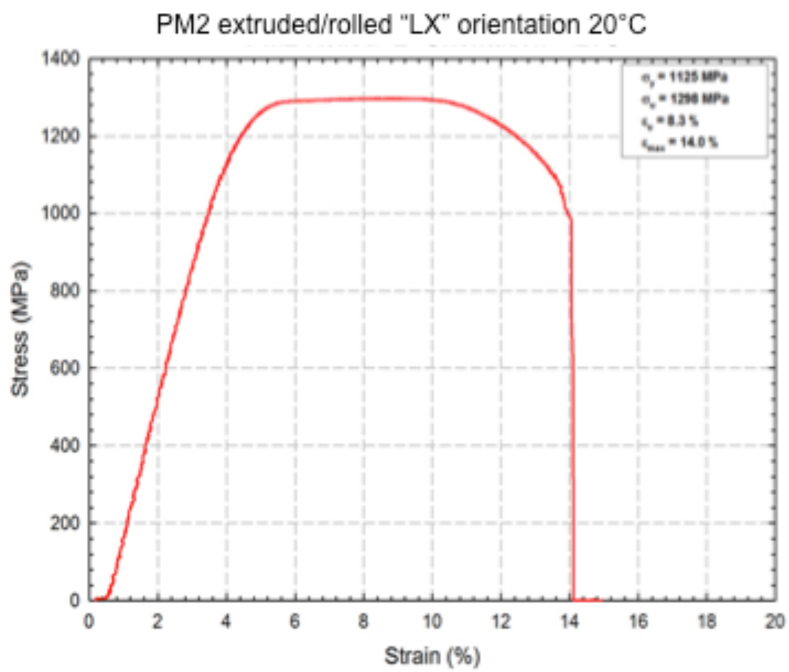
APT E. Stergar and N. Cunningham

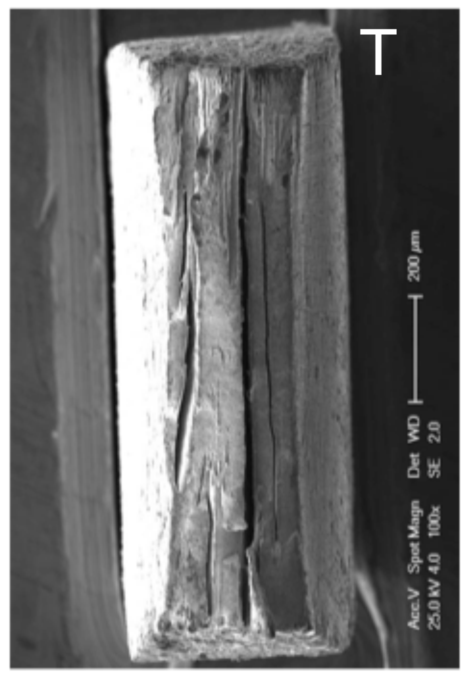
PM2 Summary Cont. – Tensile Strength

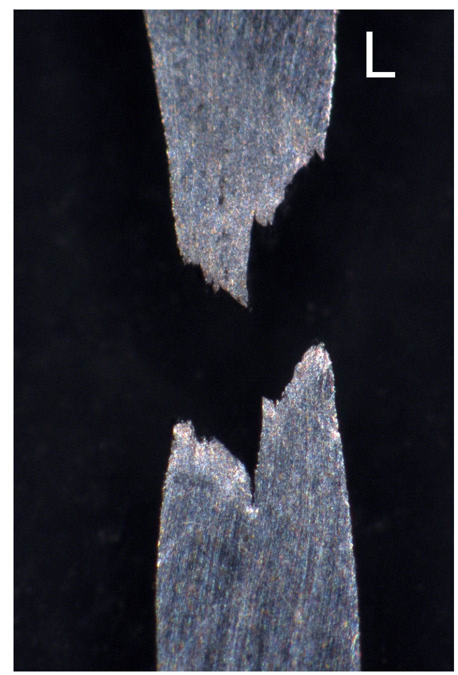
Property	L	T
σ_y (MPa)	1125	1035
σ_u (MPa)	1298	1165
ε_u (%)	8.3	7.9
ε_{max} (%)	14.0	17.5

401±15 kg/mm^2



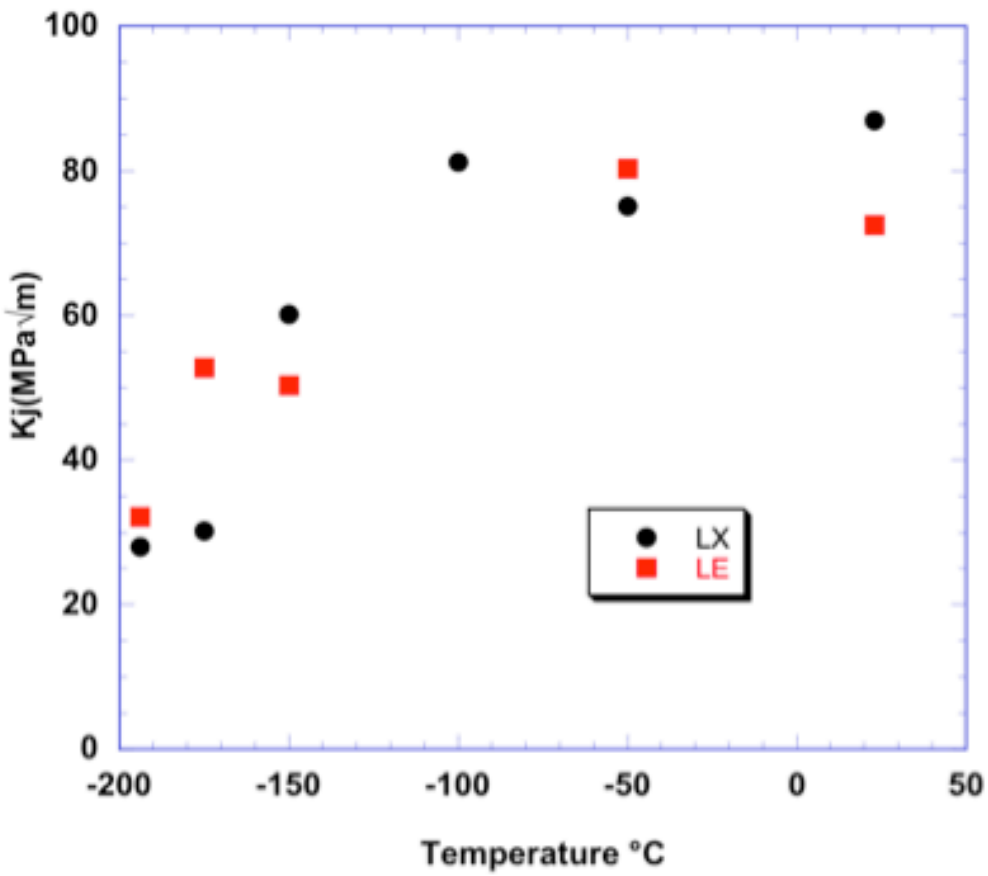


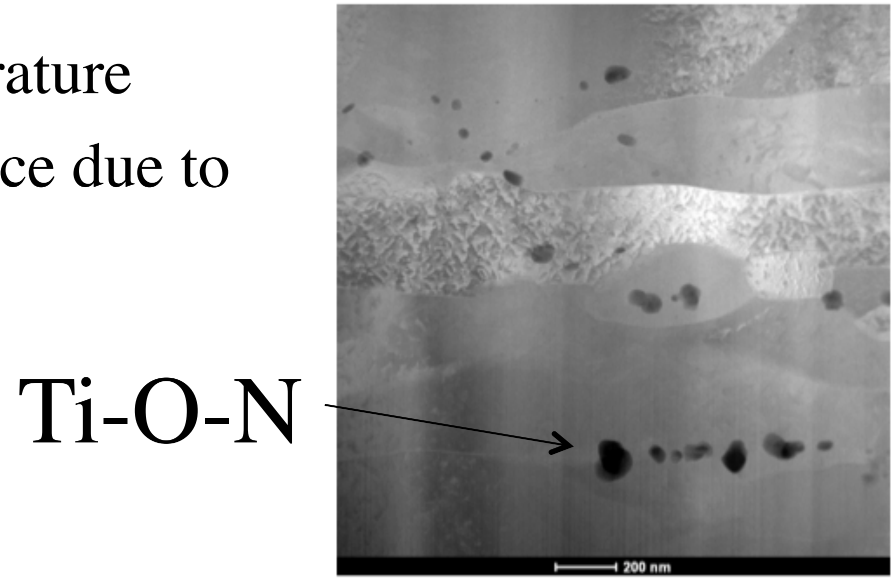


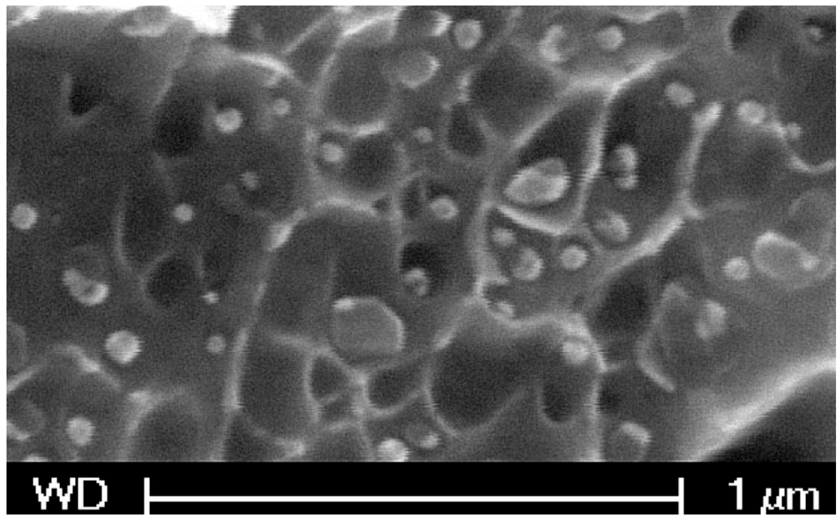


PM2 Summary – Fracture Toughness

- Excellent low transition temperature
- But low ductile tearing resistance due to coarse Ti phases

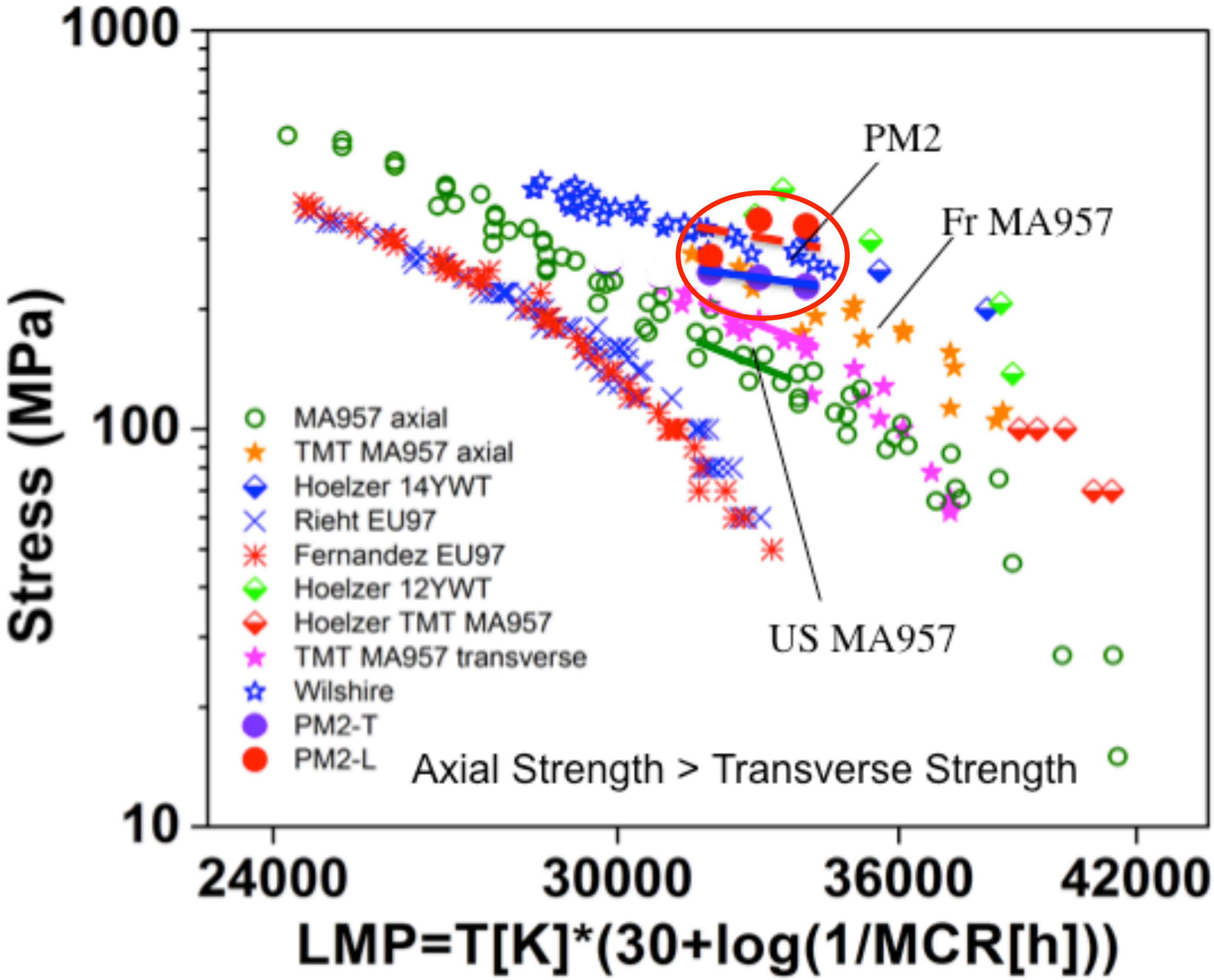






PM2 Summary– Creep Strength

- Limited data show excellent creep strength



New V540 Zoz Milled Powder

Atomized Powder Composition

Fe	Cr	W	Ti	Y	C	S	N	Si	Al	O
Bal.	13.92	3.03	0.38	0.25	0.002	0.001	0.002	0.012	0.031	0.013

Powder Production

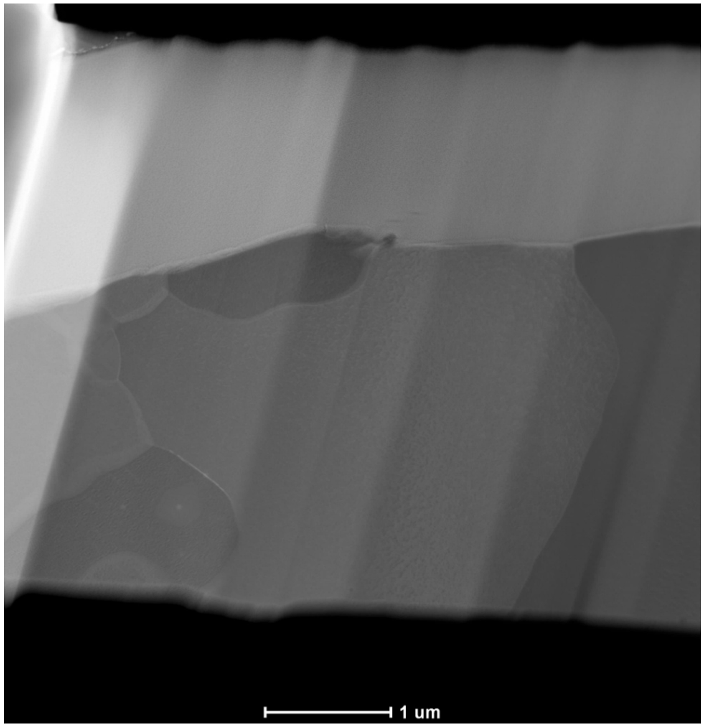
- Gas Atomized in Ar atomsphere
- FeO addition (0.35 wt.%) during milling
- Zoz milled for 40 h in CM100b Simoloyer
- 5 mm diameter grinding media
- Water cooled
- 10:1 Ball mass to charge ratio
- Process cycle 256 rpm/2 min – 150 rpm/10min

V540 Powder and Consolidated Alloys Outline

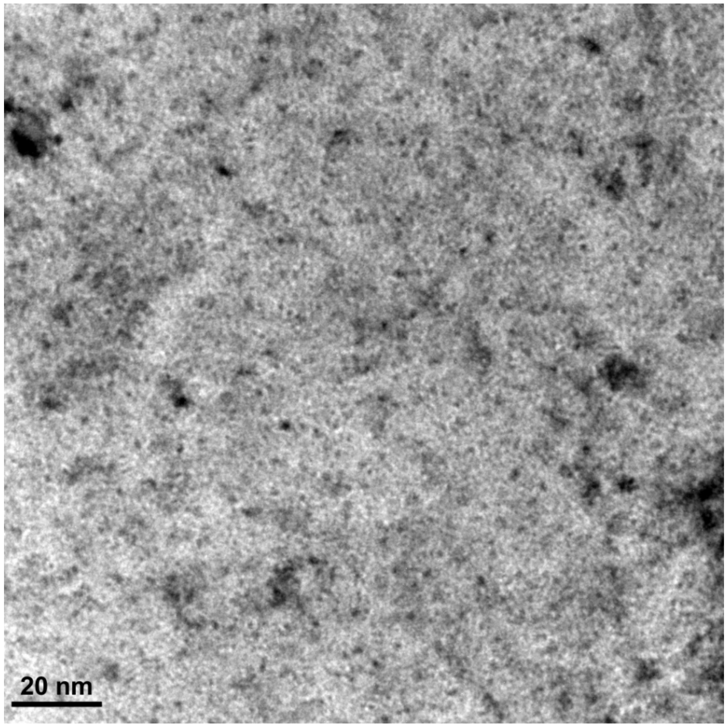
Material	Processing	Analysis
V540-02 Milled Powder	850ºC Anneal	APT
	1150ºC Anneal	APT, TEM
V540-02 HIP	850ºC HIP	SANS
	1150ºC HIP	SANS
	1200ºC Aging (48, 96, 144 and 228 h)	SANS
FCRD-NFA1	Extrude 850ºC, 1000ºC 1 h soak 50% thickness cross-roll	APT, TEM, SANS, μH

Preparation for mechanical testing: Tensile, Creep and Fracture

V540-02 1150ºC Anneal Powder - TEM



- Mixture of small grains (< 0.5 µm) and larger grains (several µm)



Precipitates

<d> (nm)	# density ($10^{23}/m^3$)	f (%)
1.5	6.5	0.18

V540-02 Annealed Powders - APT

NF Composition (Corrected for Fe and matrix atoms)

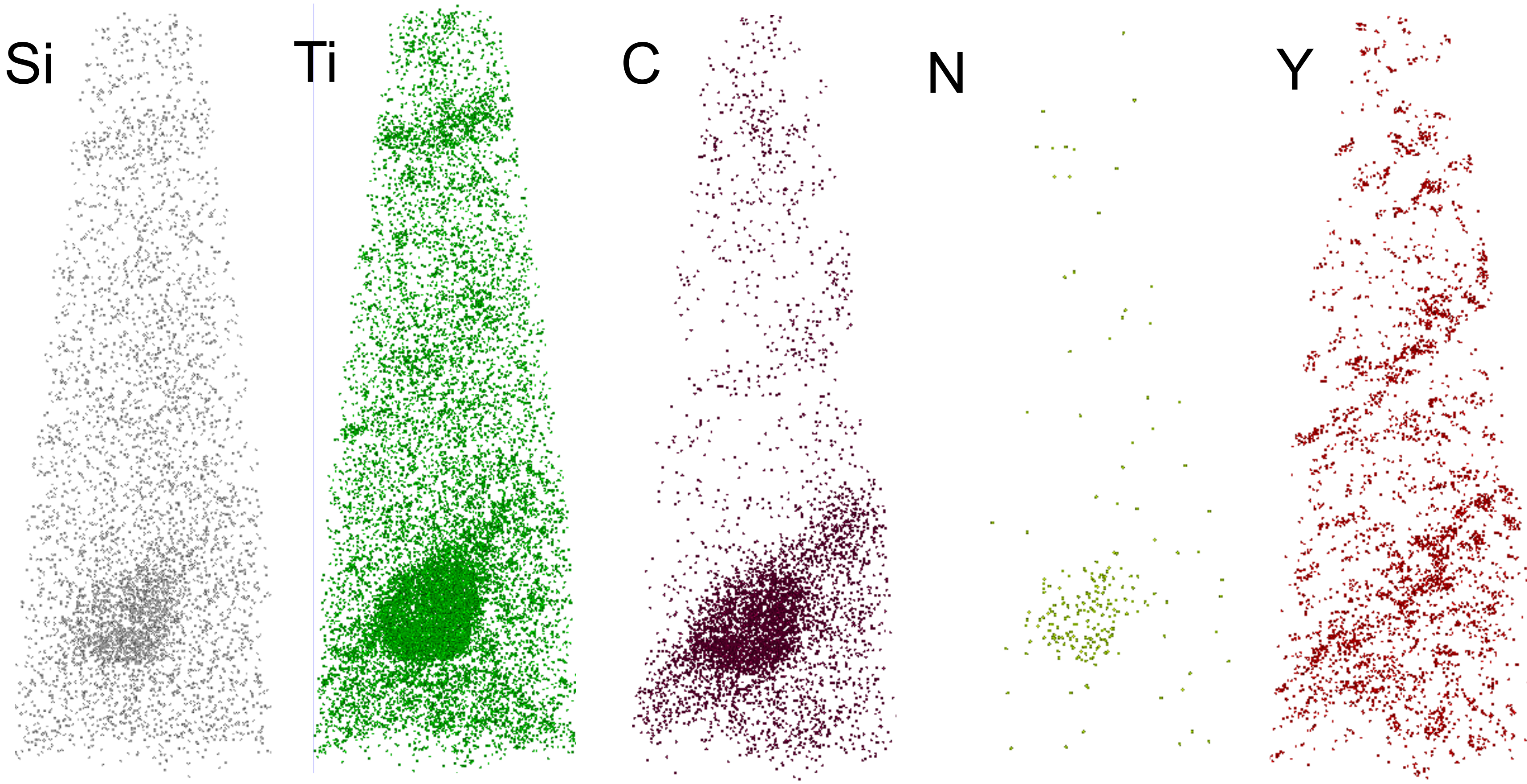
at%	Cr	W	Ti	Y	O
V540-02 850ºC					
Average	21.761	0.774	29.237	10.710	34.973
V540-02 1150C					
Average	19.911	0.244	28.986	10.195	38.274

High Cr content – Typical NFA range 0-25% - core-shell?

	Y/Ti/O	Number Density ($10^{23}/m^3$)	Diameter (nm)	Solute Fraction (%)
V540-02 850				
Average	14.2/39.0/46.8	10.0	1.80	0.51
V540-02 1150				
Average	13.6/38.5/47.9	4.8	2.05	0.38

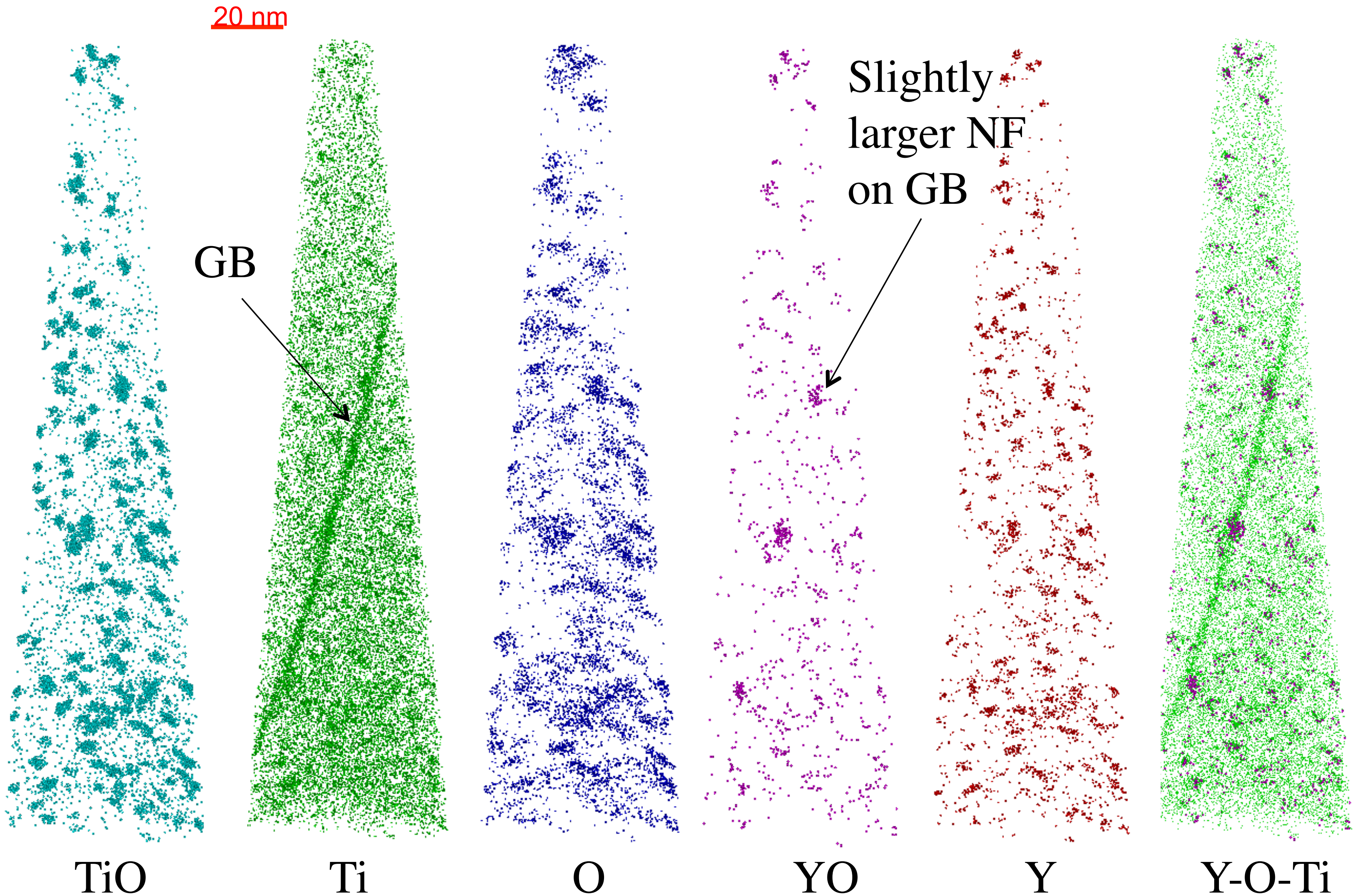
N and f decrease and d increase at higher temperatures

V540-02 Anneal 850^{o}C 3 h

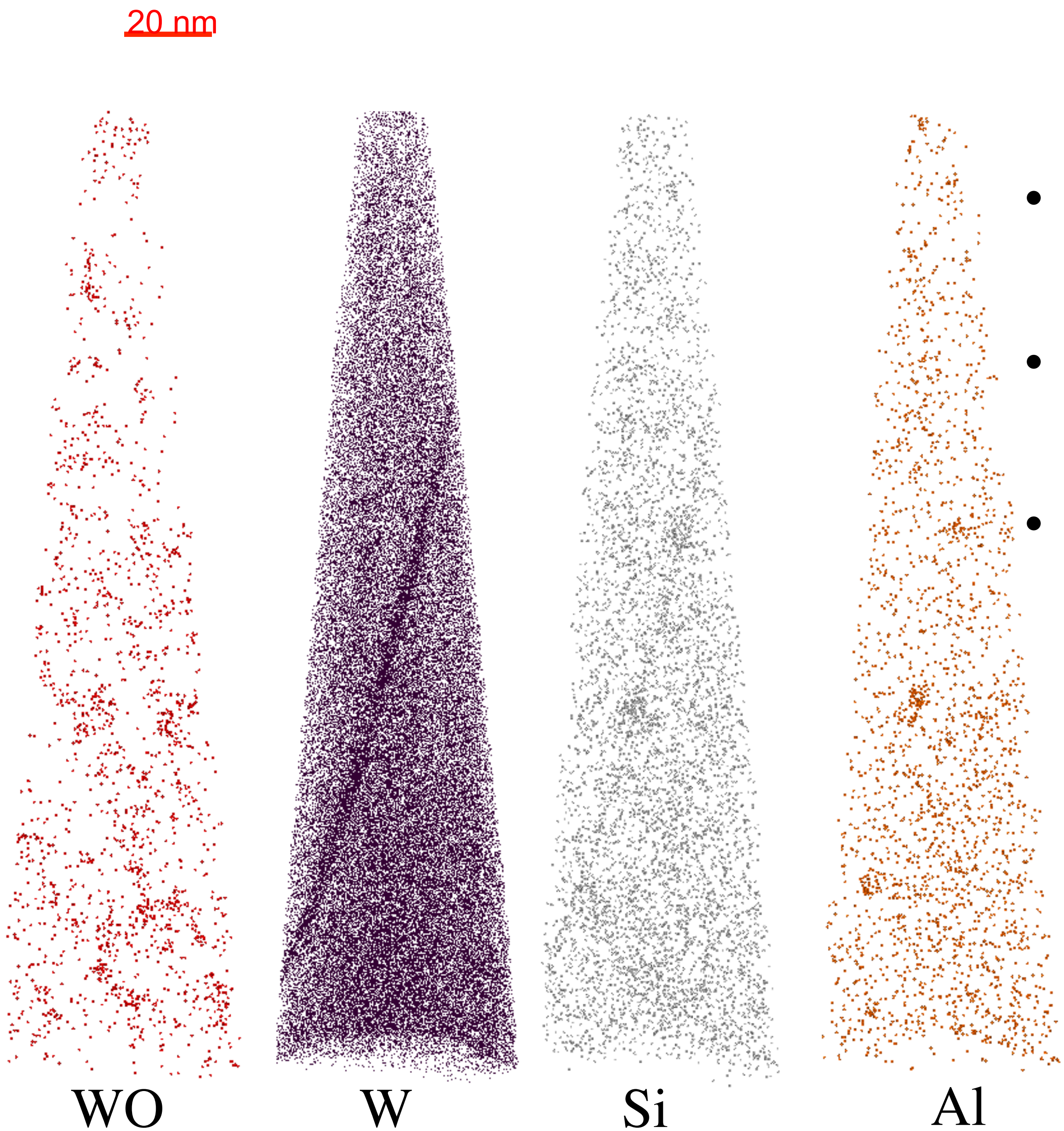


- Larger precipitate on boundary
- Ti, Si, C, O, and some N

V540-02 Anneal 1150ºC 3 h



V540-02 Anneal 1150ºC 3 h



- Ti, Cr, P and W on GB
- Si and Al found in GB NFs
- No larger precipitates observed in 5 APT samples

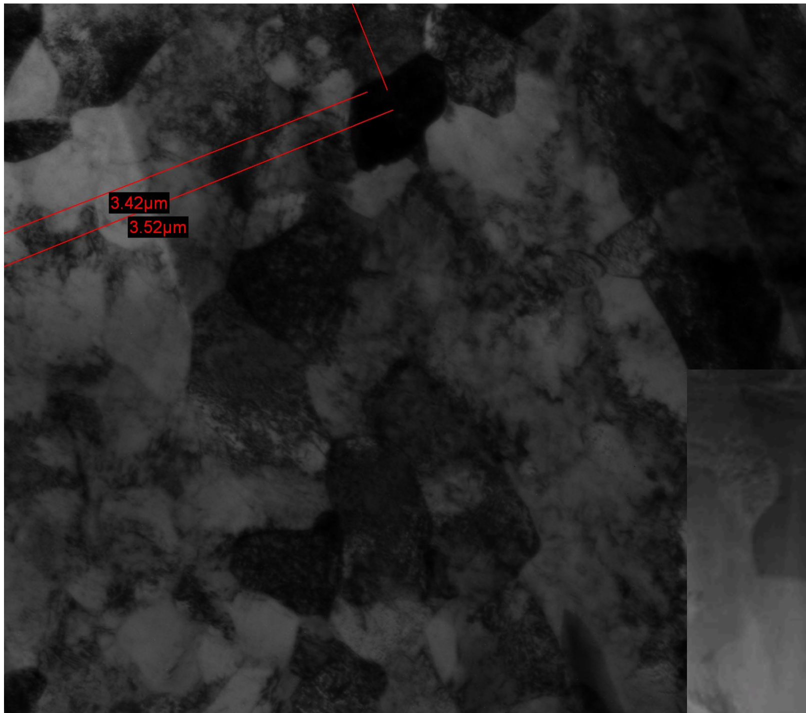
HIP Consolidated Alloys and Annealing

SANS preliminary study

Material	<r> (nm)	N ($10^{23}/m^3$)	f (%)	M/N
V540-02 HIP 850ºC	1.1	29.3	1.52	1.2
V540-02 HIP 850ºC 48h-1200ºC	1.8	3.6	0.92	1.5
V540-02 HIP 850ºC	1.5	7.2	1.00	1.1
V540-02 HIP 850ºC 48h-1200ºC	1.9	3.3	0.88	1.6

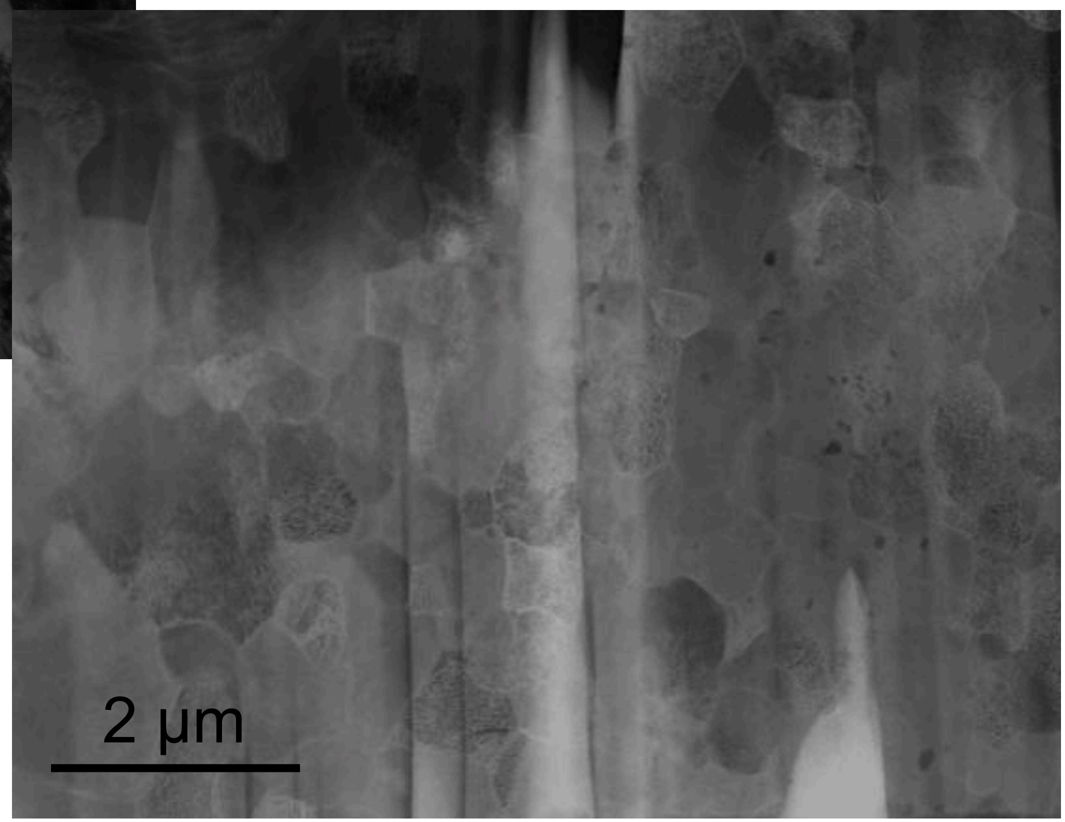
- Smallest <r> and highest N found in 850ºC HIP
- Annealing 1200°C increases r and decreases N amd f
- 850 and 1150°C NF ≈ same after 1200ºC aging at 48 h

FCRD-NFA1 Grain Structure



- Small grain size < 1μm
- Similar to PM2 alloy

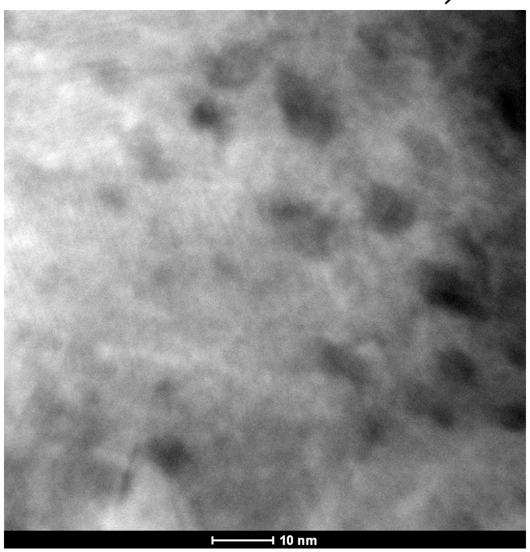
Alloy	Grain Size (nm)
MA957	Elongated 1470 X 630
PM2	424
FCRD-NFA1	454



FCRD-NFA1 TEM

EDX Larger Feature Compositions

	diameter (nm)	Ti	Y	Y/Ti
1	5	3.33	4.38	1.31
2	8	1.65	1.55	0.94
3	9	4.40	2.35	0.53
4	9	2.75	2.63	0.96
5	20	3.76	3.47	0.92

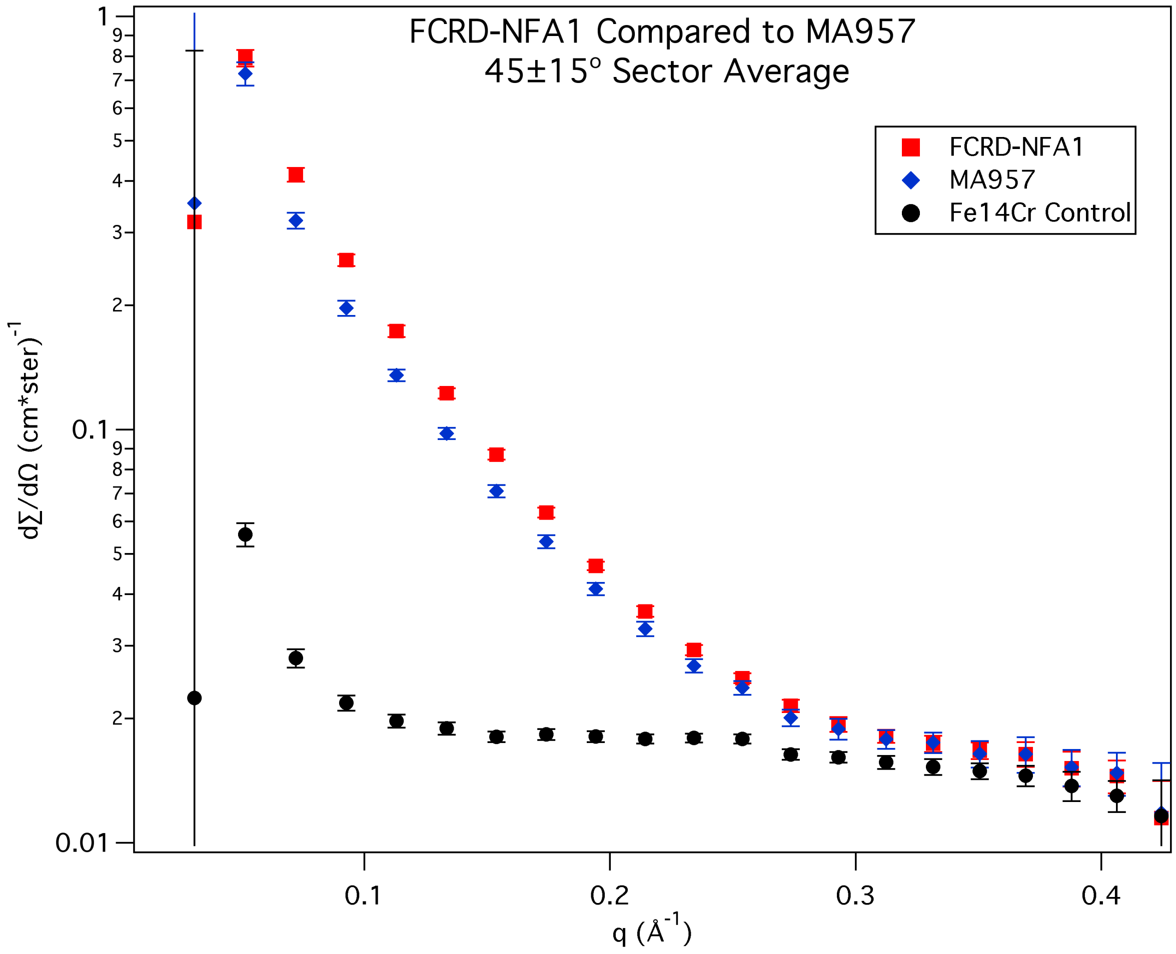


TEM d, N, and f

<d> (nm)	# density ($10^{23}/m^3$)	f (%)
2.1±1.3	1.6	0.25

FCRD-NFA1 SANS

Material	<r> (nm)	N ($10^{23}/m^3$)	f (%)	M/N
FCRD-NFA1	1.5	6.0	0.82	1.2



- Scattering similar to MA957

FCRD-NFA1 APT

Bulk Composition (at.%)

Fe	Cr	W	Ti	Y	O	C	Si	N
82.936	15.047	0.967	0.283	0.090	0.366	0.138	0.076	0.006

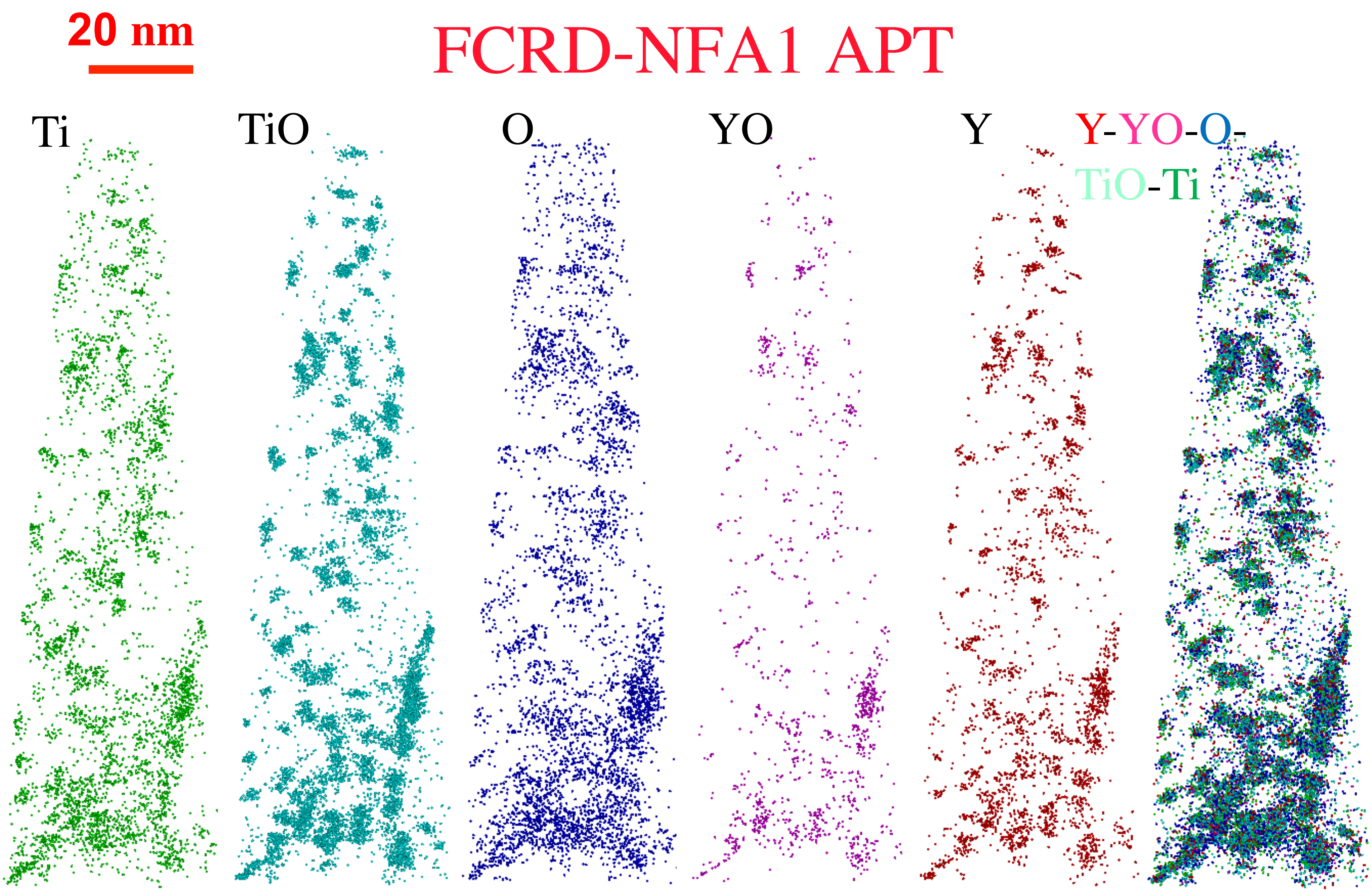
Matrix Composition (at.%)

Fe	Cr	W	Ti	Y	O	C	Si	N
83.573	14.974	0.969	0.056	0.016	0.125	0.127	0.073	0.005

- Again large reduction in Y, Ti, and O between bulk and matrix
- High Cr content >20%
- Similar Y/Ti/O ratio to previous NFAs
- High C content possibly due to sample distance from can

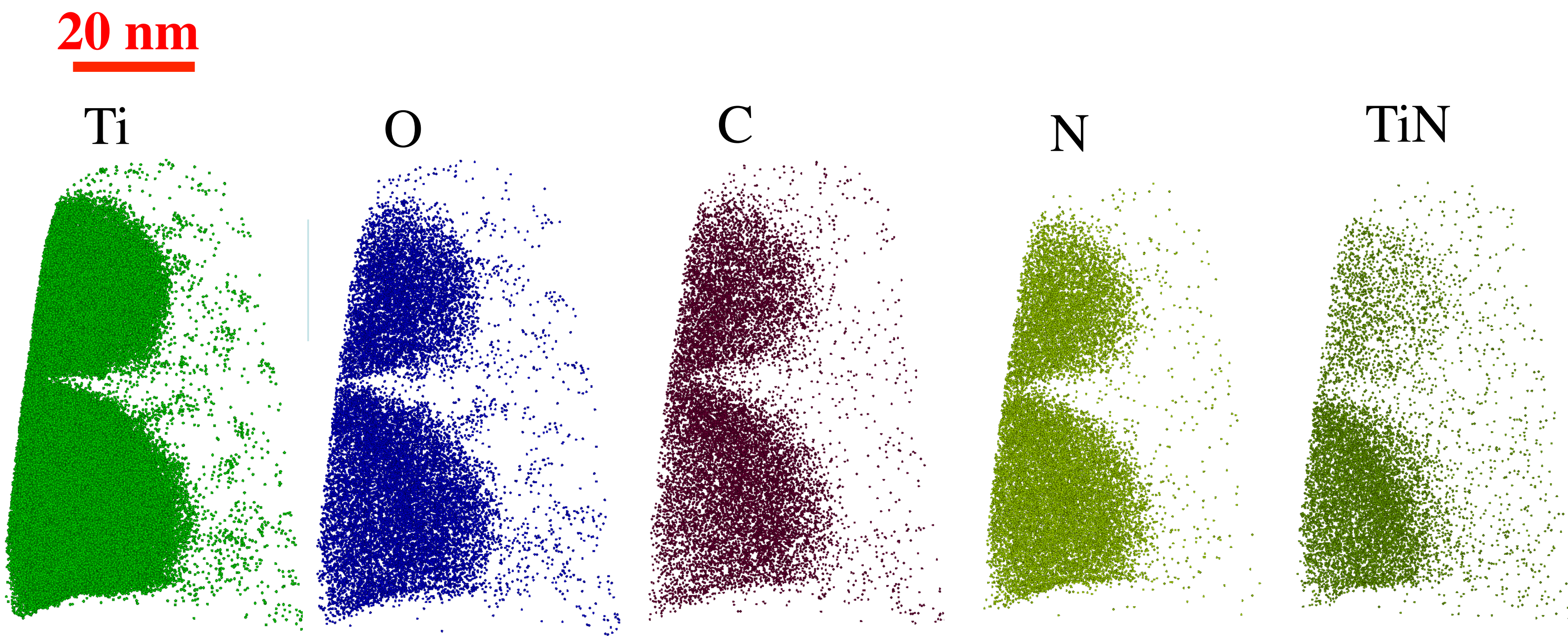
Y/Ti/O	Y/Ti/Cr/O	Number Density ($10^{23}/m^3$)	Diameter (nm)	Solute Fraction (%)
13.7/41.8/44.5	10.5/32.0/23.6/34.0	6.86	2.02±0.78	0.74

FCRD-NFA1 APT



- APT results from single LEAP run

FCRD-NFA1 APT



- Most APT runs showed large Ti, N, C, O precipitates

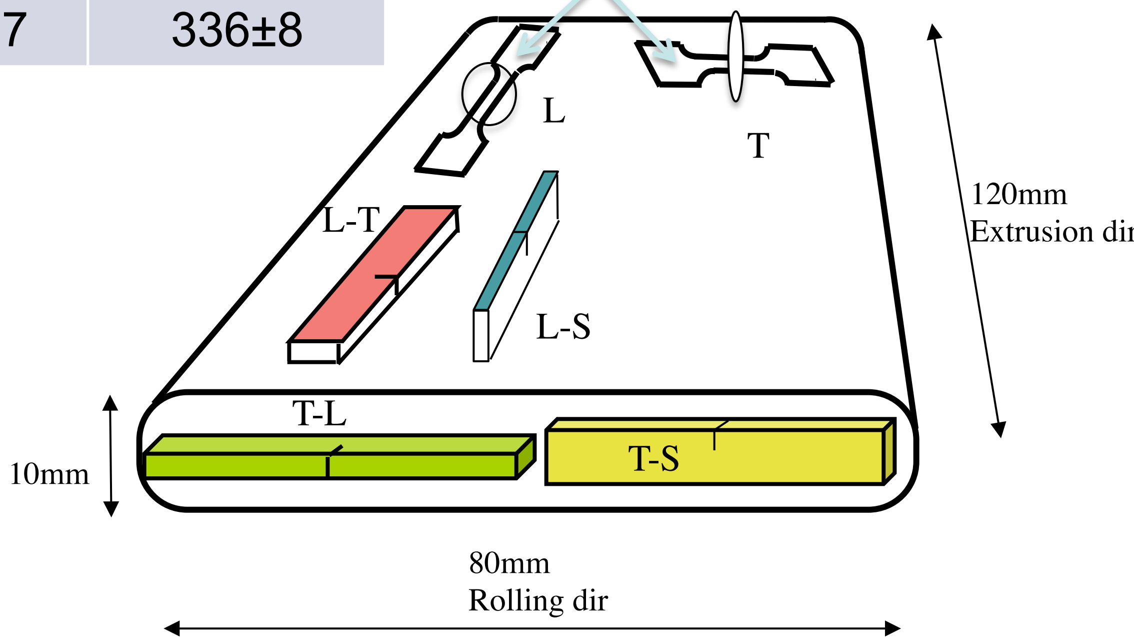
FCRD-NFA1 Results Comparison

Technique	FCRD	PM2	MA957
TEM <d> (nm)	2.1	1.4	2.8
APT <d> (nm)	2.0	2.1	2.7
SANS <d> (nm)	3.0	2.8	2.7
Average (nm)	2.4	2.1	2.7
TEM N (10^{23} m^{-3})	1.6	5.8	2.6
APT N (10^{23} m^{-3})	6.9	11.0	5.1
SANS N (10^{23} m^{-3})	6.0	8.9	9.0
Average (10^{23} m^{-3})	4.8	8.6	5.6
TEM f (%)	0.25	-	0.40
APT f (%)	0.74	-	0.51
SANS f (%)	0.82	1.00	0.90
Average (%)	0.60	1.00	0.60
APT Y/Ti/O	14/42/45	12/43/40	14/46/40
μH	359±18	401±15	336±8
Grain Size (μm)	454	424	1.47 X 0.63

FCRD-NFA1 Mechanical Testing Matrix

Alloy	µH
FCRD	359±18
PM2	401±15
MA957	336±8

SSJ2 and Round SSJ2 tensile specimens



Machined Samples

1/3-1/2 bend bars

T-S: 17
L-S: 20
T-L: 20
L-T: 20

SSJ2 Tensile

T: 36
L: 30

Round Tensile

T: 8
L: 8

Round tensile specimens will be used for high temperature strain-rate jump tests

Summary

- FCRD NFA-1 very similar to PM2
- MP to be characterized but continued presence of larger impurity containing precipitates is worrisome
- The next focus will be on post consolidation thermal mechanical treatment processing, shape fabrication & joining
- Continued progress on many other NFA topics/issues – e.g., quantitative/ physically based long term thermal aging model completed, exploring new alloys and processing routes, interface studies, characterization methods, irradiation experiments …
- The US is continuing to play a leading role in ODS/NFA research but still many unmet challenges