Low Cost Fabrication of ODS Alloys

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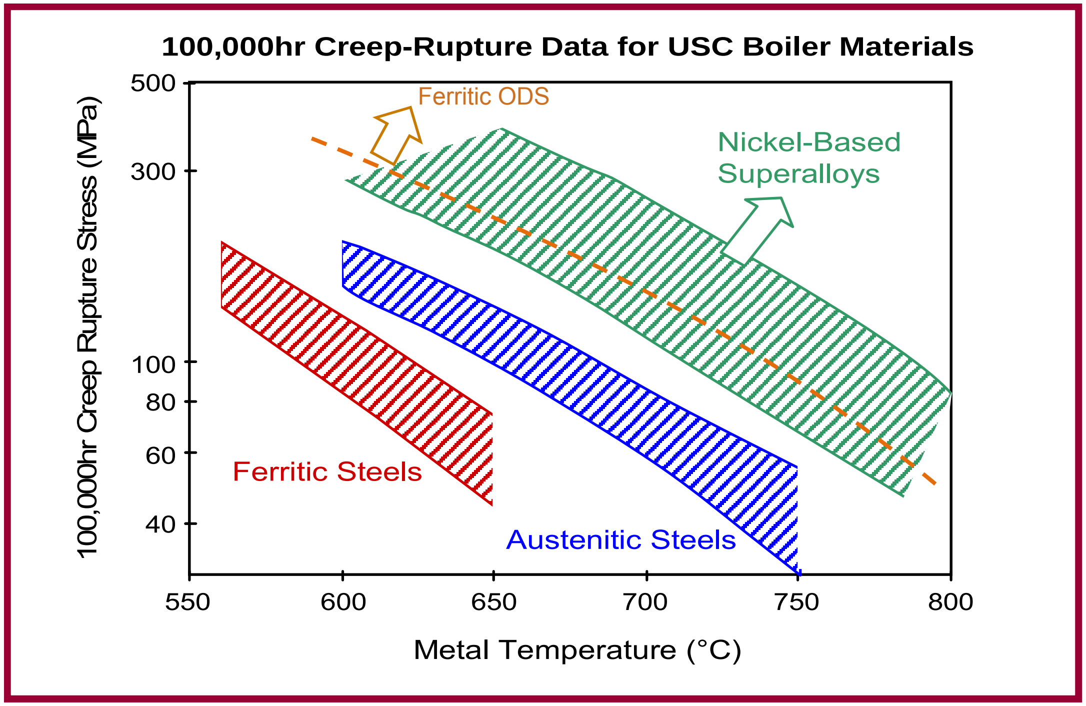
DOE-FE
27th Annual Review Meeting
Fossil Energy Materials

Pittsburgh, PA
Jun 18-20, 2013

NFA / ODS Alloys have excellent performance in both creep and oxidation resistance



From P. J. Maziasz et al., DOE-FE(ARM) 2005 proceedings

So why don't we have a myriad of ODS products ?

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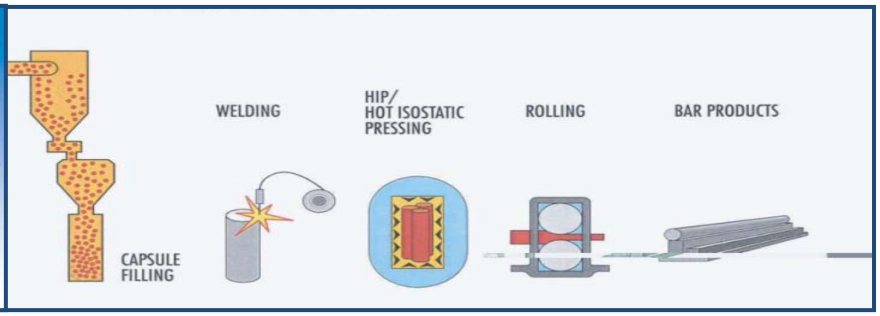
Cost

- **The high cost of ODS alloys and components is driven by the multistep process of fabrication from powder to final product form**
  - **Make the powder in the first place, mix and mill of oxide particle, vacuum canning, densification CIP/HIP, decanning, and processing to semi-finished form (extrusion or rolling), machine or roll to tube, then heat treat for microstructure**
  - **Batch Process**
  - **Machining operations produce significant waste. Many ODS materials produced in the past for pipe or clad applications are extruded and then gun drilled.**



Also affecting cost: fabrication processes

ODS alloys can be hard to form, bend, pierce, draw, pilger due to anisotropy and in some alloys low RT ductility

Cost estimates for current processing route

Front End (Powder processing): $10.00/lb to $50.00/lb
Back End (Consolidation): $30 to $80.00/lb,

Traditional ODS materials prepared by MA routes can be $60.00/lb to $150.00/lb and wrought, semi-finished products can be $200 to $400 per lb (plate / tube /pipe)

Are there alternative process routes that can remove the some of the cost when going from powder to semi-finished product ?

Friction Consolidation : Process description

1

Powder is loaded directly into cylindrical hole in can

2

Spinning tool is inserted in top half of split can and a downward axial force is applied while spinning

Process description

3

Heat is generated initially by friction between particles, but as the powder consolidates the heat is from plastic work energy dissipation

The energy released from plastic work results in significant heating, up to **700 to 900°C**. The heat and strain energy imparted to the powders causes them to fully densify and flow within the reservoir in a complex way dictated by the design of features on the face of the tool.

Process description

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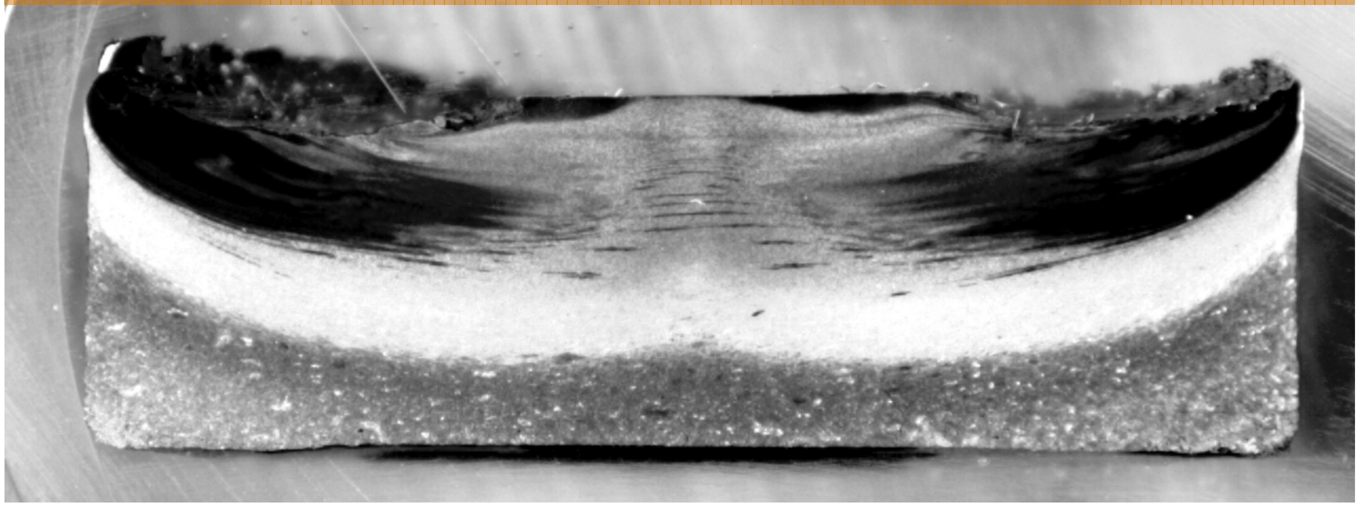
Dense compacts result



- During the plastic flow event the metal is in a state of continuous dynamic recrystallization, which can result in a wide range of microstructures and final grain sizes depending on cooling rate and chemistry

- Very high levels of total strain produce extremely good mixing of constituents and potentially diffusion rates high enough for good oxide mobility (dissolution?) and redistribution to form nano-clusters

Challenges – Process Control

FCE: What are the major process variables?

- **Die RPM** : friction between die face and the underlying material causes heat generation. Further enhanced by deformation. In general, higher RPM leads to increased temperature
- **Forging load** : increase in forging load results in higher temperature. Amount of strain (or depth of shearing can increase). May induce different levels of texture.
- **Die plunge speed** : faster die feed rates arrive at peak temperatures quicker and can reach hydrostatic loads in the early part of the deformation process
- **Die face feature** : a scroll feature on die face promotes strong flow of material at the die face but can create “turbulent” regions with inconsistent grain orientation
- **Boundary Conditions:** active cooling systems control cooling rate after the cessation of dynamic recrystallization and can help minimize grain growth, control of oxidation

FCE is a thermo-mechanical process. We can have control over the microstructure, and hence on the properties, by adjusting process variables.

Scroll feature on die tool face

Objective

- Demonstrate a low-cost method of fabricating wrought ODS ferritic billet, rod, and tube directly from oxide-doped stainless steel powder, thereby eliminating costly, batch-based MA process, and can/HIP/extrude densification steps
- Approach
  - Develop the process control, and equipment to produce fully compacted billets from metallic powder feedstocks
  - Produce lab-scale densified compacts , then, with new die designs, produce rod and tube product forms without intermediate steps such as powder canning, HIPing, and rolling or extrusion.
  - In evaluating the efficacy of the process, our initial focus will be to:
    - verify that high density (i.e. pore-free) billet and rod forms can be fabricated by this approach,
    - demonstrate that the oxide dispersoids are nanoscale (<20 nm in size) and uniformly distributed throughout the steel matrix
    - the mechanical properties (creep and strength at temperature) approximate those of the current ODS alloys being evaluated for FE applications
    - investigate scale-up issues

Materials

Starting powders

Mechanically Alloyed Powder

- Eliminates majority of "back end" cost of canning, HIPing, extruding, but still is moderate cost and time in the front end step (the MA step)

Gas Atomized Powder

- Reduces cost of "front end" MA step, but may have low yield depending on if distribution of yttria in final powder product is dependent on particle size

Steel Powder + Y_2O_3

- Further reduces cost of "front end". If the primary "mixing" occurs in the Friction Consolidation process, then the distribution of Yttria in starting powder may not be as important

Materials

- Chemical composition of the different materials

	Mechanically Alloyed Powder	Gas Atomized Powder	Steel Powder + Y_2O_3
	Special Metals	*Sandvik Osprey*	*ATI Powder Metals*
	MA956	*Fe22Cr5AlYZr*	*Custom*
Fe	Bal	Bal	Bal
Cr	19.64	22.4	18.6
Al	4.87	6	4.94
Ti	0.39	-	0.5
Y_2O_3	0.5	-	0.5
Y	-	0.07	-
Zr	-	0.42	-
Oxygen	0.25	-	-
Si	0.07	0.21	-
Mn	0.09	0.2	0.04
Ni	0.06	-	-
N	0.031	-	-
C	0.02	-	0.02
Cu	0.02	-	-
Co	0.01	-	-
S	0.007	-	0.01
P	0.006	-	-

- The mechanically alloyed powder (MA956) and the steel powder + Y_2O_3 have virtually the same global chemical composition.
- The gas atomized powder has lower amount of Y, has Zr and Si.
- The evaluation of the effects of the process is not straight forward.

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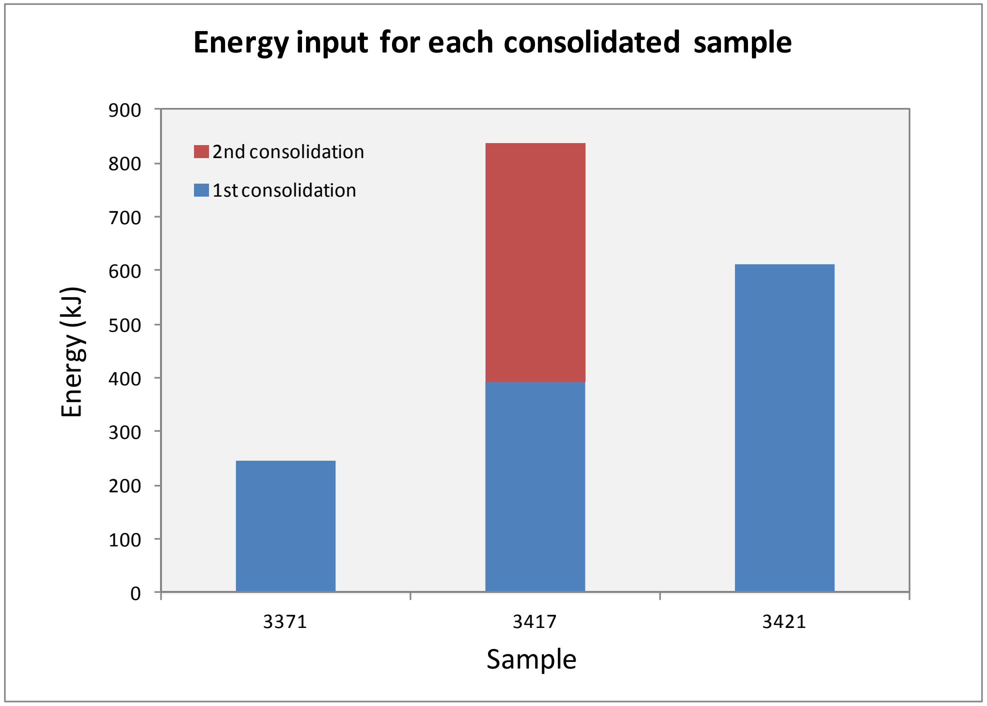
Processing conditions

Run	RPM	Force (lbf)	Comment
3371	500	5000	Smooth faced die
3417	500	5000	Double side consolidation, 0.16”/rev scroll
3421	300	10000	0.3”/rev scroll

Tool features



Mechanically alloyed powder route

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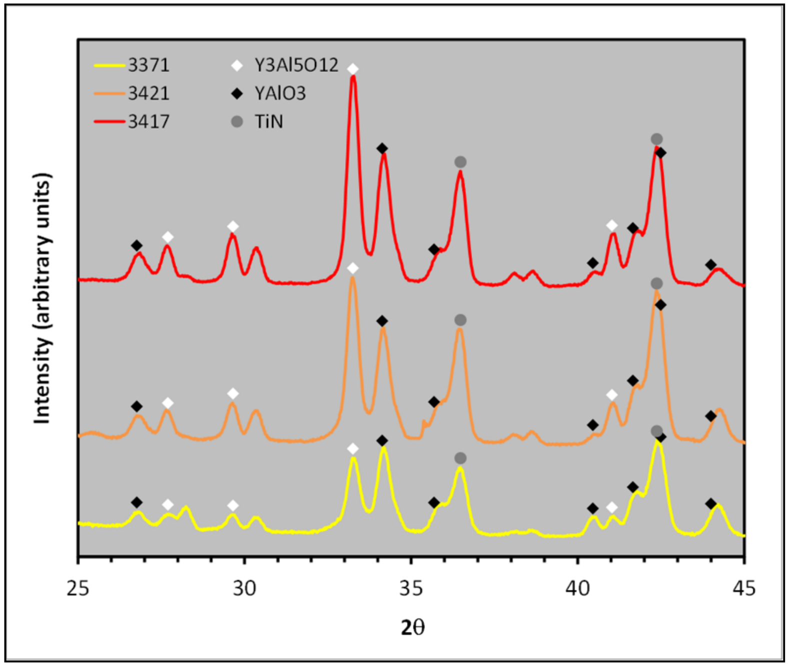
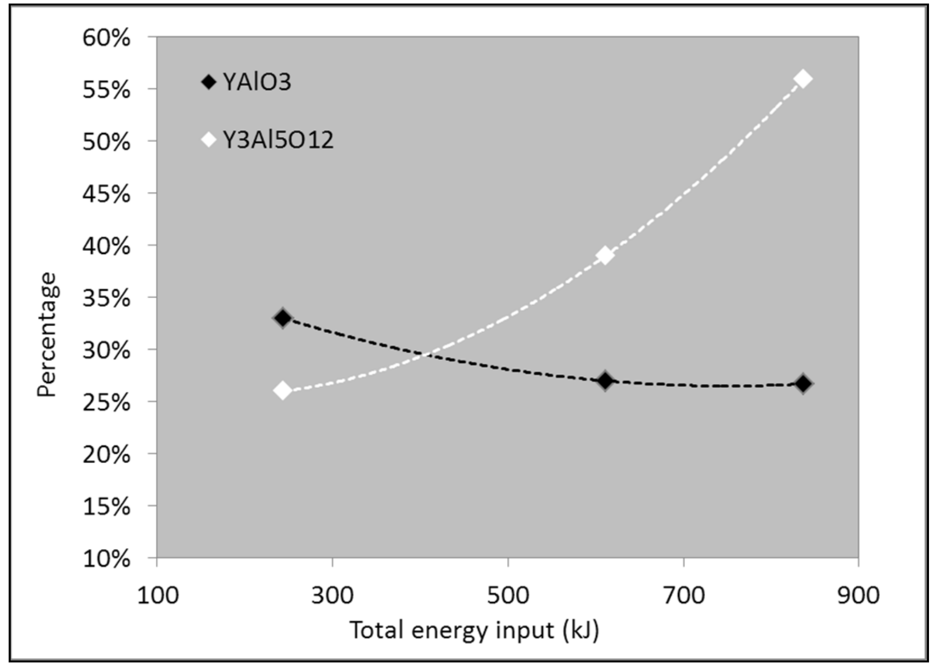




XRD made on extracted precipitates. Sample was representative of the whole. Membrane filter size, 20nm

As energy input increases

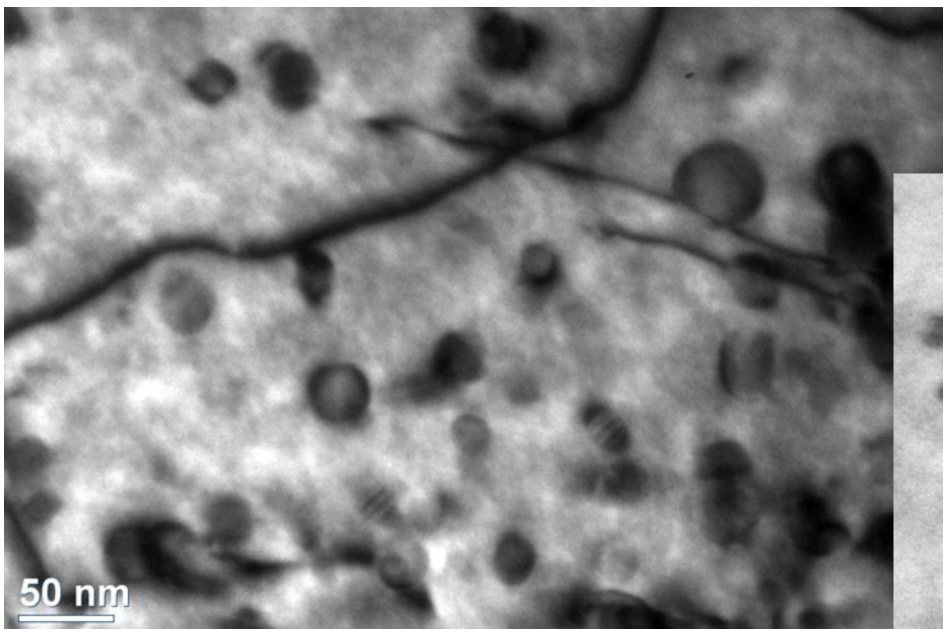
$YAlO_3$ (Perovskite-YAP) → $Y_3Al_5O_{12}$ (Garnet-YAG)

Quantitative data was obtained from XRD experiments by the RIR method. See relative intensity of main peaks as well.

Amount of each phase is dependent of processing conditions.
Microstructure could be optimized

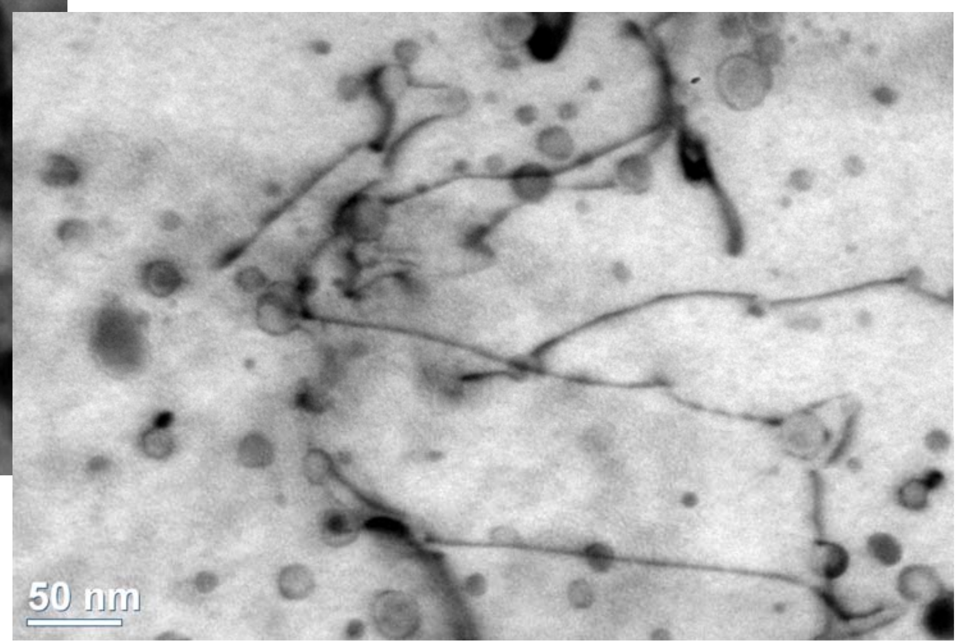
Previous TEM results

- Coarser oxides in the compacts are YAP and YAG.
- What about the fine grain parts of the compacts with relatively few particles? Still struggling to identify the small Al-Y-O clusters

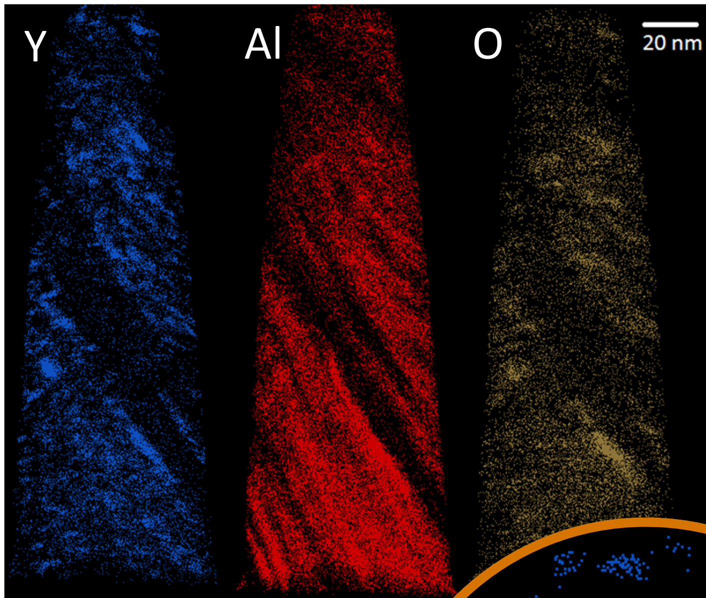


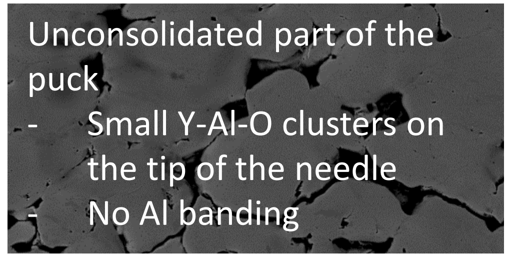
MA 956 RL

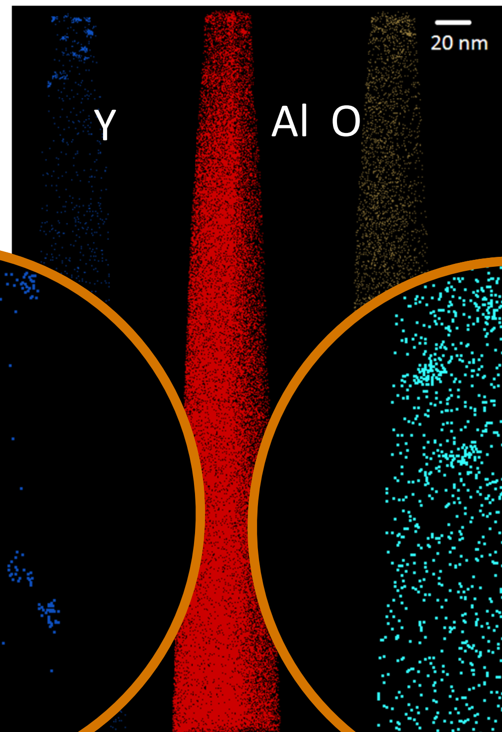
Friction Consolidation

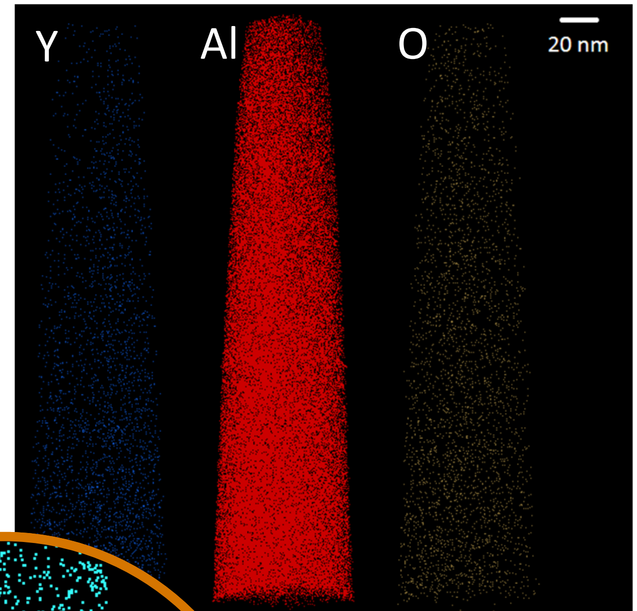


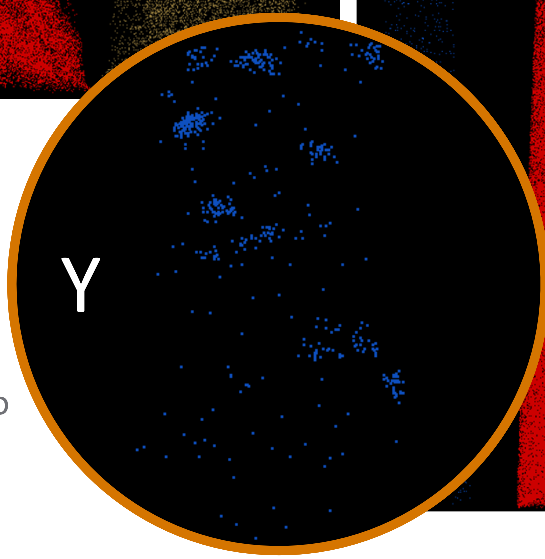
Atom Probe Tomography (APT) of Friction Consolidated MA956

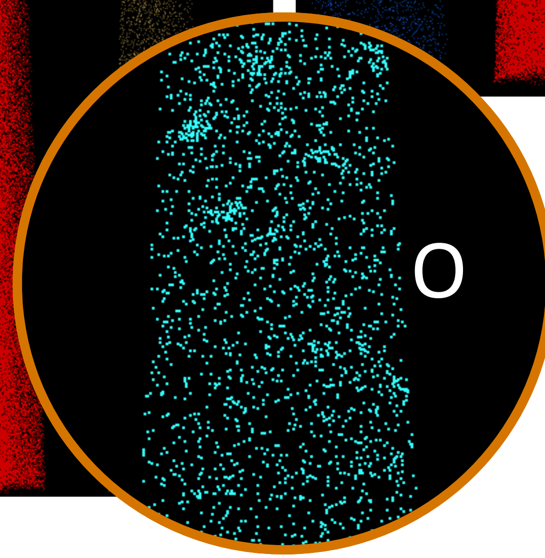












Powder before consolidation
- Y is present in clusters
- Al shows banding
- O also correlates to some Y locations

Consolidated Puck
- Y is very finely divided
- Al is homegeneously distributed
- O is very finely divided

Summary of MA work

- Small particles are precipitated from the MA powders when densified that are Y-Al-O compounds
- Particle composition of the >50nm particles trend from the perovskite to the garnet phase with increasing energy input into the puck
- < 10 nm particles are identified by TEM
- Creep testing is the macroscopic way to tell if you have the correct dispersoids!

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Steel Powder + Y_2O_3

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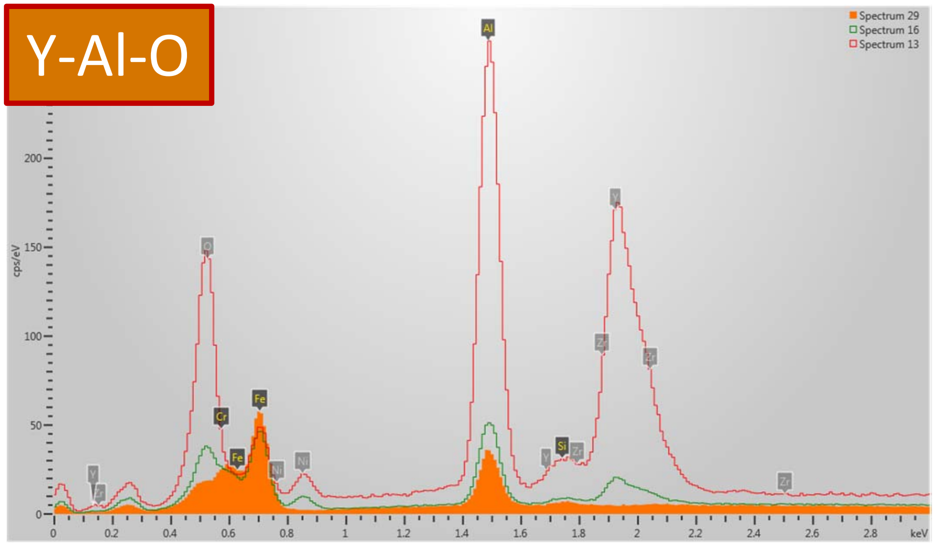
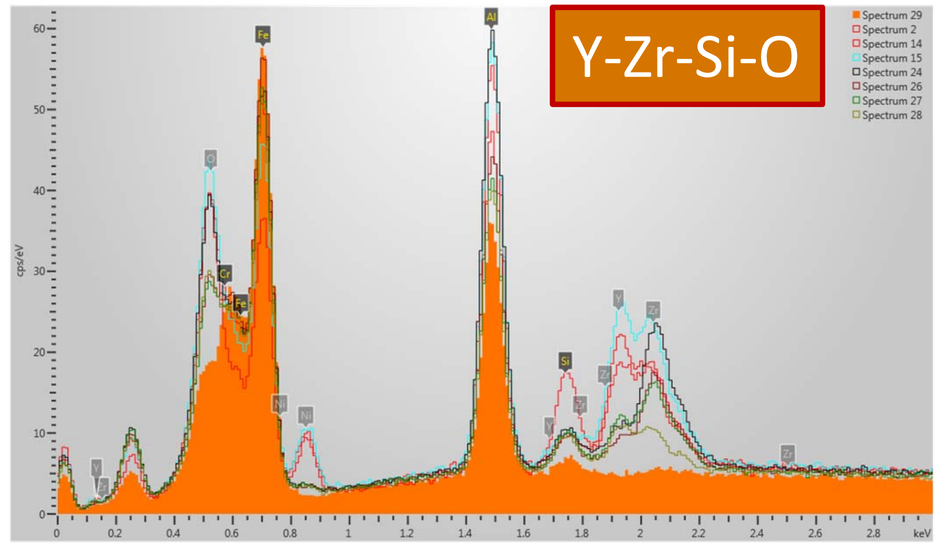
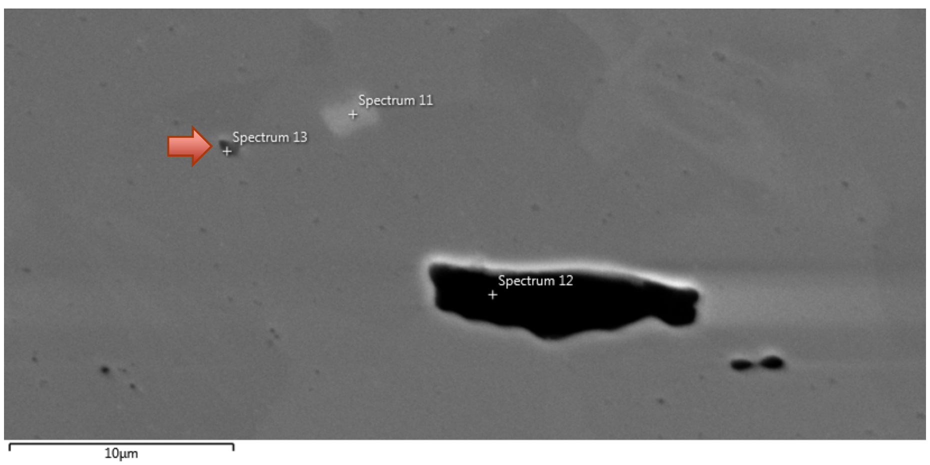
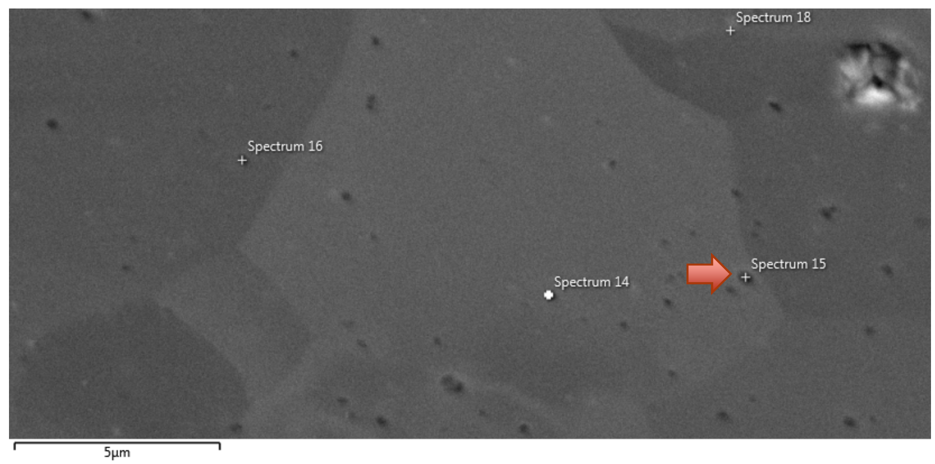
SEM and EDS of Friction Consolidated powder









SEM and EDS of Friction Consolidated powder

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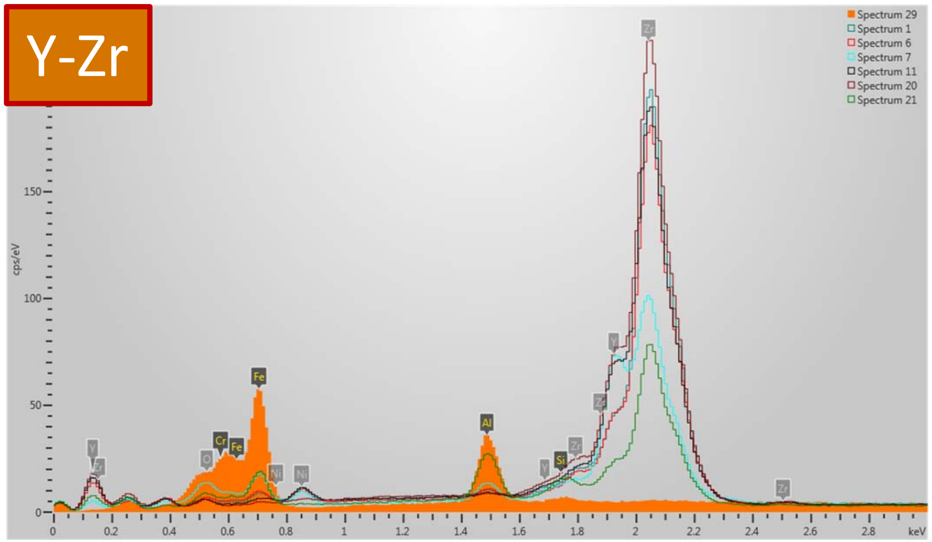

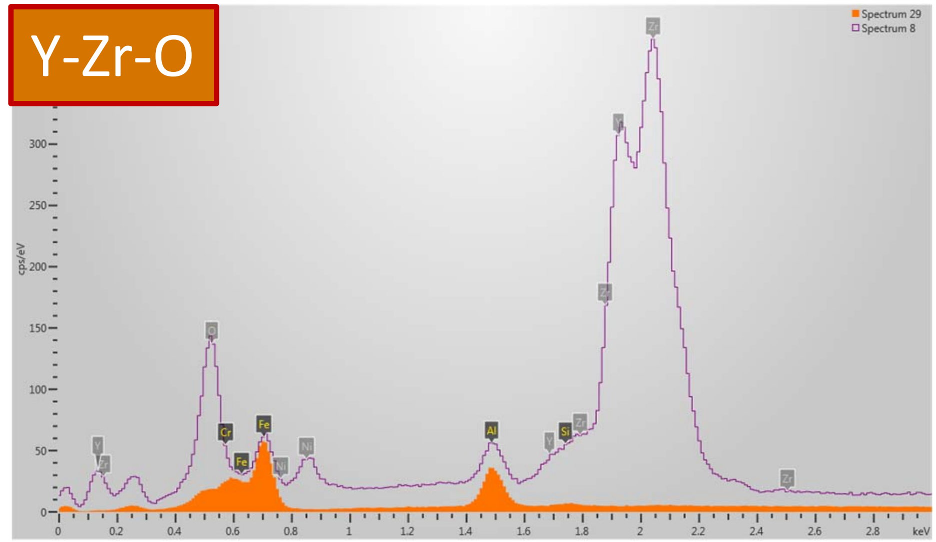

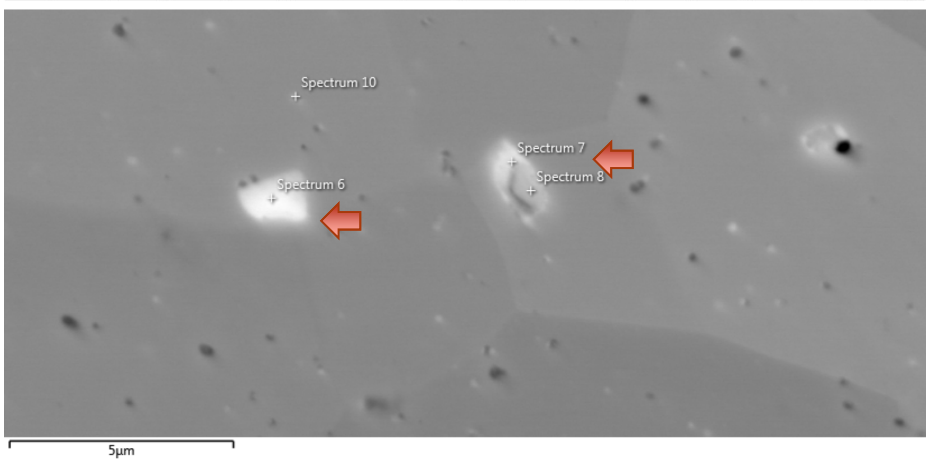

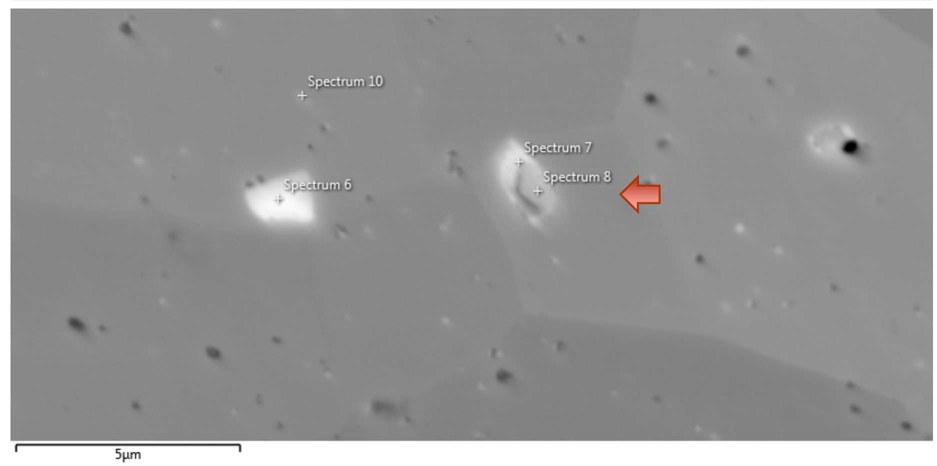

Materials

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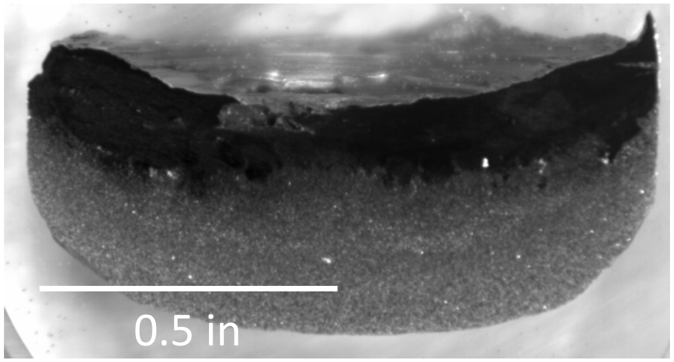
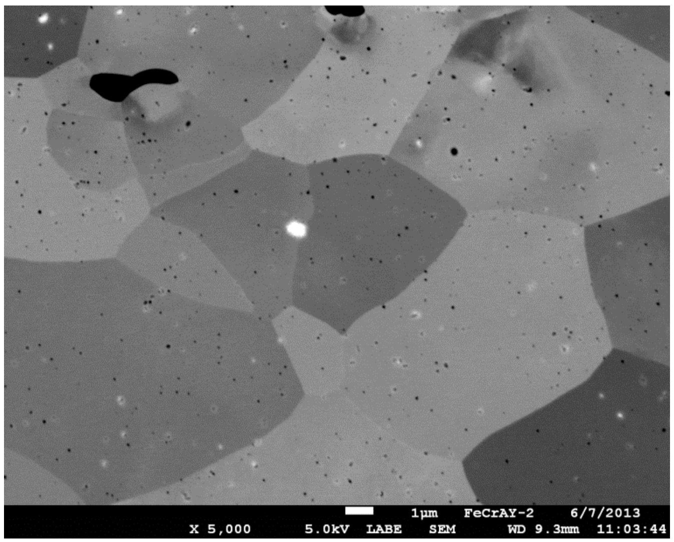
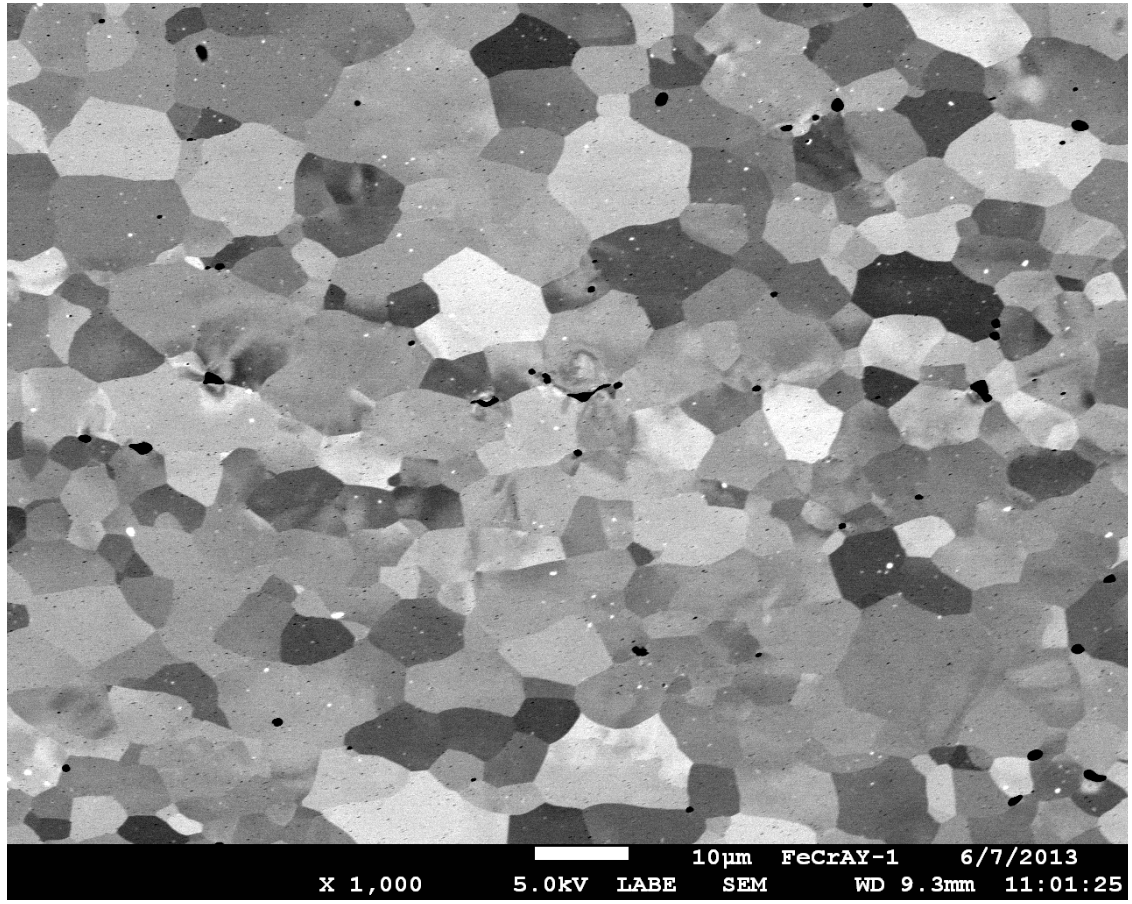


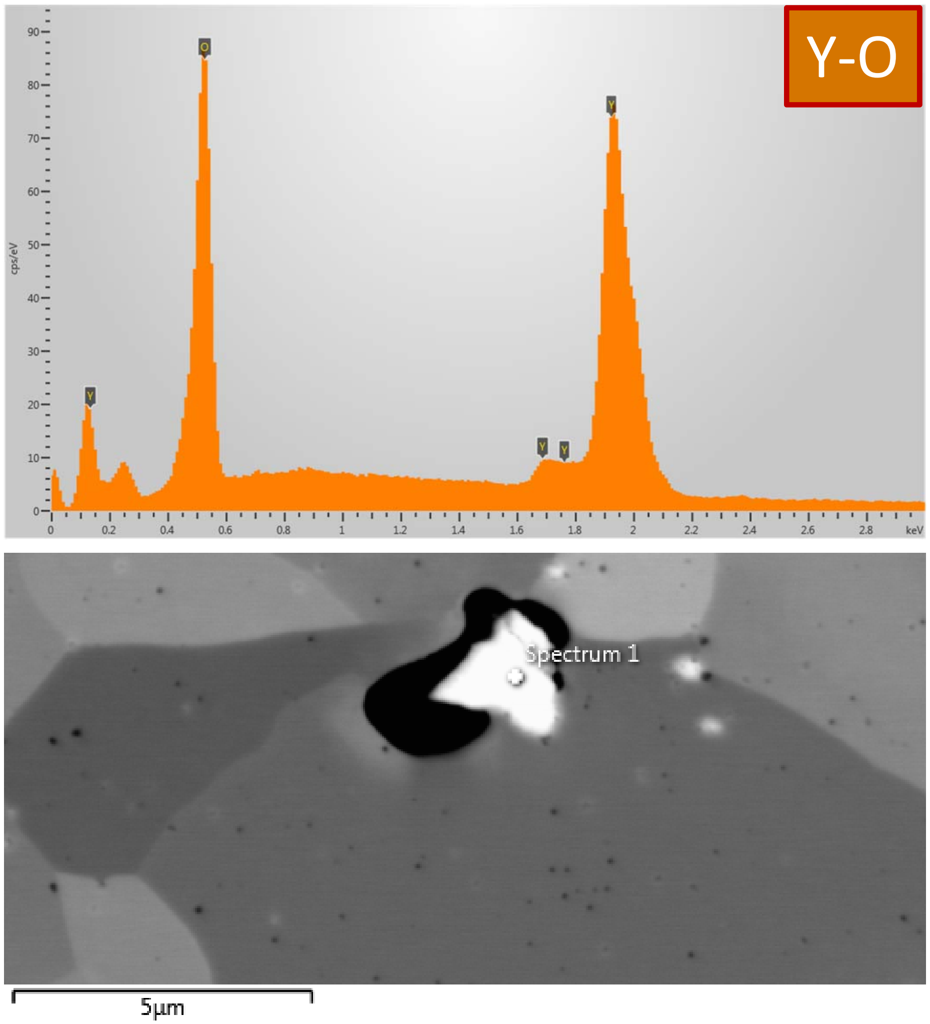


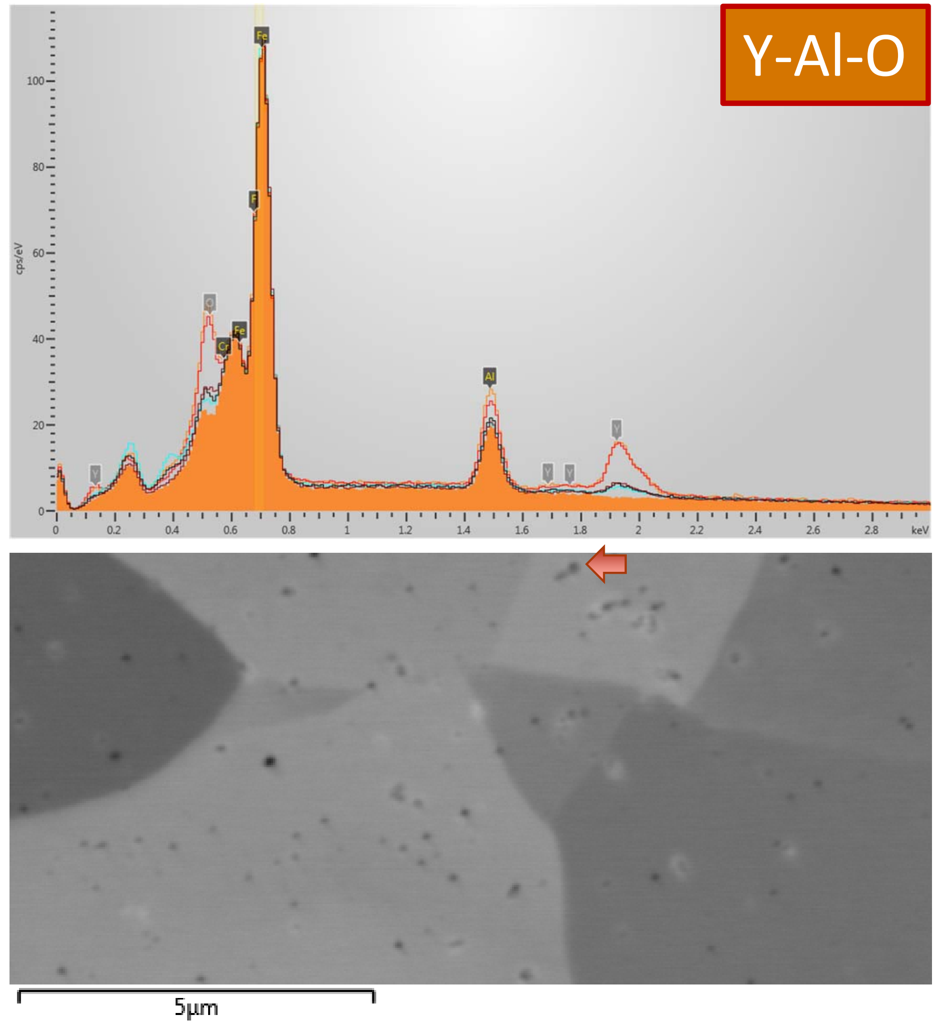


- Equiaxed grain structure
- Bright particles and dark precipitates can be observed
- EDS analysis showed four different kinds of particles

Y-O | Y-Al-O
Al-O | Si-O

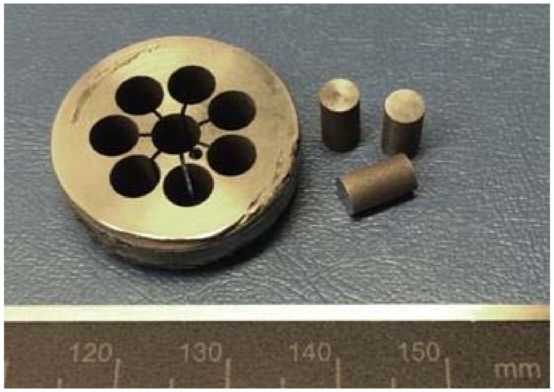
SEM and EDS of Friction Consolidated powder





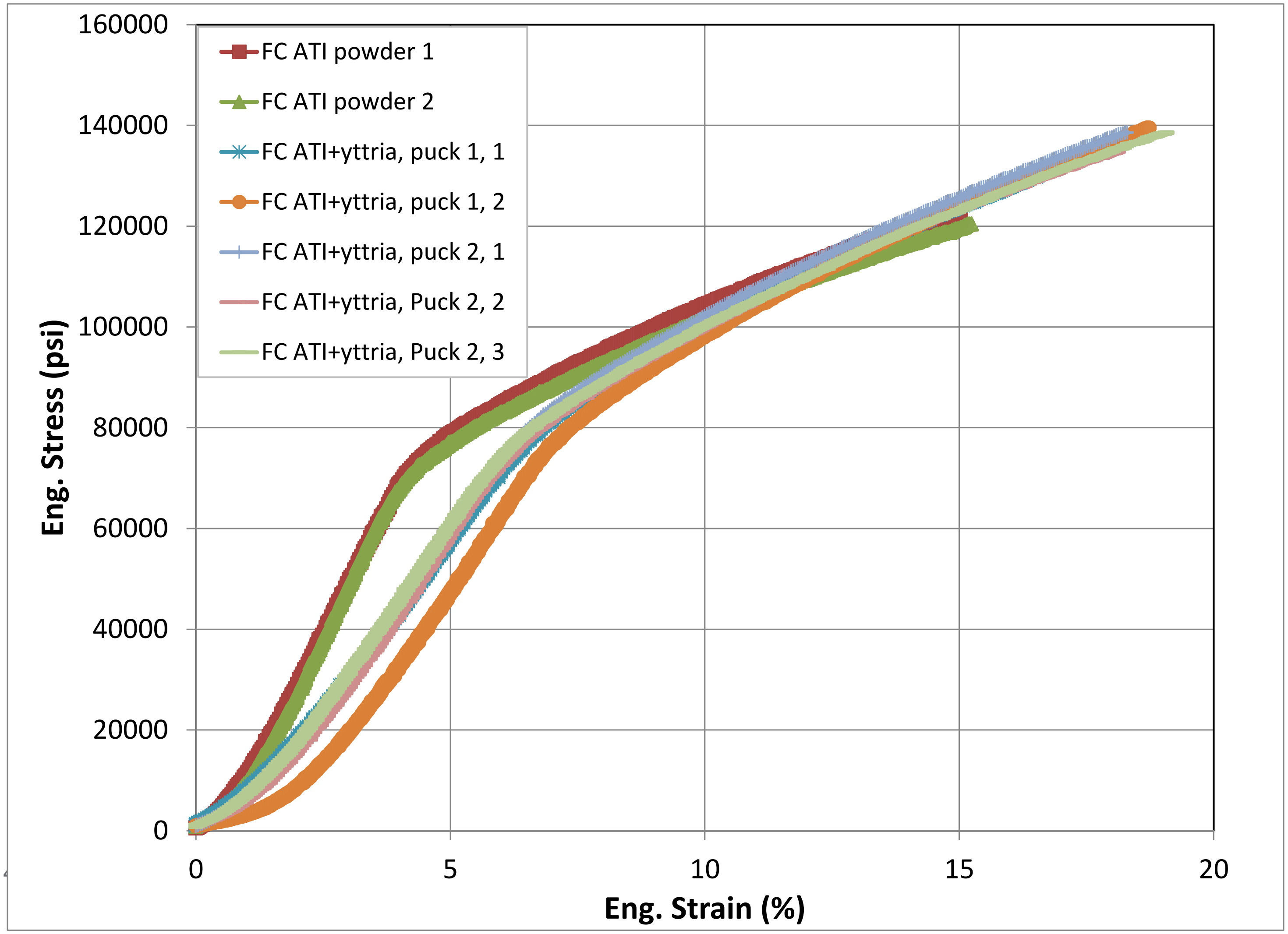
Mechanical evaluation of friction consolidated pucks

- Steel powder and steel + yttria consolidated into pucks at 300RPM, 12,500lbs, to 1000C
- Compression samples 4mm x 7mm EDM machined from pucks
- Compression testing with IN718 tension to compression fixture and following ASTM E9 – 09
- Used Ni antiseize on the top and bottom load pads – no significant barreling



Mechanical evaluation of friction consolidated pucks

- Room temperature compression, results uncorrected for machine compliance



Compressive Yield Strength of Friction Consolidated Pucks

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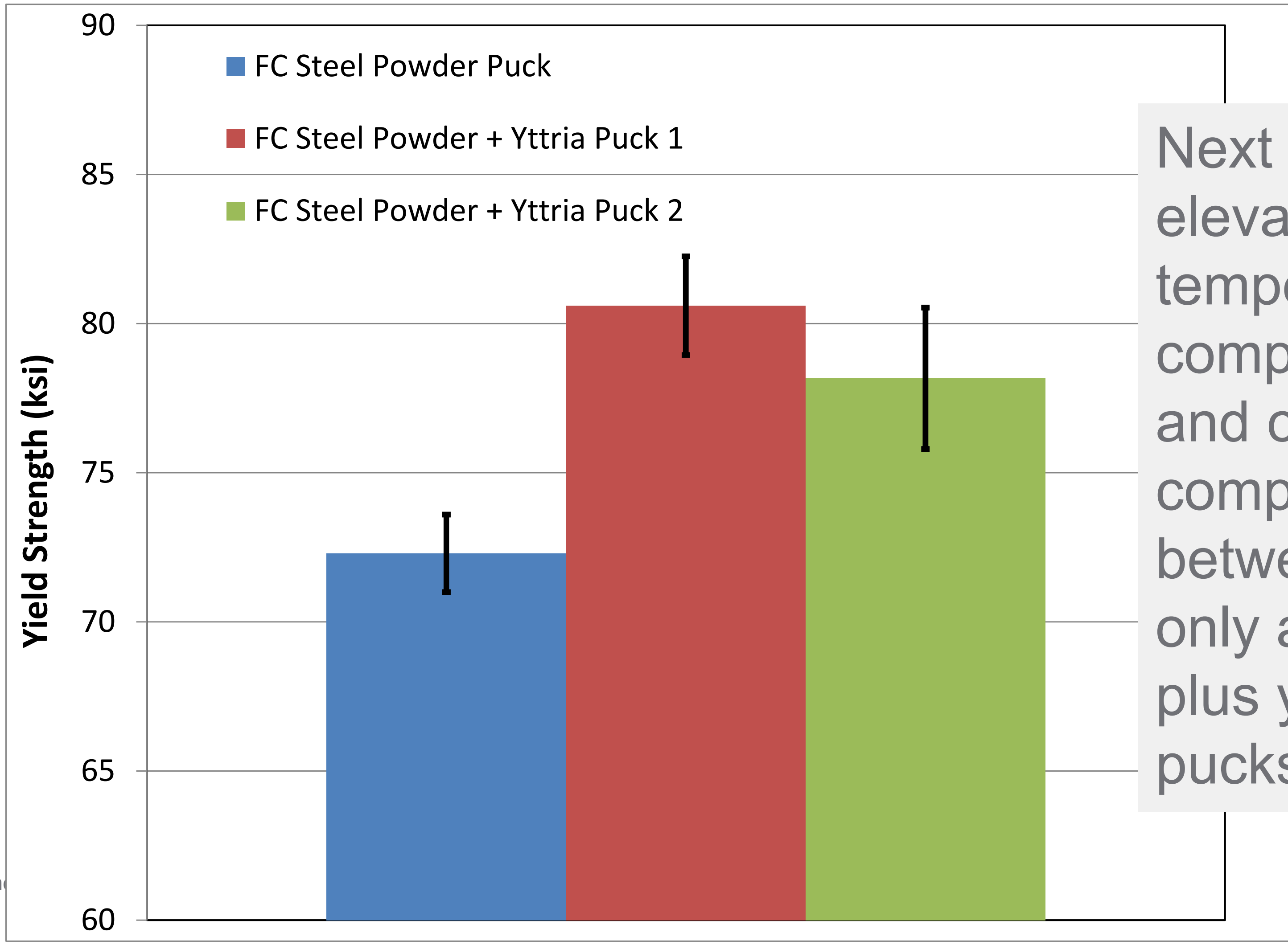
- Addition of 0.5wt% yttria increases the yield strength by 6-8ksi at RT



Next tests: elevated temperature compression and creep comparison between steel only and steel plus yttria pucks

Summary of steel plus yttria work

- Yttria did dissolve and react during the Friction Consolidation Process
- Some small yttria particles in the matrix still exist, but new phases are formed between Y-Al-O.
- Small Y-Al-O are distributed primarily intergranular and are very evenly dispersed.
- Dispersed particles are not broken down large Y_2O_3 particles, but reacted particles formed from a dissolution and reprecipitation process
- Question of sub 10nm dispersoids is still open
- R.T. yield strength similar to MA956
- Addition of yttria increases R.T. yield strength by 6-8ksi over the same steel powder without yttria

Process Development Review

We have demonstrated that FC&E can:

- fully densifies MA, gas atomized and ss+Y powders to crystalline solids with complex and process parameter dependent microstructures
- Sub 10nm dispersoids were observed in the FC processed MA compacts where no dispersoids were originally present.
- Al-Y-O phases developed are process parameter dependent, especially formation of YAP and YAG in the solid allowing for tailoring of the Al, Y, or O available to form nano YAM (the dispersoid of interest)
- Can recrystallize and refine the microstructure
- Can create nanoscale Y-O and Y-Al-O dispersions from coarse precursors

Challenges

- Process control
  - Temperature, management of the flow in the shearing solid, cooling rate, oxidation, machine limitations form torque and force control when die and can are fully engaged
- Homogeneity of properties
- Homogeneity of the microstructure in the puck
  - need to develop the extrusion process

Rod extrusion

Example plunger rod with hole in center

Depending on the geometry and dimensions of the die; billets, rods and potentially back-extruded tube can be produced by this process directly from powders.

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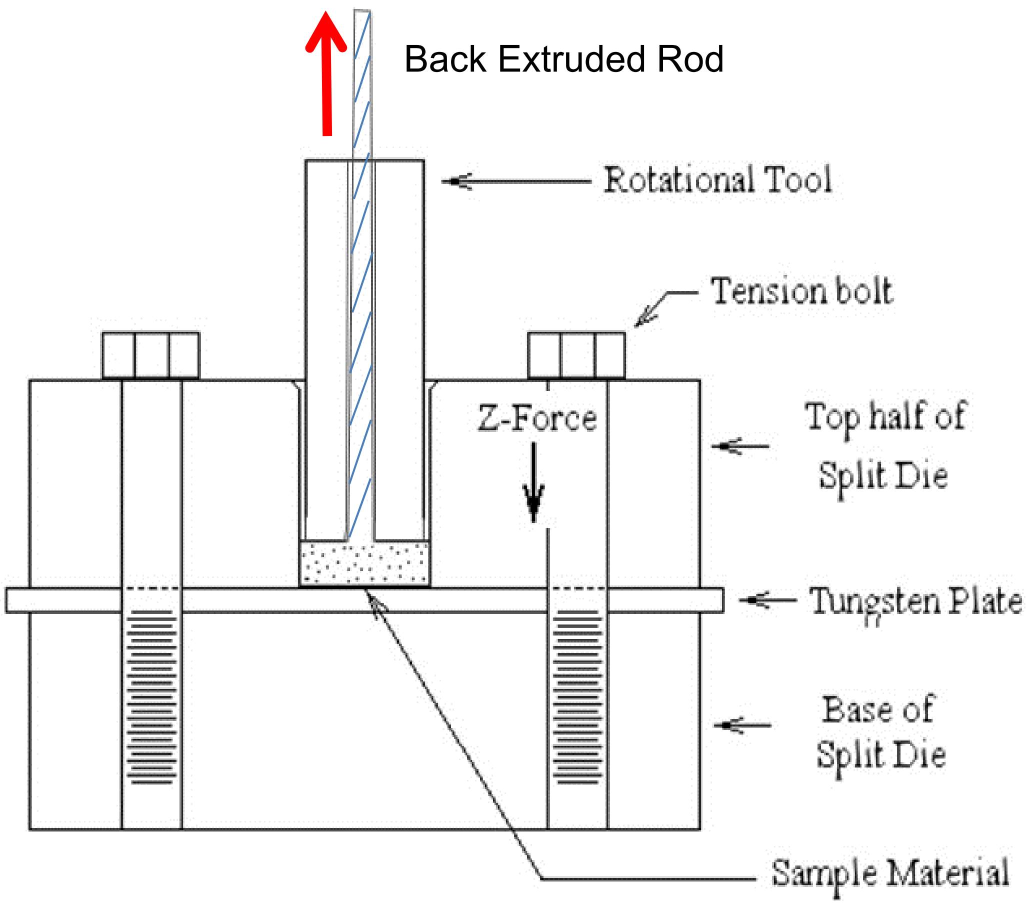

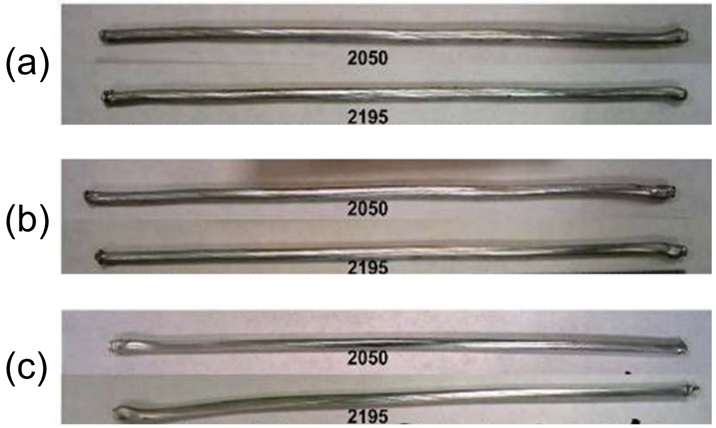

Solid Al rod fabricated directly from powder via a friction stir rod extrusion process. 2050 and 2195 rod extruded at: (a) 150, (b) 200, and (c) 250 rpm rotational speed.

A. Reynolds, USC, 2008

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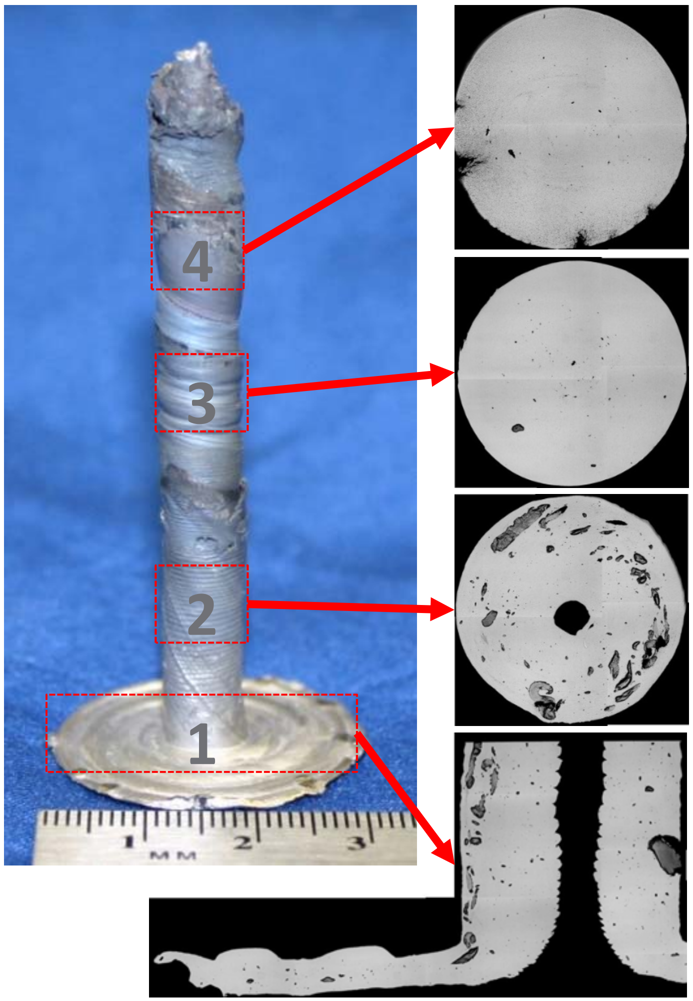
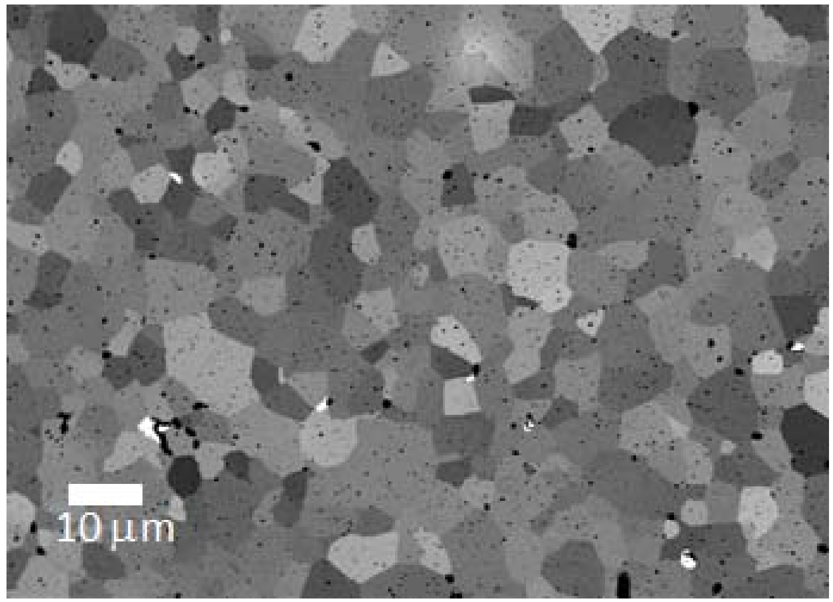
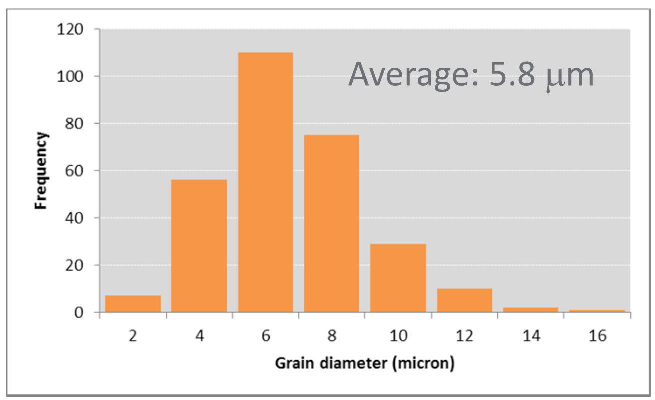
SEM and EDS of Friction Consolidated and Extruded MA956





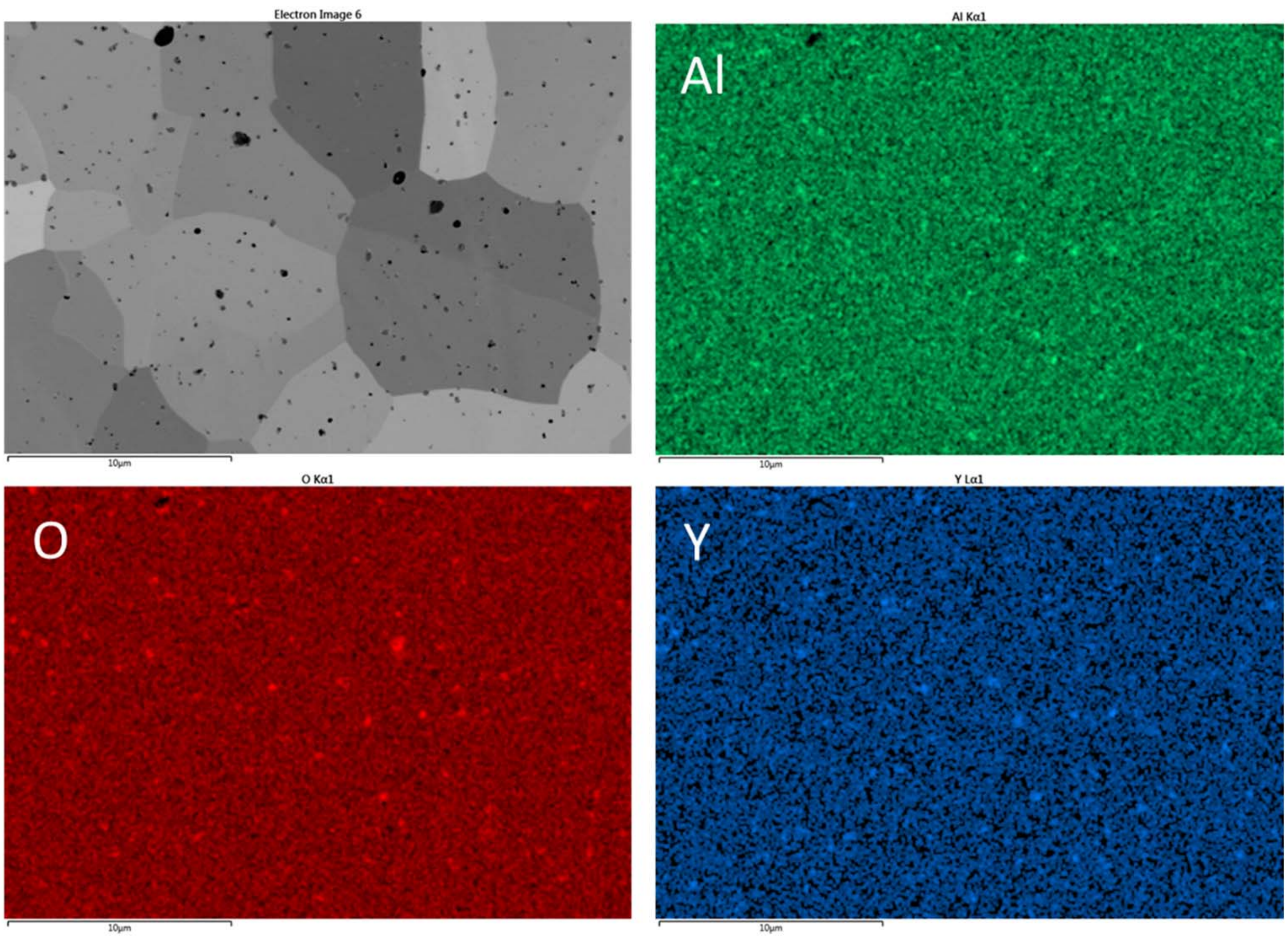
Tool used for extrusion, with a hole in the center.



- Equiaxed grain structure
- Grain are NOT elongated in the longitudinal direction.

SEM and EDS of Friction Consolidated and Extruded MA956

EDS mapping reveals the presence of Y-Al-O



SEM and EDS of Friction Consolidated and Extruded MA956 and 14YWT

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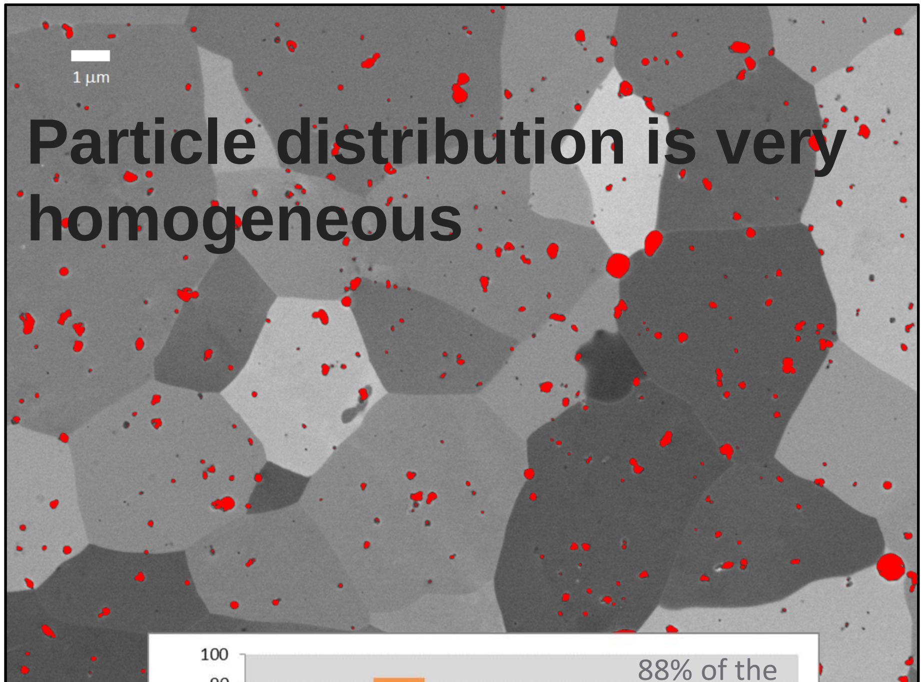
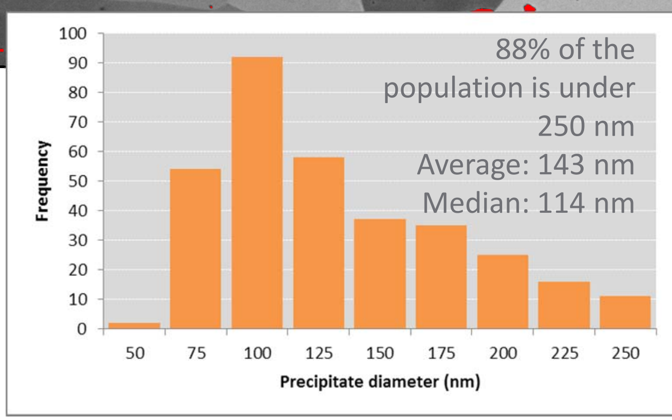
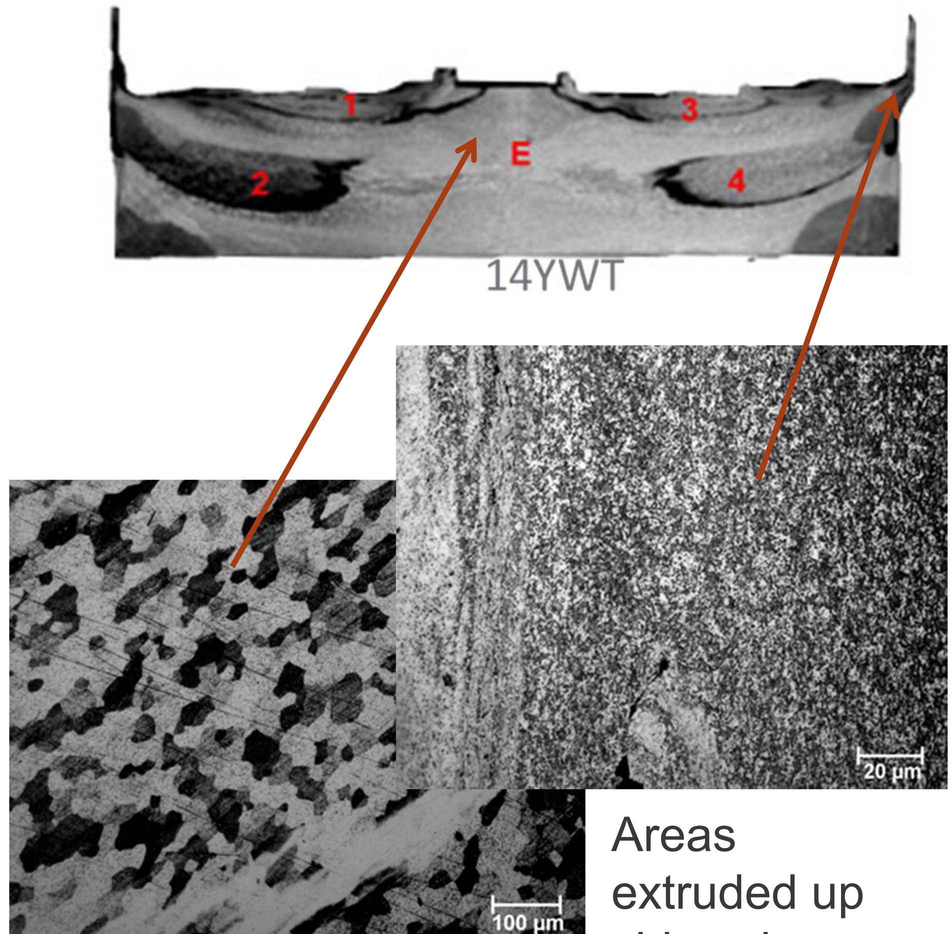


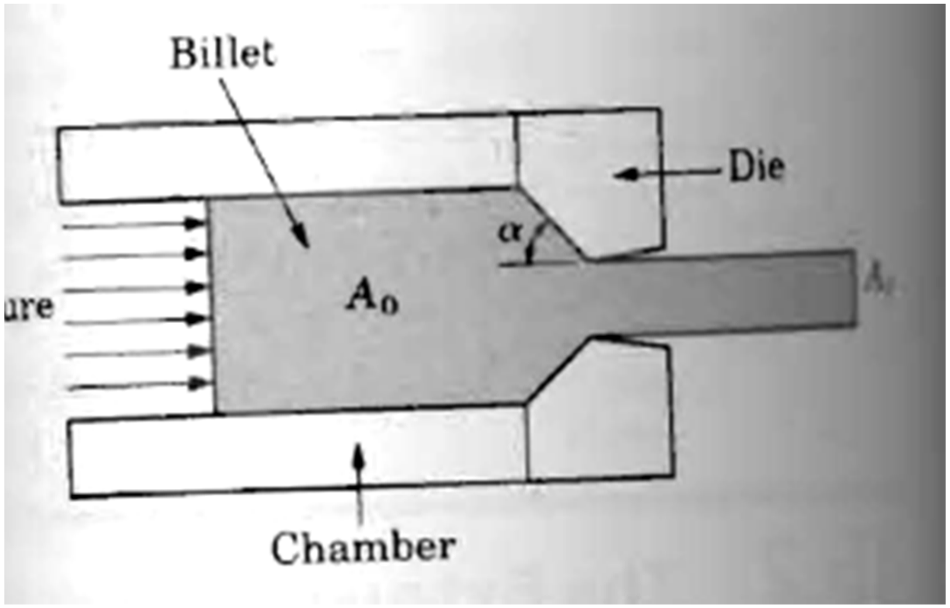


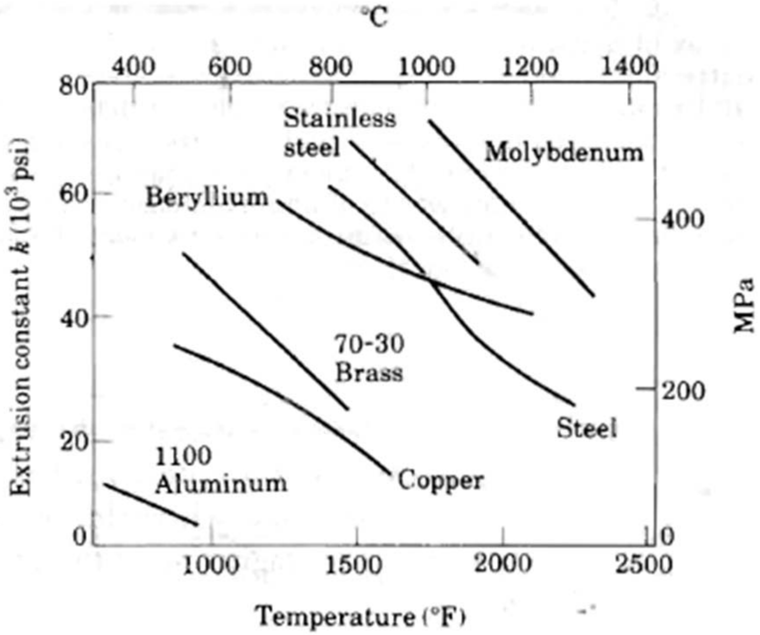


Areas extruded up sides show sub micron grain size

friction-extrusion vs. normal extrusion

- Do normal extrusion guidelines apply? No
- Ram loads are 1/10 that estimated from conventional extrusion
- This is extremely good for scale up considerations



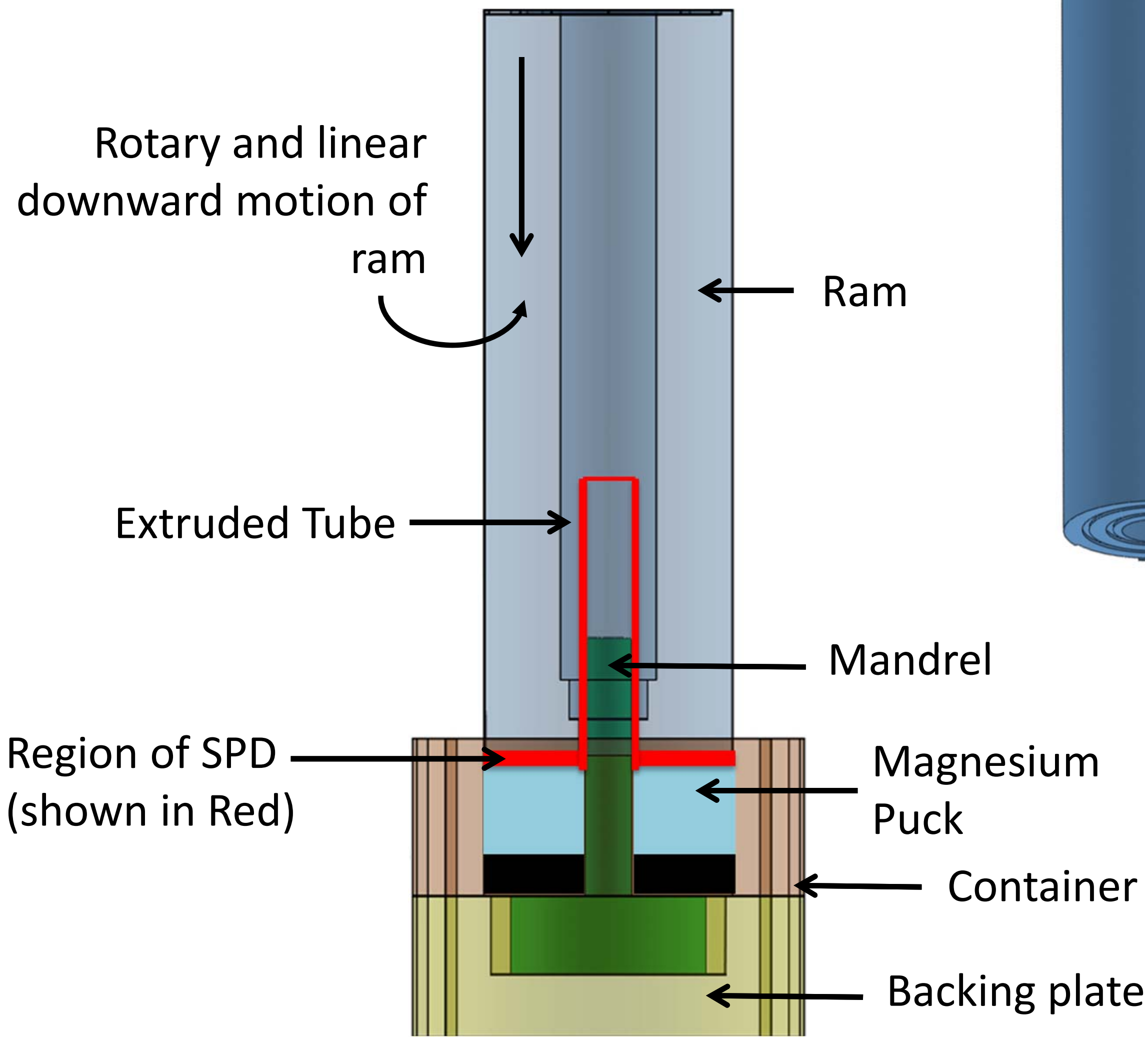


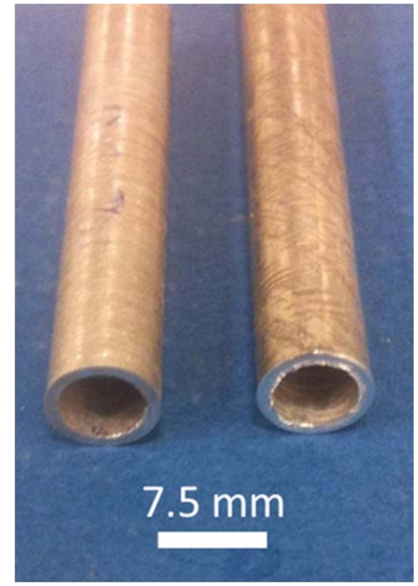
F = Ao*k*ln(Re)

Re = Ao/Af

K = extrusion constant

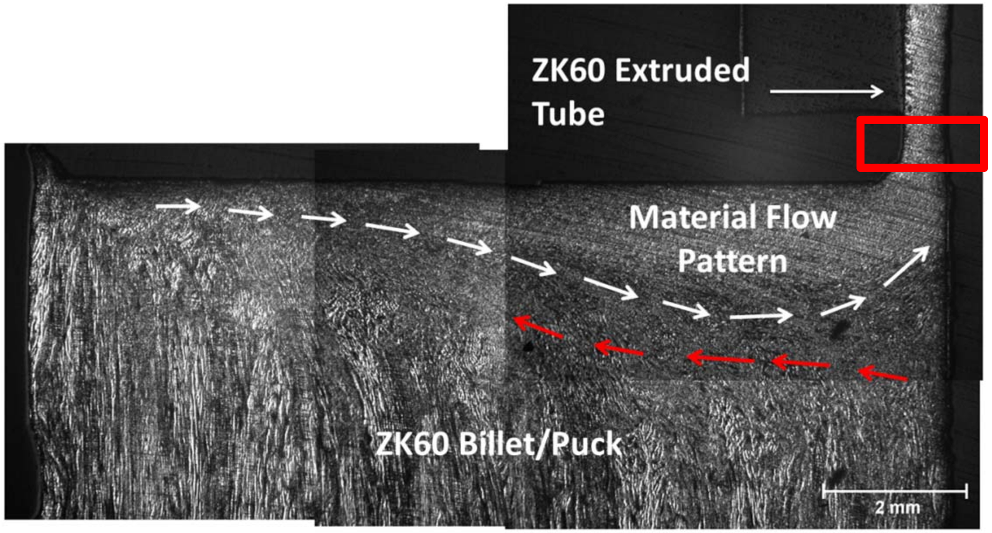
Shear Assisted Indirect Extrusion Process
Evidence of efficacy from Magnesium trials

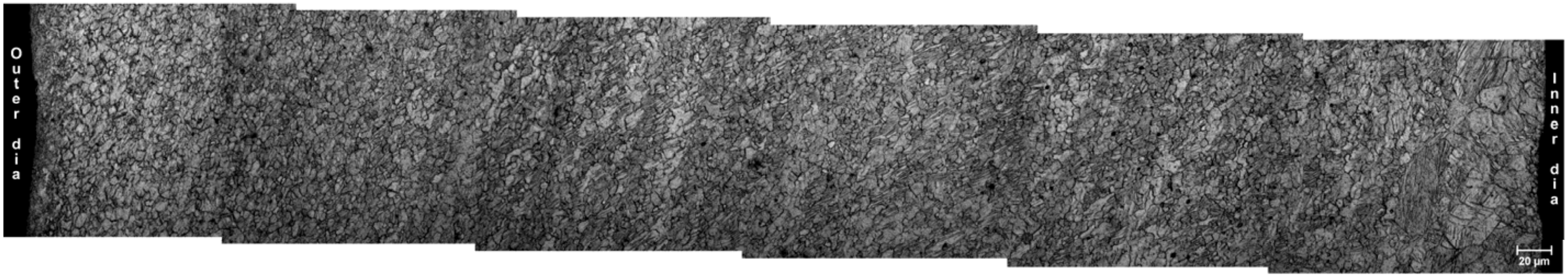




Flute/ Scroll profile on the Ram

Microstructural Characterization of the Extrusion-ZK60





Tube cross-section montage, near tool mandrel, at 500 X

Promise from the Processing Side ZK60

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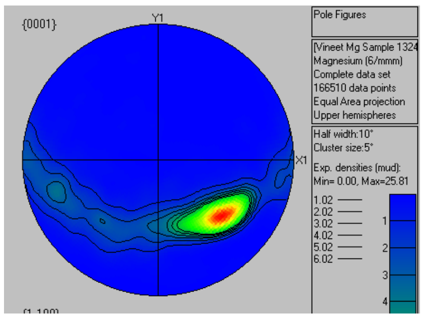

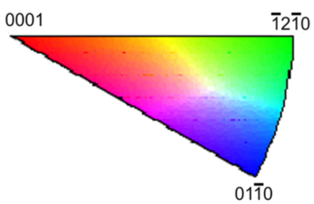

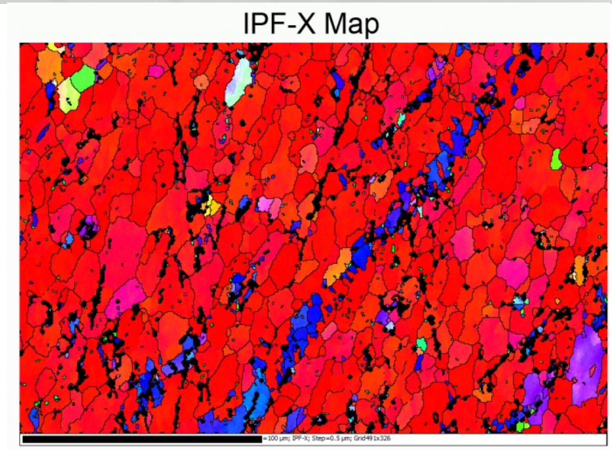

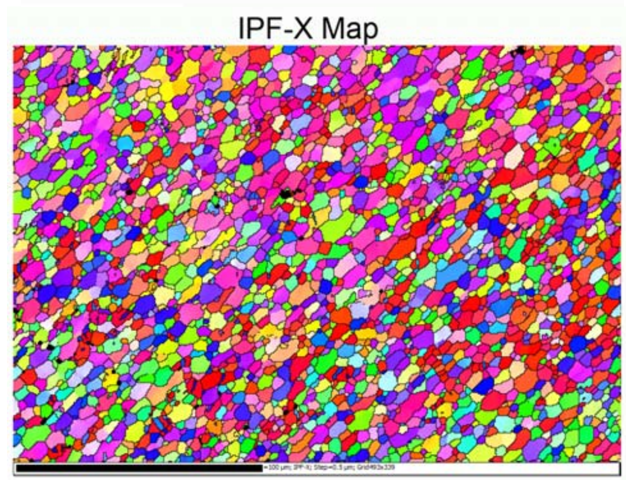

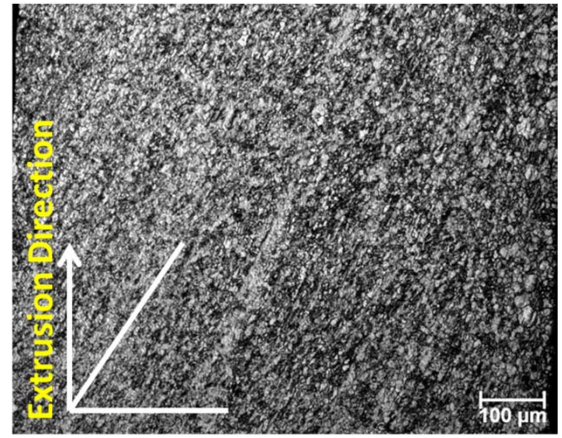

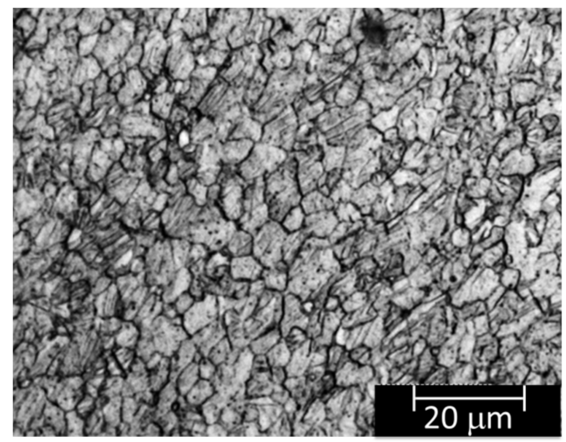

GS less than 5 microns and oriented 45deg to the extrusion axis…… (related to the scroll pattern!!!)

Next Steps

- More Process Development
- Die design optimization
  - Scroll depth and pitch
  - Active control of die can temperature
- Billet or Compact Characterization
  - Microstructural
  - Mechanical Performance
    - Creep performance similar to MA alloys?
    - What is the toughness of this microstructure (fatigue, etc)
- Rod Extrusion
  - Extrusion die design
  - Mechanical properties of the rod or tube
- Continued work on using low-cost un-alloyed powders

Potential Applications and Benefits

- **Ability to produce product forms directly from powder, eliminating numerous /costly processing steps (e.g. mechanical alloying, canning, HIPing, extrusion, etc.**
  - **Application to near-net shape processes (Rod or shape?)**
  - **Application to tubing and piping**
  - **Production of hollow billets for tubular extrusion**
  - **Potentially change from batch to continuous process**
- **Process has the potential to produce appropriate microstructures**
  - **Process can create equiaxed microstructure**
  - **Process also has the potential to break up stringers allowing for reduced roll processing and reduction in probability of defects and low fracture toughness due to stringers**
  - **Strain induced mixing may allow even poorly mixed Fe-Cr-AL-Y powders to be used as feed stocks**
- **Ability to process novel alloy compositions and microstructures without melt solidification steps - critical to ODS alloys and other non-equilibrium systems**

These features are anticipated to lead to a substantial reduction in the cost of producing ODS alloy products

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end