FWP 60098

Low-Cost Fabrication of ODS Materials

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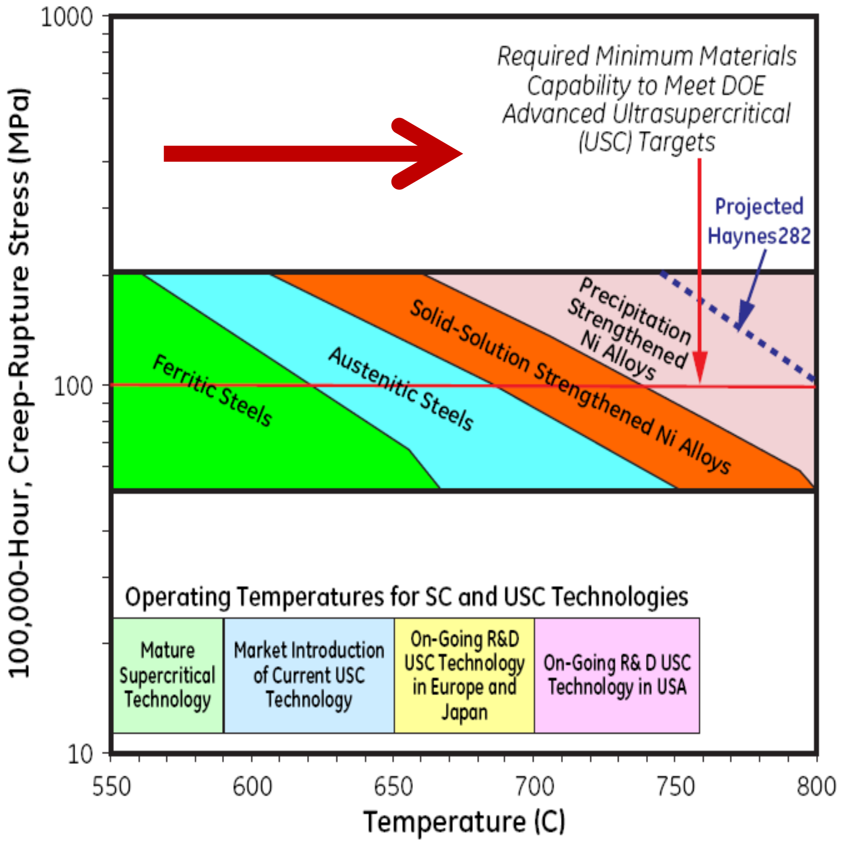
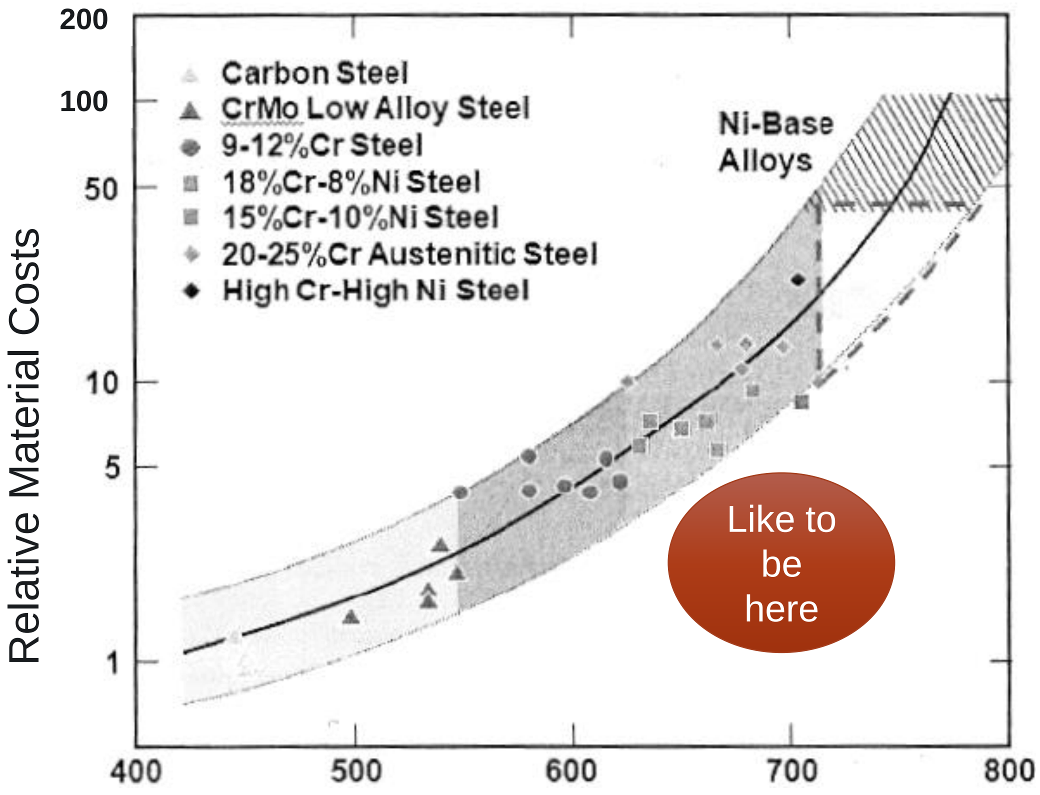
The Problem: There are no low-cost material solutions that can reach the elevated temperatures and pressures we need

The next generation of heat exchangers need to operate at higher temperature and pressure





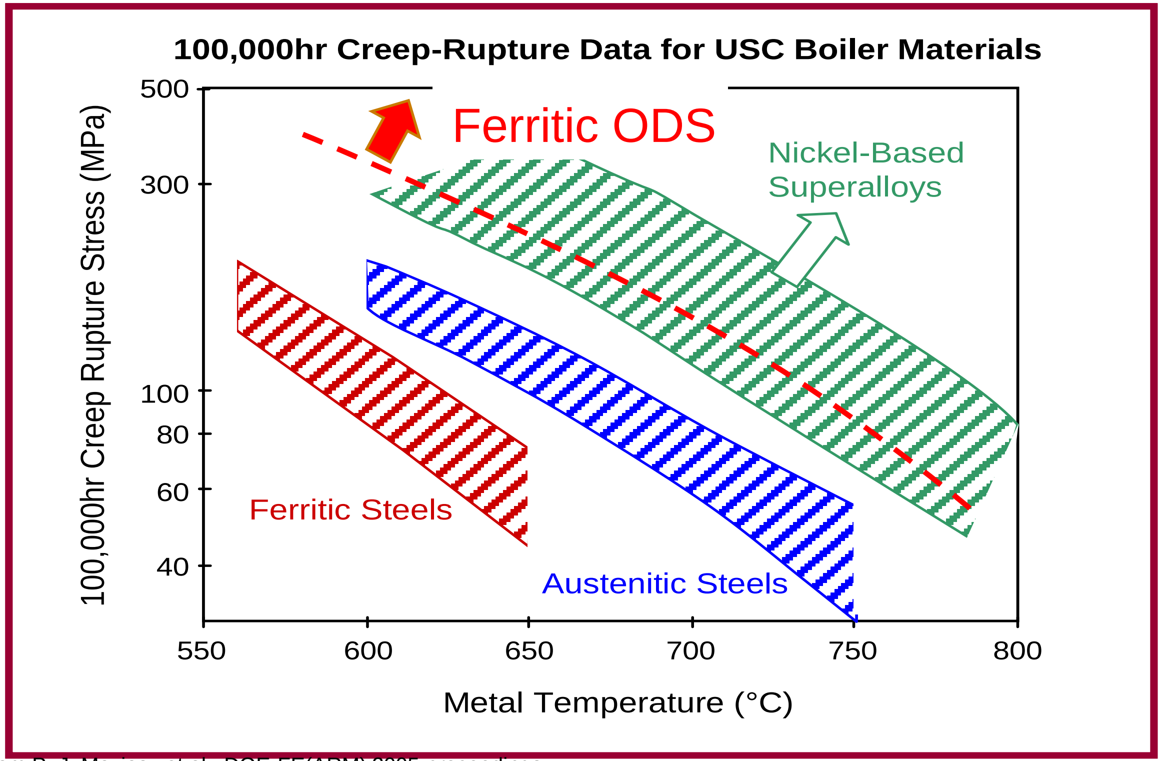
Allowable Temperature at 49 MPa Maximum Stress

NETL Overview & Crosscutting Technology Research, Vito Cedro, 2016

We need better creep and creep-fatigue performance above 700C, and we need it at a lower cost than Nickel alloys

NFA / ODS Ferritic Alloys, at first glance, have potential

1) NFA / ODS Ferritic and F/Ms have excellent performance in both creep and oxidation resistance



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

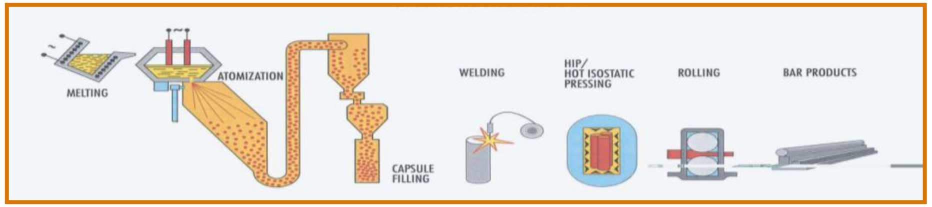
2) <u>AND</u> the ingredients (20Cr steel and 0.2% Yytria) are low cost

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

Barriers to ODS Commercialization

- **Manufacturing Cost - multistep powder process to make semi-finished products**
  - **The high cost of ODS alloys and components is partially driven by the multistep, batch process of fabrication from powder to final product form**
  - **Traditional ODS materials prepared by MA routes are expensive because of sequential small batch processing. If volume demand was there, parallel processing could lower cost but the last ODS available, PM2000, was $400/ lb., reflecting the basic chicken – egg problem.**

or

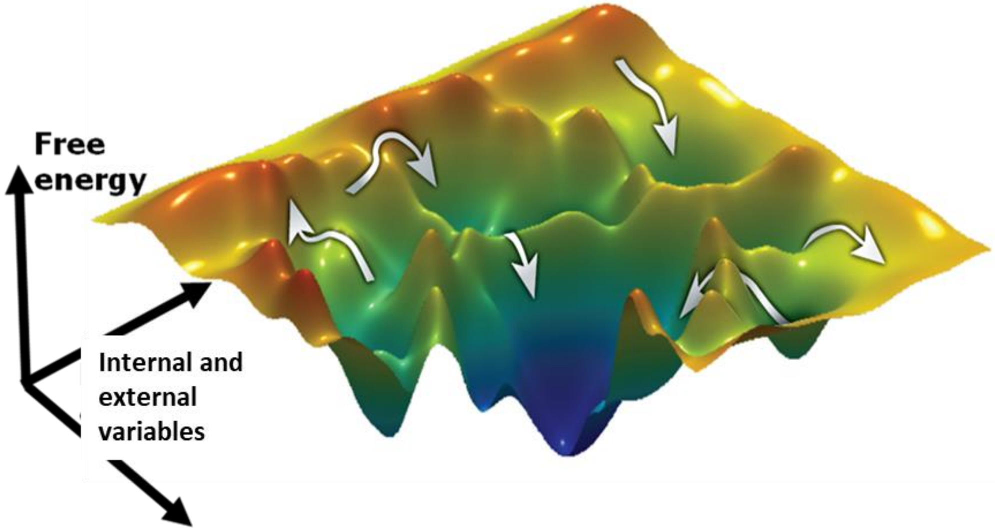


- **Properties require homogeneity and high number density of dispersoids**
- **There are challenges associated with post processing, fabrication, microstructure control / stability**
  - **ODS alloys can be hard to form, bend, pierce, draw, or pilger due to inhomogenaity that results in anisotropy, oxide stringering, and , in some alloys , low RT ductility**

Are there alternative process routes that can remove the some of the costs and produce the right microstructure and workability when going from powder to semi-finished product ?

A common route in the fabrication process of a nanostructured material can involve inducing extreme shearing

- Many nanostructured, high performance materials get their properties by taking advantage of a non-equilibrium or metastable state, either retained in the end product, or in one of the manufacturing steps.
- That state can be created by extreme straining under controlled temperatures
- The straining creates extreme microstructural modification through deformation, subgrain formation and recrystallization. In addition, rapid non-equilibrium solute distribution can occur due to advection.
- Extreme straining can provide for defect formation and high energy states at interfaces providing nucleation sites for precipitates, dispersoids or important 2nd phases.

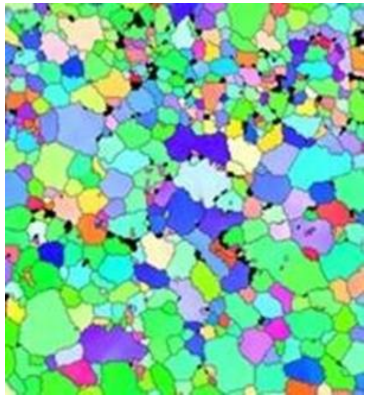


However

- Most methods of inducing shear in a bulk sample are small scale processes
  - ECAE, High Pressure Torsion, etc.

If severe plastic deformation could be translated to a high volume industrial process, we might be able to recreate the lab scale microstructures in a bulk product.

- We are developing a process that combines severe plastic deformation with a conventional, high-volume extrusion process

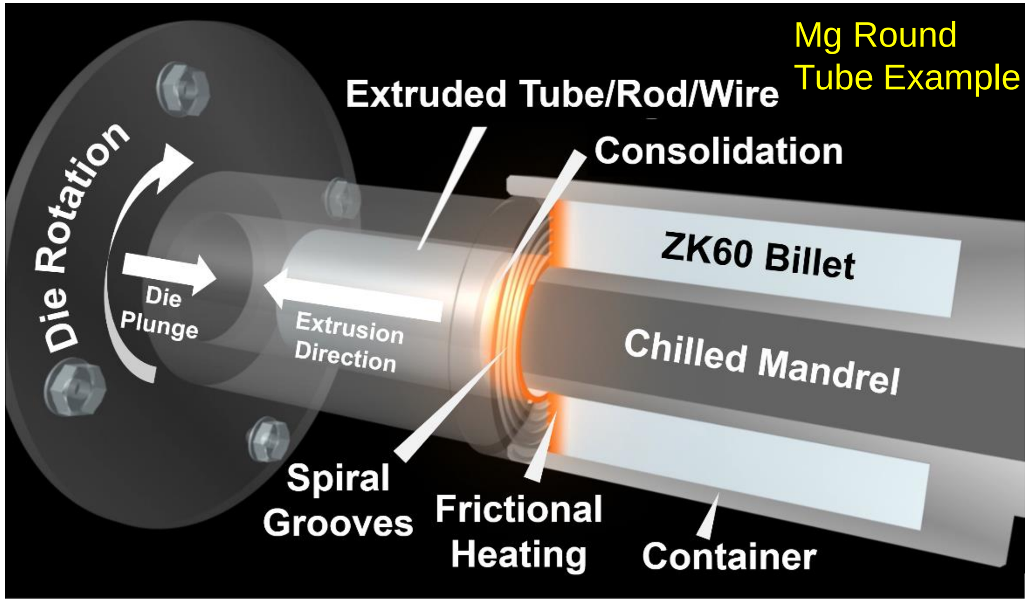


Shear Assisted Processing and Extrusion or the ShAPE process

Shear Assisted Processing and Extrusion

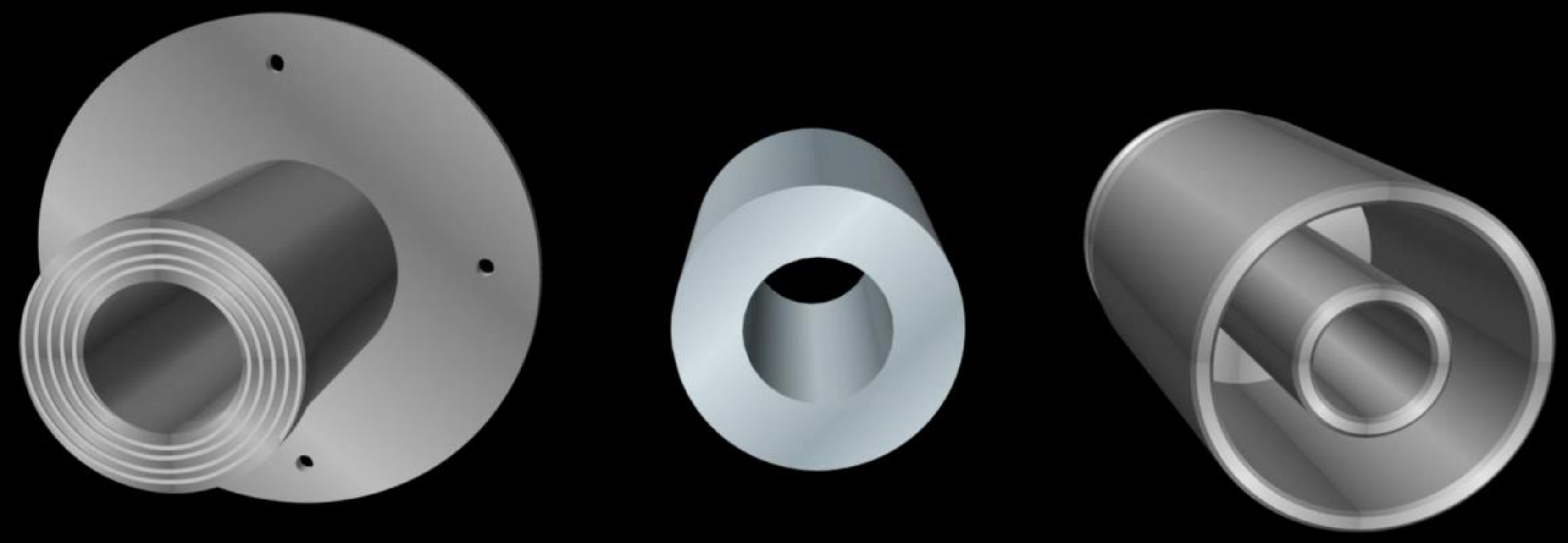
- **What is ShAPE?**
  - Method of extruding solid wire, rod, shapes and hollow profiles that combines rotational shear with axial extrusion. The process imparts extreme levels of shear strain to billets, powders or chips to form tubular structures that extrude off a mandrel.

- **What are the benefits?**
  - Flexibility of feedstock, solid billet or directly from unconsolidated powder
  - Grain refinement and texture alignment
  - Breakdown of 2nd phases
  - Extreme mixing and homogenization
  - Can eliminate PM process steps
  - Lower force and power
  - Potential for manufacturing scale
  - Potential for reduced total energy embedded in product

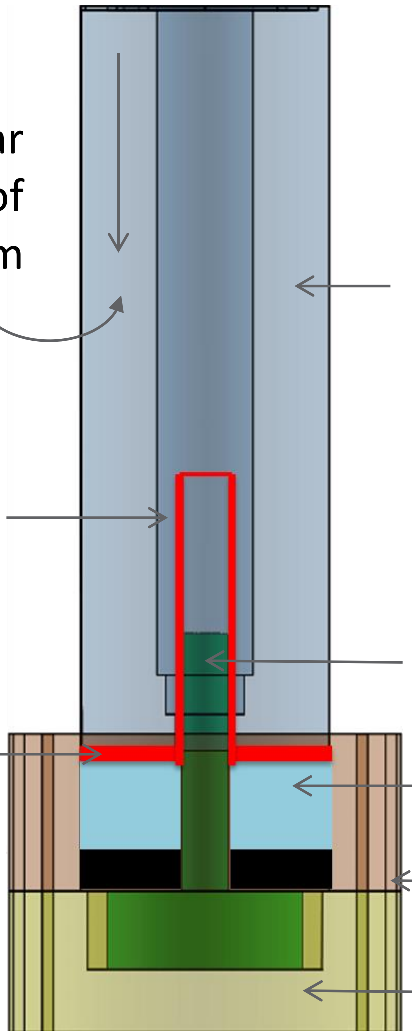


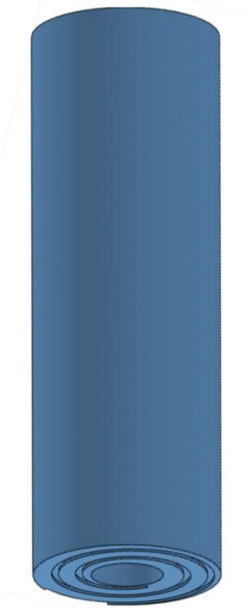
ShAPE: How Does it Work?

Hollow billet, tubular extrudate example

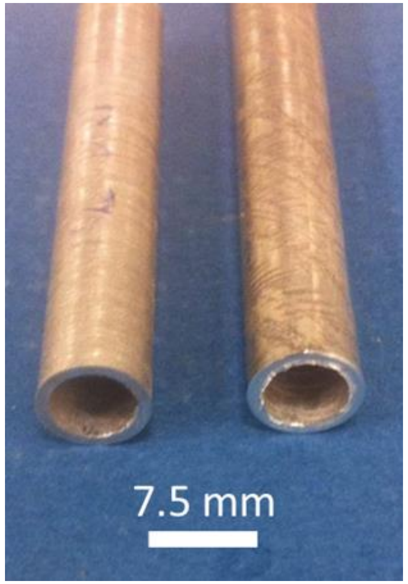


Shear Assisted Indirect Extrusion Process
-Evidence of efficacy from Magnesium trials





Flute/ Scroll profile on the Ram



Can we make ODS materials this way?

Feedstock Materials Investigated

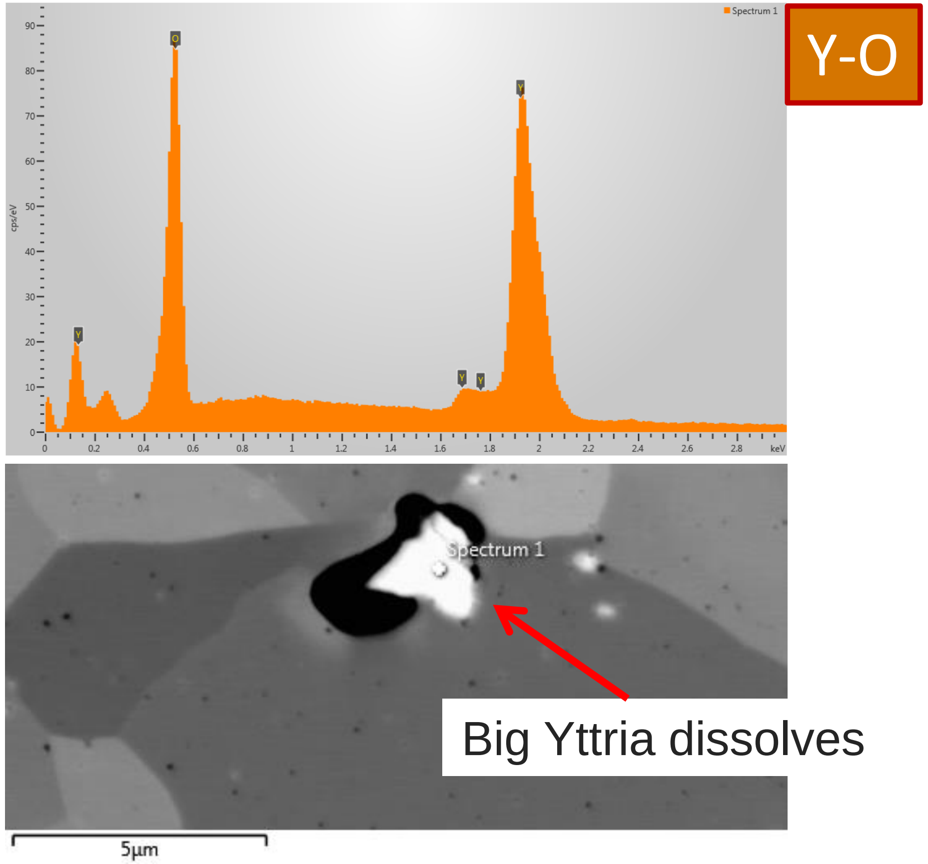
- 3 Different feedstock powders

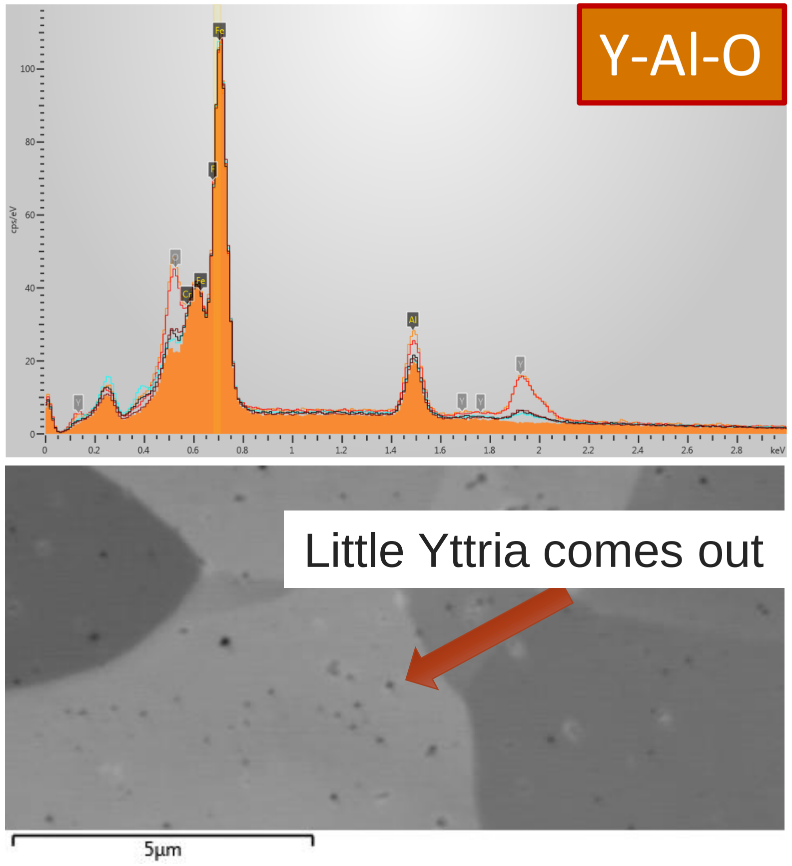
	Mechanically Alloyed Powder	Gas Atomized Powder	Steel Powder + Y_2O_3
	Special Metals	*Sandvik Osprey*	*ATI Powder Metals*
	MA956	*Fe22Cr5AlYZr*	*Custom*
Fe	Bal	Bal	Bal
Cr	19.64	22.4	18.6
Al	4.87	6	4.94
Ti	0.39	-	0.5
Y_2O_3	0.5	-	0.5
Y	-	0.07	-
Zr	-	0.42	-
Oxygen	0.25	-	-
Si	0.07	0.21	-
Mn	0.09	0.2	0.04
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 + Y2O3 have virtually the same global chemical composition.

- <u>MA</u> Powder– FCE can eliminate the downstream process costs but MA precursor still includes the up front MA cost
- <u>GA</u> Powder- Reduces cost of “front end” powder step, but distribution of yttria may be a challenge (dependent on particle size?)
- <u>Steel + Y</u> Powder- 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

Shear Assisted Extrusion of Stainless Steel powders + Yttria powder

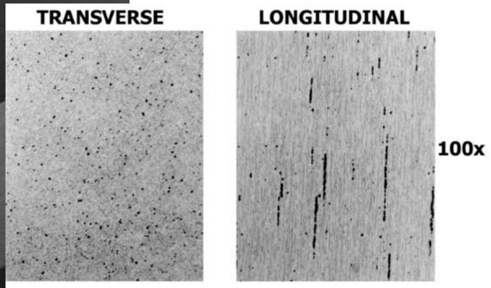


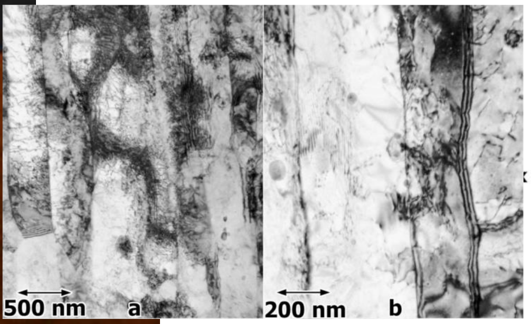


- This powder started as a 40µm steel powder mixed with 0.5% 1 to 5 µm yttria particles. Final compact shows almost no original yttria.
- Process dissolves coarse yttria and re-precipitates dispersoid Y-Al-O…in 30 seconds

Results of Extrusion Trials on ODS powders

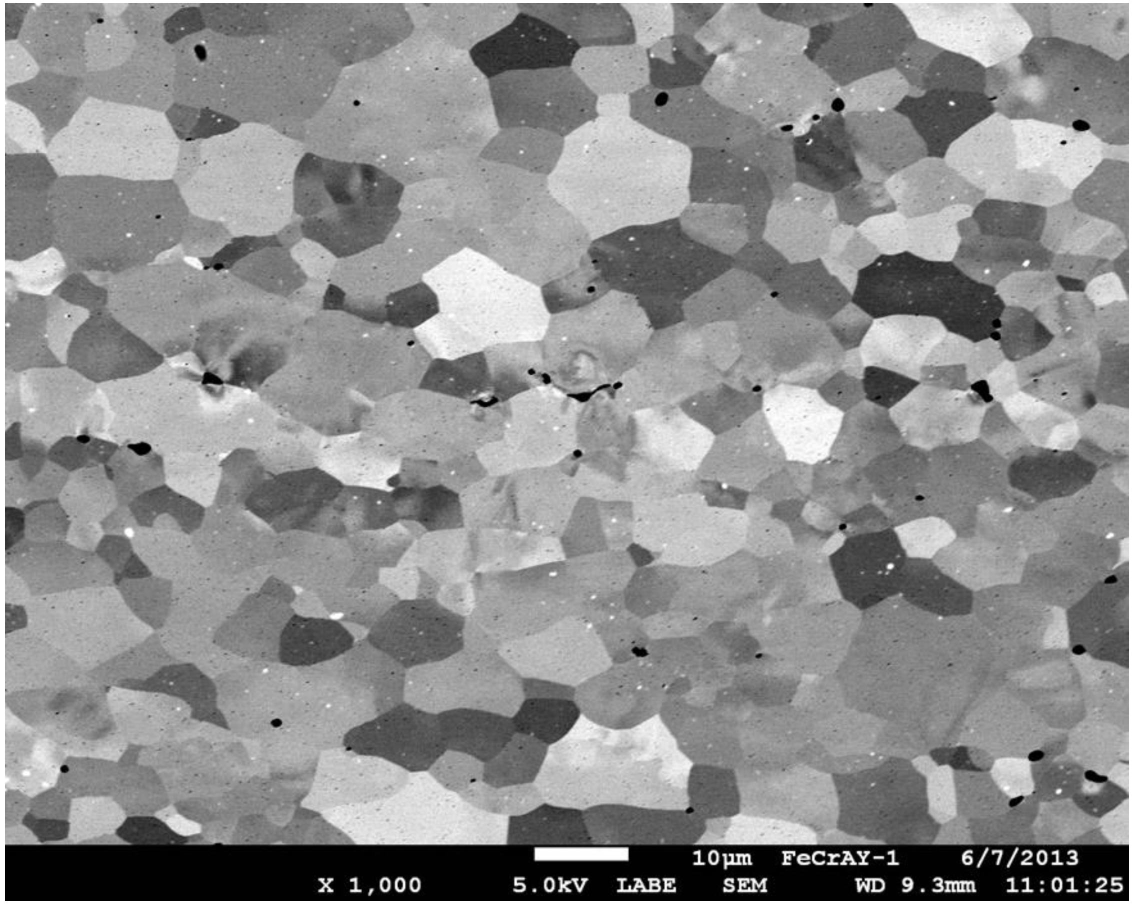
- For all three powder compositions and product forms, we were able to make fully dense compacts and rod extrudates approx. 0.25” in diameter
- Equiaxed 6 micron grain size
- Particles 10nm to 50nm are Al-Y-O clusters
- Larger particles 100nm to 0.5 micron are Al-Y-O compounds: YAP and YAG





Conventional extrusion creates highly elongate subgrain structure and long stringers of Y-Al-O (or Al_2O_3) (in those alloys with Aluminum)

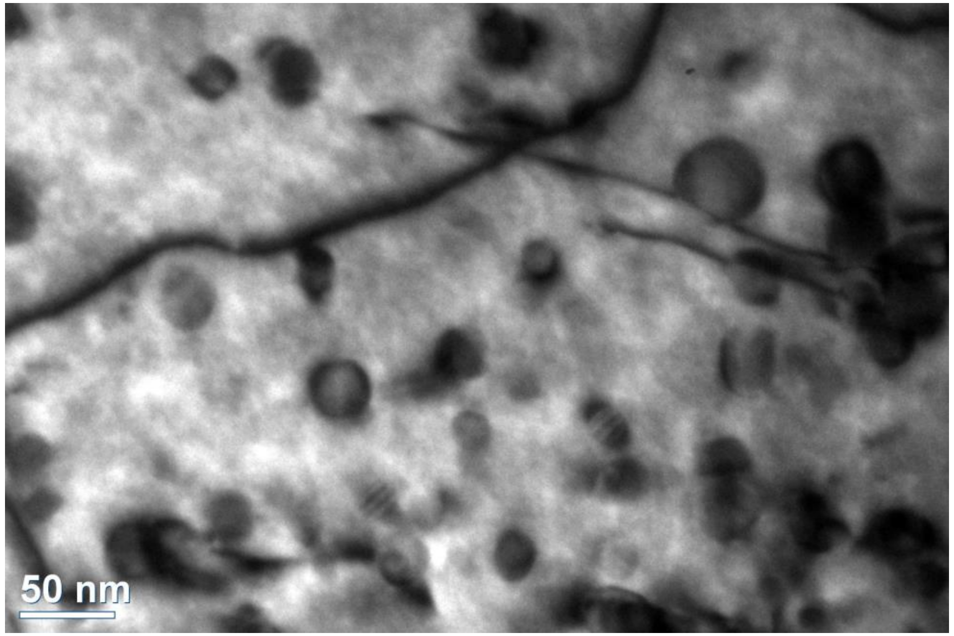
This can have a negative effect on properties needed for post processing (rolling) and in-service performance. (creep anisotropy)



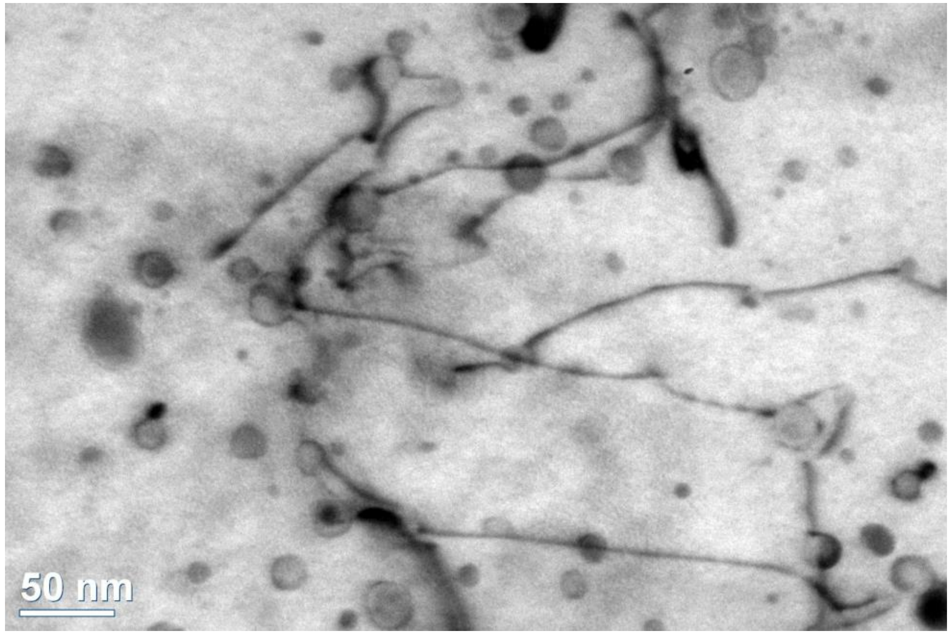
ShAPE Extrusions produce homogeneous particle distribution without stringering

Is it an ODS?

- TEM shows a similar size dispersoids in the ShAPE processed pucks as in the conventionally processed MA956. (MA, conventionally extruded, then heat treated)
- Small 5 to 20 nm dispersoids are Al-Y-O and Y-O



MA 956 RL Conventionally Processed



Shear Assisted Extrusion

- **The ShAPE process may be able to make the right microstructure in one process step, putting ODS alloys back on the map of practical materials**

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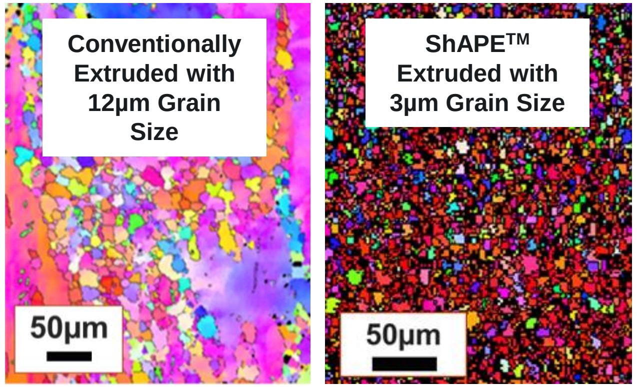
Unique Textures and Microstructures are possible (mag ZK60 example)

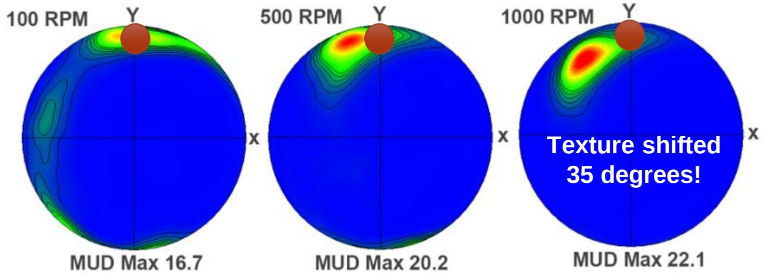
Solid Phase
PROCESSING

ShAPE can customize texture orientation in HCP alloys

Grain Size Comparison (ZK60)
Conventional vs. ShAPE™



Basal Texture Comparison (ZK60)
Conventional vs. ShAPE™



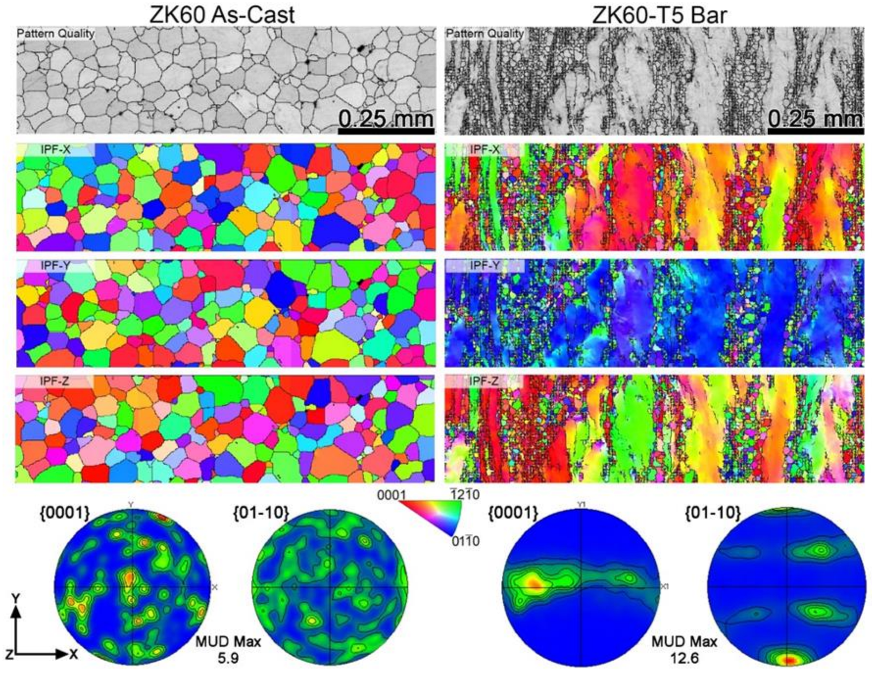
0001 Pole Figures

● = Texture in Conventionally Extruded ZK60 Mg Tubing

- Co-extruded multimaterial tubes are possible
- The texture direction is influenced by the ratio of the die rotation rate to the extrusion rate
- In ODS alloys that have secondary recrystallization behavior, this may allow for growth of elongated grains in a unique (spiral) direction in the tube/pipe

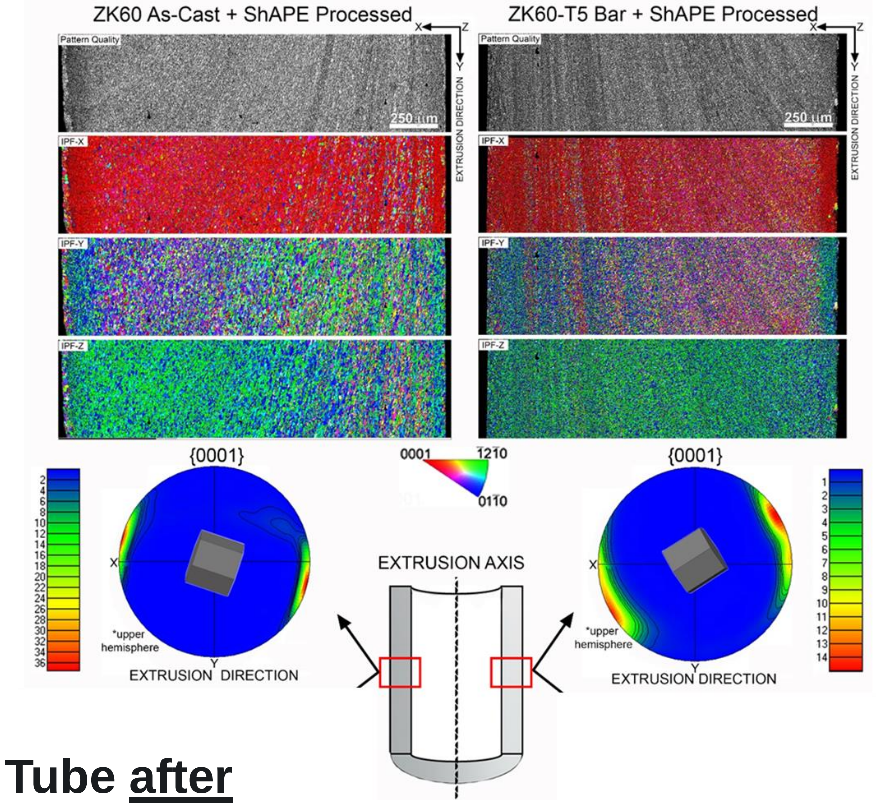
ShAPE can homogenize any starting microstructure, especially mixed powders and nonhomogeneous castings

ZK60 As-Cast | **ZK60 T5 Bar**



Billet before ShAPE extrusion

ZK60 As-Cast + ShAPE | **ZK60 T5 Bar + ShAPE**



Tube after ShAPE extrusion

Mixing is critical to ODS alloys

Shear Assisted Extrusion

- **We have demonstrated that Shear Assisted Extrusion can:**
  - fully densify MA, gas atomized and ss+Y powders to crystalline solids with complex and process parameter dependent microstructures
  - Sub 10nm dispersoids were observed in the ShAPE processed pucks where no dispersoids were originally present in the powders
  - The process can recrystallize and refine the microstructure
  - The process can create equiaxed microstructures without oxide stringering

Next step: Scale up

Can we make relevant, full-sized product forms?

Scale up - World's first Shear Assisted Extruder installed at PNNL

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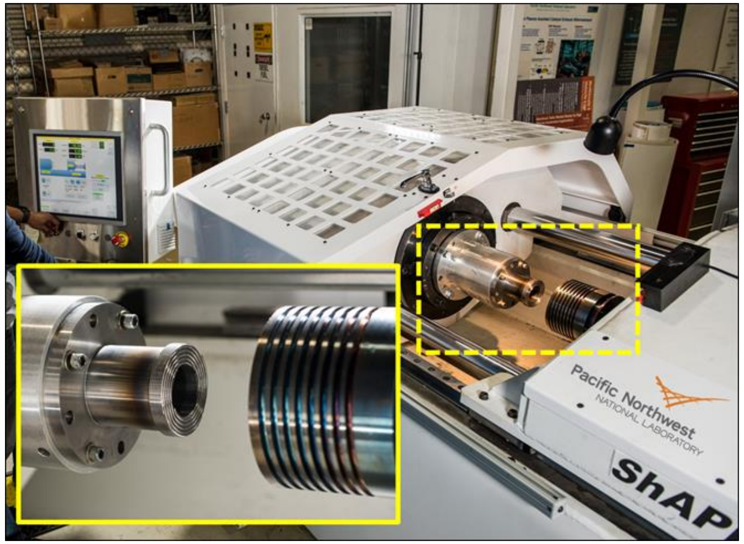
Proudly Operated by Battelle Since 1965

- 100 Ton axial feed
- 2000 ft-lb rotation torque
- 10 inch stroke
- Controlled / programmable on rpm, forge rate, force, torque



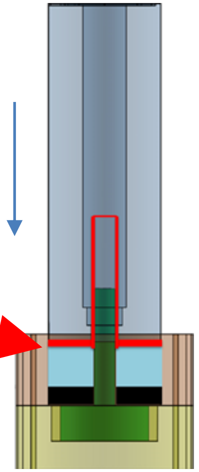
- Adaptive, closed-loop control system to maintain fixed die face/ extrudate temperature

2nd ShAPE machine will be installed in FY22
Much larger! ShAPE 2.0

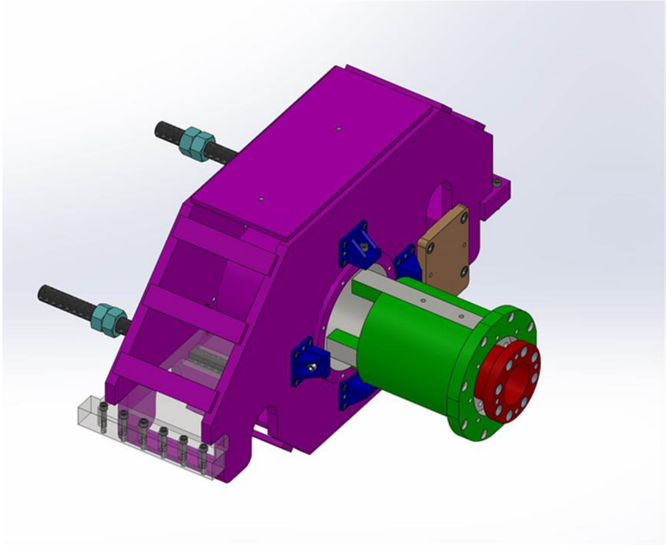
Efforts in FY19-20-21

Converting machine from a back, or “Indirect” extrusion configuration to forward, or “Direct” extrusion

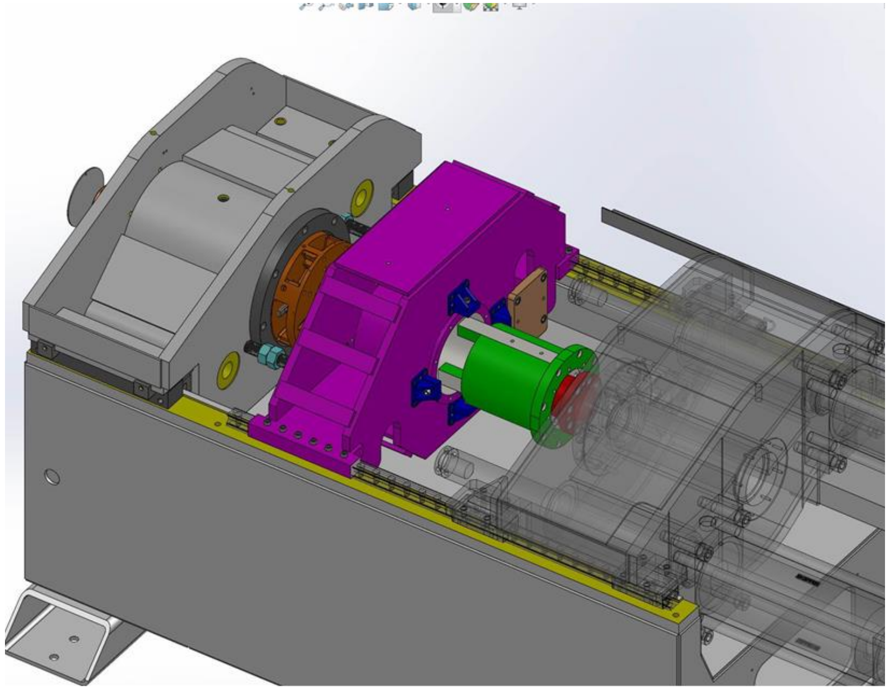
- To reach larger diameter ODS extrusions our current setup was breaking tools due to galling between the rotating W-Re die and the can

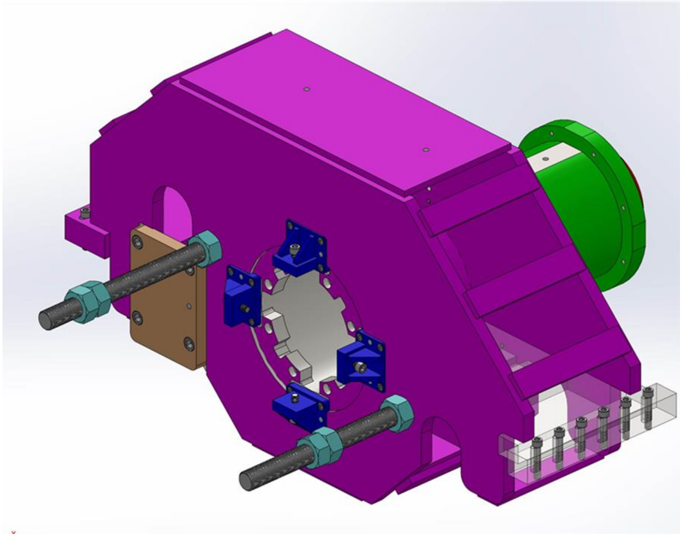


The problem with indirect extrusion at temperatures above 900C…



- Solution is to fix the die/can relationship and push the billet or powder charge from the back (Direct Extrusion)
- This necessitates building a “stationary can”
- Efforts are underway to build and test this by the end of the FY, and end of the project





Summary - Lowering the cost of ODS Alloys

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 shaped extrudate)
  - Application to tubing and piping

- Process has the potential to produce appropriate microstructures for post processing or application
  - Process can create equiaxed microstructure – reduction of anisotropic behavior?
  - Process does not produce oxide stringers
    - reduced problems in roll processing
    - reduction in defects and low fracture toughness due to stringers
  - Strain induced mixing allows even poorly mixed Fe-Cr-AL-Y powders to be used as feed stocks, lowering feedstock cost
  - Process can produce customized texture in the extrudate not possible by other methods

Summary - Lowering the cost of ODS Alloys

Potential Applications and Benefits

- Ability to process novel alloy compositions and microstructures without melt/solidification steps - critical to ODS alloys and other non-equilibrium systems

- The Shear Assisted Extrusion process loads are far below those of conventional extrusion allowing for:
  - Reduced equipment size (process intensification)
  - Reduced energy embedded in the semi-finished product and Reduced Carbon Footprint

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

Thank You

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