

***Project 40-L: Evaluation of Processing Path
Effects on Microstructure and Properties of
Powder-Based Al-TM alloy***

Fall 2021

Student: Stuart Shirley (Mines)

Faculty: Kester Clarke (Mines)

Industrial Mentors: Rob Mayer (Queen City Forge)



Project 40-L: Evaluation of Processing Path Effects on Microstructure and Properties of Powder Al-TM alloy



- Student: Stuart Shirley (Mines)
- Advisor(s): Kester Clarke (Mines)

Project Duration
Masters: August 2019 to December 2021

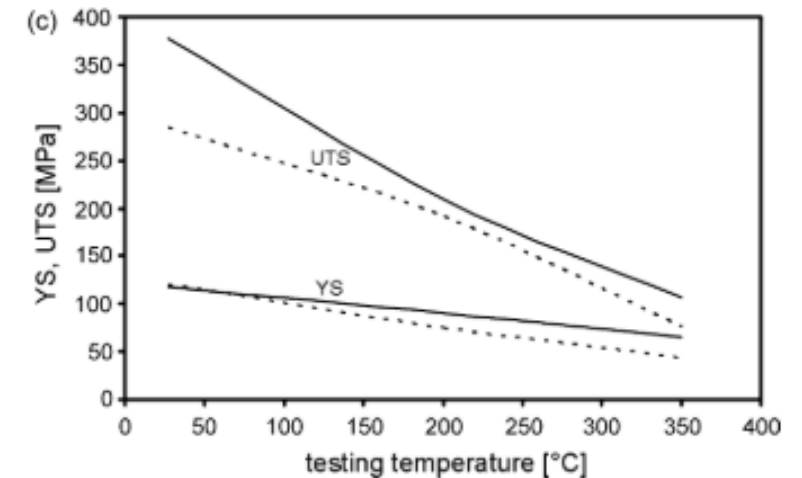
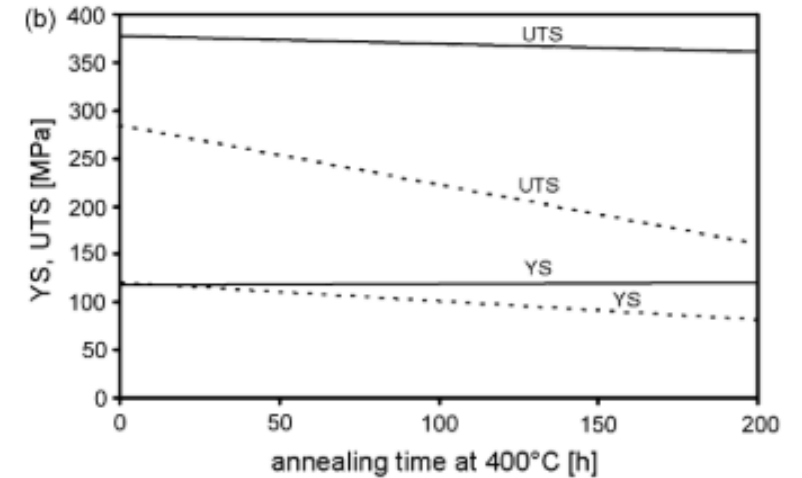
- **Problem:** Al-TM alloys have excellent performance, but can be challenging to process via conventional processing pathways.
- **Objective:** Evaluate the effect of processing path on the microstructure and mechanical properties of Al-TM alloy.
- **Benefit:** Improved understanding of processing path effects on microstructure and properties Al-TM powders.

- Recent Progress**
- Low Temperature Extrusions
 - Hot Hardness Testing
 - Powder Characterization
 - Completion of Thermal Stability Testing
 - Microstructural Characterization
 - Start of Thermomechanical Testing

Metrics		
Description	% Complete	Status
1. Literature review	75	●
2. Microstructure Characterization of Al-TM	20	●
4. Microstructure Characterization of ShAPE Al-TM alloy	40	●
5. Thermal Stability Testing	100	●

Al-TM Background

- Produced as a powder via melting and gas atomization
- Aluminum alloyed with Fe, Cr and Ti
 - Other alloys are Al-Fe-Cr-X
 - X; Ti, Nb, Ta, V [2]
- High temperature thermal stability
 - Annealing and room temperature hardness
 - Hot hardness testing
- Ductility
 - 15% elongation via ShAPE processing [3]
 - 4-9% elongation via extrusion [4]

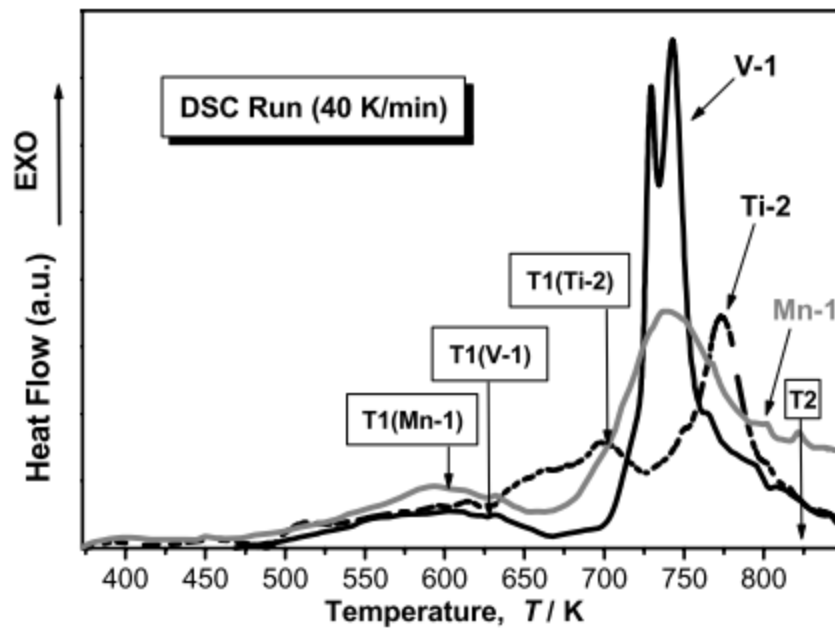


Solid line: PM extrusion consolidated Al-Cr-Fe-Ti
Dashed line: Al-12Si-1Ni-1Cu-1Mg casting alloy
Sourced: Adapted from [1]

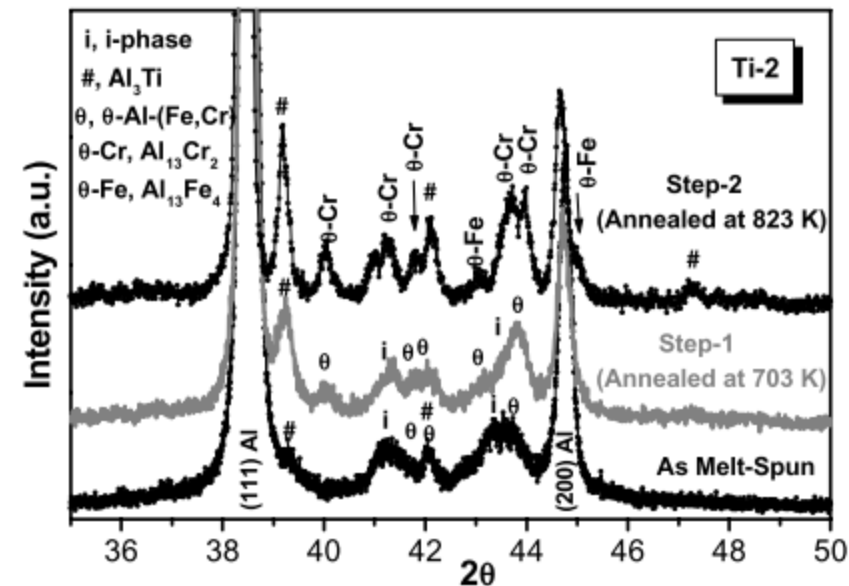
Al-TM Phase Evolution

Strengthening Precipitates

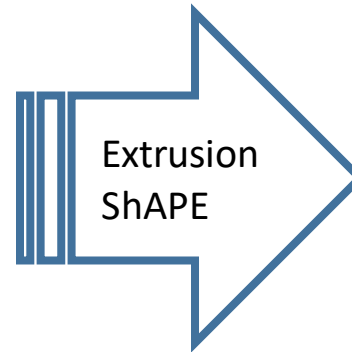
- Quasi-crystalline particles 50-80 nm [5]
- Coarsening at $\sim 427^\circ\text{C}$ (800°F) and transformation at $\sim 500^\circ\text{C}$ (932°F) [5,6]



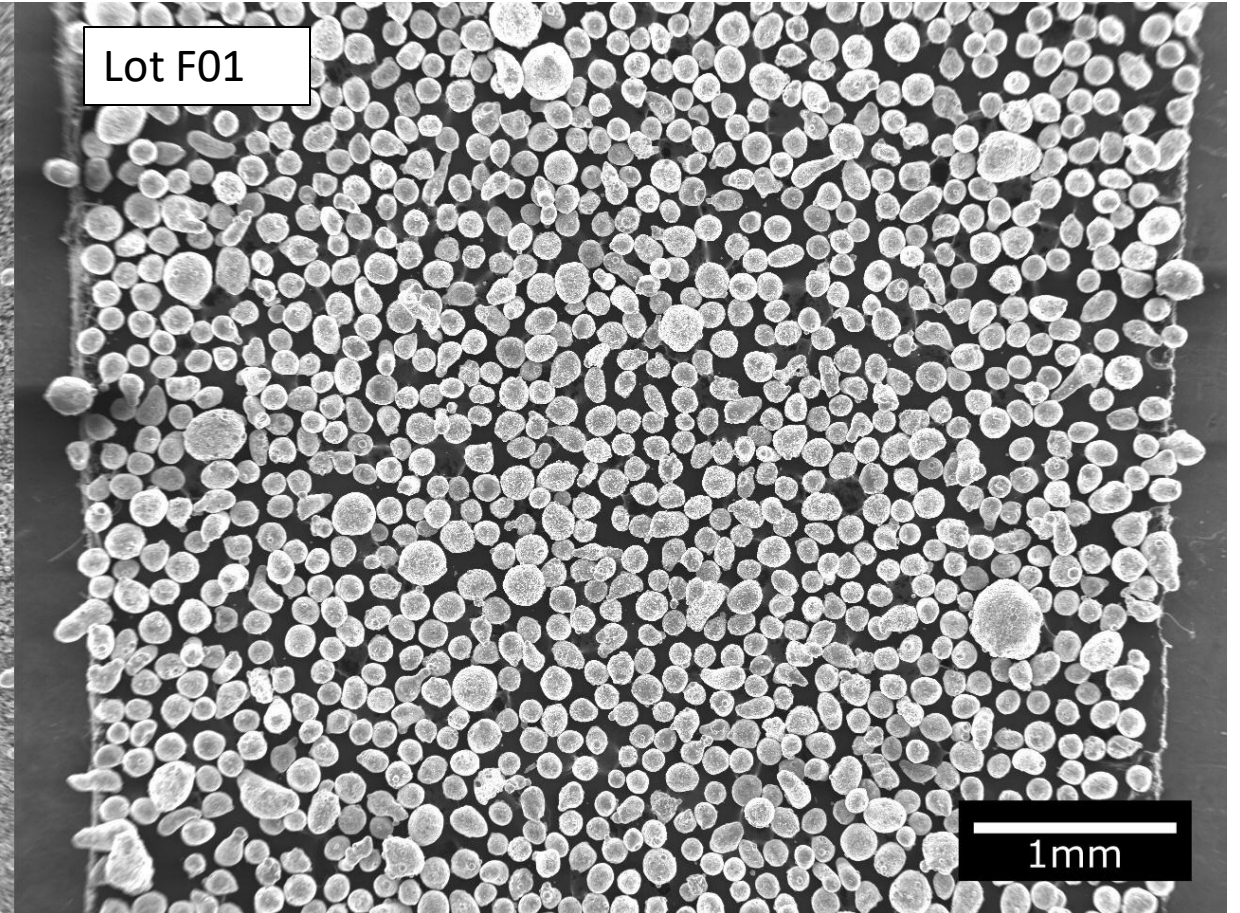
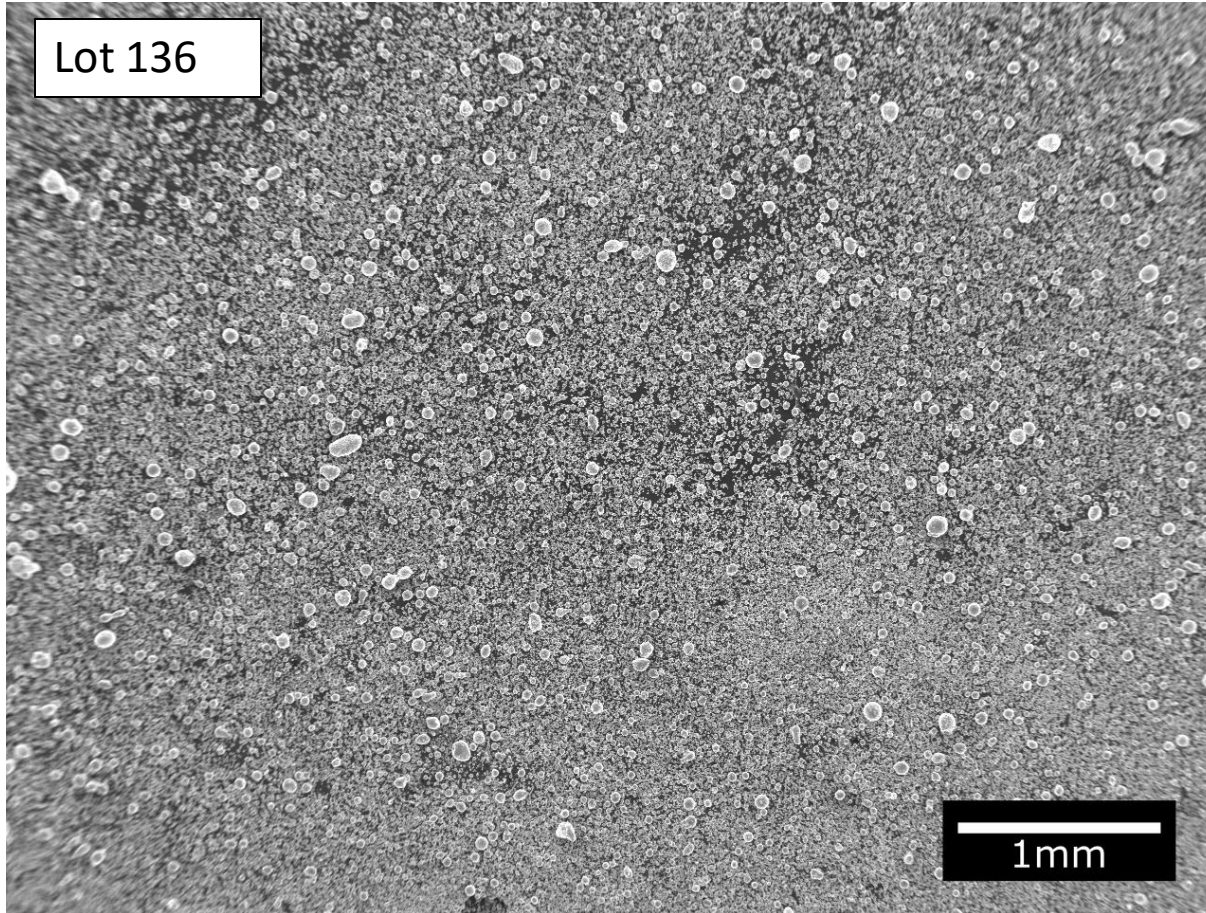
Adapted from [5]



Motivation



The case of two powder lots

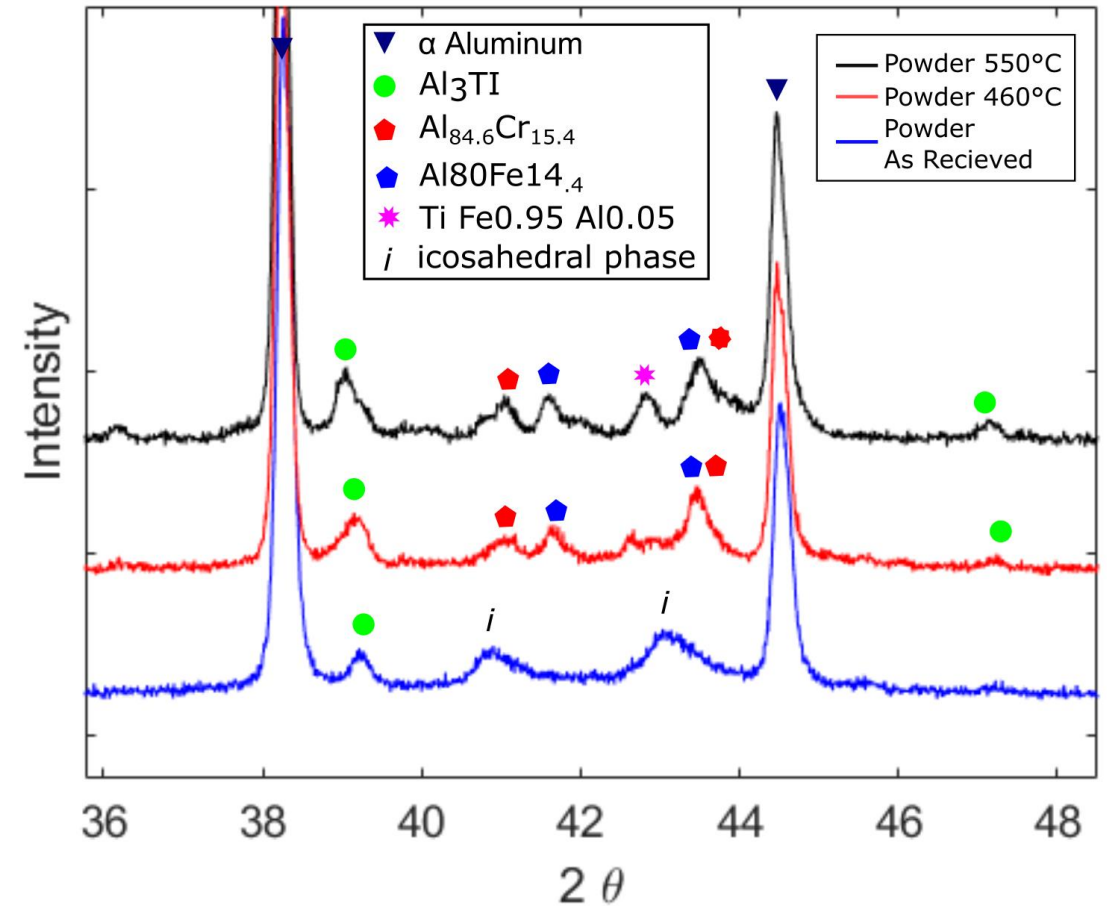
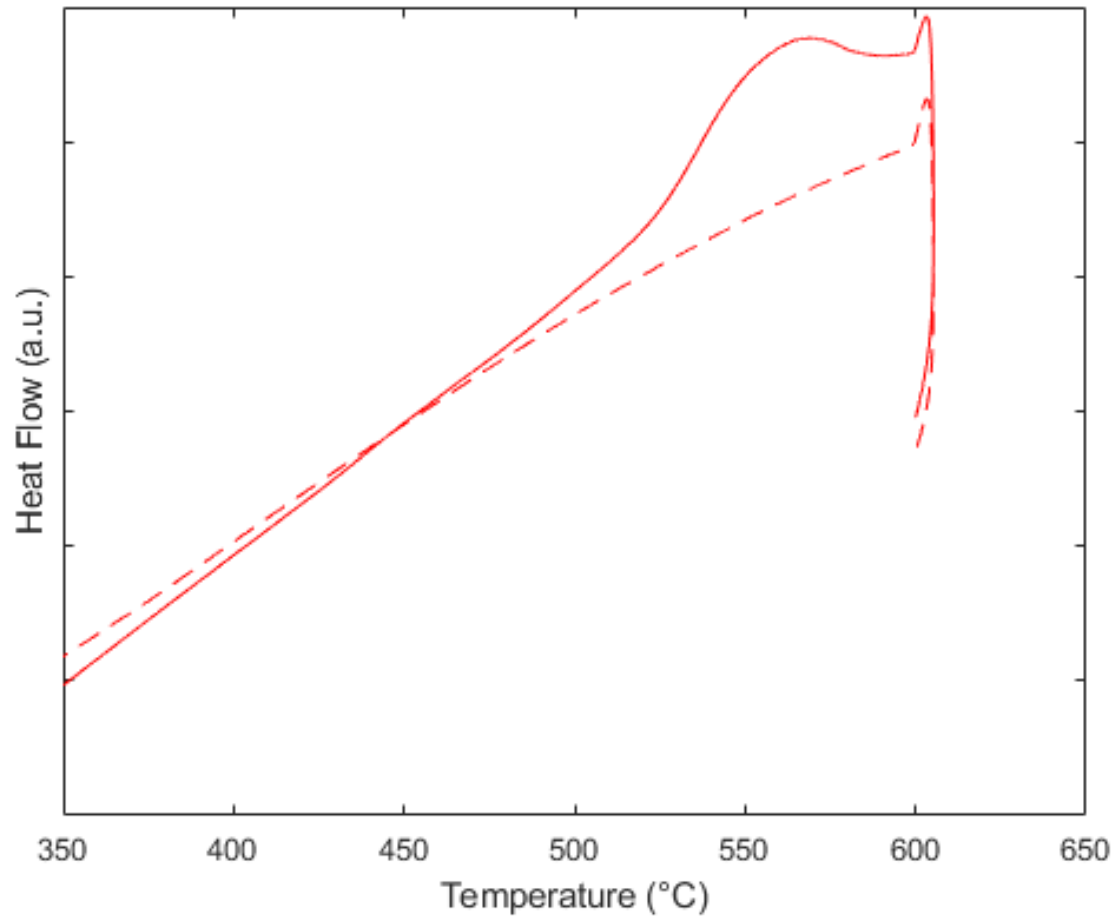


— 20 μm powder

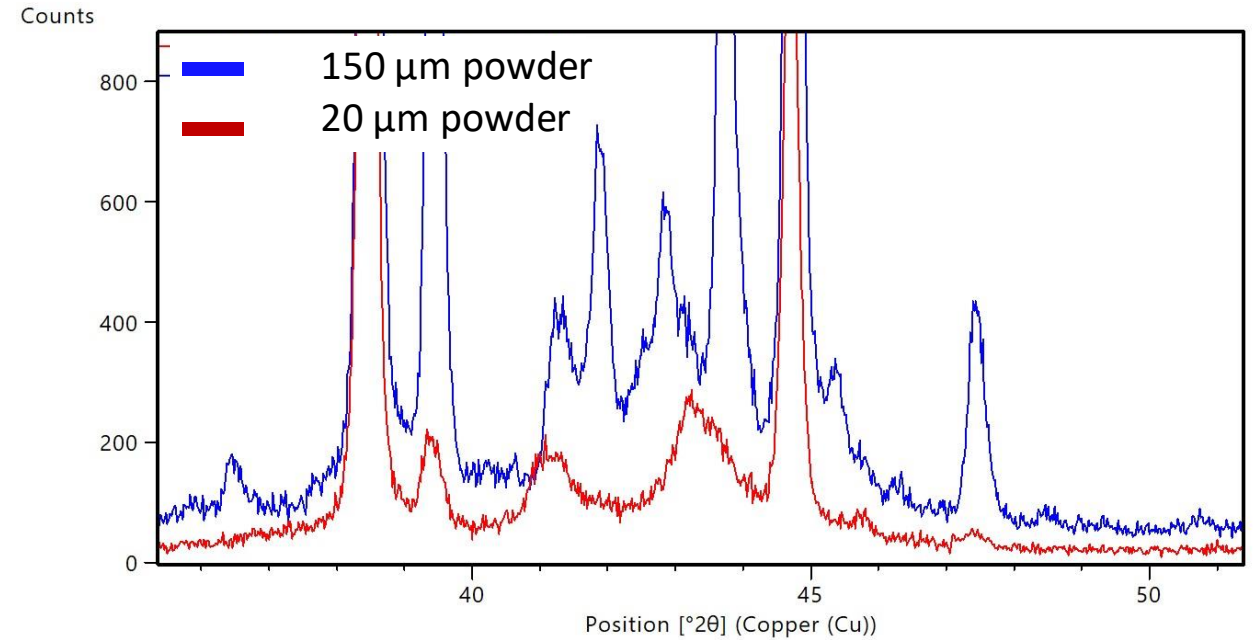
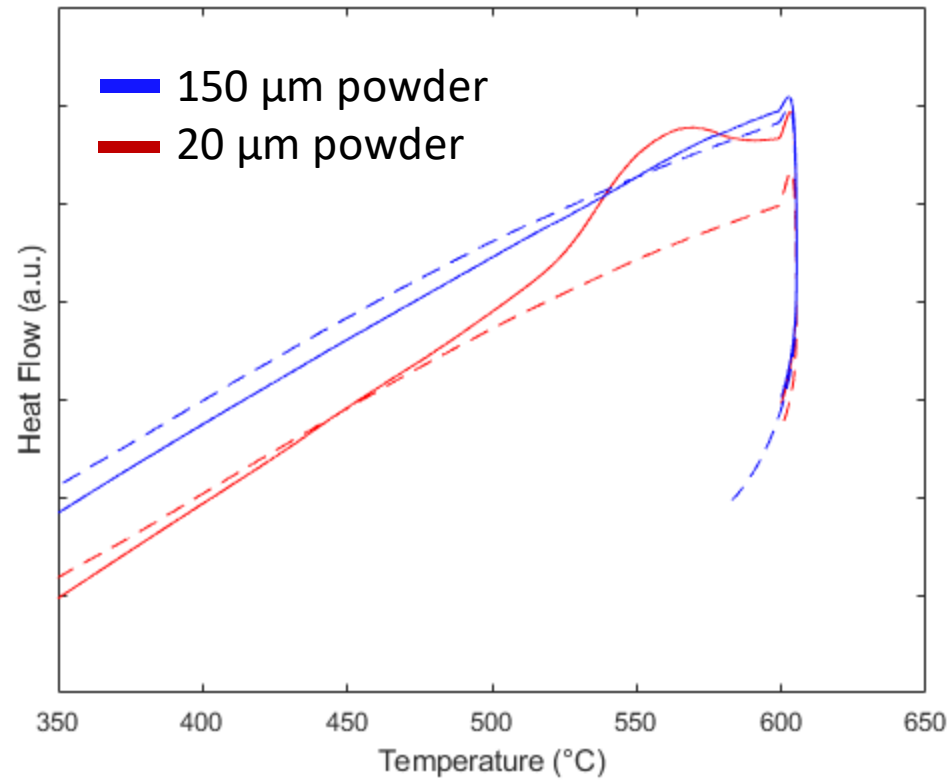
— 150 μm powder

Phase Evolution in this Study

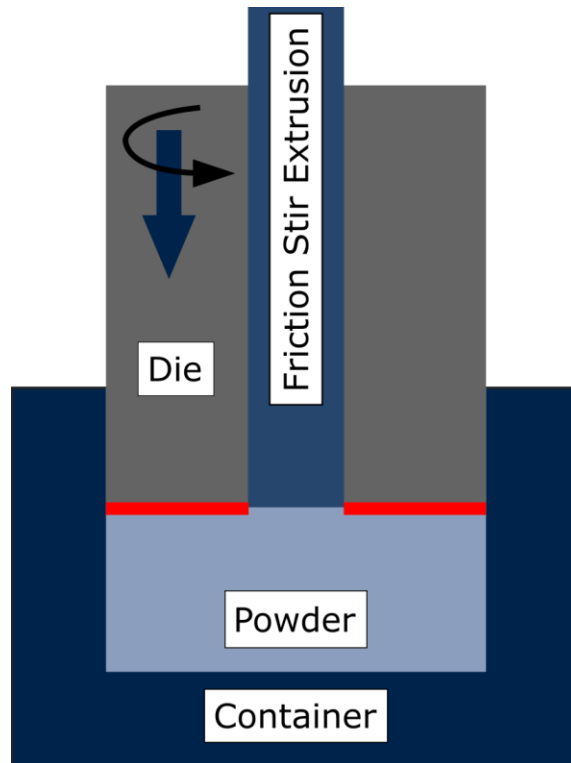
Lot 136



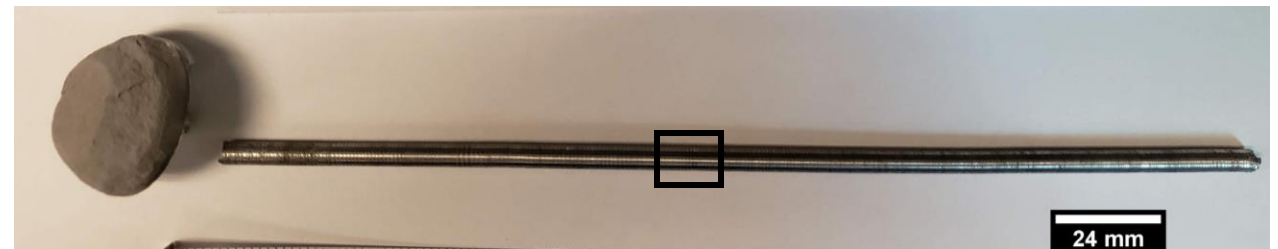
Icosahedral Phase Powder Size Dependent



Friction Stir Extrusion

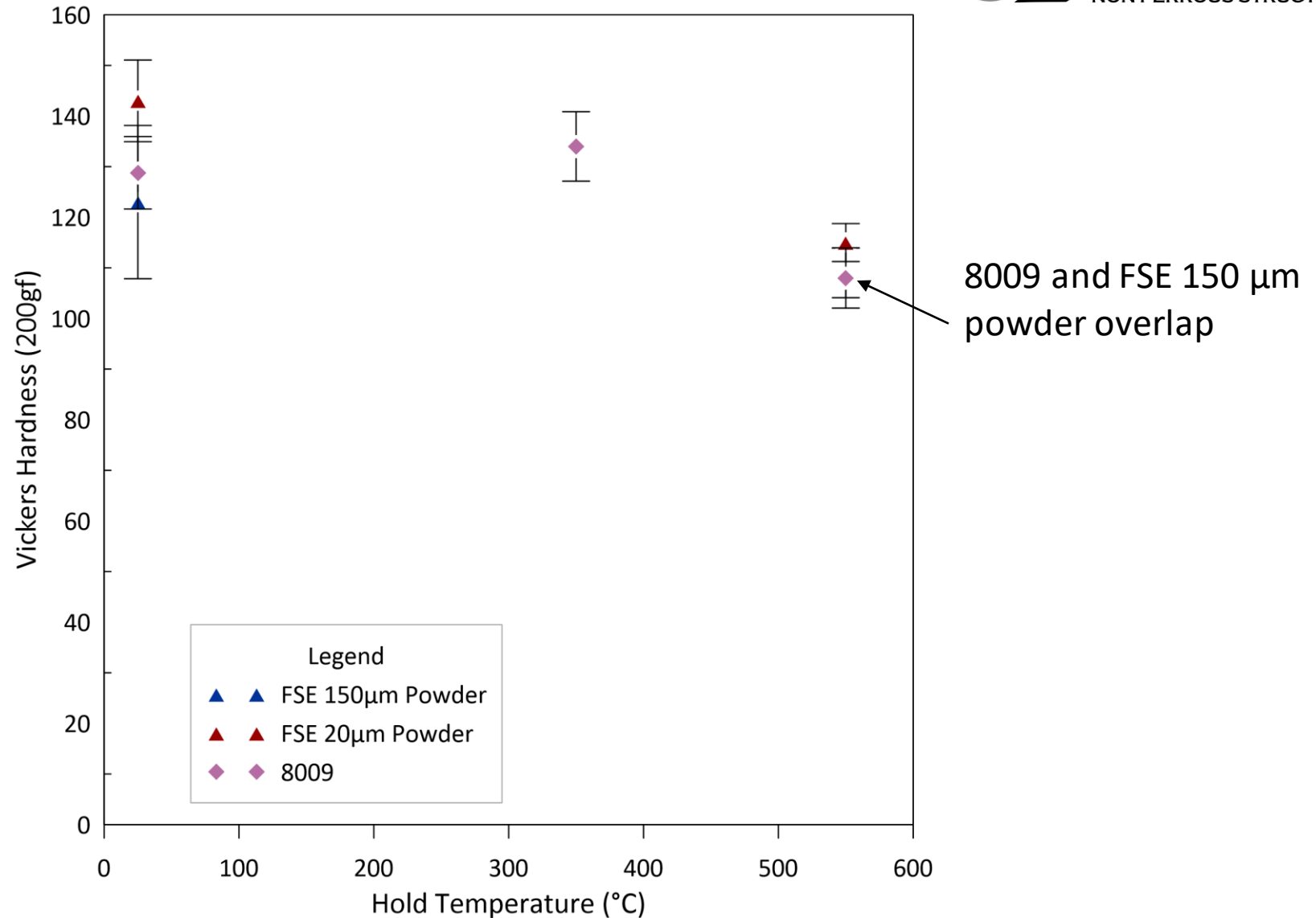


- Powder loaded open to atmosphere
- Aluminum, Copper, Magnesium [7-9]
- ShAPE™

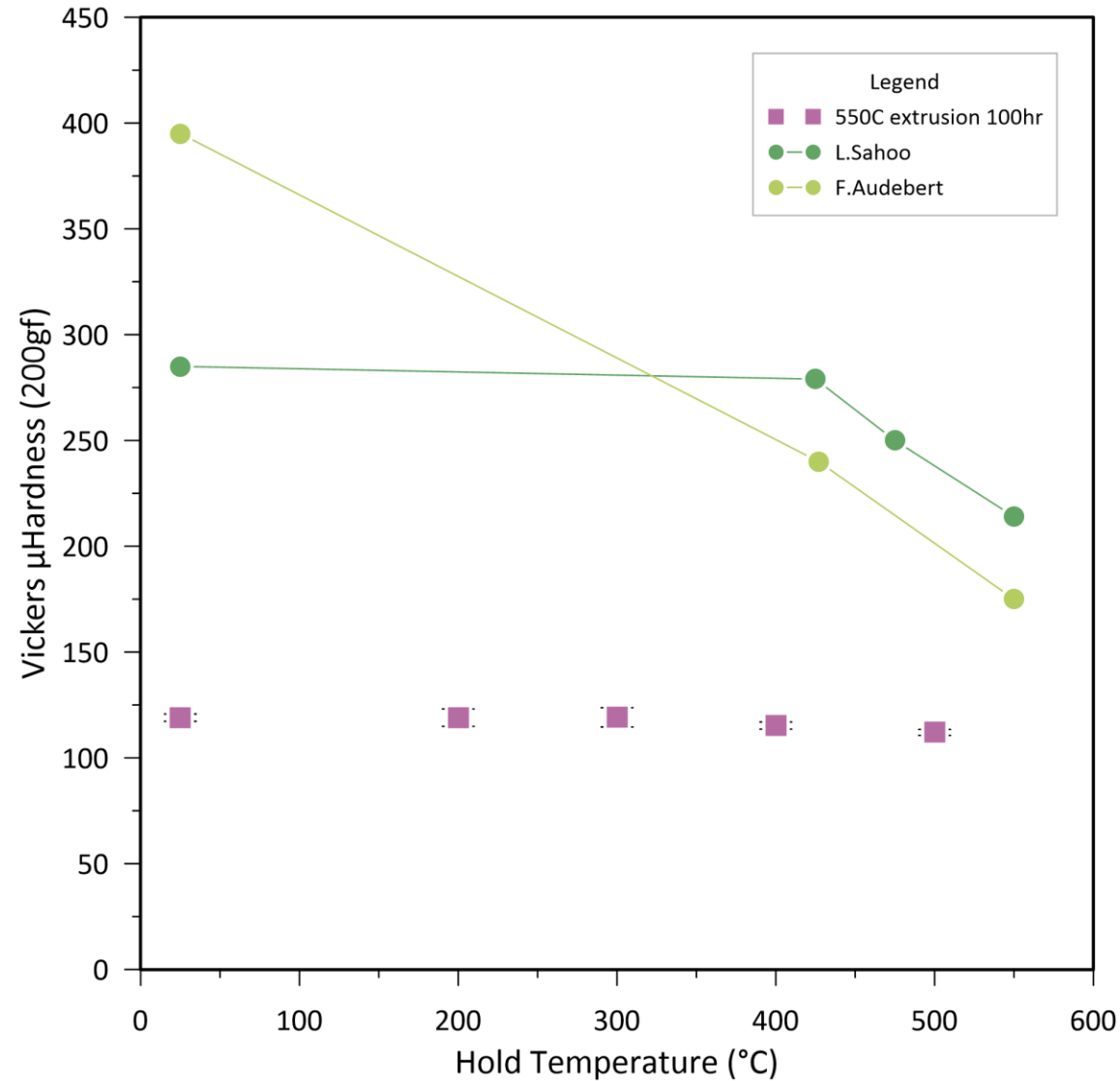


FSE graciously provided by Scott Whalen of PNNL

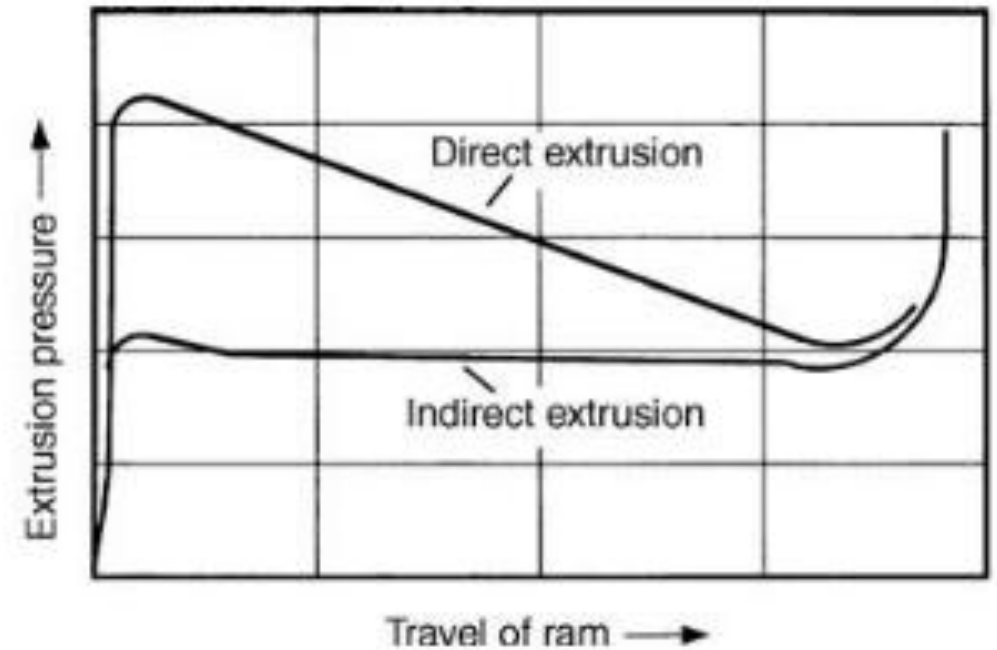
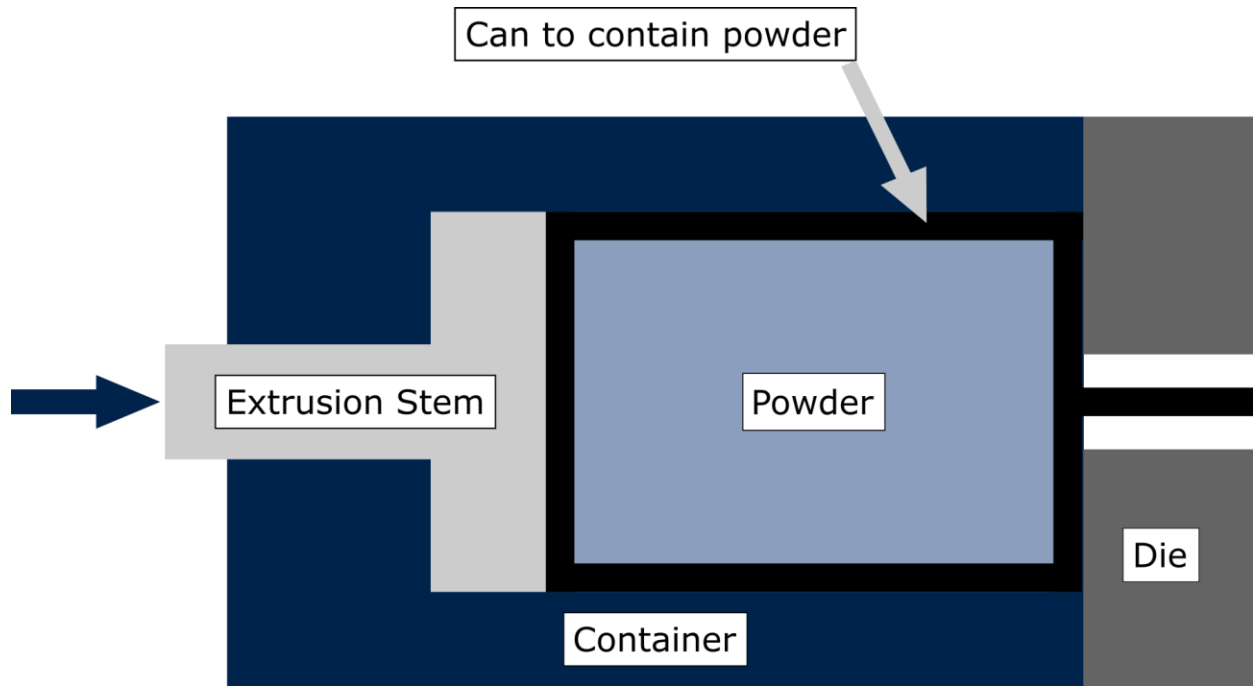
FSE Shows Equivalent Hardness to 8009



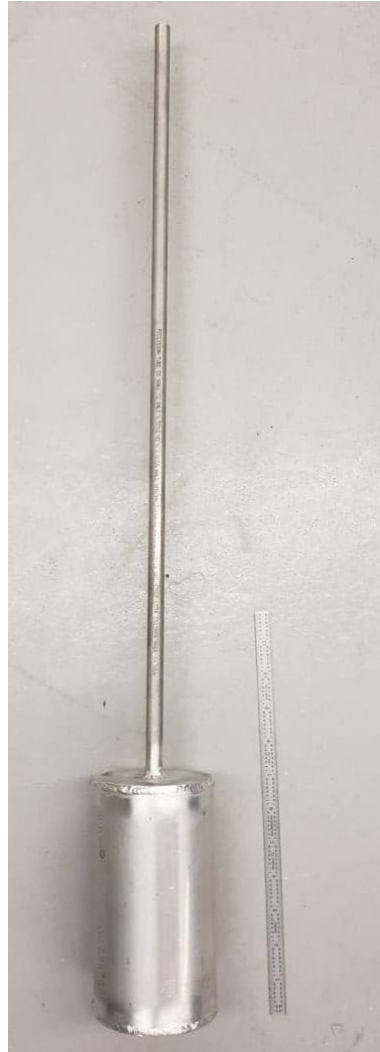
Thermal Stability



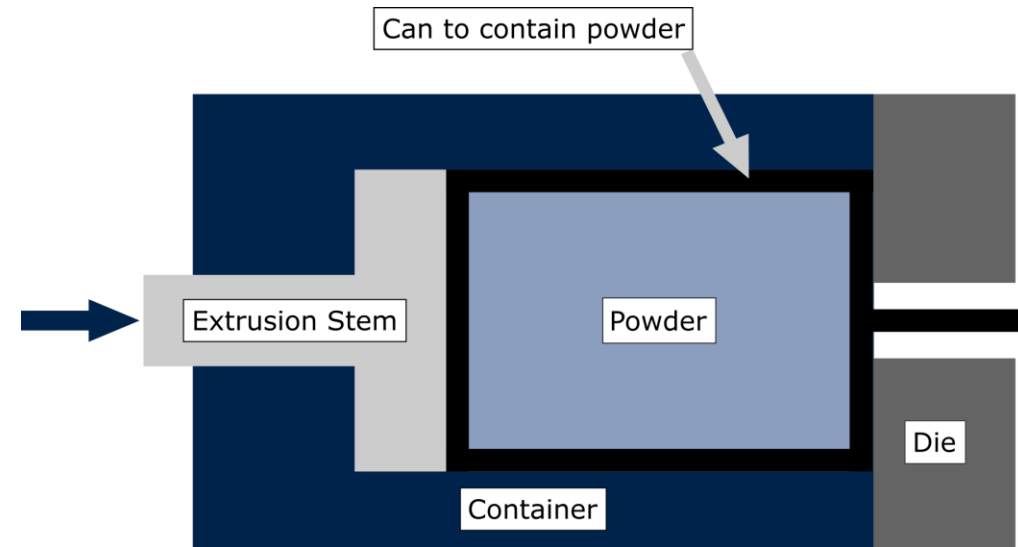
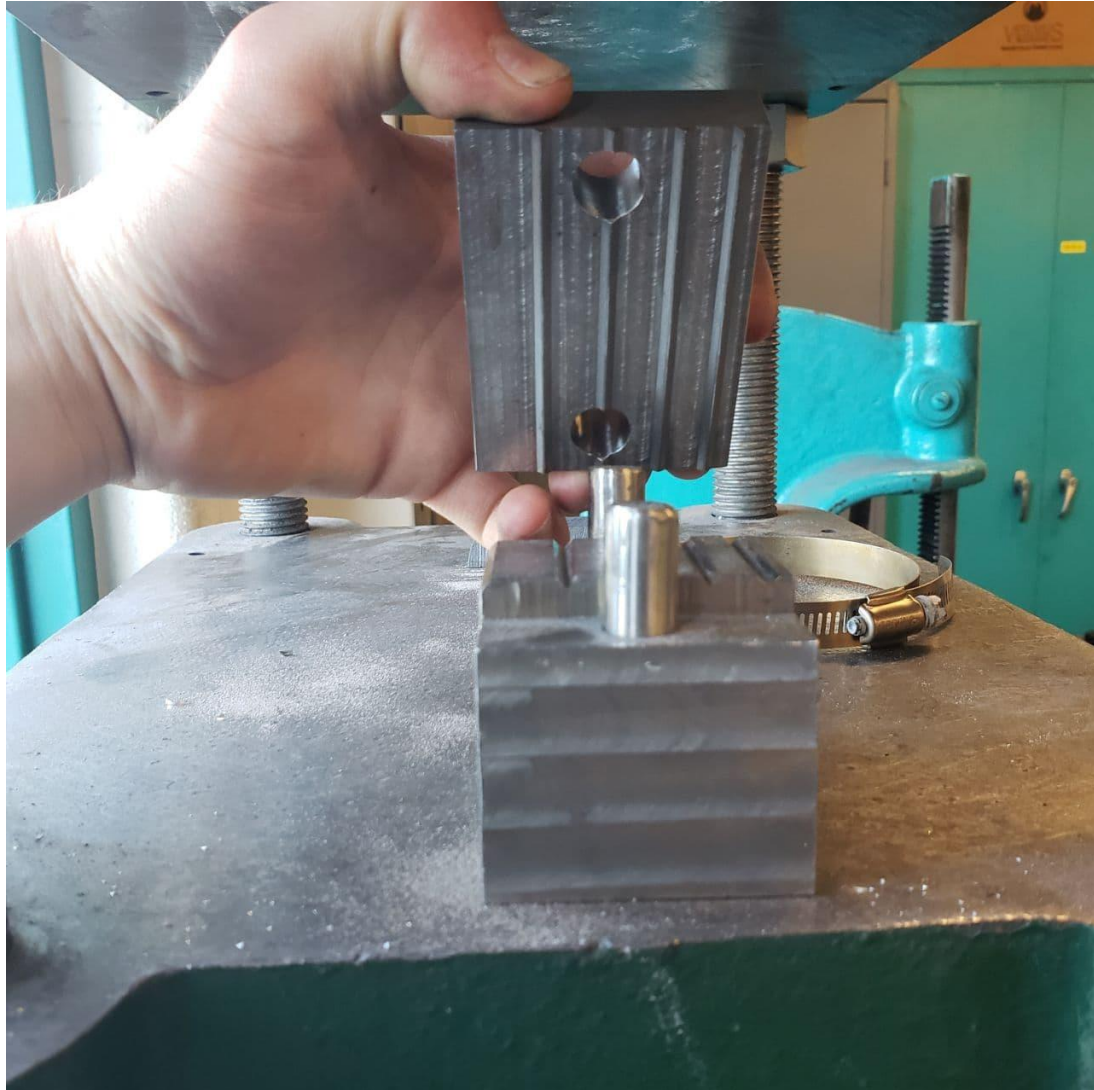
Direct Extrusion



Direct Extrusion



Direct Extrusion



550 Ton Press



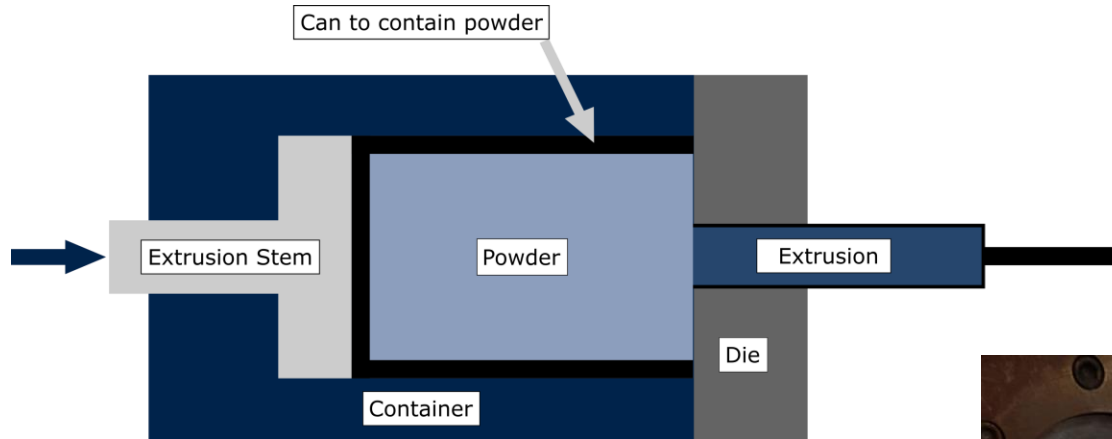
Direct Extrusion Conditions



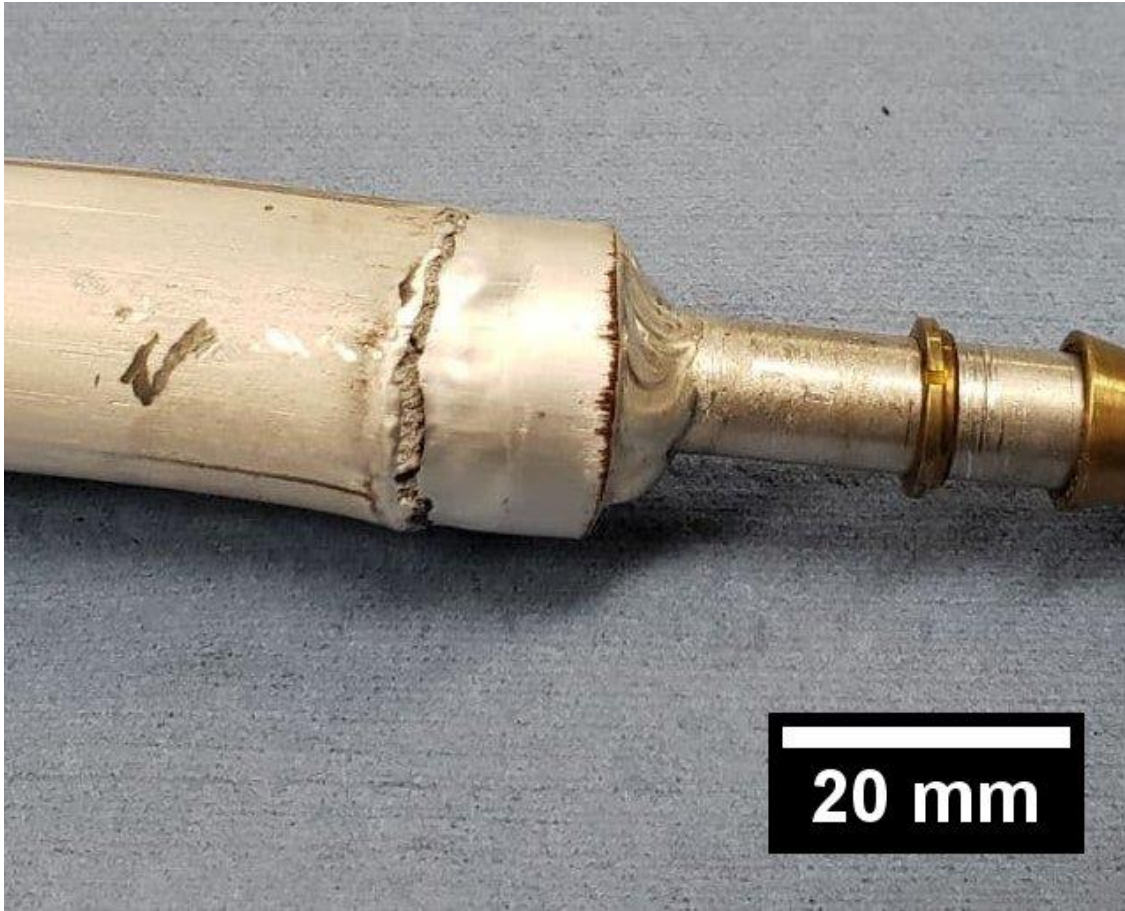
Temperature (°C)	Extrusion Ratio	Exit Temp Contact Thermocouple	Exit Temp IR
350	10:1	360	390
400	16:1	390*	420*
425	16:1	410	440
450	16:1	410*	440*

*Difficulties loading can into extrusion chamber, external thermocouple dropped 50C before loading

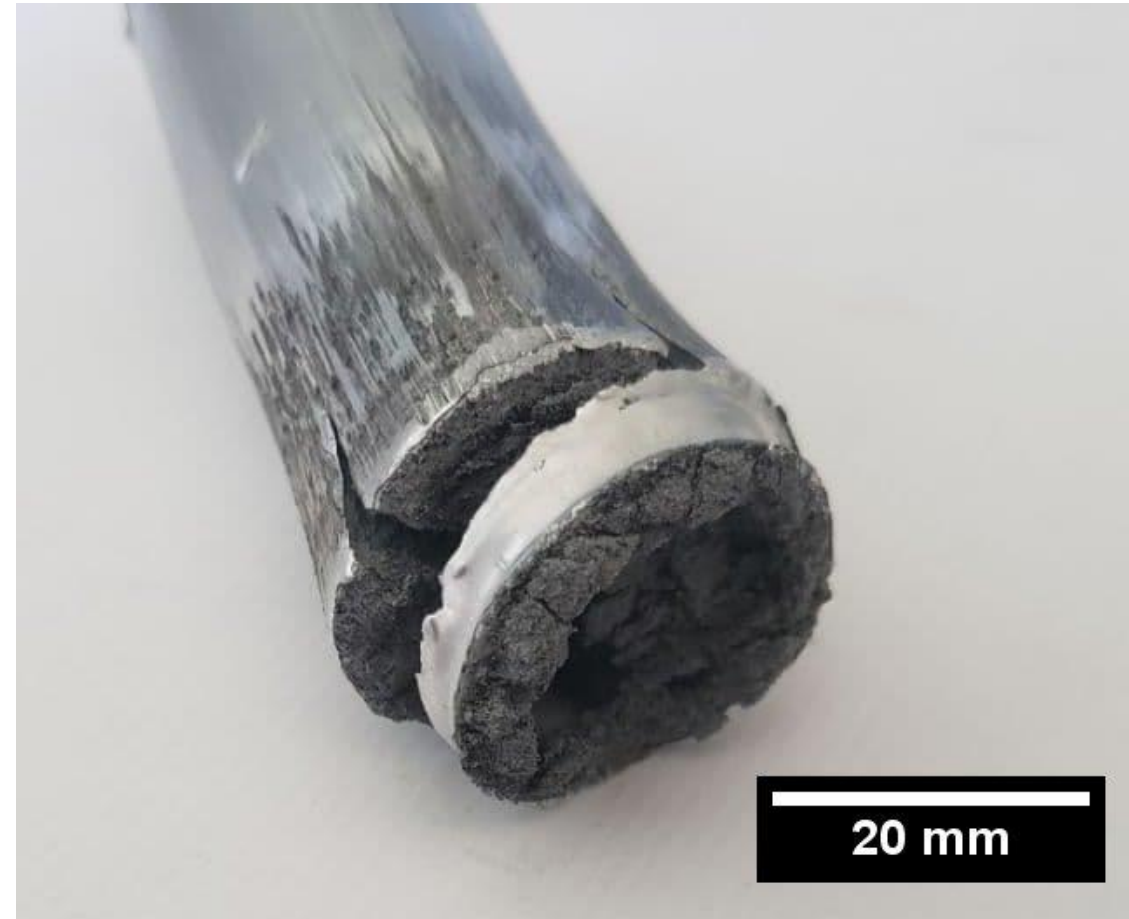
Direct Extrusion



Extrusion Start

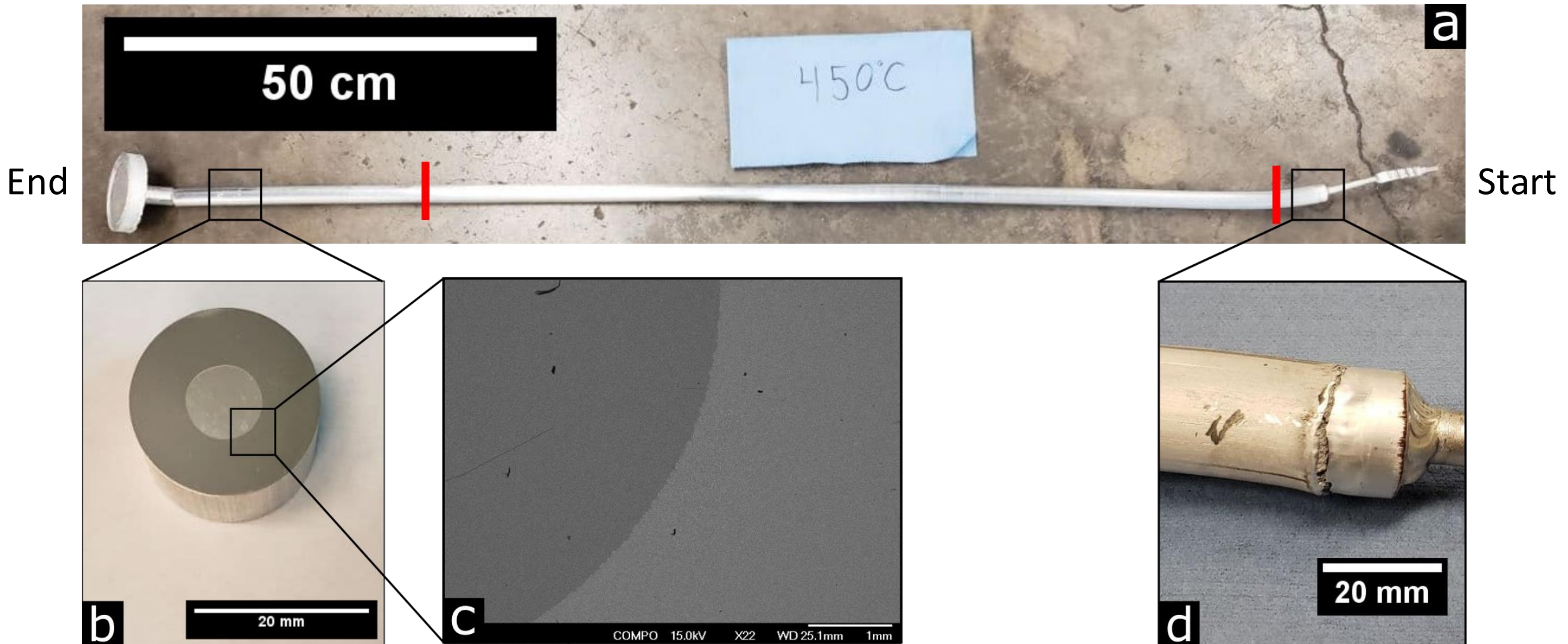


425°C extrusion temperature 16:1 ER

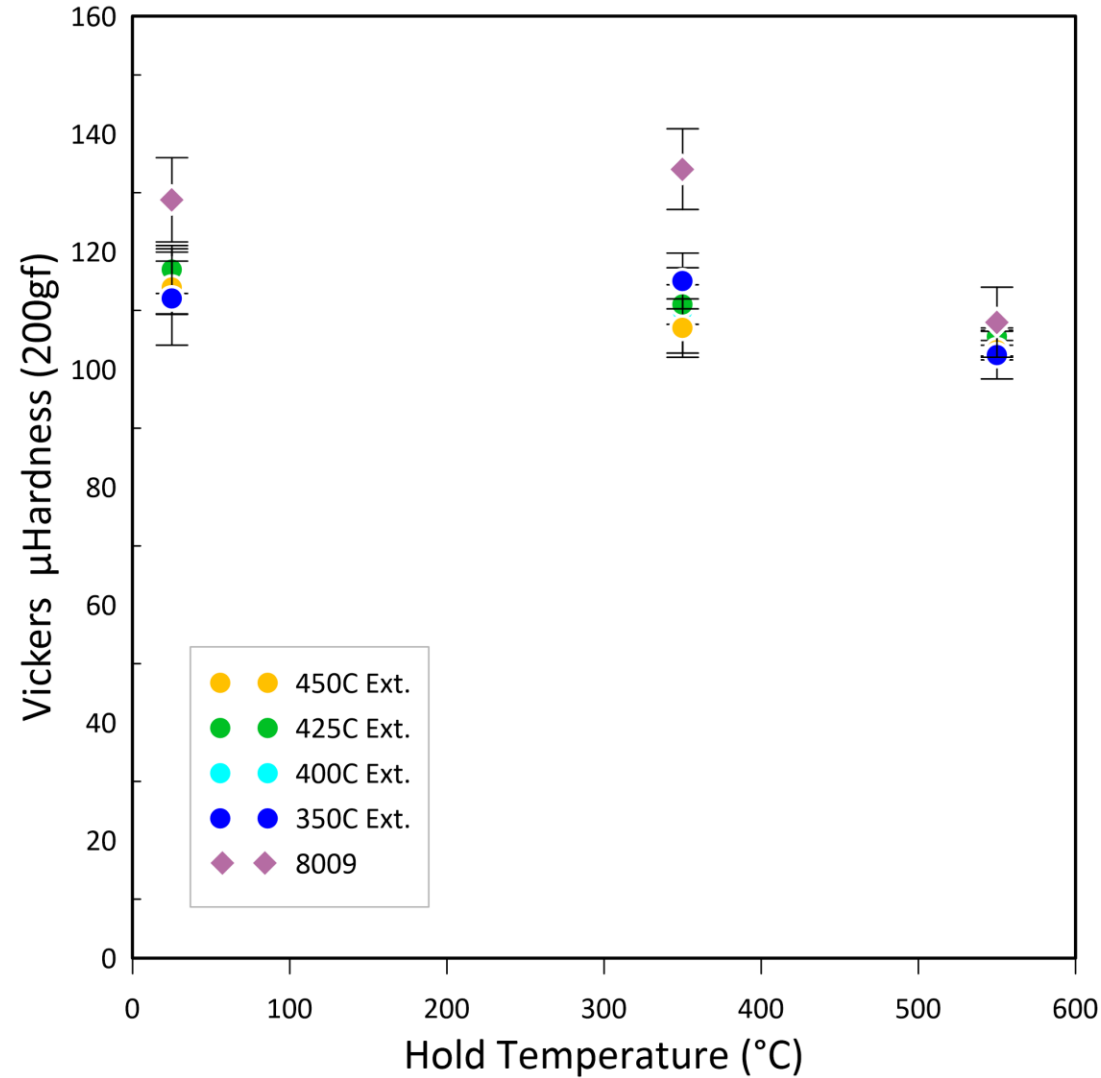


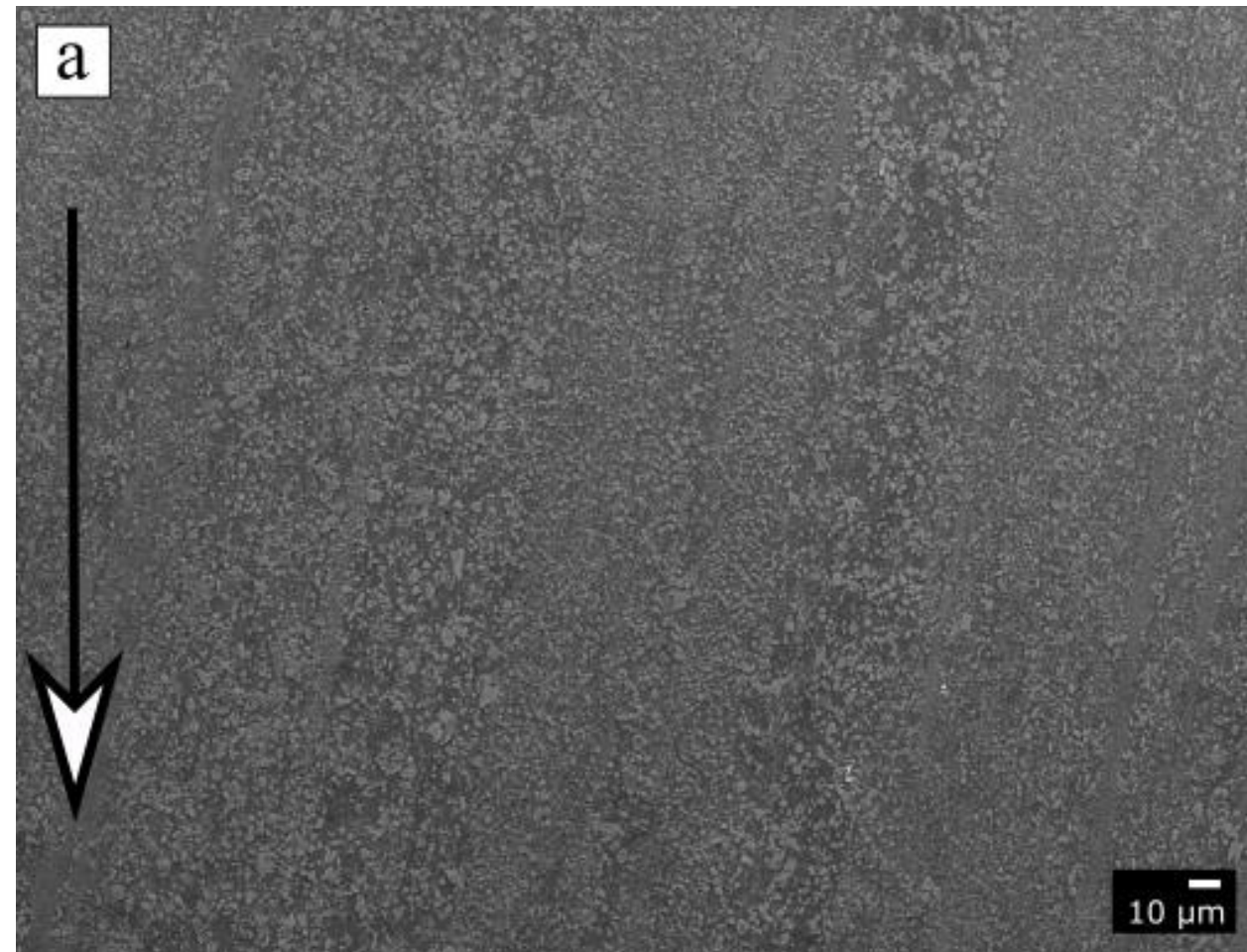
350°C extrusion temperature 10:1 ER

Sound Extrusions

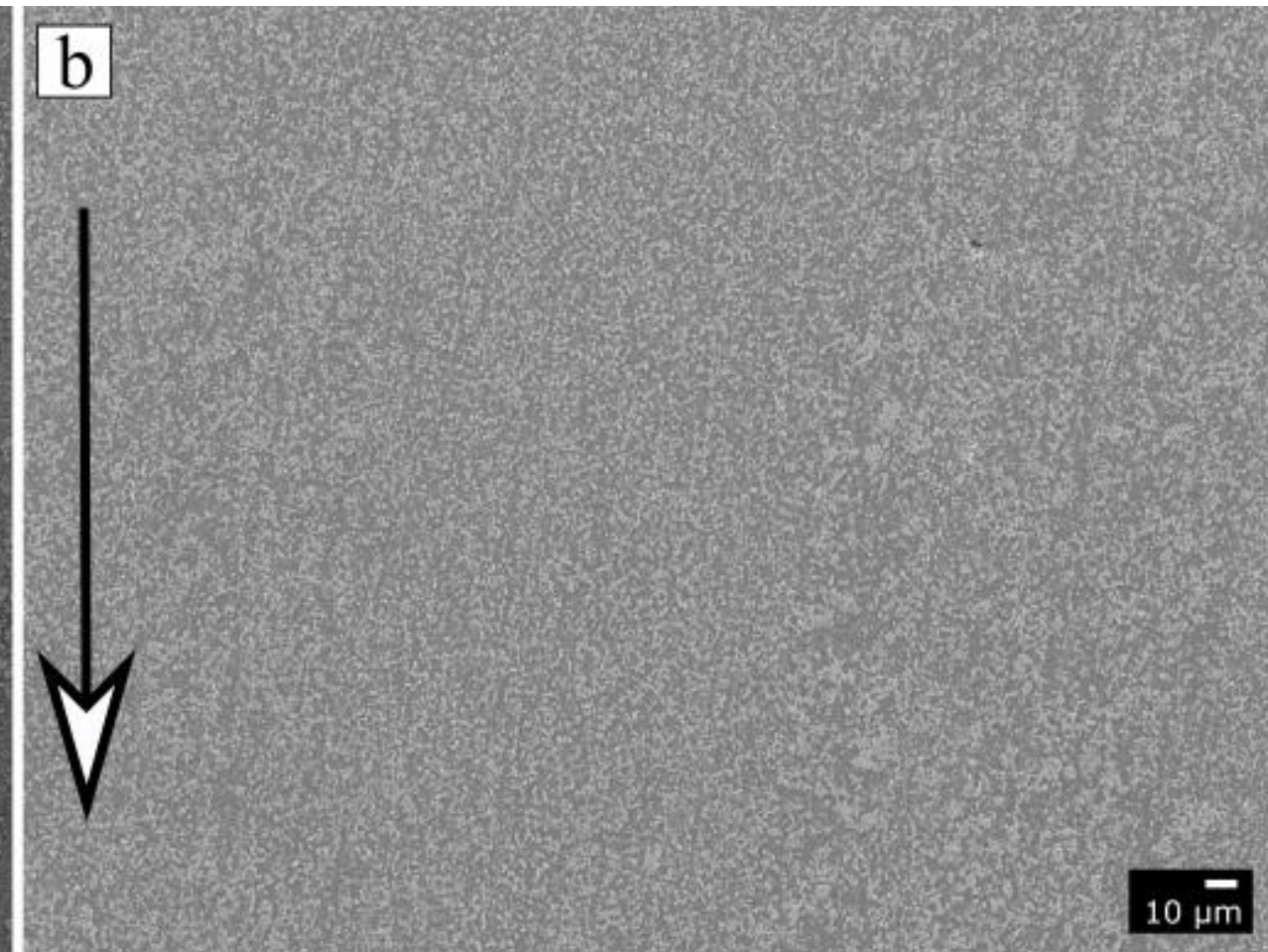


Al-TM Alloys Maintain Hardness After Annealing at 550C



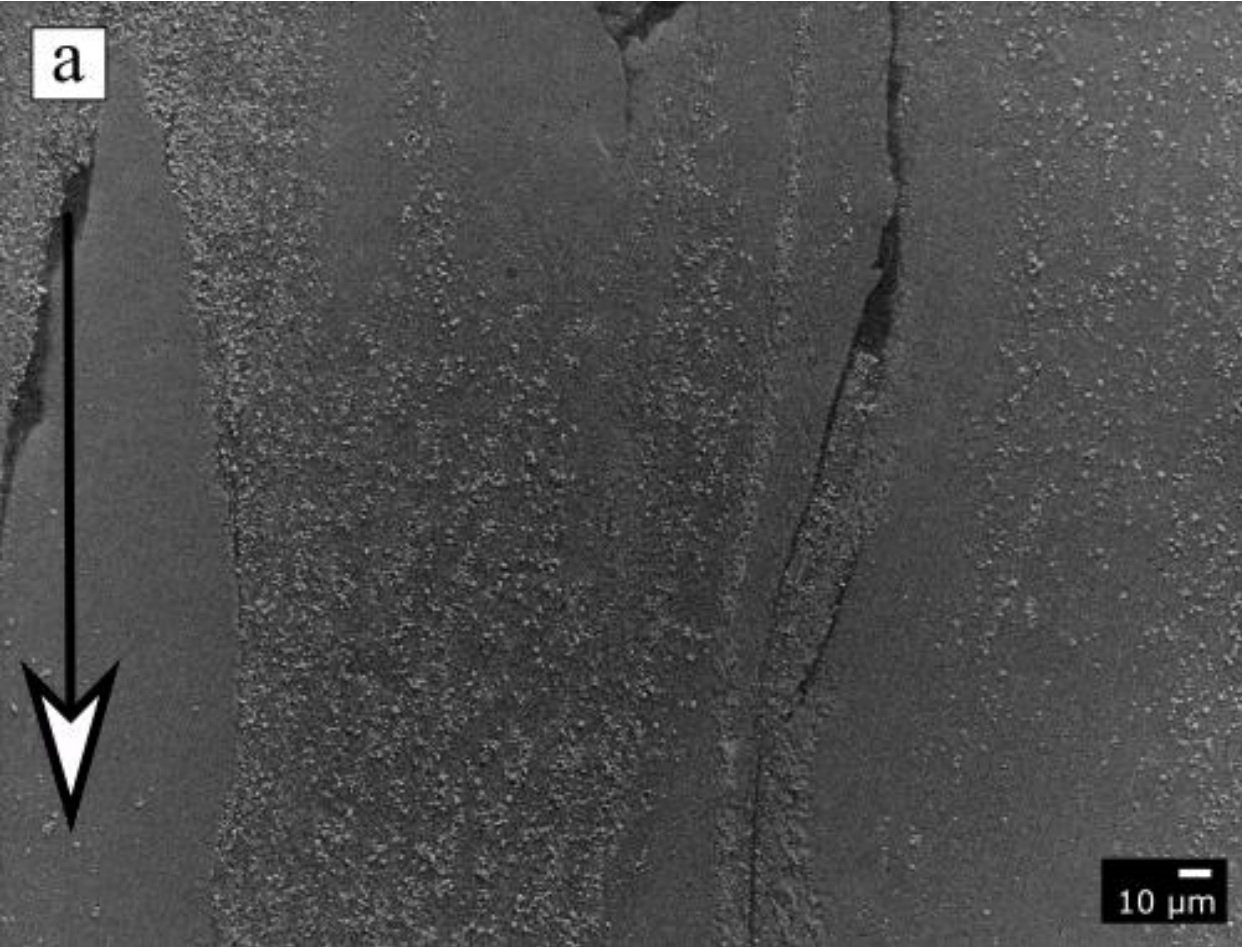


As Extruded

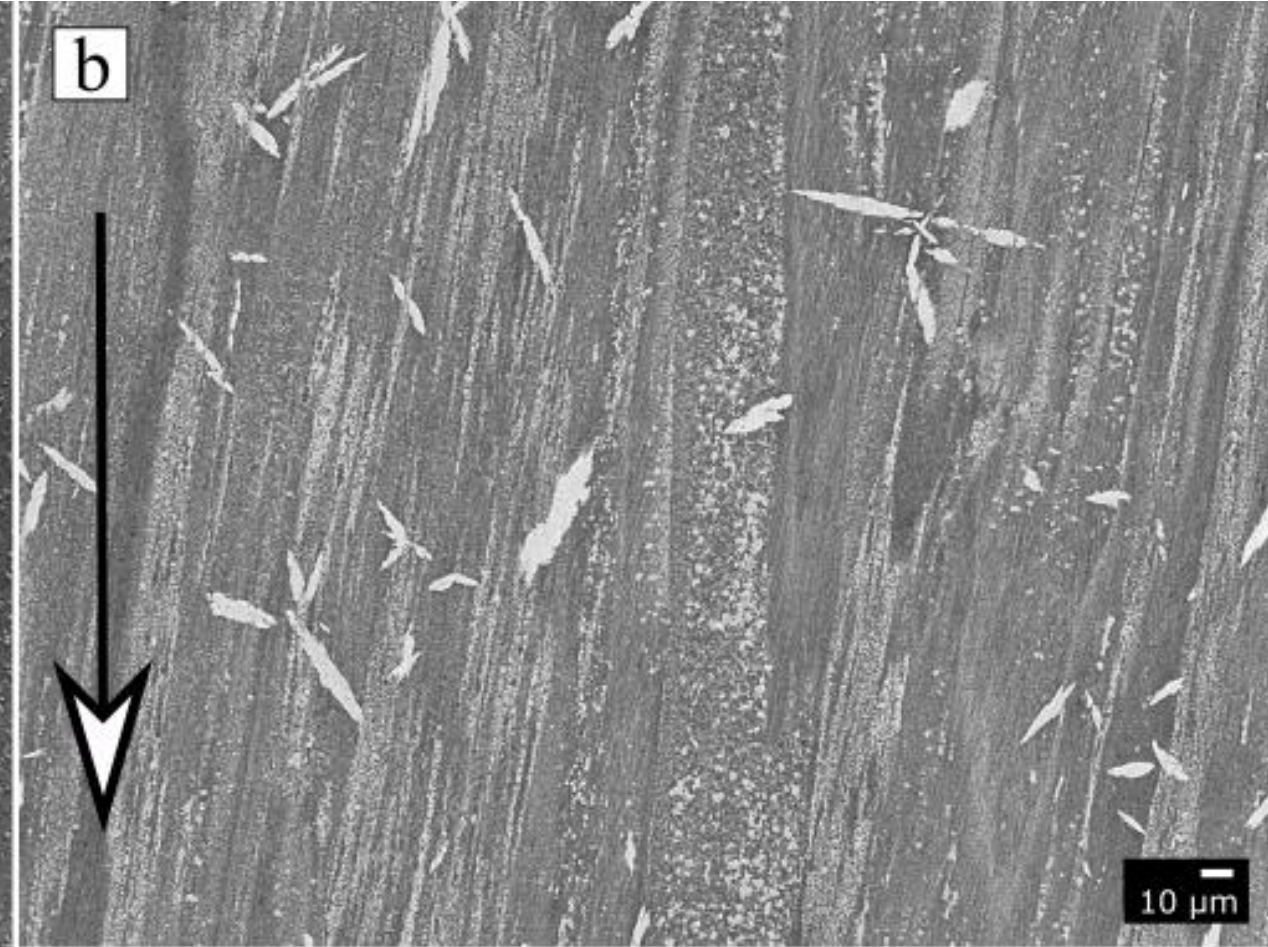


550°C hold 100 hours

8009 Secondary Phases Significantly Coarsen



As Extruded



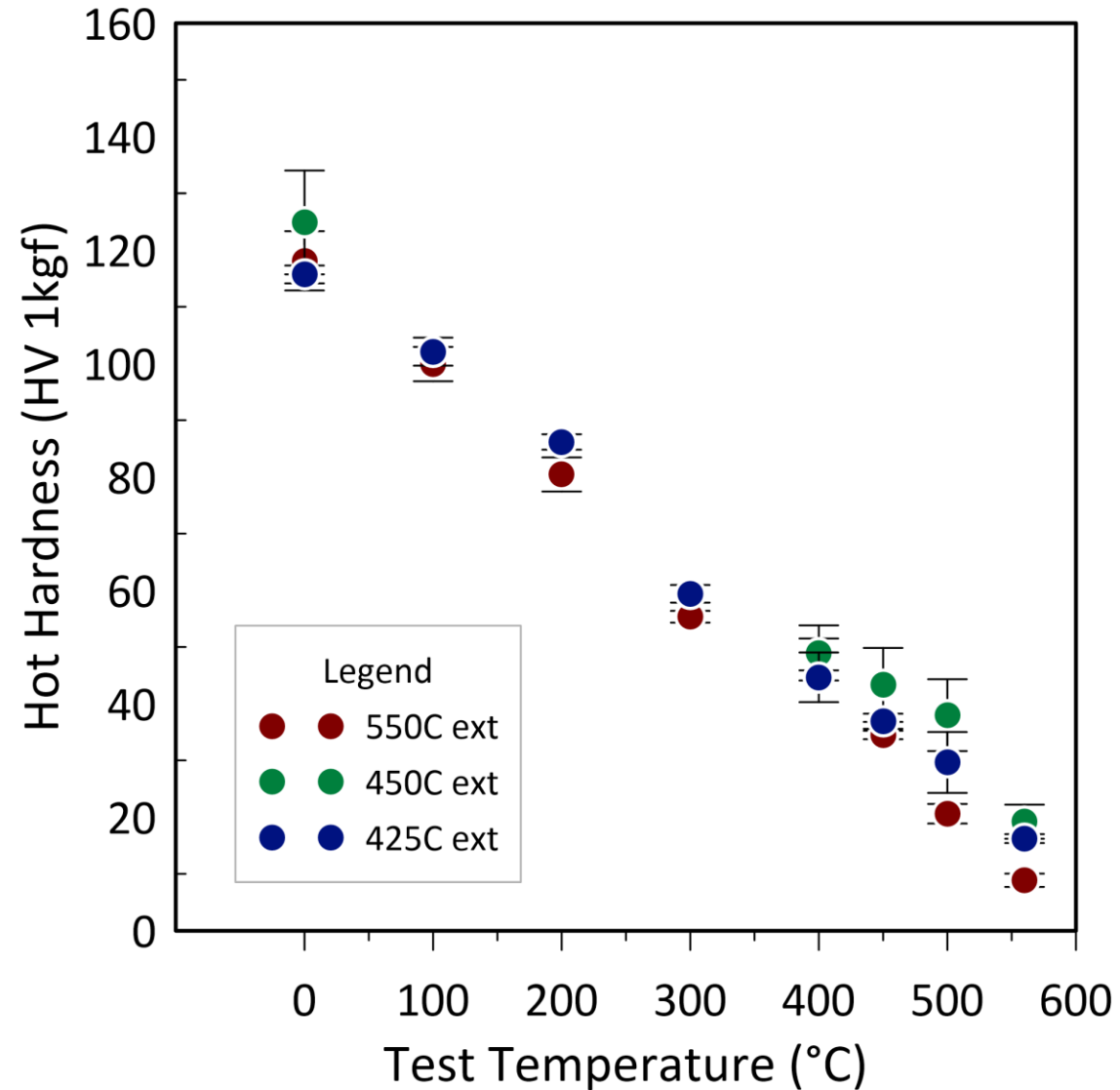
550°C hold 100 hours

Vickers Hot Hardness



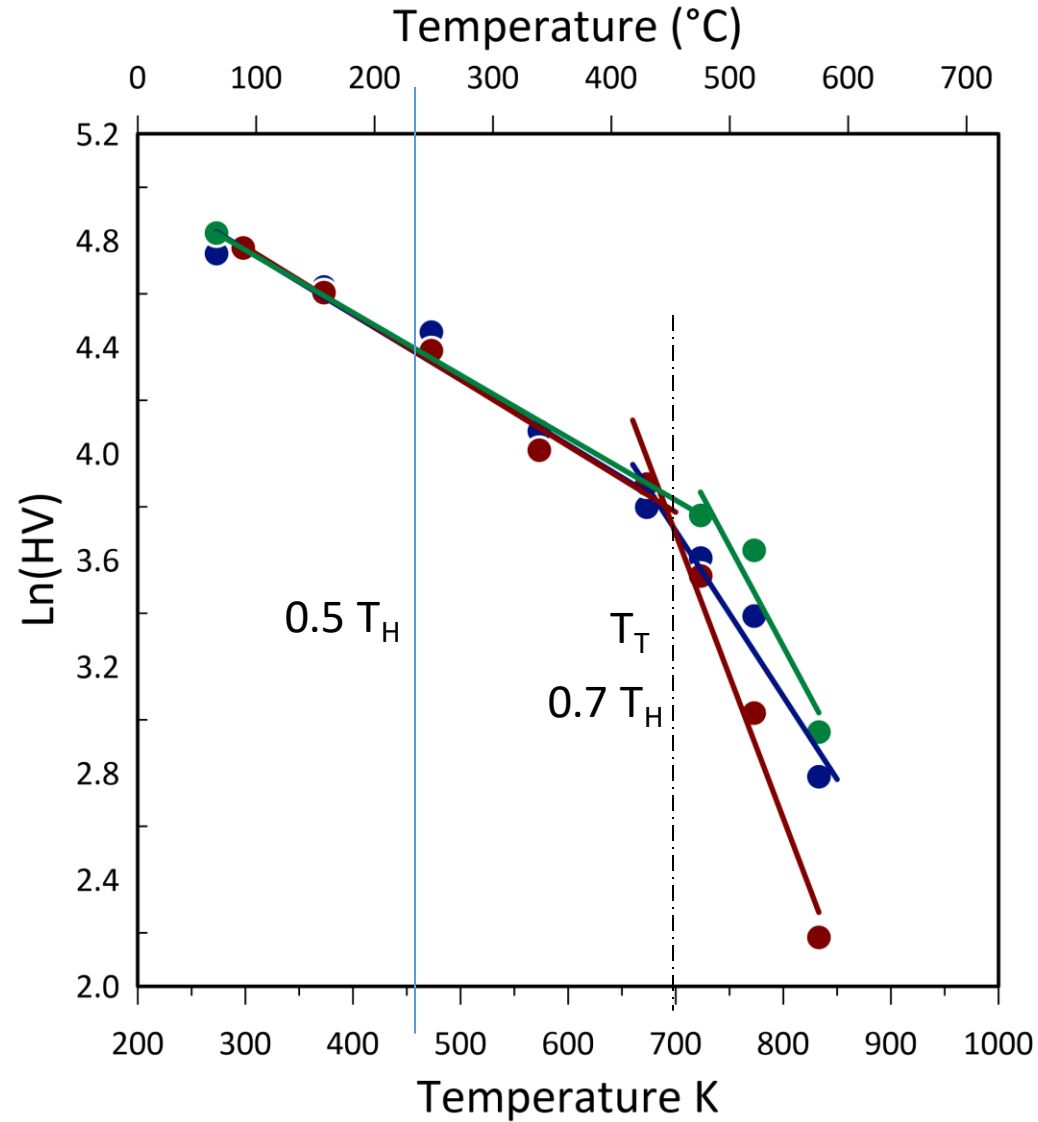
- 25°C to 600°C open atmosphere
- K-type Thermocouple attached to sample
- Sample held for 10 minutes at temperature
- Minimum of 3x indent diameter between indents and edge of sample

Hot Hardness



- Average of 10 indents
- One standard deviation

Al-TM Delays Transition to Dislocation Climb



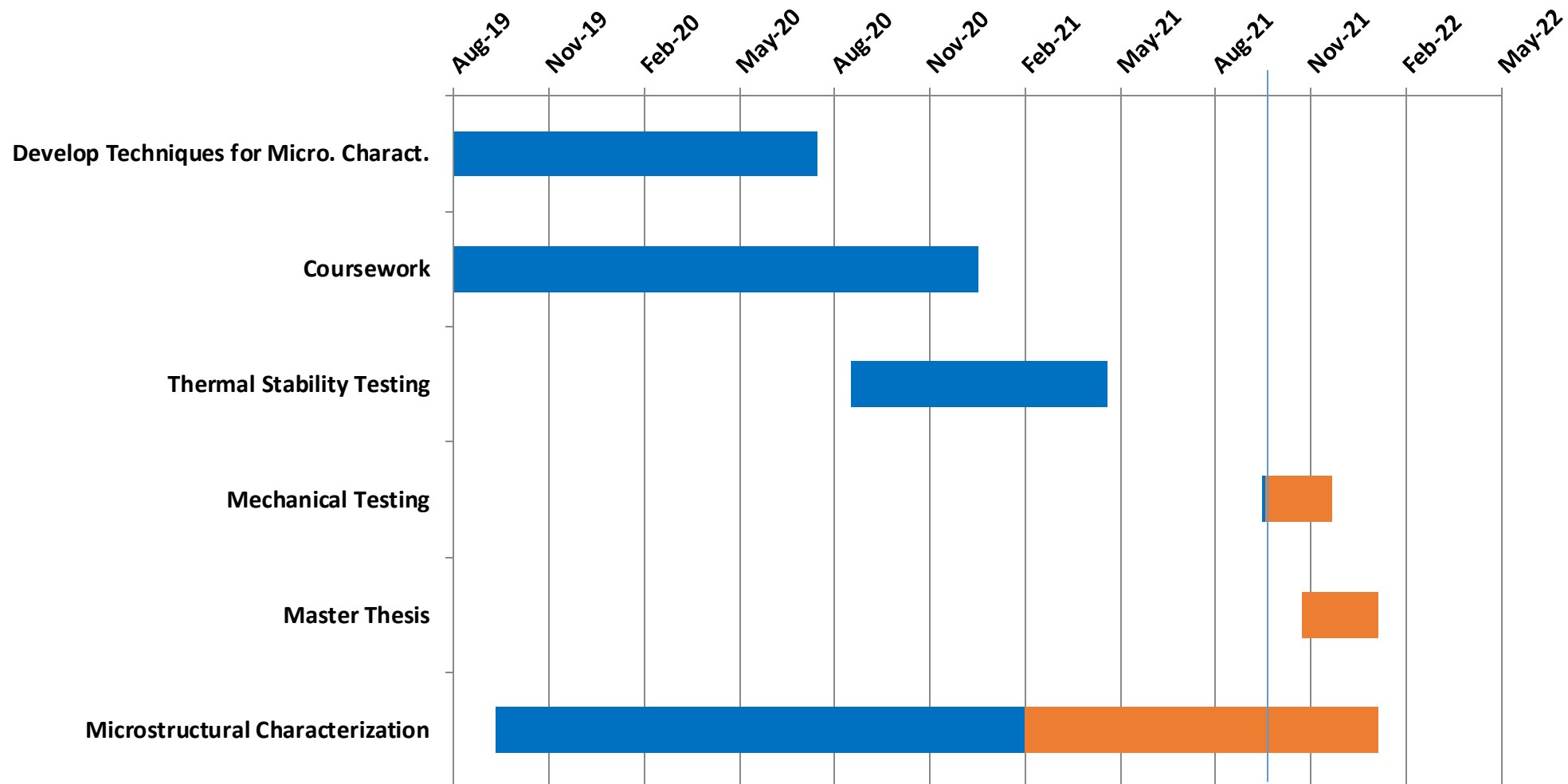
Melting temperature for Al-TM determined via DSC

Challenges & Opportunities

- Elevated temperature Tensile and Compression testing
- Thesis writing



Gantt Chart



Summary



- Extrusion
 - Successful extrusion from 350-450°C
 - Minor coarsening occurring in Al-TM alloy leading to a retention in hardness

- Hot Hardness indicates good tensile and creep properties

Acknowledgements



- Kymera International provided Al-TM extrusions and powder for this research project. Special thanks to Tom Pelletiers and Wayne Daye for technical information and project support.
- Michigan Technological University for extrusion and hot hardness testing
- Friction stir extrusion material was provided for this project by Scott Whalen, Pacific Northwest National Laboratories.
- Forgings and project support provided by Rob Mayer, Queen City Forge.
- DeformTM simulations provided by Suzanne Tkach, Tkach Metal Forming Consultants. Jim Miller & Scientific Forming Technologies for the use of DeformTM.

Questions?

Thank you!
Stuart Shirley
sshirley@mines.edu



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- Kymera International provided Al-TM extrusions and powder for this research project. Special thanks to Tom Pelletiers and Wayne Daye for technical information and project support.
- AFSD billets are to be produced by research groups at Virginia Tech under Dr. Hang Yu and University of Alabama under Dr. Paul Allison.
- Friction stir extrusion material was provided for this project by Scott Whalen, Pacific Northwest National Laboratories.
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- DeformTM simulations provided by Suzanne Tkach, Tkach Metal Forming Consultants. Jim Miller & Scientific Forming Technologies for the use of DeformTM.
- Michigan Technological University for the use of facilities and technical expertise

References



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- [4] W.K. Daye, T.W. Pelletiers II, PROPERTY DEVELOPMENT OF NEW GENERATION PM ALUMINUM MATERIALS VIA Innovative Processing, *Int. J. Powder Metall.* 54 (2018).
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References Cont.



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- [8] S. Whalen, N. Overman, V. Joshi, T. Varga, D. Gra, and C. Lavender, “Magnesium alloy ZK60 tubing made by Shear Assisted Processing and Extrusion (ShAPE),” *Mater. Sci. Eng. A*, vol. 755, no. March, pp. 278–288, 2019, doi: 10.1016/j.msea.2019.04.013.
- [9] X. Li et al., “Microstructure and Mechanical Properties of Pure Copper Wire Produced by Shear Assisted Processing and Extrusion,” *JOM*, vol. 71, no. 12, pp. 4799–4805, 2019, doi: 10.1007/s11837-019-03752-w.

Thermal Stability

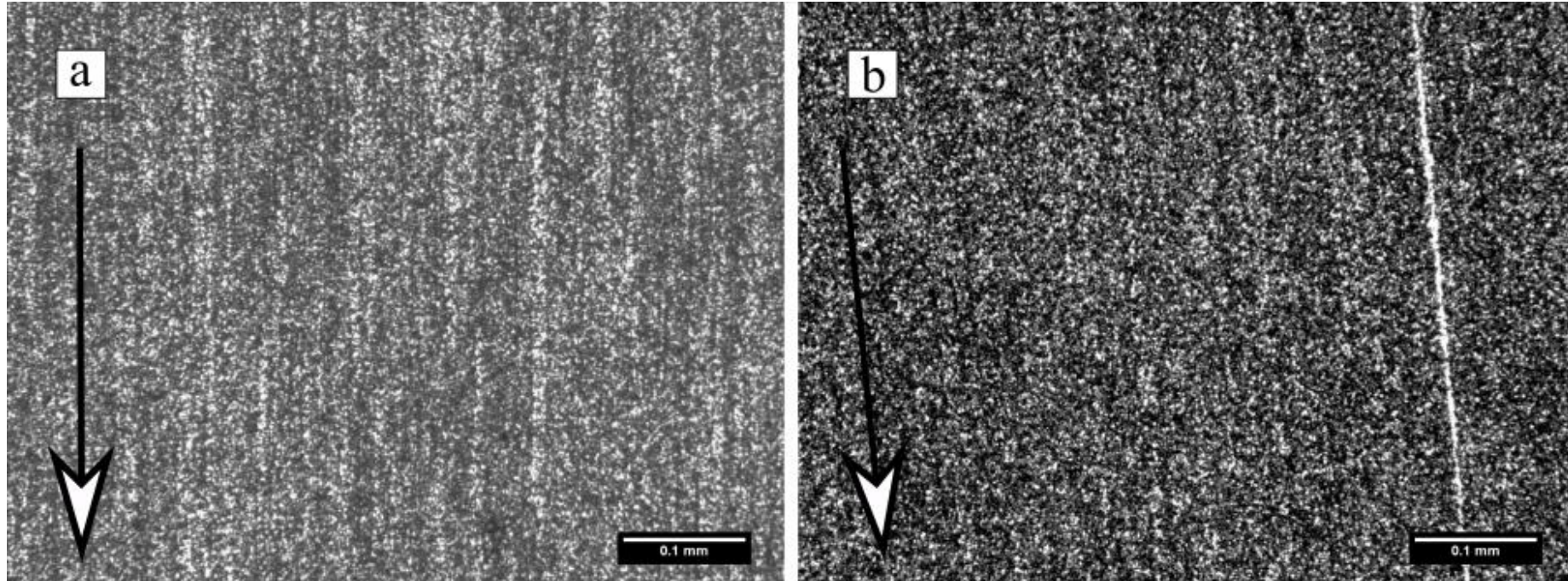
- 1 hour thermal stability test

TEMP FOR 1HR (°C)	200	300	350	400	450	500	550
EXTRUDED	[Green bar]						

- 100 hour long time treatment

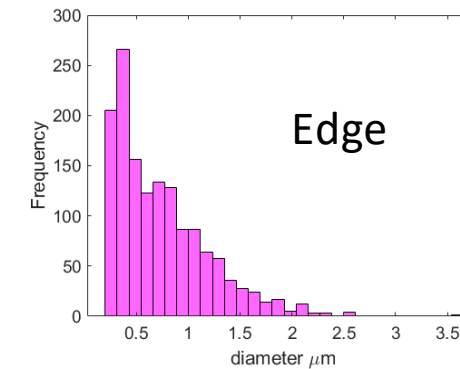
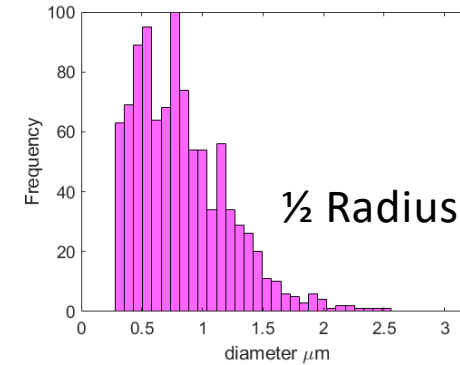
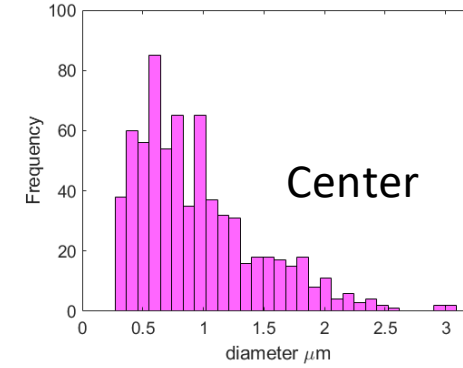
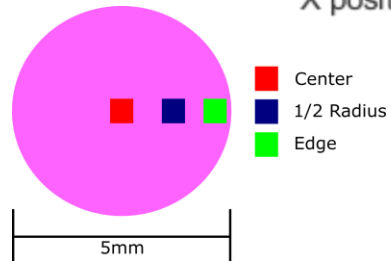
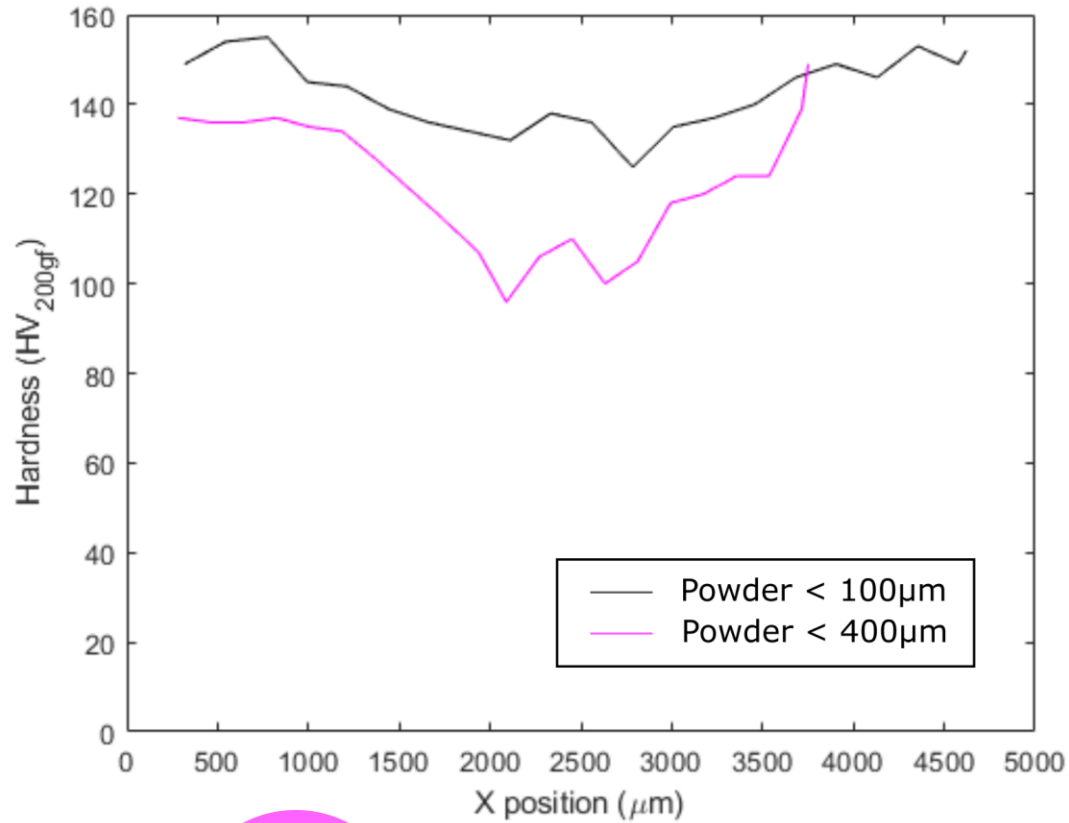
TEMP FOR 100HR (°C)	200	300	400	500
EXTRUDED	[Green bar]			

Thermal Stability Microstructure



Light optical microscope image of 17:1 extrusion etched with Keller's reagent; extrusion direction indicated with arrow. A) Microstructure of the extrusion in the as received condition. B) Microstructure of the extrusion after 100 hours at 500°C showing little change in grain size or morphology. Scale bar 0.1mm

FSE hardness



Direct Extrusion Conditions



Temperature (°C)	Extrusion Ratio	Tension (quasi-static)	Compression
350	10:1	25C,300C	$\dot{\epsilon}$:0.01,0.1,1,10 Temp: 400°C-550°C
400	16:1		$\dot{\epsilon}$:0.01,0.1,1,10 Temp: 400°C-550°C
400, 350 pre compact	16:1	Temp:25°C*not enough material	Could make a few compression
425	16:1	Temp:25°C,200°C,300°C,400°C,500°C	
450	16:1	Temp:25°C,350°C,450°C	
550	25:1	Temp:25°C,200°C,300°C,400°C,500°C	$\dot{\epsilon}$:0.01,0.1,1,10 Temp: 400°C-550°C