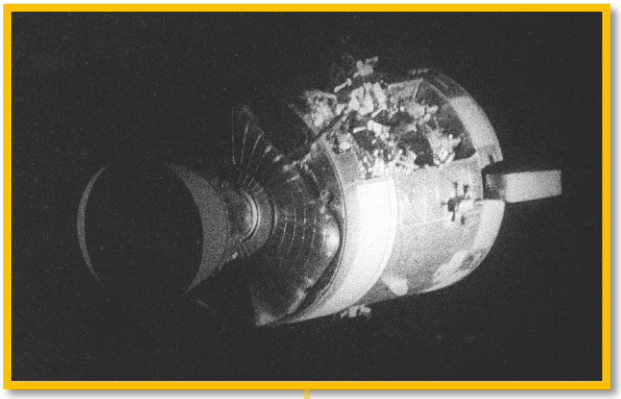
Update: Evaluation of Additively Manufactured Metals for Use in Oxygen Systems

Presented By: Jonathan Tylka

NASA Johnson Space Center

White Sands Test Facility

Aerospace Fire History



1965 | 1975 | 1985 | 1995 | 2005 | 2015

Apollo 1
1/27/1967

Apollo 13
4/14/1970

The EMU Fire
9/15/1980

MIR Fire
2/24/1997

Cygnus CRS Orb-3
10/28/2014

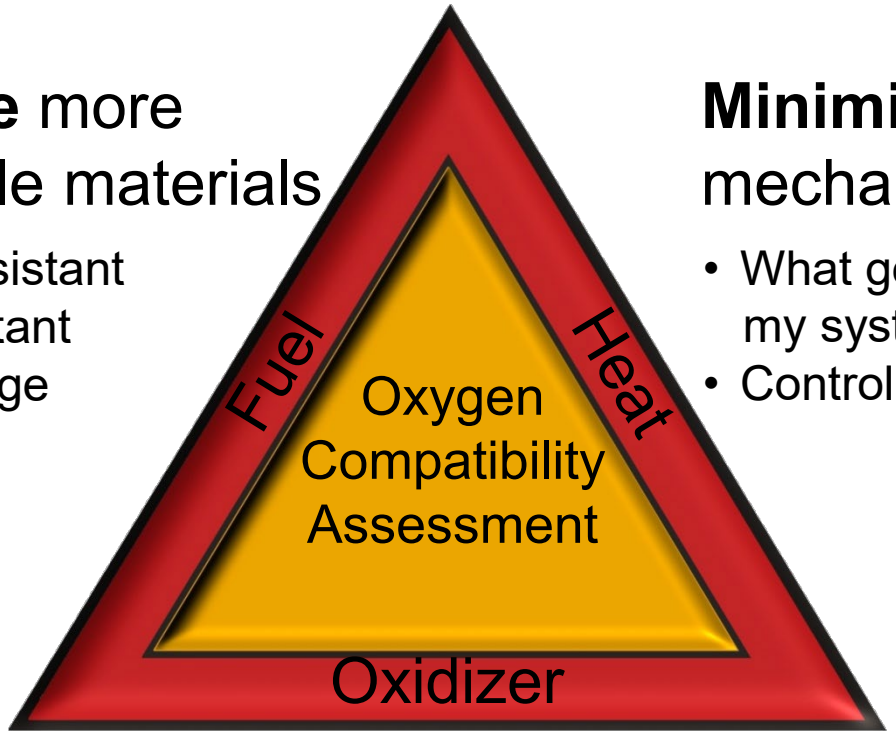
Oxygen Compatibility

- Additive Manufacturing (AM) is currently and will continue to be, used in oxygen systems
- Compatibility studies are a necessity
- Risks if not pursued
  - Equipment Damage, Loss of Mission, Loss of Life
- NASA Centers of Excellence leading efforts
  - White Sands Test Facility (WSTF)
    - Oxygen Compatibility Testing
  - Marshall Space Flight Center (MSFC)
    - Additive Manufacturing
  - Glenn Research Center
    - Metals characterization
  - NASA Engineering Safety Center (NESC)
    - Statistical Design of Experiments

We must manage the risks…

Maximize more compatible materials

- Ignition resistant
- Burn resistant
- Low damage potential



Minimize ignition mechanisms

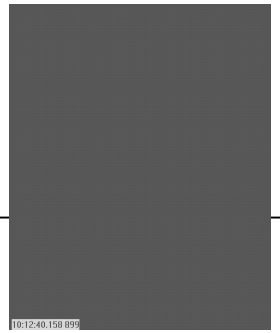
- What generates heat in my system?
- Control or eliminate

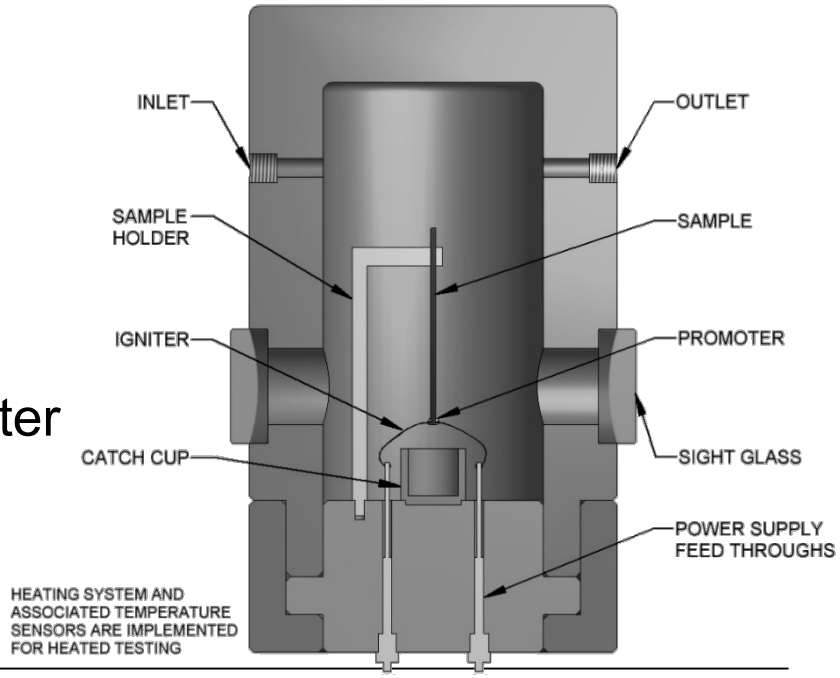
Utilize good practices

- Implement all aspects of oxygen system safety

Maximize

- Testing determines AM flammability performance
  - Note: Flammability is configurationally dependent, not a material property.
- NASA-STD-6001B Test 17/ ASTM G124
  - Upward flammability test
  - 1/8-in. diameter x 6-in. long
  - Unheated
  - Static Pressure
  - >99.5% Oxygen
  - Magnesium/Pyrofuse Promoter

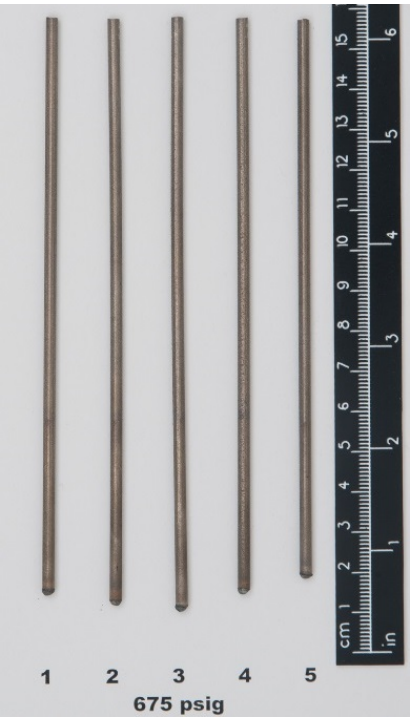




Preliminary Flammability Testing

- Experiment conducted between:
  - Wrought Inconel 718
  - Selective Laser Melting (SLM) Inconel 718 (IN718)
- Statistically designed, efficient, and randomized
- Test specimens manufactured at MSFC
- Material flammability differences noted
  - Result statistically significant but counterintuitive
- SLM IN718 post-build processes need investigation
  - Stress relief (SR)
  - Hot isostatic pressing (HIP)
  - Solutionizing and aging heat treatments (HT)

Preliminary Flammability Results



Printed, HT, no HIP

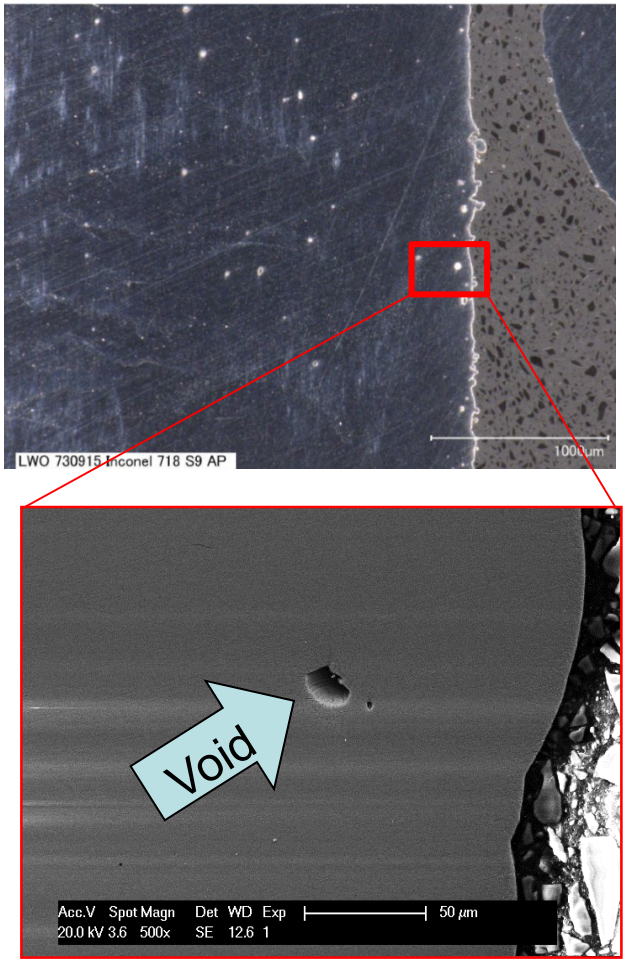
Printed, HIP, and HT

Wrought and HT

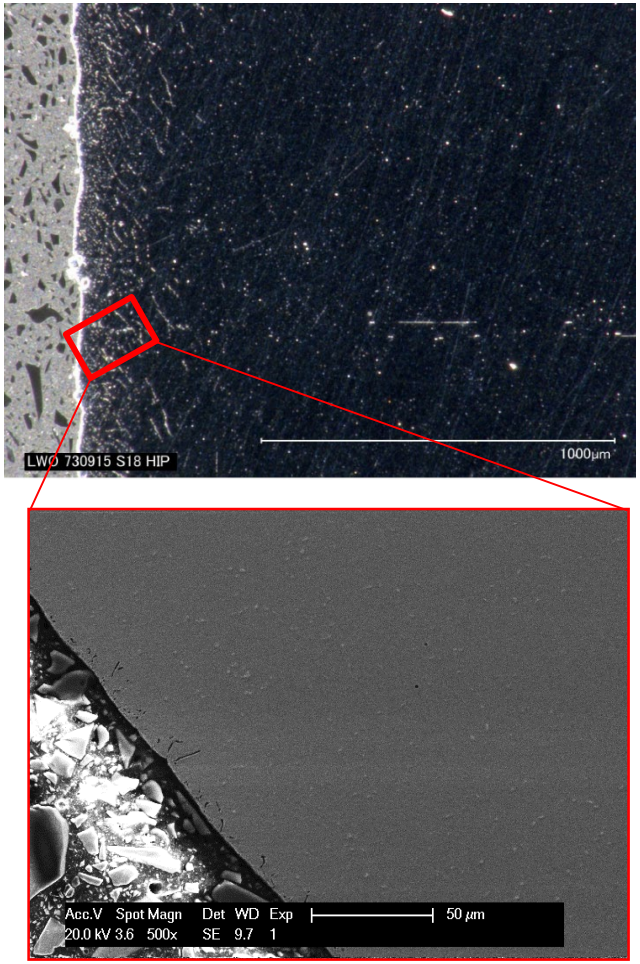
- SLM IN718 with/without HIP vs Wrought
- All materials had AMS 5664 HT

Various Nb Precipitate Formation

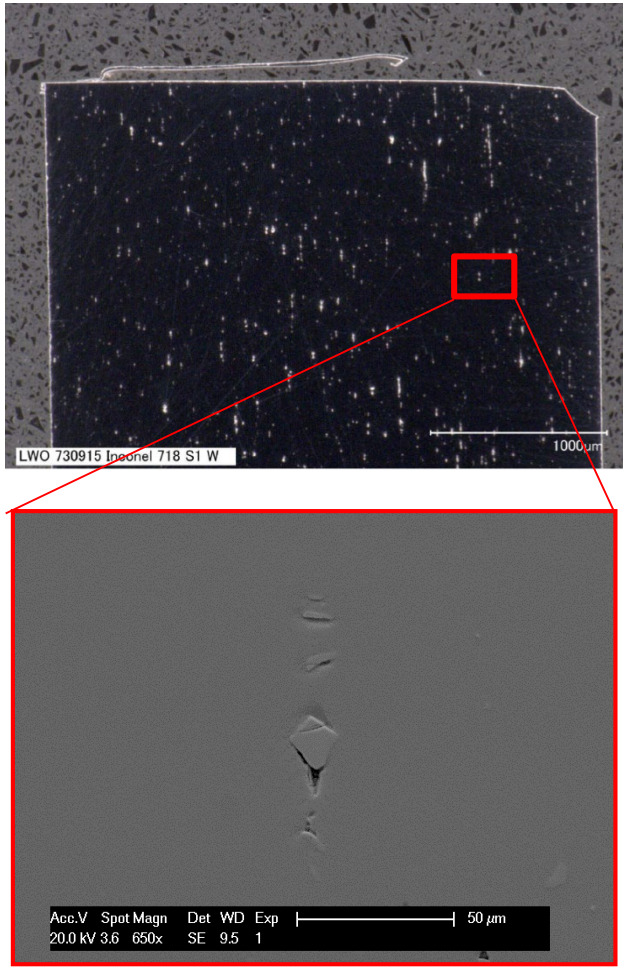
As-Printed/HT



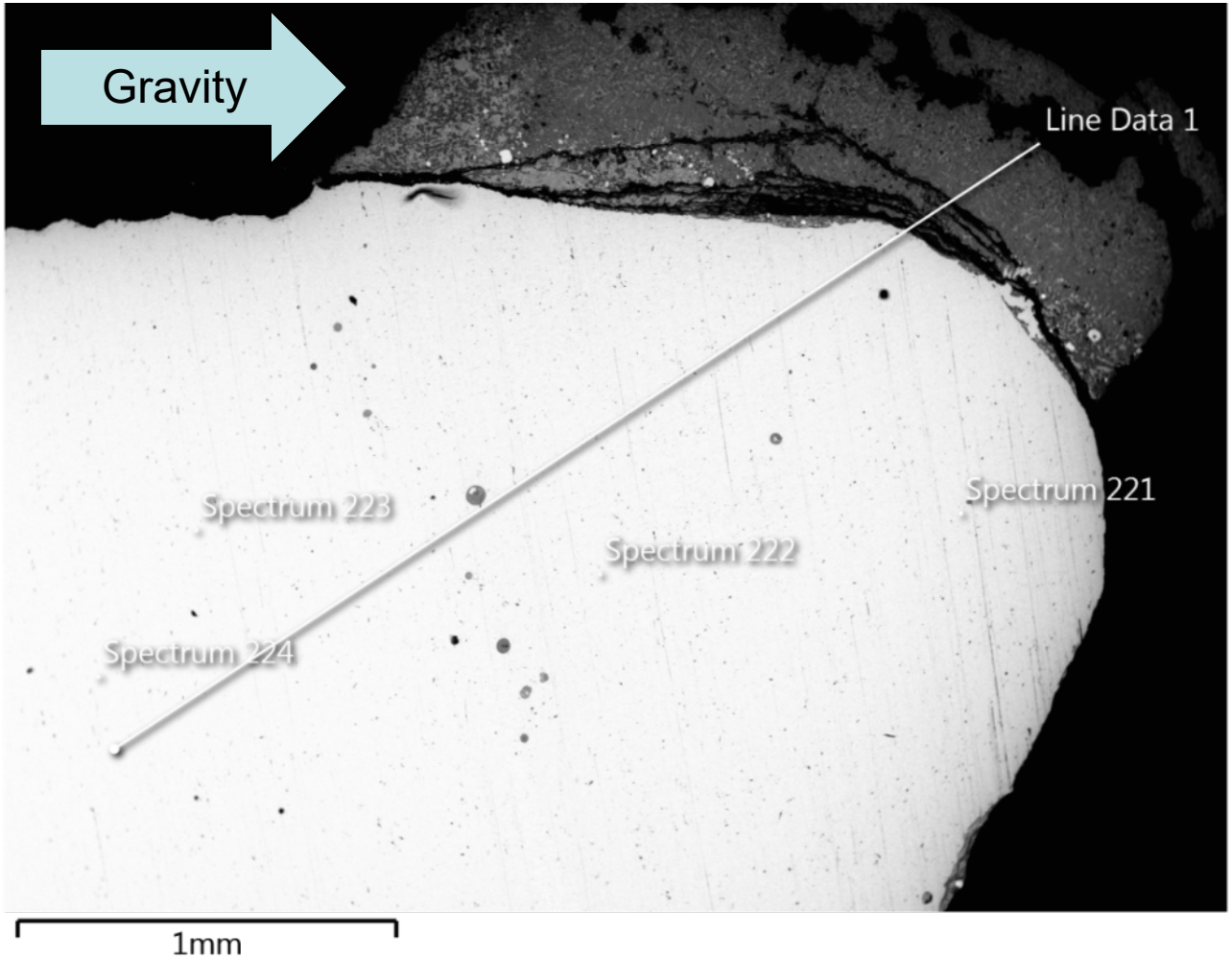
HIP/HT



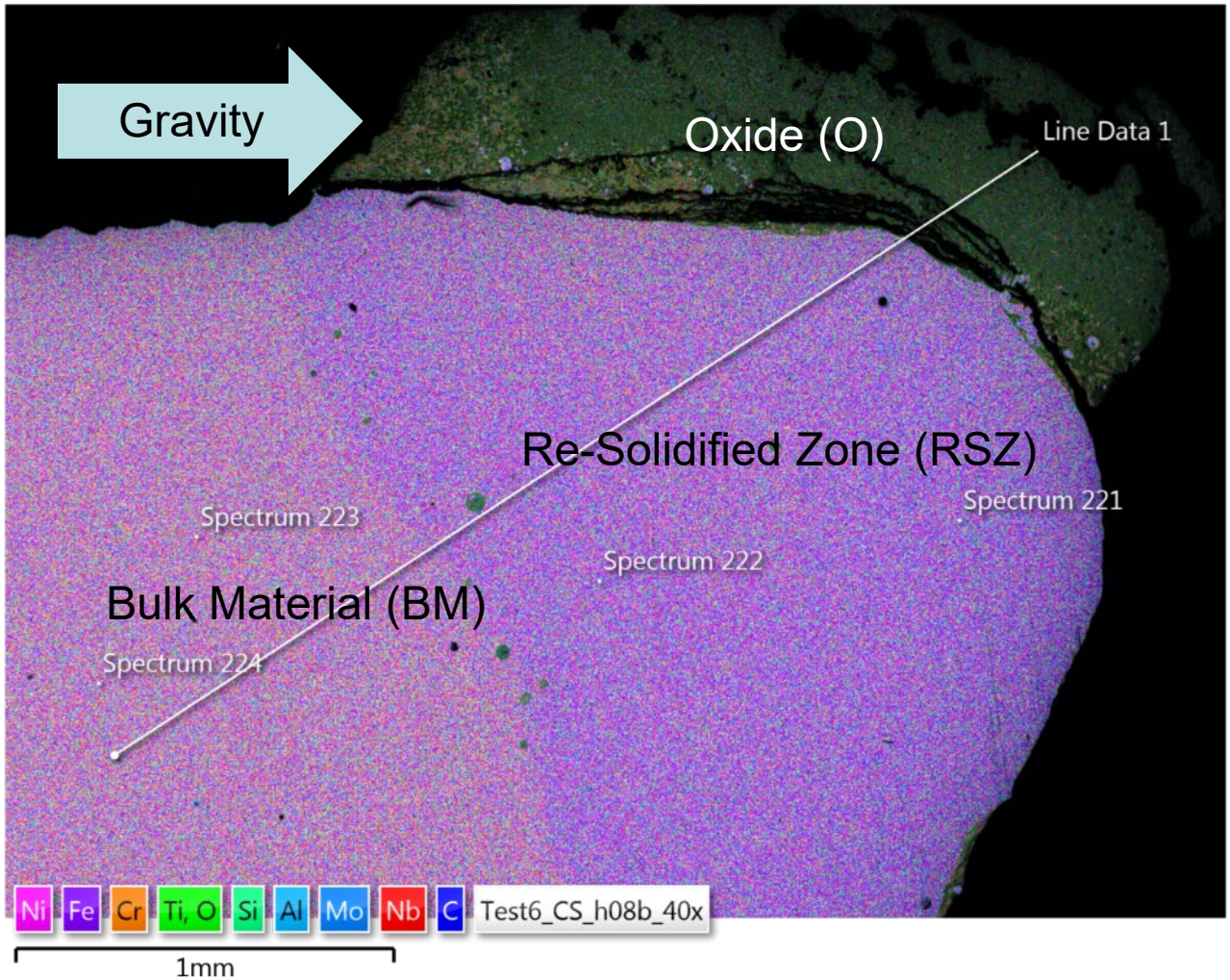
Wrought/HT



Axial Burning Interface of HIP Sample

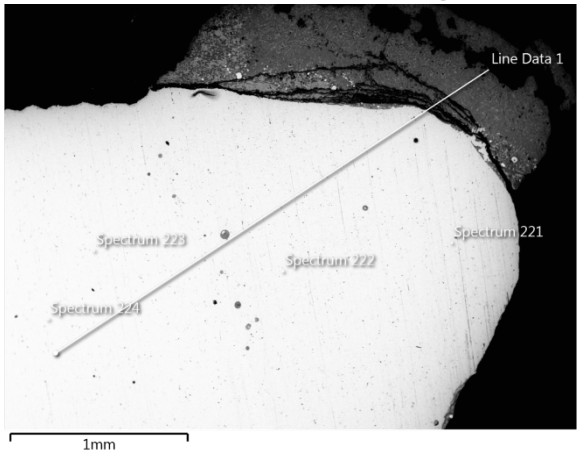


Composite Energy-Dispersive Spectroscopy (EDS)

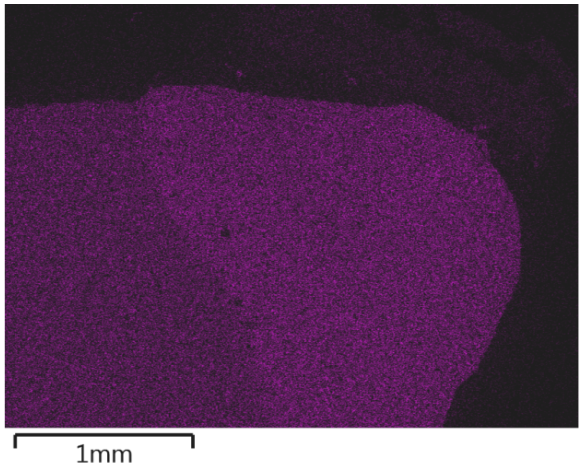


EDS Mapping of Individual Elements

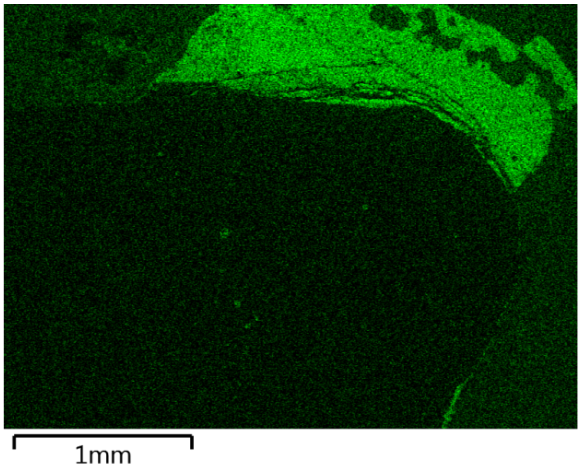
Reference Image



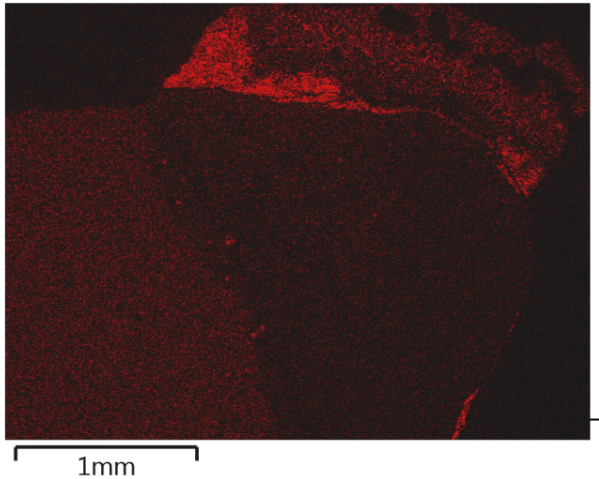
Ni



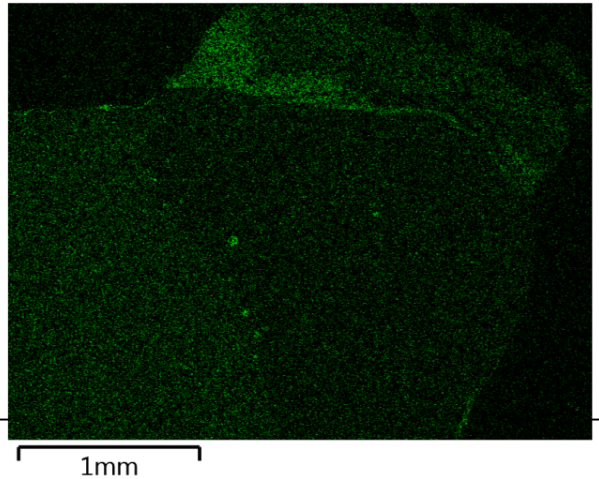
O



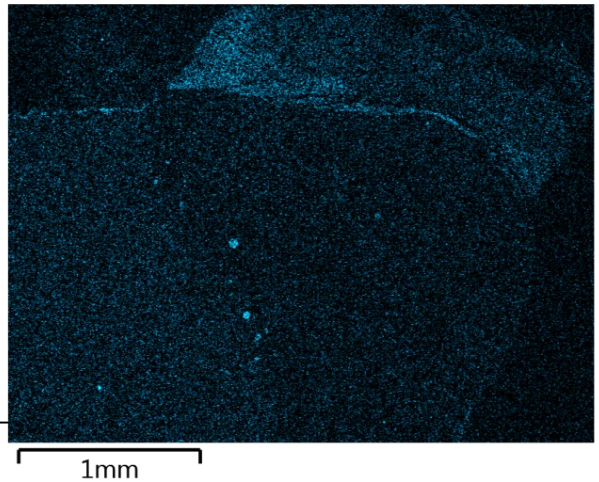
Nb



Ti

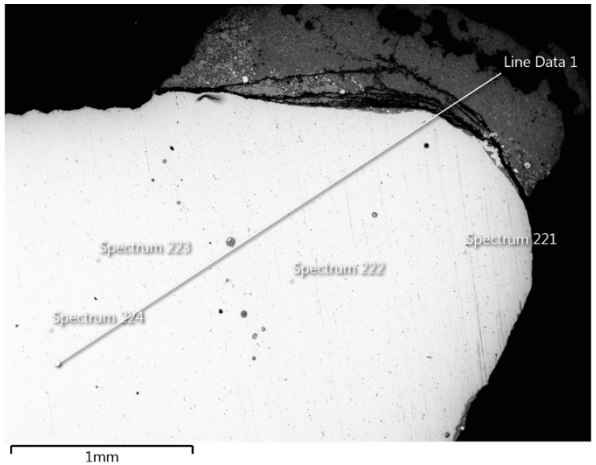


Al

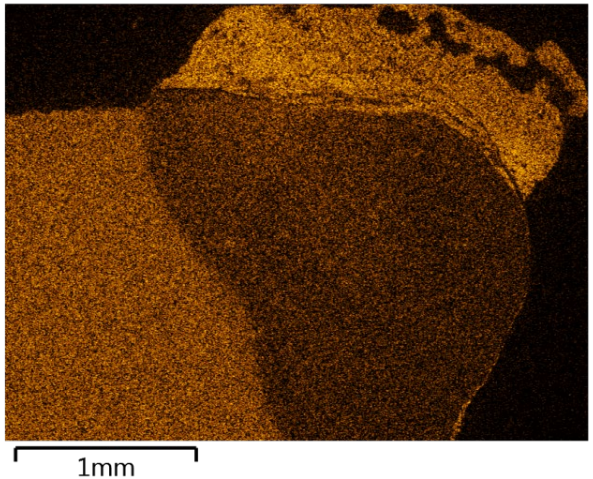


EDS Mapping of Individual Elements

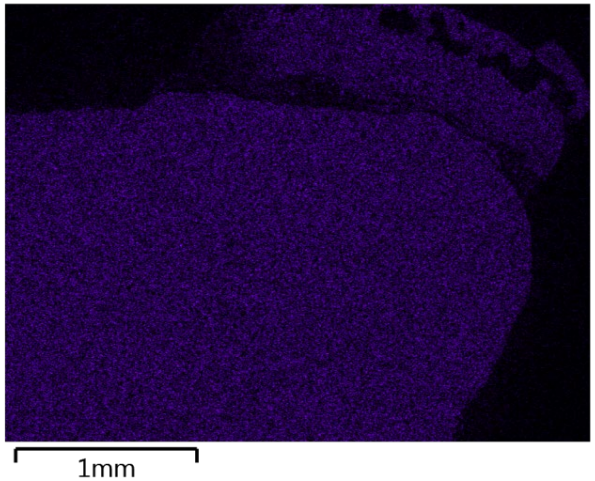
Reference Image



Cr



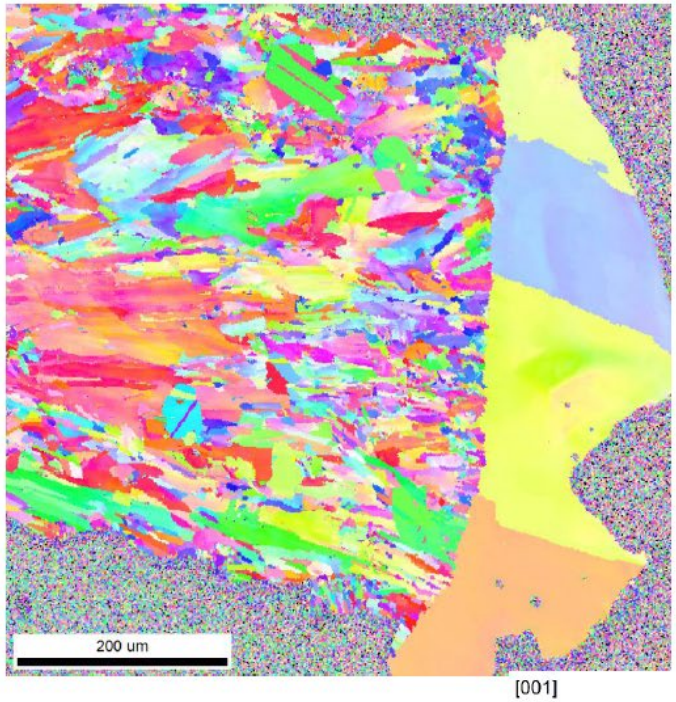
Fe



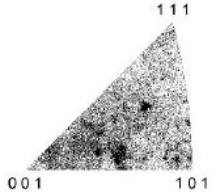
- Scavenging of flammable constituents in RSZ
  - Cr, Al, Ti, Nb (interesting segregation)
- Concentration of non/less flammable constituents in RSZ
  - Ni
- Fe remained distributed in BM, RSZ, and O Zones

EBSD – Burn Area

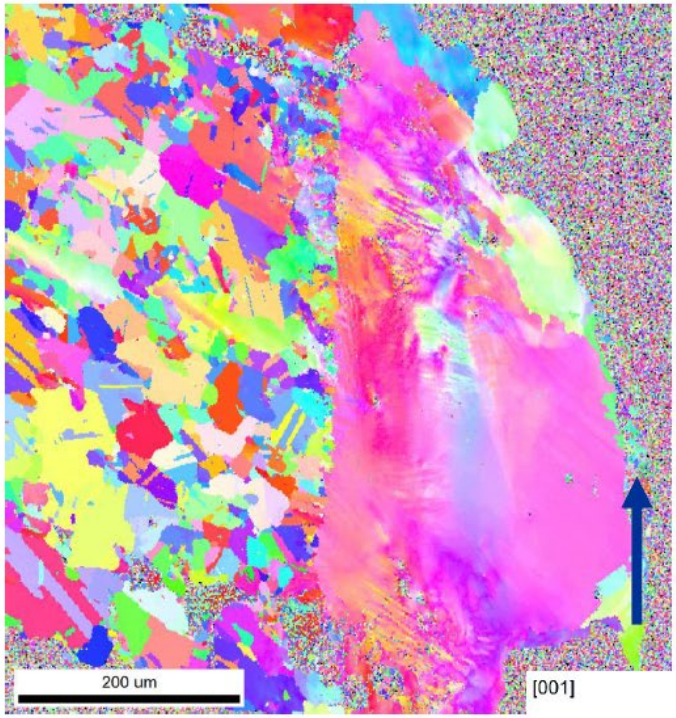
V1-1 (Printed w/SR)



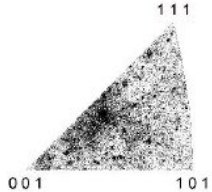
Recrystallization near melt area



V2-9 (HIP)



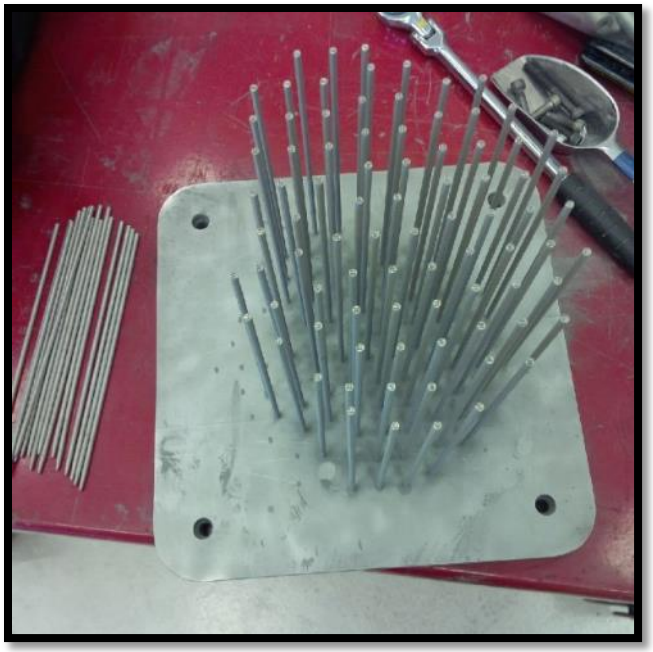
Microstructure stays same up to melt area



Analysis: Tim Smith- GRC

Flammability Study - Ongoing

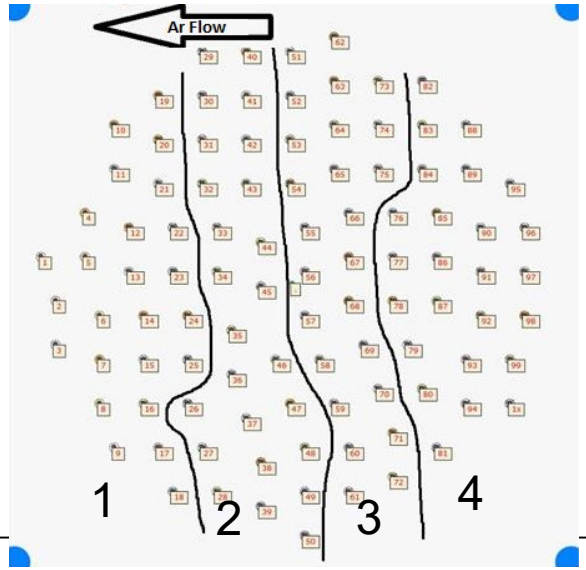
- SLM IN718
- Replicate and expand experiment
- Print parts in same build
- Synchronously SR and HT
- Factors
  - HIP (with/without)
  - Effect of HIP temperature excursion
    - Performed in vacuum furnace
    - Furnace cool vs. quench
  - AMS 5664 HT (with/without)
  - Location on build plate

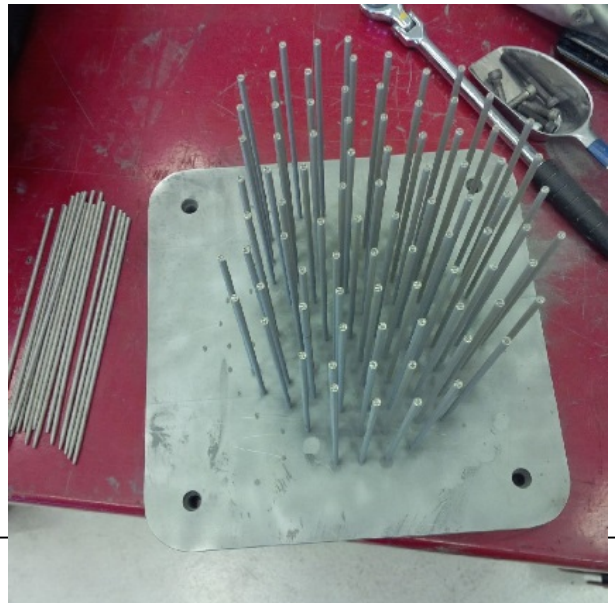


FY16 Matrix

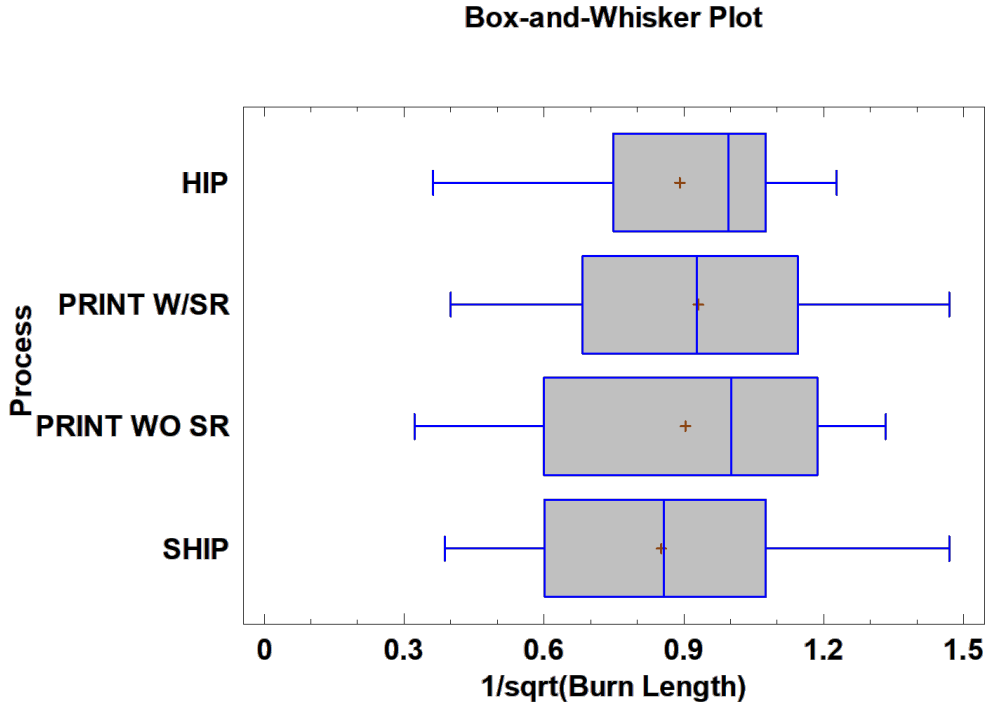
Process	Cooling Rate From Process	Heat Treatment	Sample Numbers
Printing	N/A	None	13,25,36,37,52,58,80,91
Printing	N/A	AMS 5664 (Sol/Age)	20,21,30,45,63,72,78,95
Hot Isostatic Pressing	Furnace Cool	None	12,16,39,50,53,62,79,84
Hot Isostatic Pressing	Furnace Cool	AMS 5664 (Sol/Age)	18,23,46,49,56,60,81,85
Vacuum HIP (HIP Heating profile no pressure)	Furnace Cool	None	3,8,32,47,57,64,94,98
Vacuum HIP (HIP Heating profile no pressure)	Furnace Cool	AMS 5664 (Sol/Age)	19,24,44,48,74,75,76,92
Vacuum HIP (HIP Heating profile no pressure)	Gas Quench	None	1,4,29,33,59,61,83,87
Vacuum HIP (HIP Heating profile no pressure)	Gas Quench	AMS 5664 (Sol/Age)	15,17,26,35,55,71,90,100

Test Samples Build





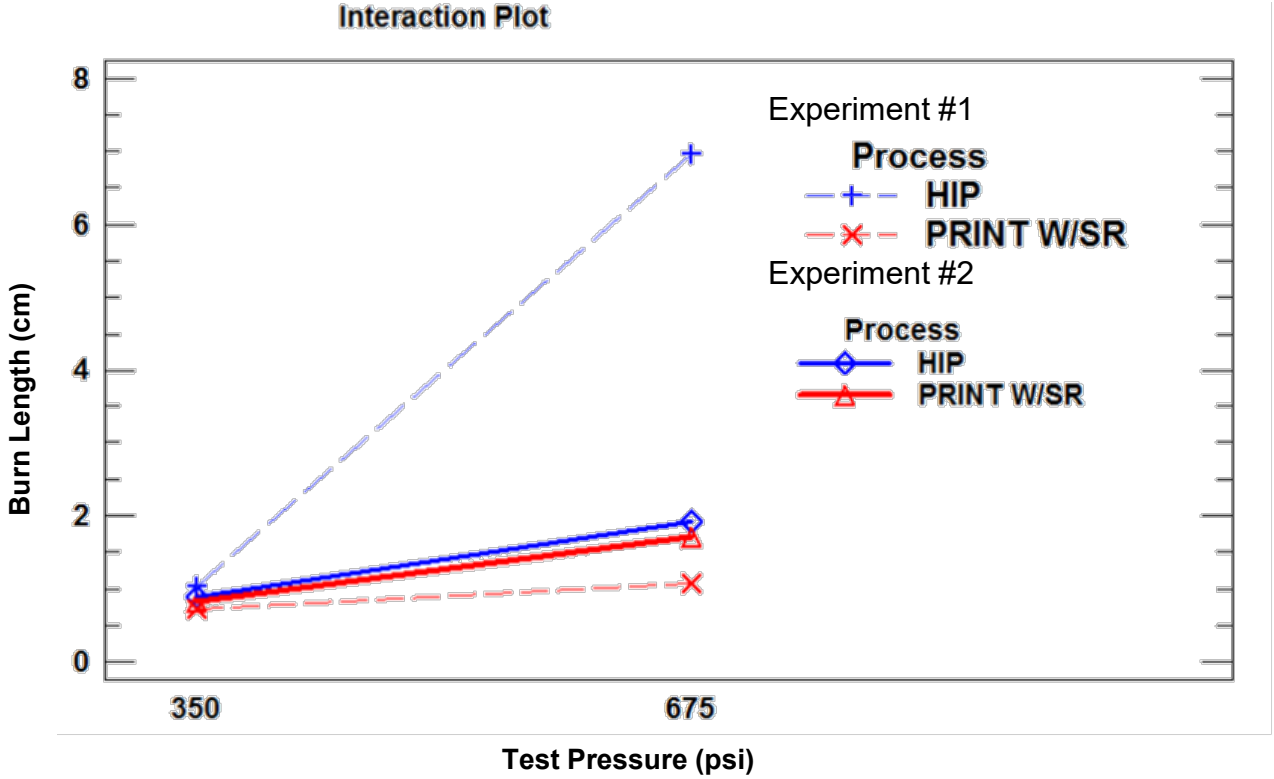
FY16 Experiment Results



- None of process factors studied in the FY16 experiment have a statistically significant effect on flammability performance.
- Pressure only significant factor for all treatments.

16

Comparison to Previous Experiment



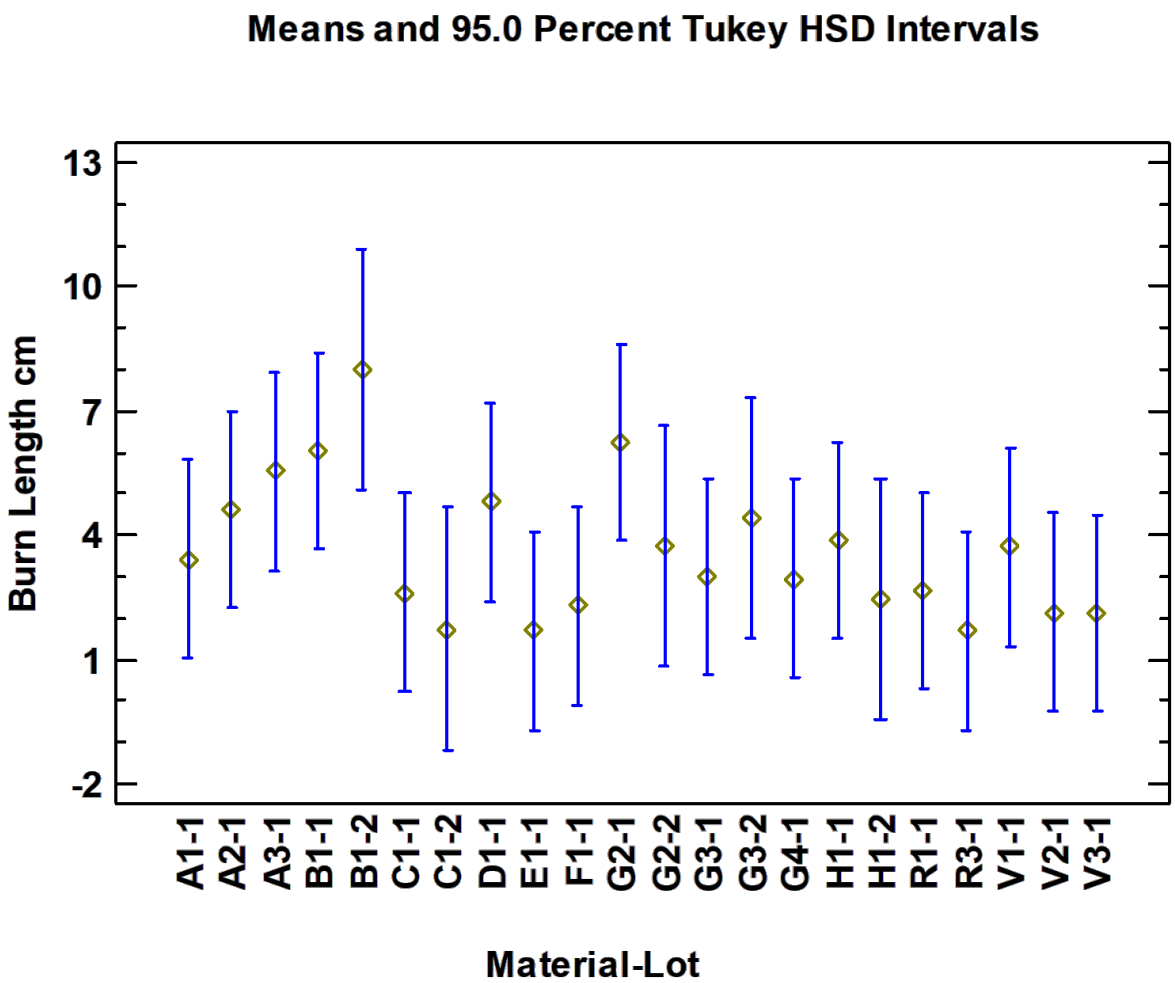
- Significant difference in performance between HIP #1 and HIP #2
- Data from preliminary test and second test show comparable data quality

AMSII Flammability Summary

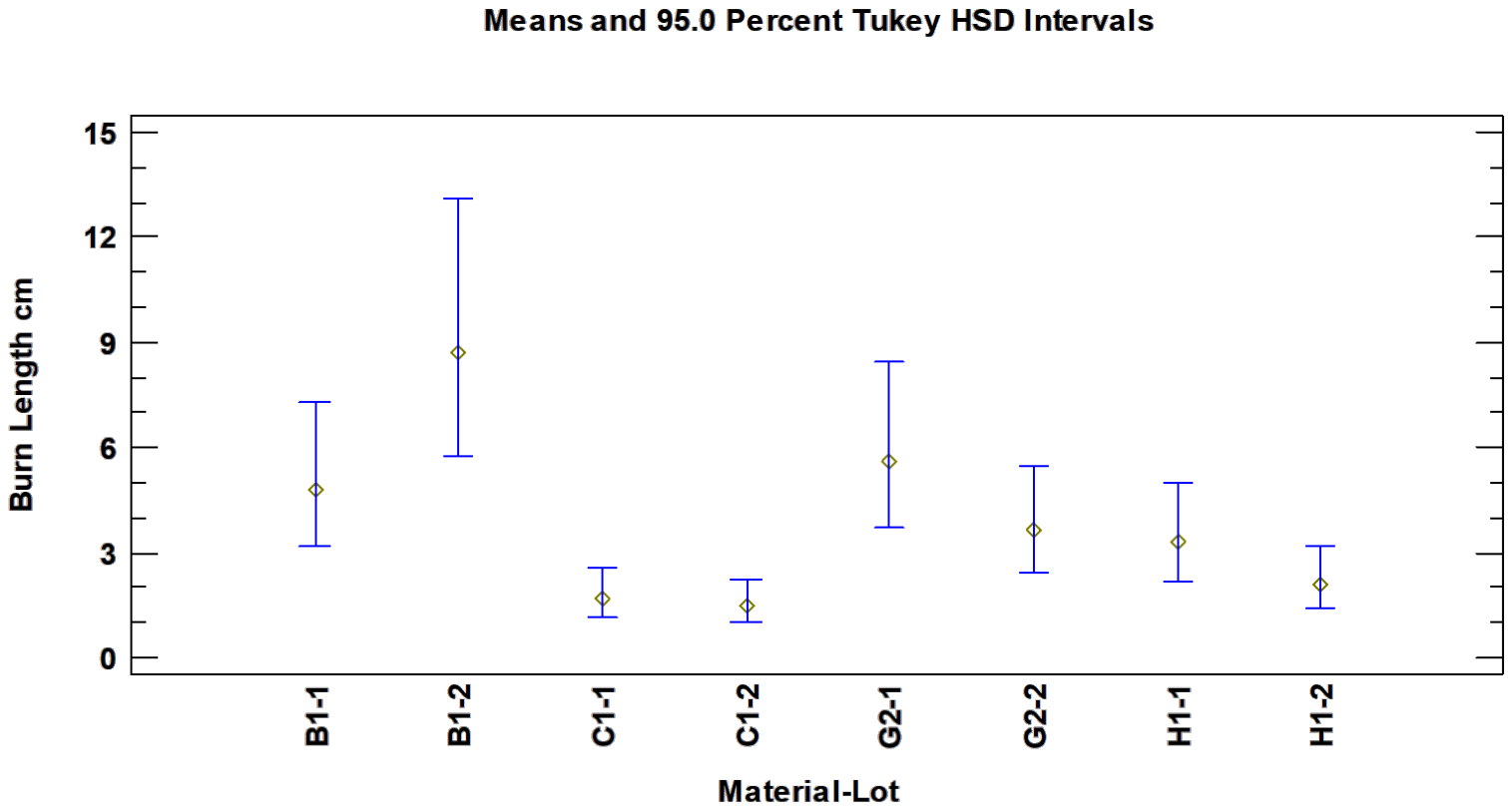
- Additive Manufacturing Structural Integrity Initiative (AMSII)
  - Included flammability performance
- Factors
  - 18 different Inconel 718 powders (HIP Wrap, Full HT)
- Covariates
  - Zone
  - Powder production method
  - Machined vs as printed
  - Green State
  - Chemical composition
  - Virgin vs recycled powder
- Findings
  - Different powders had significant differences in flammability at constant pressure.
  - Composition may matter
    - TiN volume fraction may influence flammability

AMSII 2 Summary

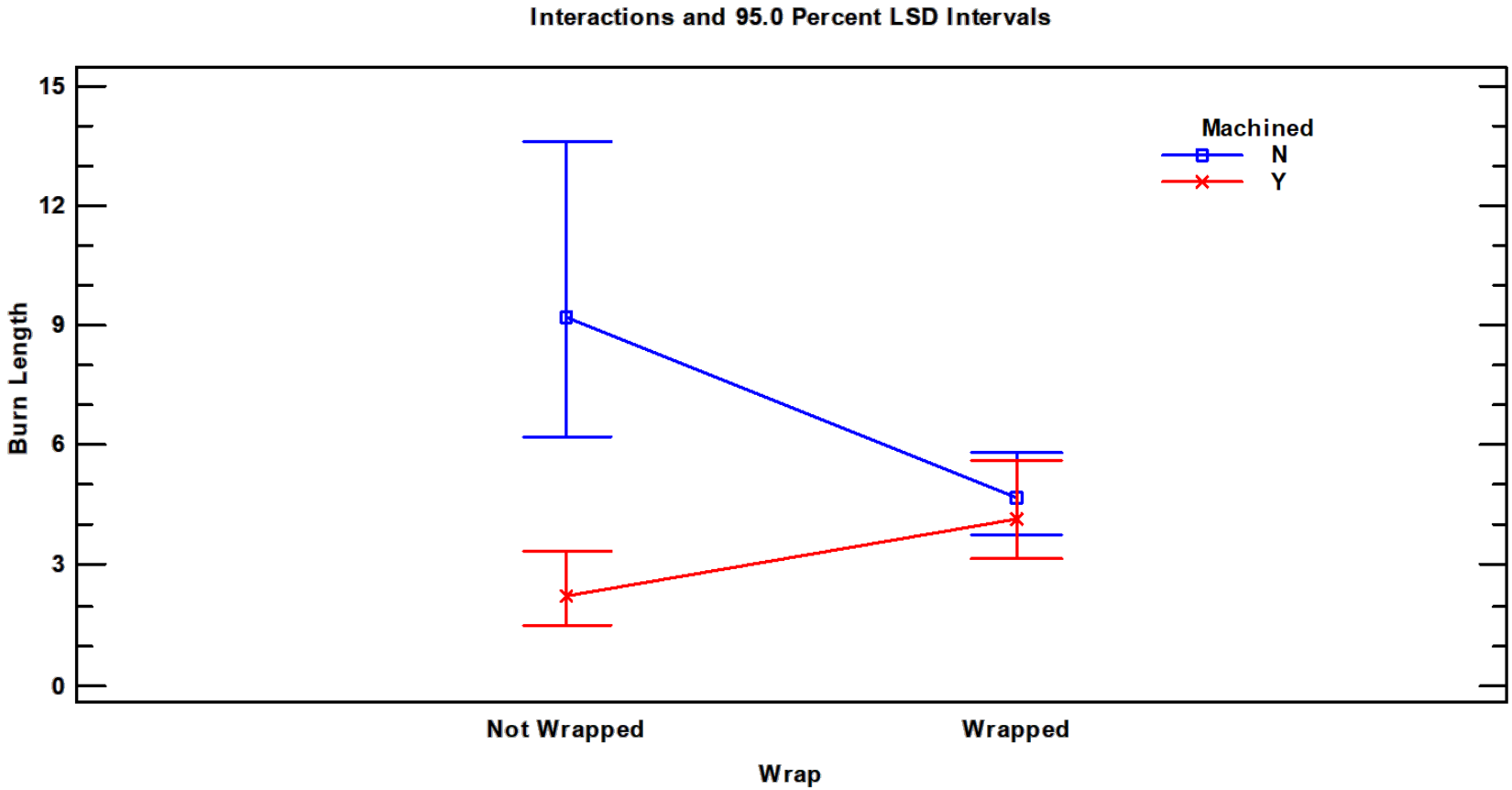
- Factors
  - Second lot of 5 AMSII 1 powders
  - HIP Wrap vs No Wrap
  - Machined vs Not Machined
- Covariates
  - Composition
  - Lot to lot comparisons
- Findings
  - Lots and composition probably matter… a lot… ☹
- Regression model selection with AMSII 1&2 data


Means and 95.0 Percent Tukey HSD Intervals
Burn Length cm
13
10
7
4
1
-2
A1-1
A2-1
A3-1
B1-1
B1-2
C1-1
C1-2
D1-1
E1-1
F1-1
G2-1
G2-2
G3-1
G3-2
G4-1
H1-1
H1-2
R1-1
R3-1
V1-1
V2-1
V3-1
Material-Lot

Direct Comparison between AMSII 1&2

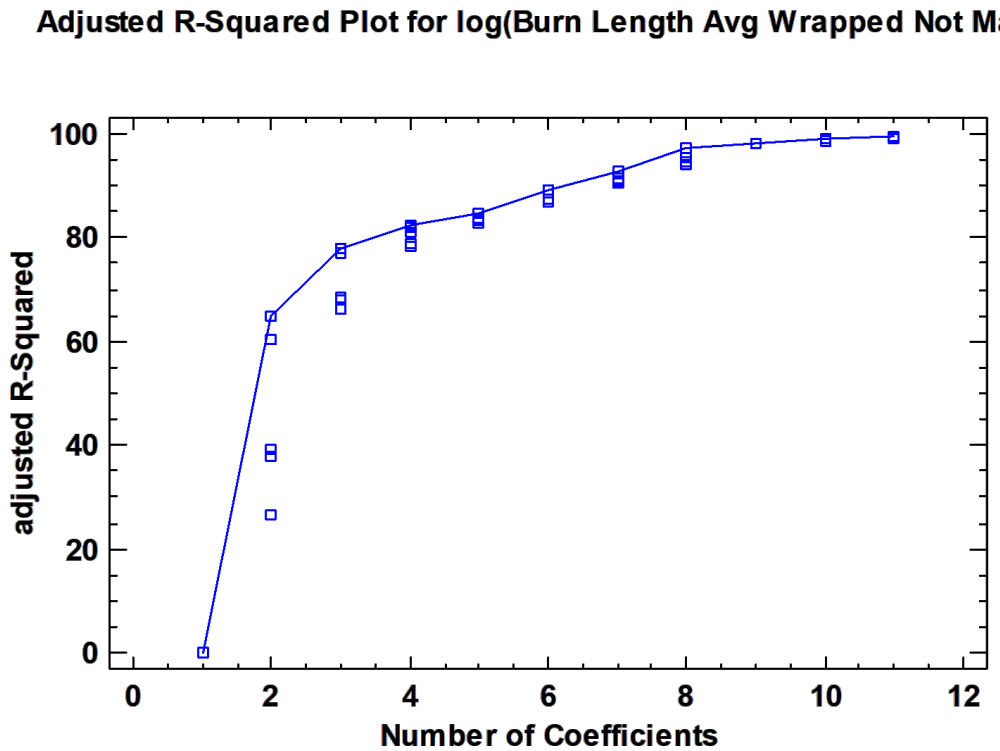


AMSII 2 Material G-2



- Apparent interaction with wrapping during HIP and machining. Did we catch first experiment observation?

Regression Model Selection- Composition



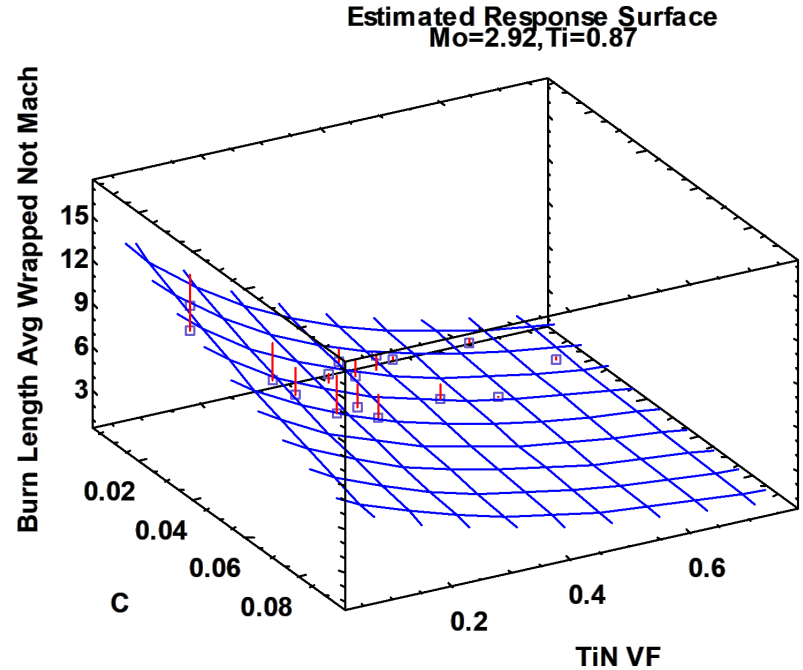
Type III Sums of Squares

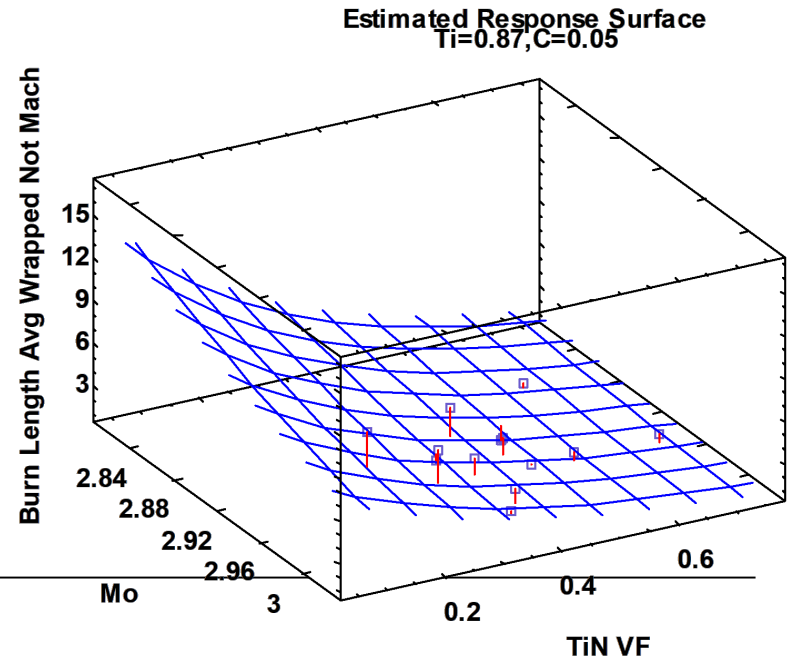
Source	*Sum of Squares*	*Df*	*Mean Square*	*F-Ratio*	*P-Value*
Mo	2.39042	1	2.39042	5.18	0.0404
C	5.73464	1	5.73464	12.43	0.0037
TiN VF	19.0101	1	19.0101	41.21	0.0000

R-Squared (adjusted for d.f.) = **82.3814 percent**

Flammability Model

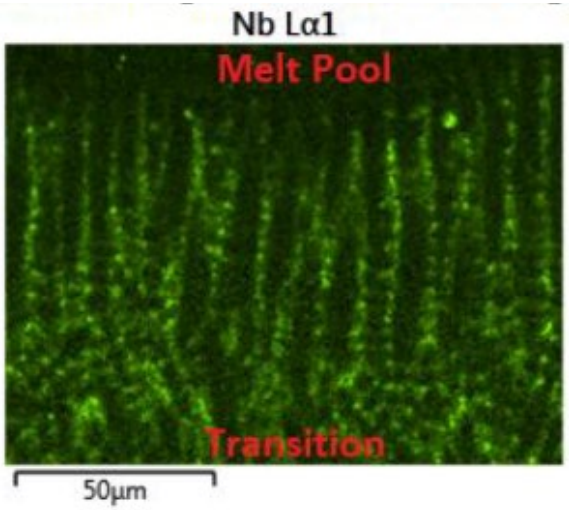
- All HIP wrapped, all full HT, for all AMSII 1 & 2 data.
- Three factors (TiN volume fraction, Carbon, and Molybdenum) describe ~80% of flammability response.
- TiN and C seem to heavily influence flammability.
  - Possible NbC and TiN tie up flammable alloying consituents.
  - Appear to account for 80% of flammability in IN718.
  - DISCLAMER: Data mining caveat. Covariate analysis is not as robust as a designed experiment.
- Mo may be tied up in carbides as well…





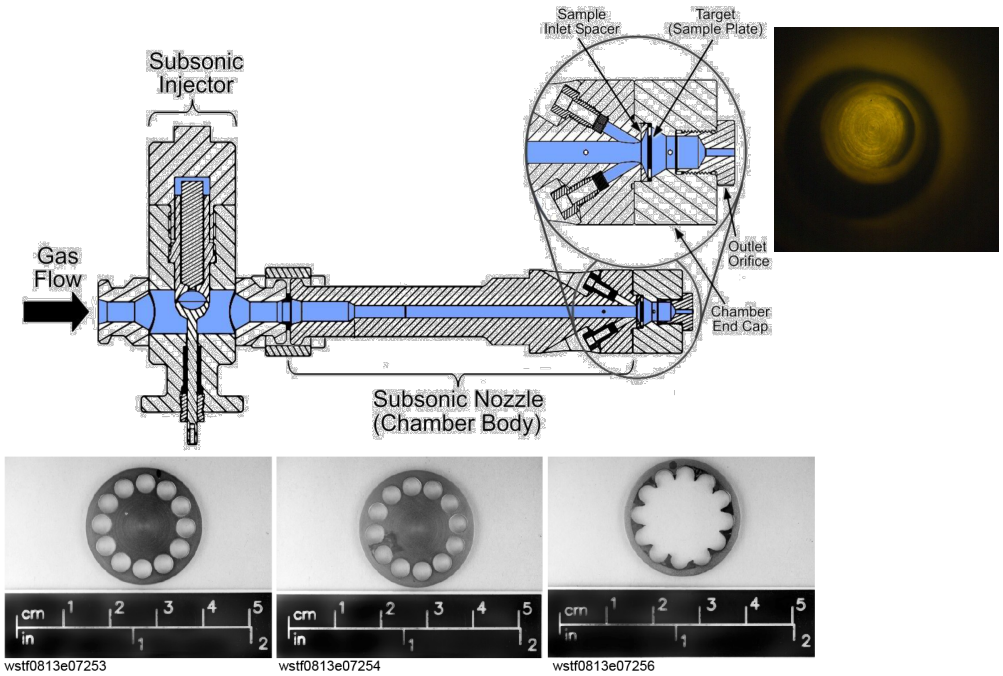
Future Flammability Work

- Perform additional materials characterization on tested samples
  - Determine if Nb in transition region is still tied up as NbC
- See if material G2-2 reveals HIP observation
- Independently verify identified flammability factors
  - Design orthogonal experiment to understand composition TiN and C effects on flammability.
- Characterize flammability performance of more common AM materials and build methods
- Publishing papers on current AM flammability findings to date in ASTM STP
- Reach out to computational materials experts for help modeling flammability of alloys.
- Test more materials and factors…
- Help to advance state of the art materials for performance in severe oxygen service.

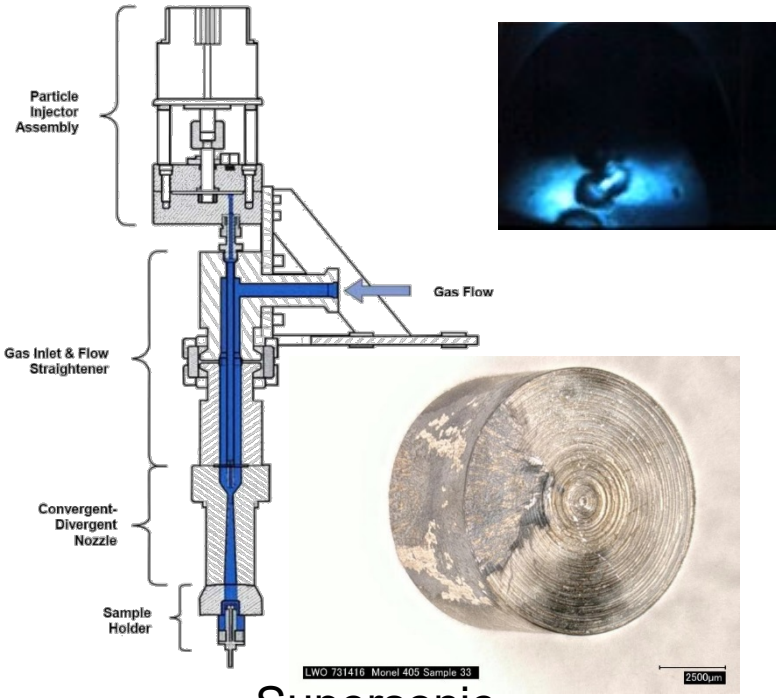


Minimize

- Particle Impact
  - Most common direct igniter of metals
  - Hazards increase with:
    - Pressure, temperature, velocity, flammable particles
  - SLM Components shed metal particles (Lowrey 2016)



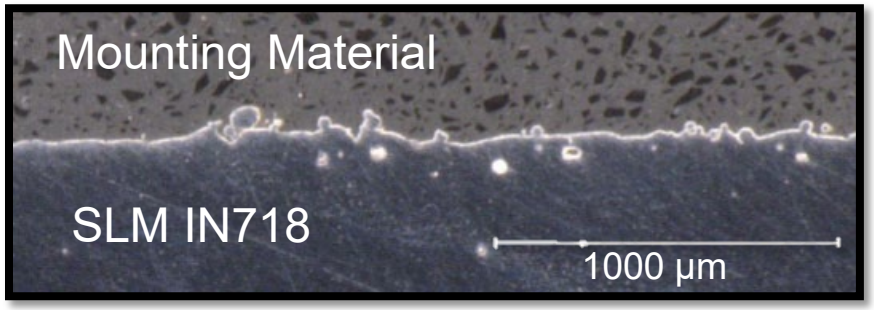
Subsonic



Supersonic

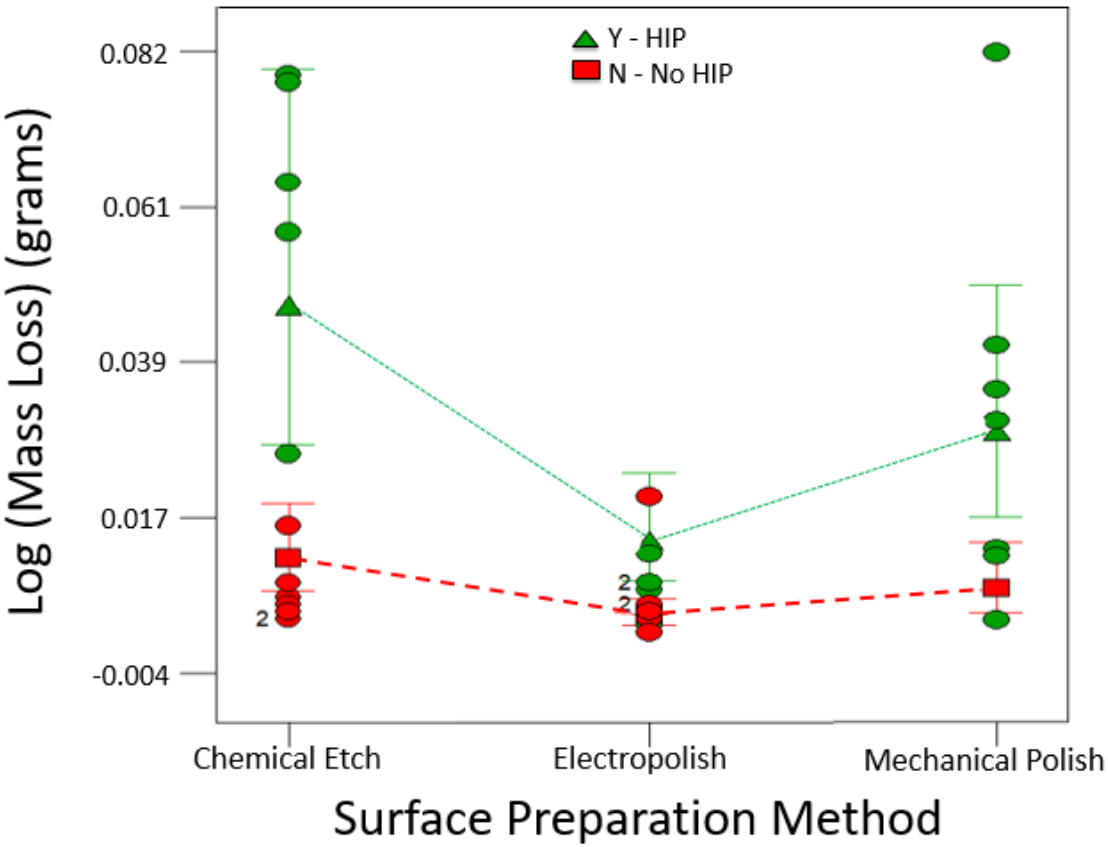
Ignition Study

- Subsonic & Supersonic Impacts on SLM IN718
  - Pressures, temperatures, velocities
- Study effect of AM characteristics on ignition sensitivity
- Factors
  - Wrought vs. SLM
  - Presence or lack of hot isostatic pressing (HIP)
  - Heat treatment (AMS 5664 vs. Annealing)
  - Surface preparation (chemical etching, electropolishing, electric discharge machining, mechanical polishing, rough machined surface)
  - Particulate type (Aluminum, IN718 powder, Sapphire)
  - Particle Velocity (Subsonic, Supersonic)
  - Temperature (300-950 °F)
  - Pressure (1,300 psia-4000 psia)



Selected Supersonic Testing Results

- SLM samples that received HIP and electro polishing lost less mass than HIP samples with either mechanical polishing or chemical etching when impacted.
- SLM HIP samples lost significantly more mass than samples that were not HIP when impacted.
- Heat treatment and annealing was not observed to affect the ignitability of any Inconel 718 sample type.

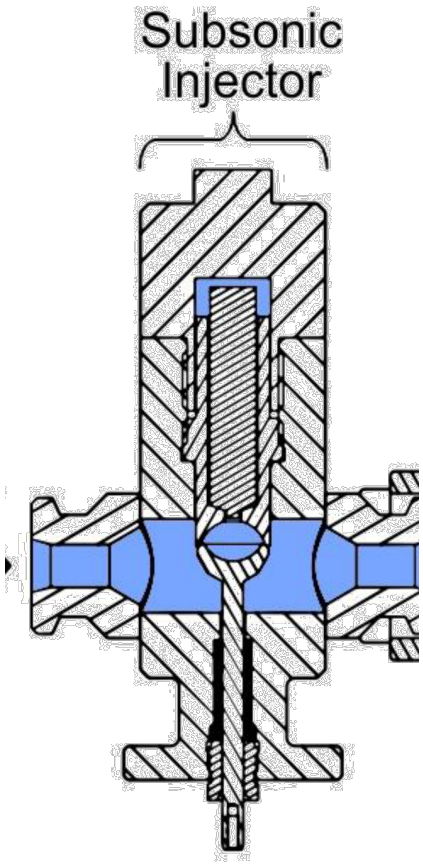


- *Results of a 30 test supersonic PI surface preparation experiment using only SLM IN718 comparing surface treatment and HIP at a static pressure of 1300 psia, and an average temperature of 562° F, and a single 2000 µm aluminum ball.*

Selected Subsonic Testing Results

- Even without ignition, SLM samples lost more mass than wrought samples.
  - This is likely due to particle silting from the SLM samples during exposure to high flow even after aqueous cleaning.
- SLM powder is highly flammable. When contained in the subsonic particle injector, the powder ignited before injection into the heated flowing gas.

AM feed stock is extremely flammable…



Future Ignition Work

- Replicate results of previous experiment.
- More fully characterize factors affecting ignition in AM materials.
- Perform testing on more AM aerospace materials.
- Perform ignition testing at a component level.
- Quantify representative contamination likely to be generated from SLM components.
  - Perform particle impact tests with representative contamination quantaties.

Utilize

- AM production
  - Dedicated machine(s) for each material
  - Prevent cross contamination
- Precision cleaning
  - What is the best method.
- AM component/system design recommendations specific to oxygen systems.
- Assembly
- Operations
- Maintenance

Long-Term Goals

- Identify and characterize major factors that effect AM ignitability and flammability. Including modeling.
- Test more representative aerospace AM metals and methods.
- Test additional ignition mechanisms.
  - Friction, cavitation
- Develop guide for the use of AM in oxygen systems
  - Design
  - Manufacturing
  - Cleaning
  - Assembly
  - Operations
  - Maintenance

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Questions?

Back Up

Scatter plot for log(Burn Length) and TiN VF