Powder Metallurgy Molybdenum Accelerator Target Materials and Assemblies

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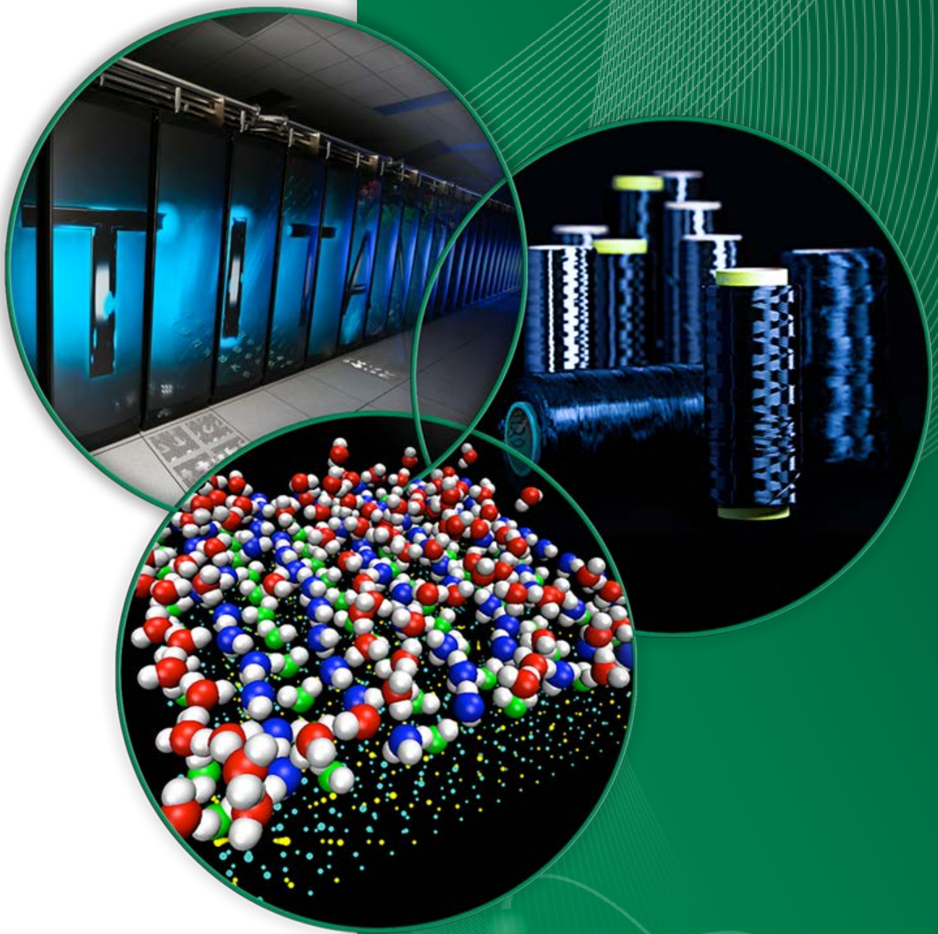


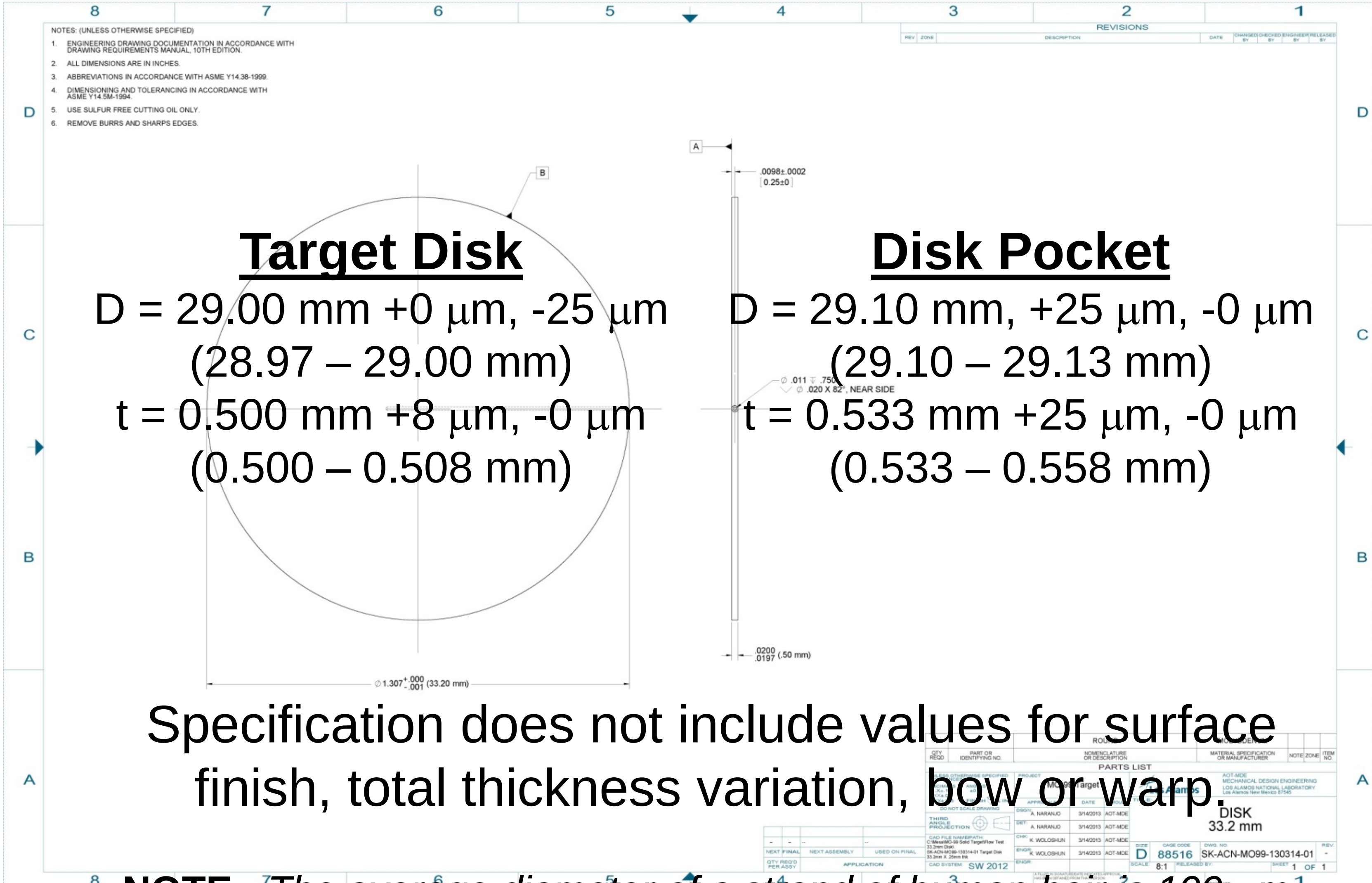
Mo-99 Topical Meeting
St. Louis, MO
September 14, 2016

Target Disks and Assemblies for NorthStar's Accelerator Production of Mo-99 Are Being Fabricated Employing Powder Metallurgy Techniques

The goals of this effort:

- Understand the **requirements for and fabrication of molybdenum target disks and assemblies** that will be used in the accelerator production of Mo-99.
- Develop a **process for fabricating accelerator target disks and other structural components with a density of 90% or greater and acceptable thermomechanical properties**.
- Identify **characteristics that affect the dissolution rate** of target materials.
- Assist in developing a process for **recycling isotopically-enriched molybdenum**.

The Proposed Target is a Thin Metal Disk with Quite Stringent Dimensional Tolerances



Target Disk

D = 29.00 mm +0 μm, -25 μm
(28.97 – 29.00 mm)
t = 0.500 mm +8 μm, -0 μm
(0.500 – 0.508 mm)

Disk Pocket

D = 29.10 mm, +25 μm, -0 μm
(29.10 – 29.13 mm)
t = 0.533 mm +25 μm, -0 μm
(0.533 – 0.558 mm)

Specification does not include values for surface finish, total thickness variation, bow or warp.

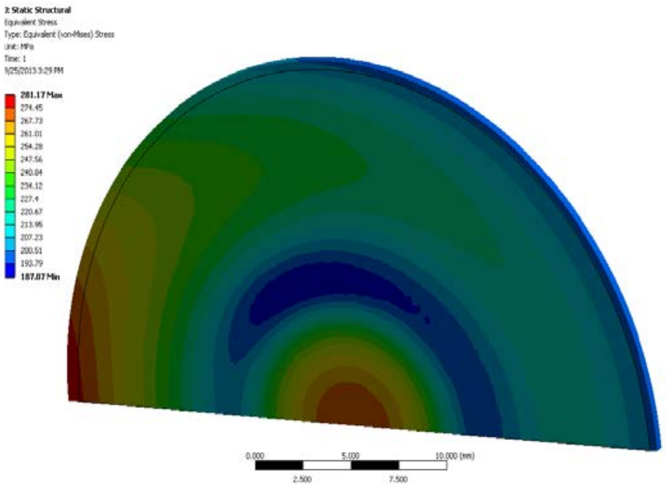
NOTE: *The average diameter of a strand of human hair is 100 μm.*

Target Disks Have Been Fabricated Using Traditional Powder Metallurgy Approaches

- Powder metallurgy has been used to produce accelerator target disks that meet dimensional specifications with minimal waste.
- A combination of lubricated, spray-dried powders and good tooling has been used to produce uniform disks that dissolve quickly.
- Thermophysical and thermomechanical properties of the powder metallurgy Mo materials have been measured.
- The properties and behavior of materials and components after exposure to simulated operational environments are being characterized.

Thermomechanical Models Predict Disk Distortion Due to the Thermal Gradients

Resulting stress:



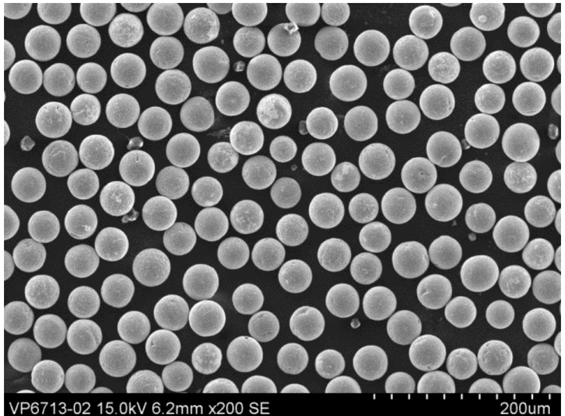
Bowing was verified experimentally.

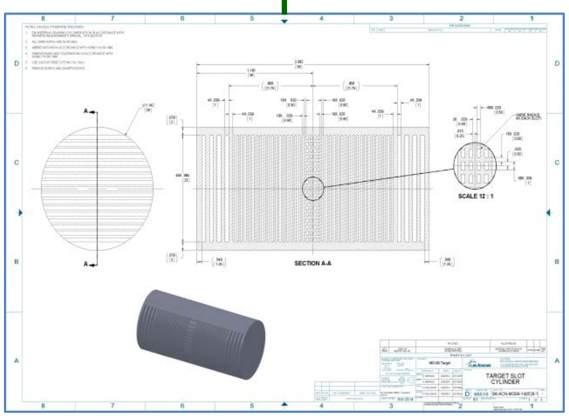
Specimen	Power (W)	Pulse (msec)	Frequency (Hz)	Time (sec)	Δ (μm)
SM-1	92	0.5	100	30	+18
SM-2	120	0.5	100	30	+70
SM-3	200	1	50	60	+60
PMA-28	120	0.5	100	30	+6
PMA-29	193	1	50	30	+10
PMA-30	193	1	50	60	+16
PMA5-22	200	1	50	75	+17
PMA-26	200	1	50	150	+20
PMA5-21	250	1.5	50	30	+40

Δ = increase in bow, SM = sheet metal

The Hot Center Expands While the Cold Periphery is Constrained Resulting in Non-Uniform Stress and Strain Across the Disk

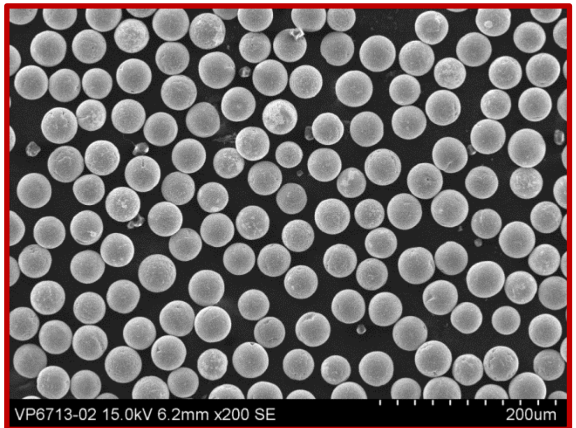
Targets Assemblies That Accommodate Thermally Induced Stresses and Strains Could Be Designed and Fabricated Employing Additive Manufacturing

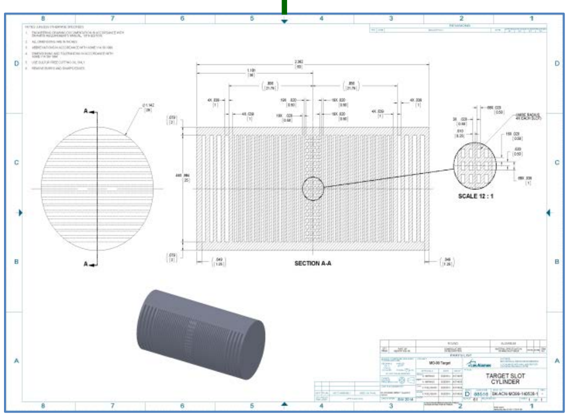




Notional 3D target design printed in stainless steel

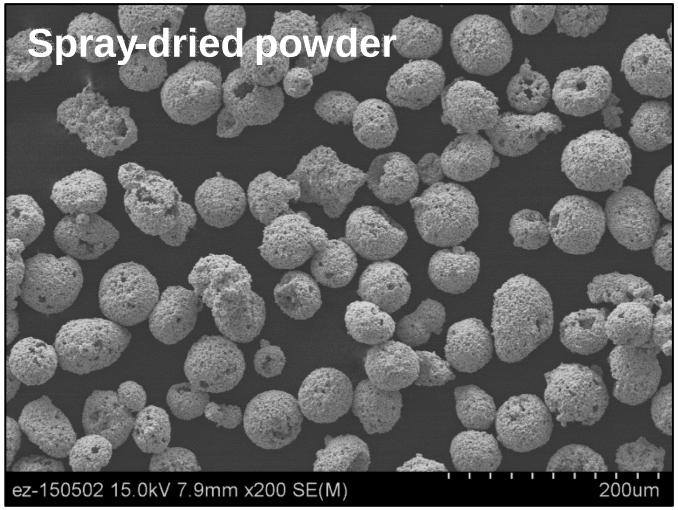
Dense, Spherical Powder is Required for Additive Manufacturing



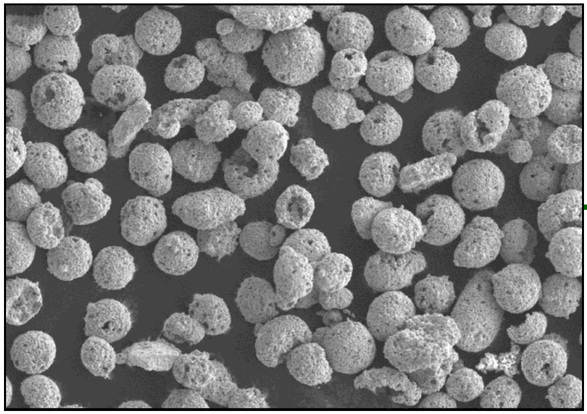


Notional 3D target design printed in stainless steel

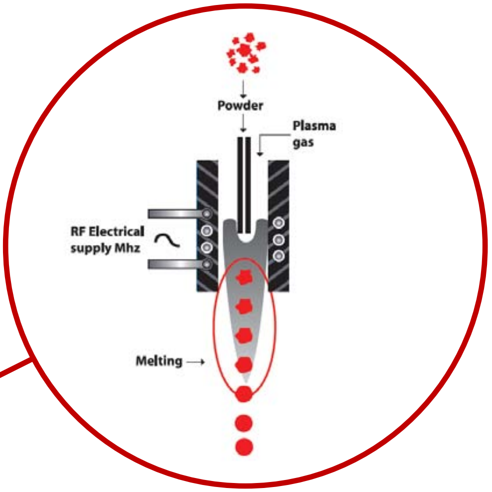
Reduced Powder Is Milled and Spray-Dried to Produce "Flow-able" Feedstock



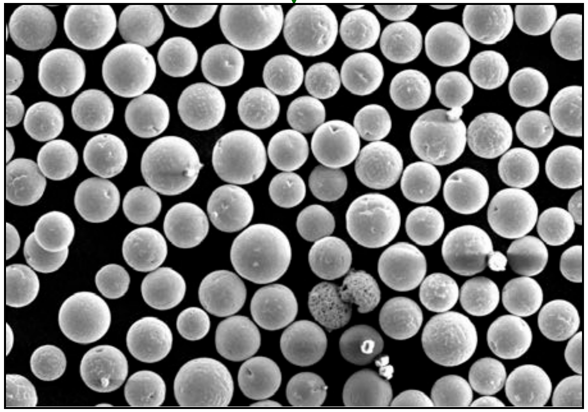
Spray-Dried Powders Can Be Spheroidized for Use in the Additive Manufacture of Targets



55 ± 13 um
(-200/+325 mesh)



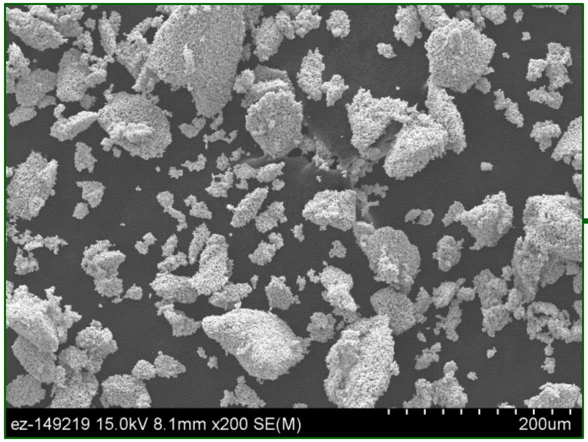
TEKNA 15 kW Plasma Spheroidization System



48 ± 9 um

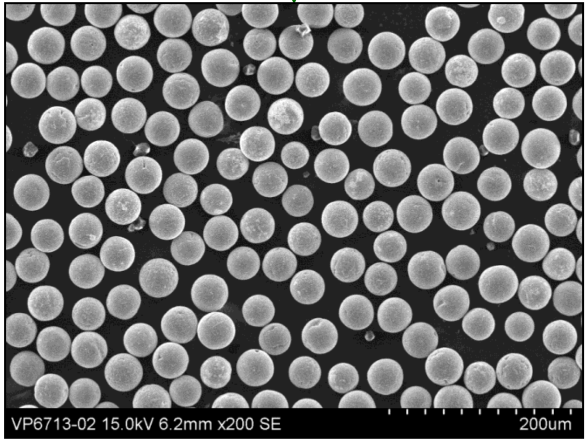
Target powder particle size:
+15/-45 μm (30 ± 15 μm)

As-Reduced Trioxide Powder Has Also Been Spheroidized



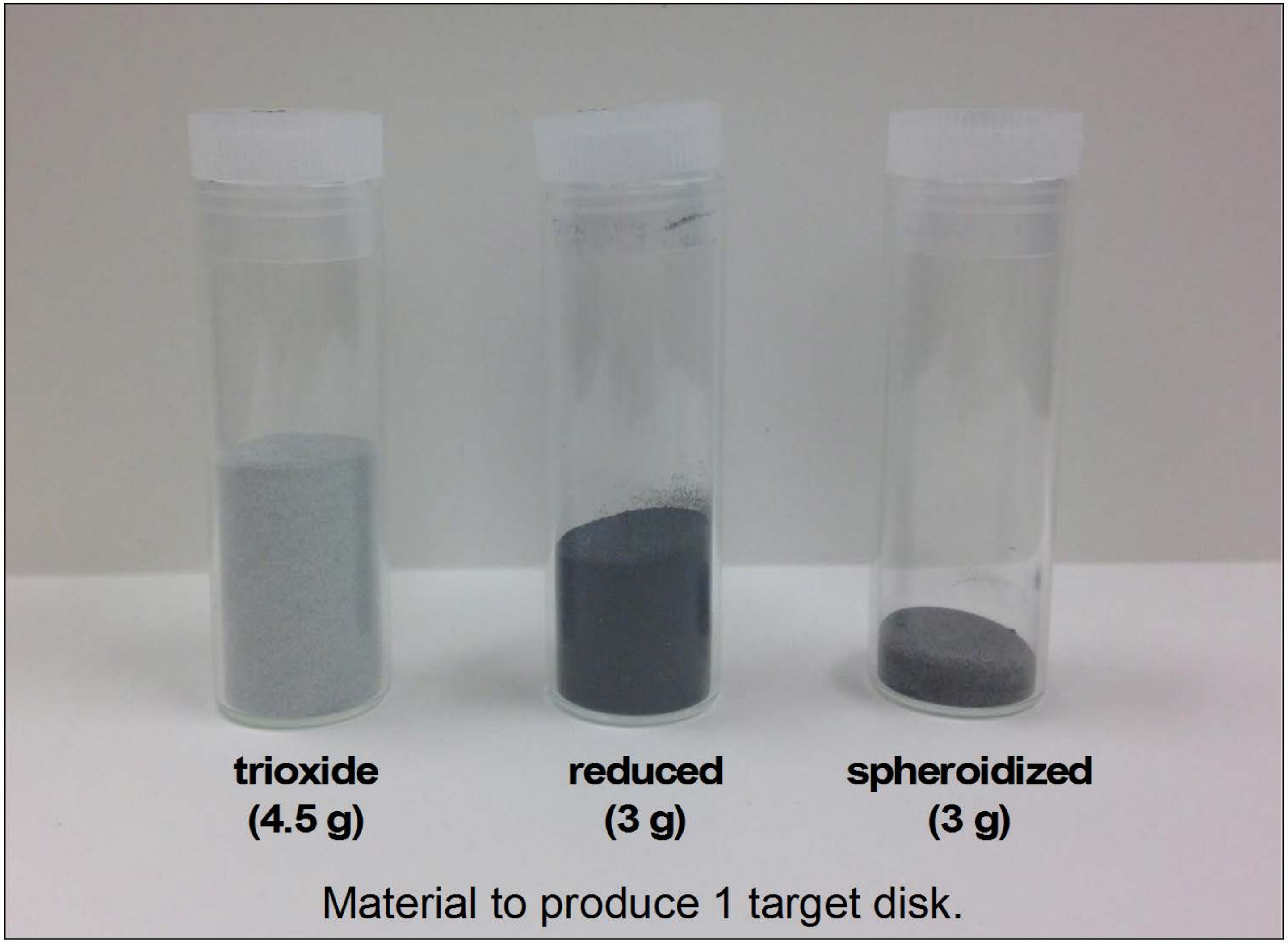
126 ± 15 um
(-100/+200 mesh)
96 ± 34 um
(-140/+325 mesh)

TEKNA 15 kW Plasma Spheroidization System

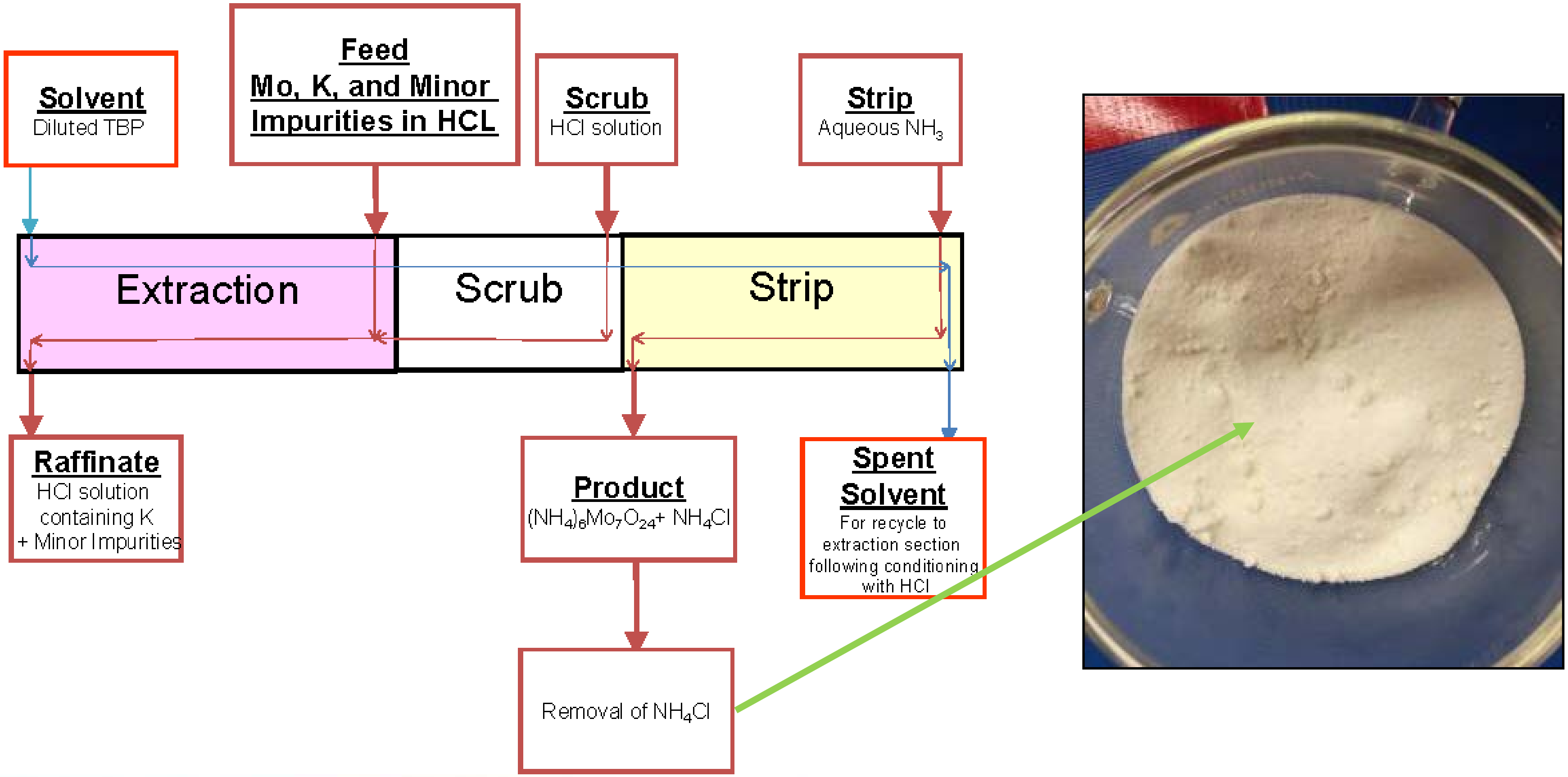


51 ± 20 um
41 ± 14 um

Trioxide and Its Reduction Product Are Friable and Produce Unwanted Fines During Processing



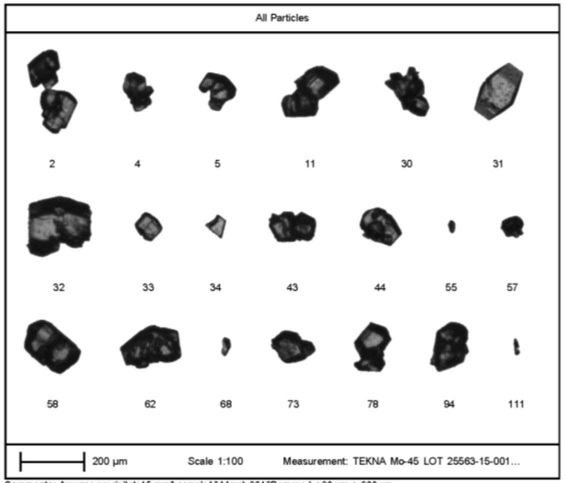
ANL is Developing a Solvent Extraction Process for the Recovery/Recycle of Mo



Ammonium Heptamolybdate Tetrahydrate - $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$ or AHMTH

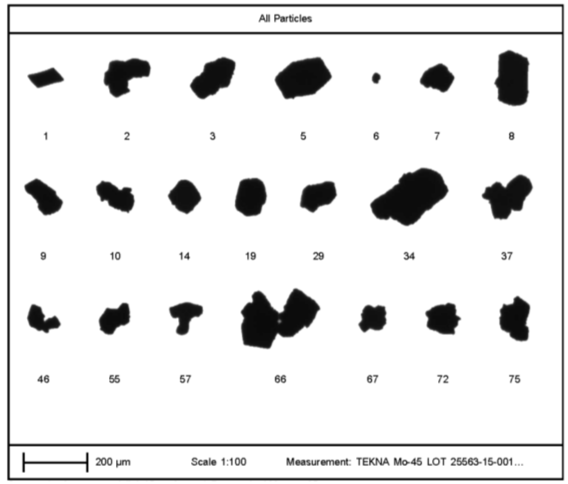
AHMTH is Readily Reduced and Spheroidized

Sieved



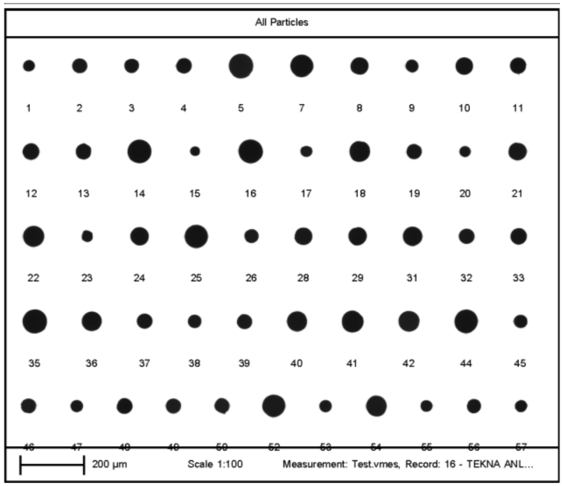
156 ± 62 um
(-100/+200 mesh)

Reduced



129 ± 41 um

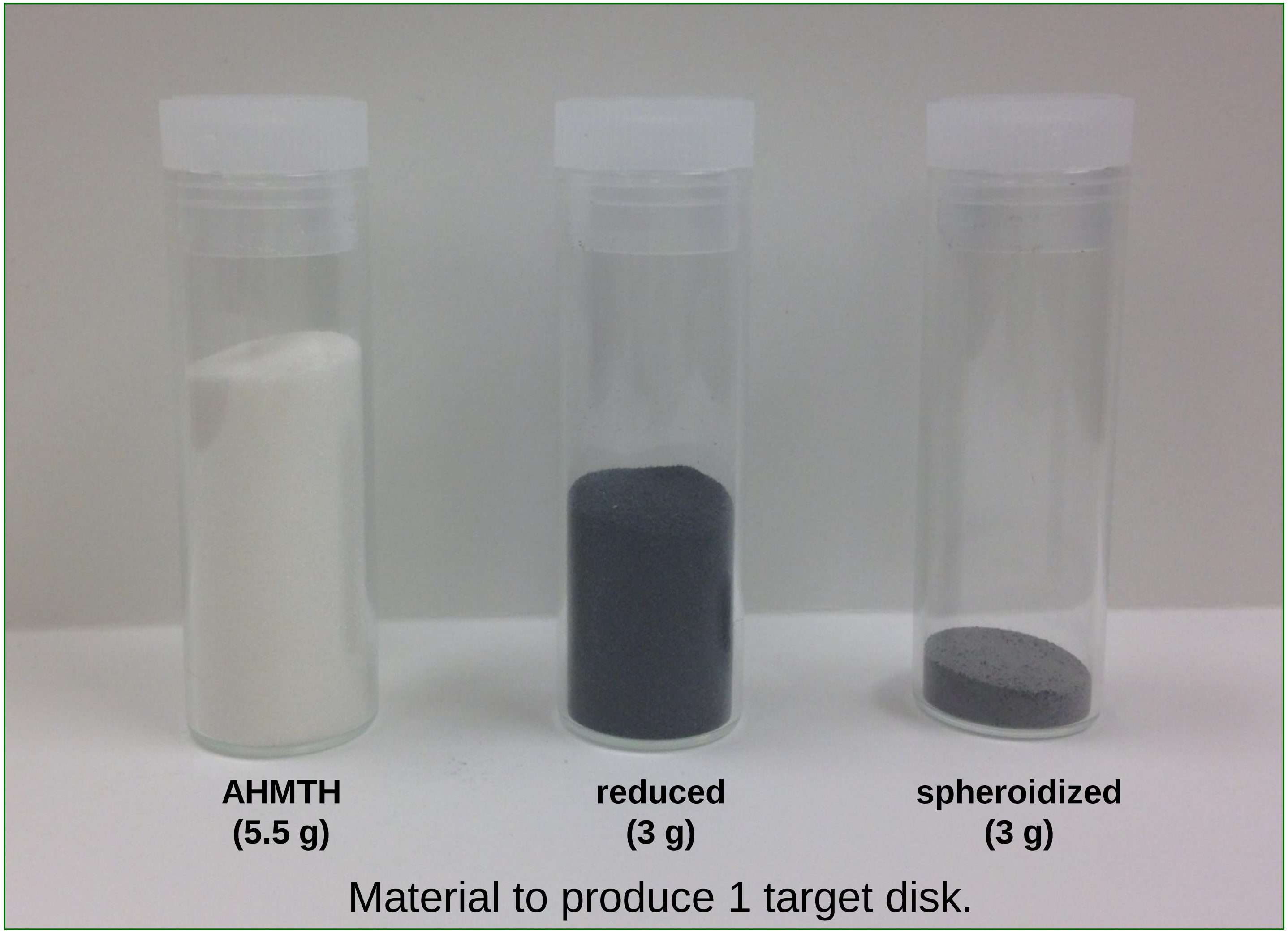
Spheroidized



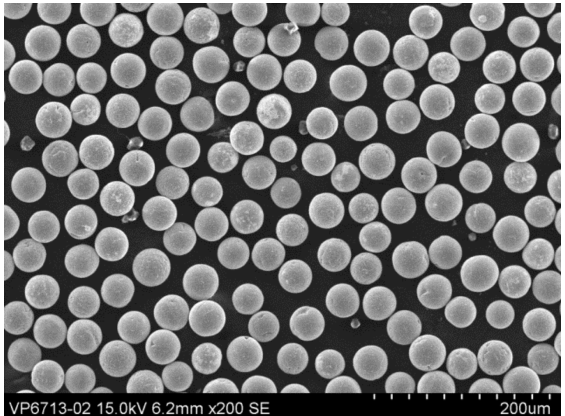
61 ± 14 um

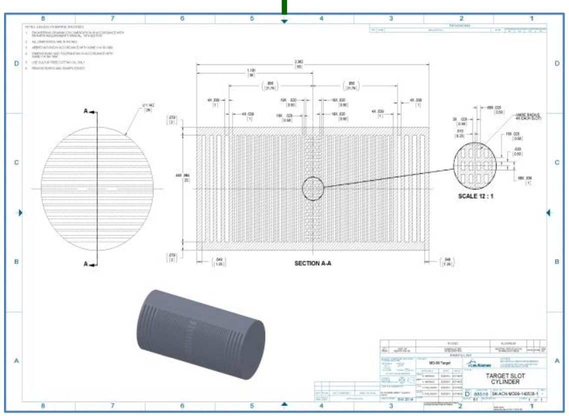
AHMTH is much more robust than trioxide resulting in significantly fewer fine particles!

Shrinkage During Conversion from AHMTH to Metal is Significant



Targets & Assemblies Are Being Fabricated Employing a Selective Laser Melt Technique





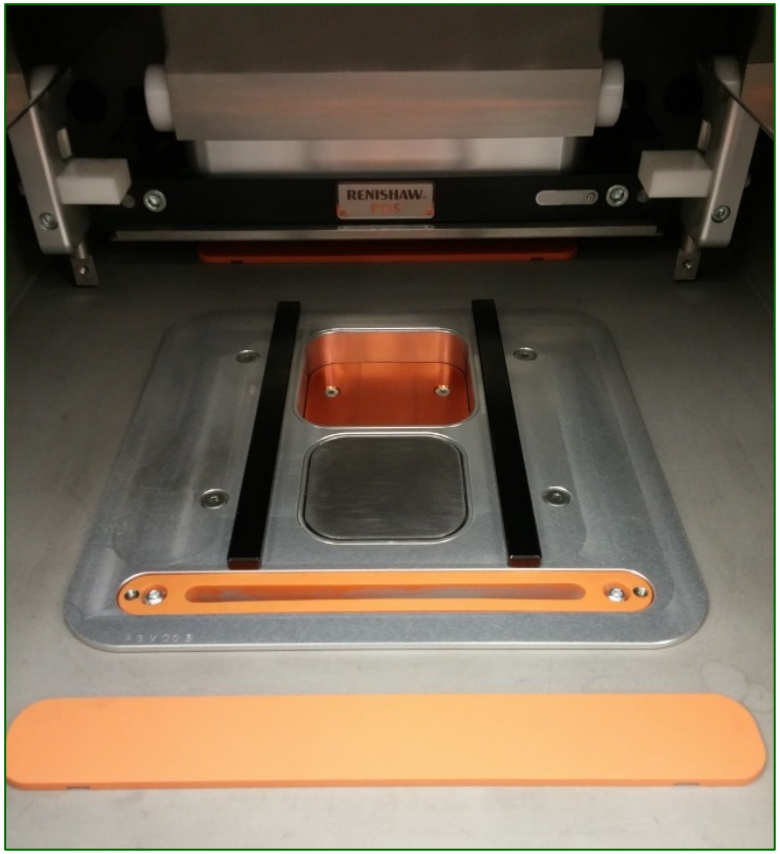
Notional 3D target design printed in stainless steel

Laser Melt Processing of Molybdenum is Quite Challenging

Material	Melting Point (°C)	Thermal Conductivity (W/m-K)
Titanium 6Al/4V	1600 – 1660	7
316 Stainless	1370 – 1400	16
Inconel 625	1290 – 1350	10
Inconel 718	1260 - 1340	11
Tool Steels	~ 1400	Up to ~ 25
Molybdenum	**2620**	**140**

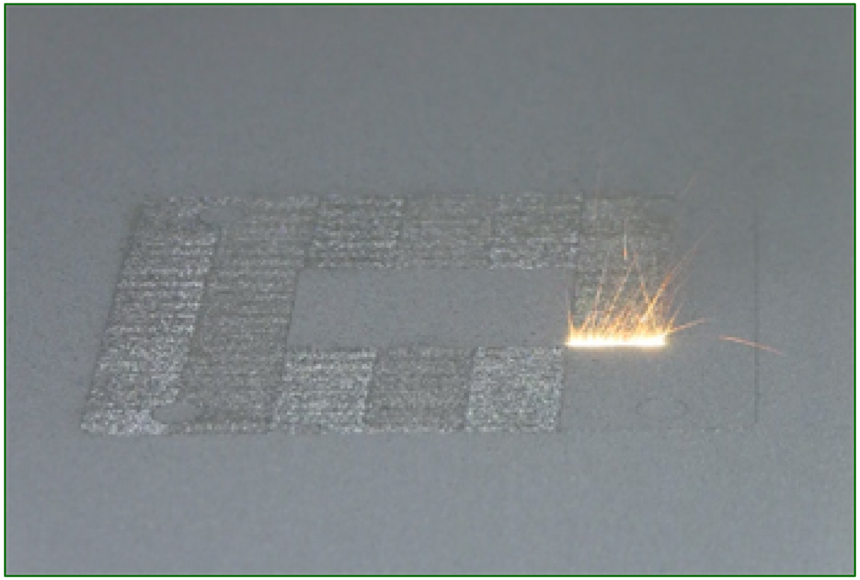
Unique Processing Capability Was Established to Examine SLM AM of Molybdenum

Renishaw AM250 400 watt selective laser melting system

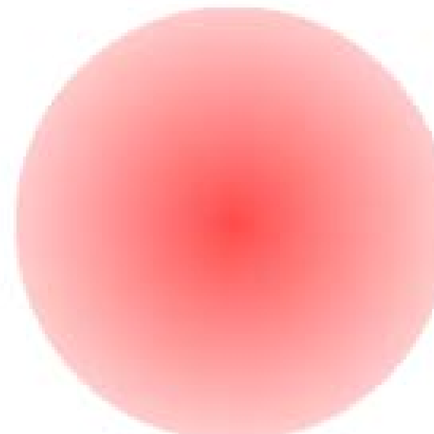


Reduced build volume insert for small-scale experiments

A Laser is Used to Selectively Melt Successive Layers of Metal Powder



Laser Spot = 130 μm

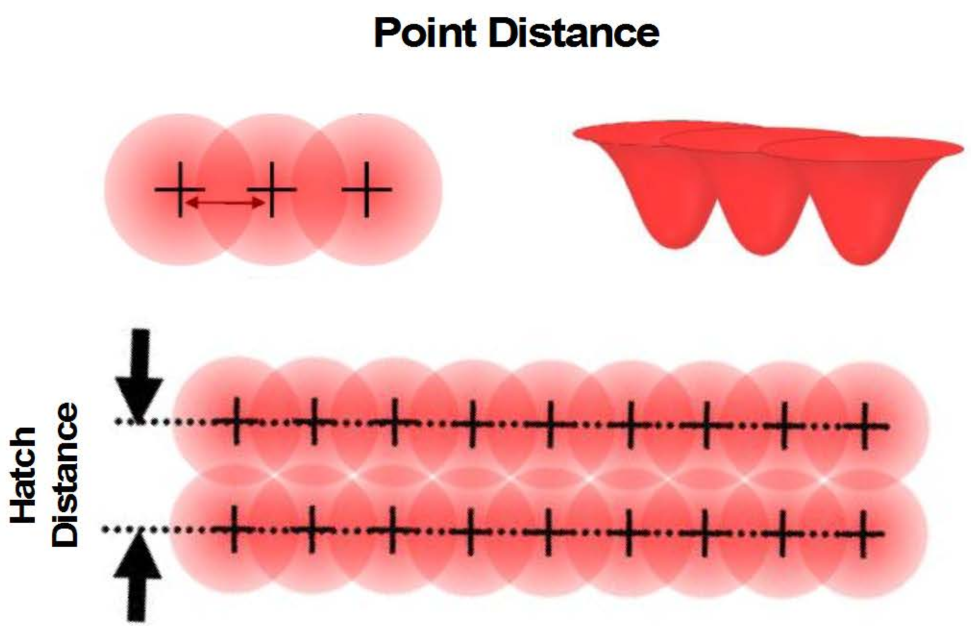


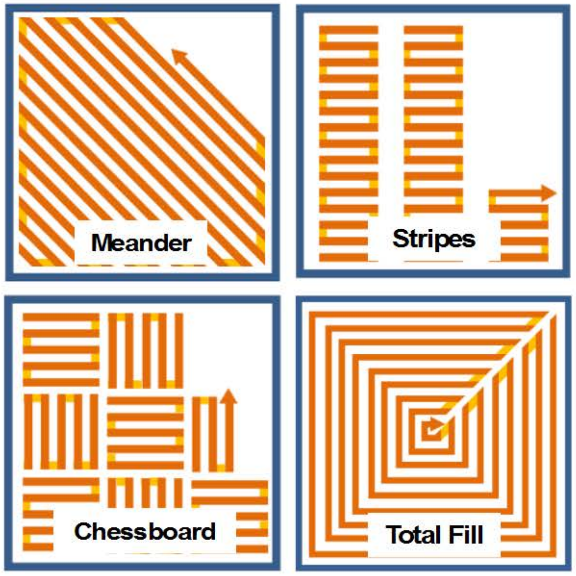
Melt Pool is Dependent Upon Exposure Time and Power

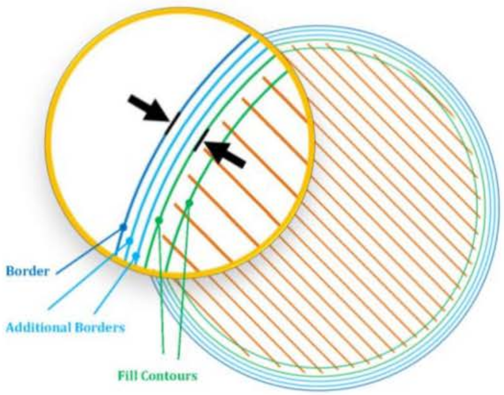
<u>Primary SLM Process Variables</u>

Point Distance (μm)
Exposure Time (μs)
Power (W)
Hatch Distance (μm)
Layer Thickness (μm)
Powder Particle Size (μm)

Scan or Build Strategy Influences Critical Features Such as Porosity, Microstructure, and Surface Roughness





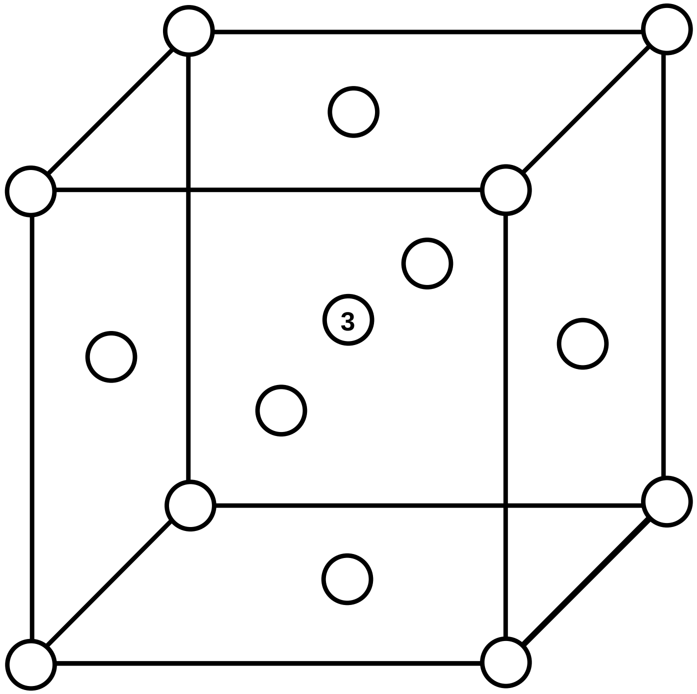


There are ~ 180 different process variables associated with SLM AM build strategies.

(Note: 1st Mo run was very entertaining!)

A Set of Experiments Was Conducted to Develop an Understanding of the Primary SLM Process Variables for Molybdenum

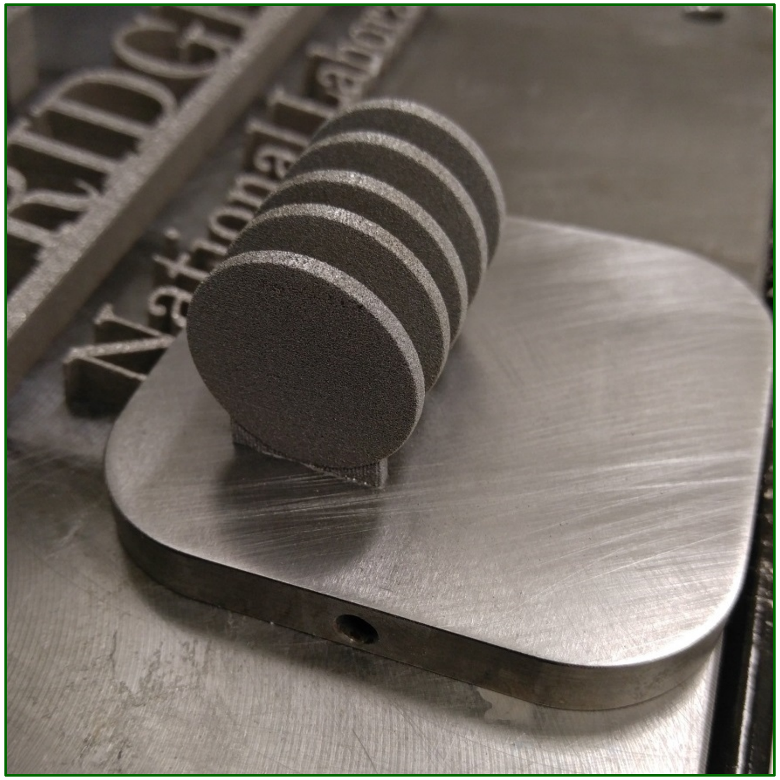
- **Independent Variables**
  - Spot Distance: 50, 75 and 100 μm
  - Hash Distance: 50, 75, and 100 μm
  - Exposure time: 200, 300 and 400 μs
- **Fixed**
  - Power: 400 watts
  - Spot size: 150 μm (130 μm)
  - Powder layer: 30 μm
  - Particle size: 30 ± 15 μm (TEKNA Mo45)
- **Dependent Variables**
  - Density (**88 to 95%**)
  - Open porosity (**4 to ~ 10%**)
  - Thickness
  - Weight
  - Width

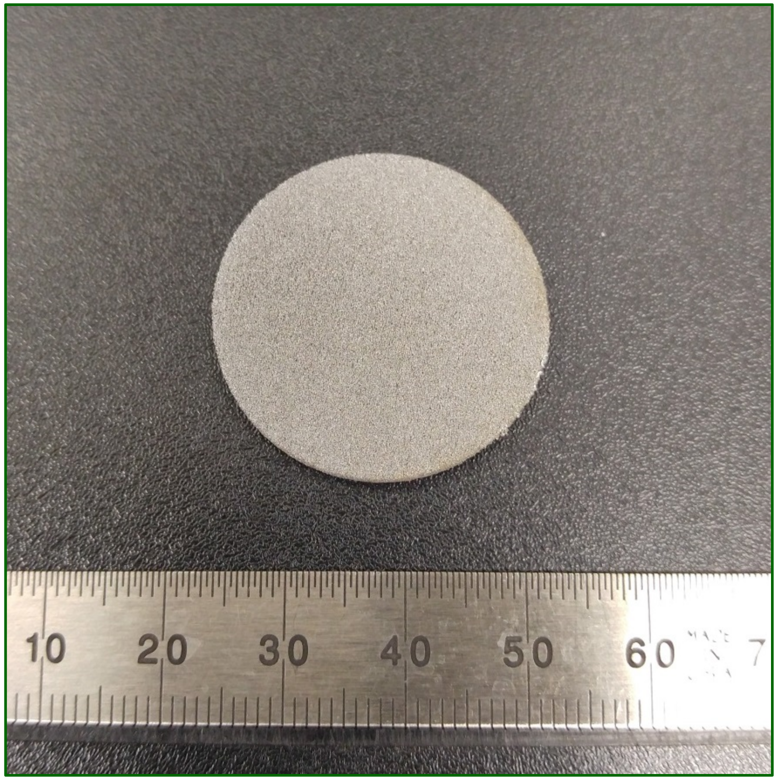


17 sets of independent variables including 3 repetitions of the center point

Test specimen: 10 x 10 x ~ 1 mm coupon

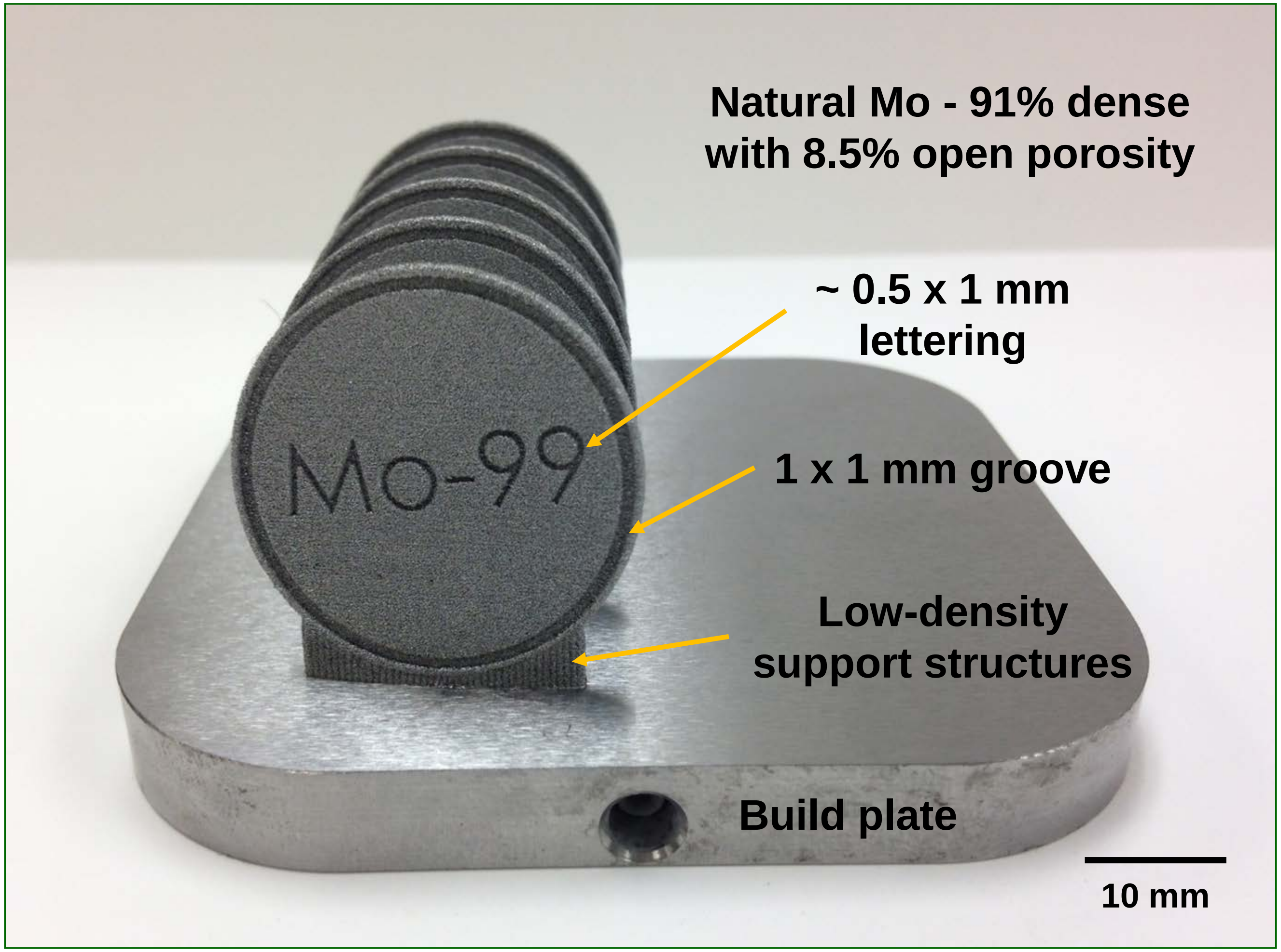
Once the Study Was Complete, Emphasis Switched to Fabricating Representative Shapes





Build parameters, part orientation and process atmosphere are being explored and optimized using 12 and 29 mm diameter disks of different thicknesses.

Build “Resolution” is Also Being Evaluated



Blanks and net-shape test specimens are being fabricated for characterization of microstructure and testing material properties.

Summary

- Target disks that meet specification and dissolve quickly have been produced employing traditional "press and sinter" powder metallurgy techniques.
- Laser melt additive manufacturing is being examined for the fabrication of molybdenum target materials, components and assemblies.
- To minimize waste and processing steps, spherical powder for AM is being produced directly from compounds being evaluated for the recycle/recovery of Mo from spent solutions.
- SLM AM materials with desired densities have been produced, the properties and behavior of which are now being investigated.