OAK RIDGE National Laboratory

Fabrication of Molybdenum Target Materials Employing "Recycled" Powders

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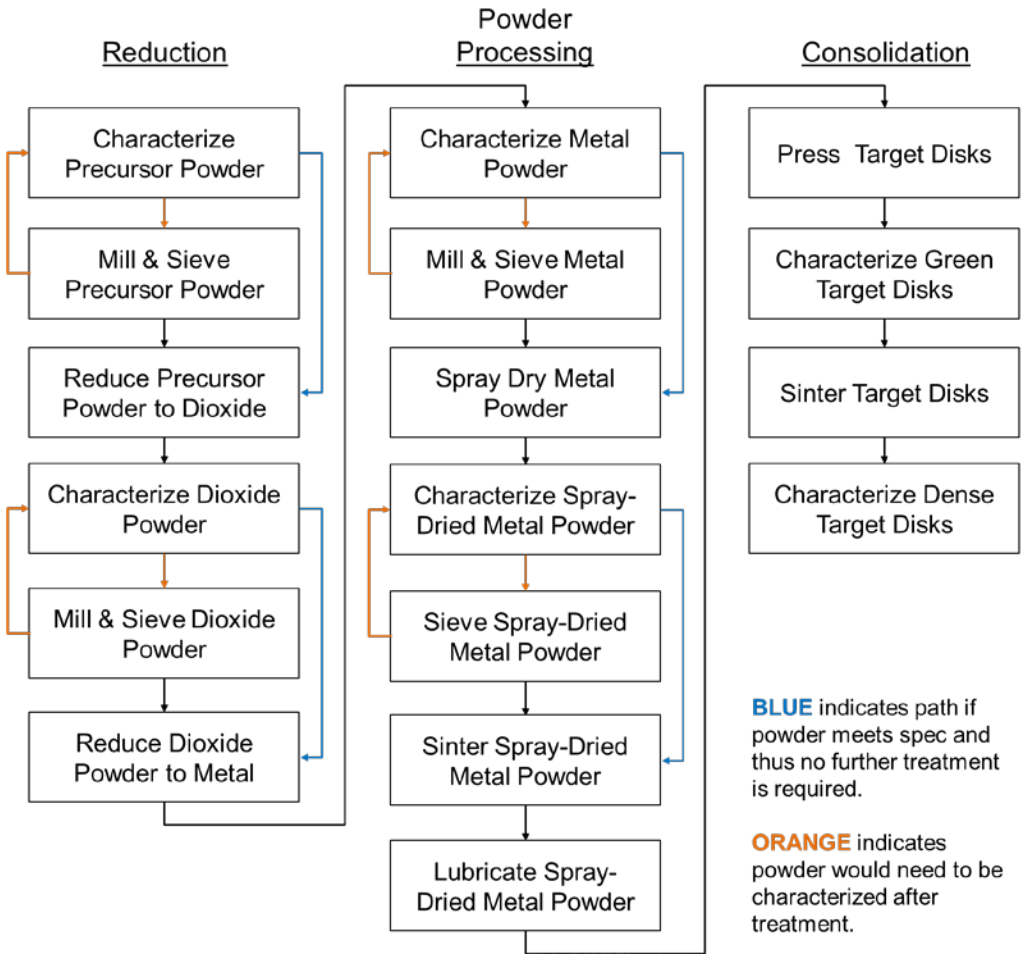
Target Disks and Assemblies in Support of NorthStar's Production of Mo-99 Are Being Fabricated Employing Powder Metallurgy Techniques

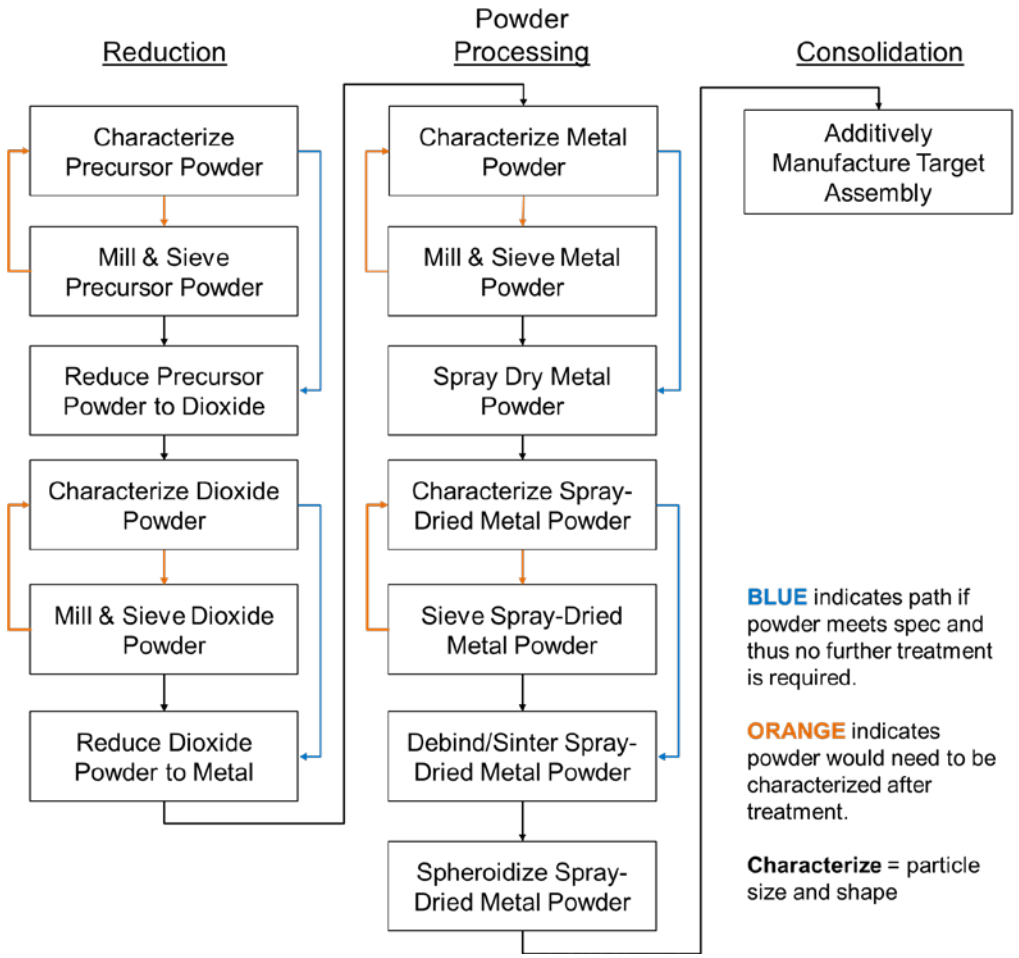
The goals of this effort:

- Understand the requirements for and fabrication of molybdenum target disks and assemblies that will be used in the production of Mo-99.
- Develop a process for fabricating 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**.

Initially, Fabricating Target Disks from Recycled Molybdenum-Containing Compounds Required Numerous Processing Steps

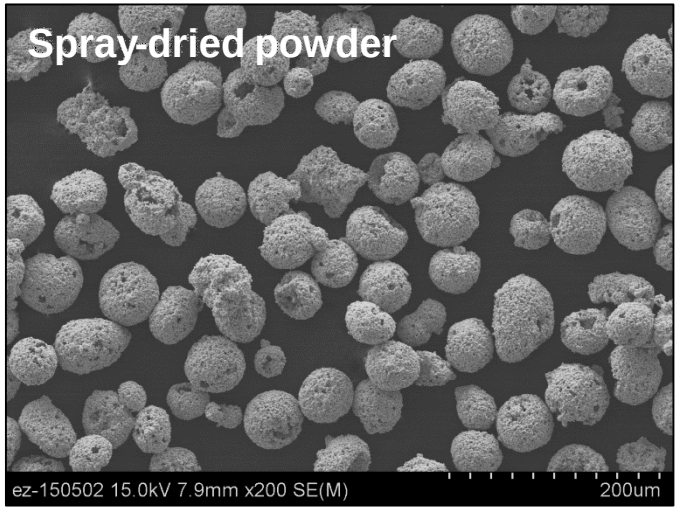


Press-and-Sinter PM Process

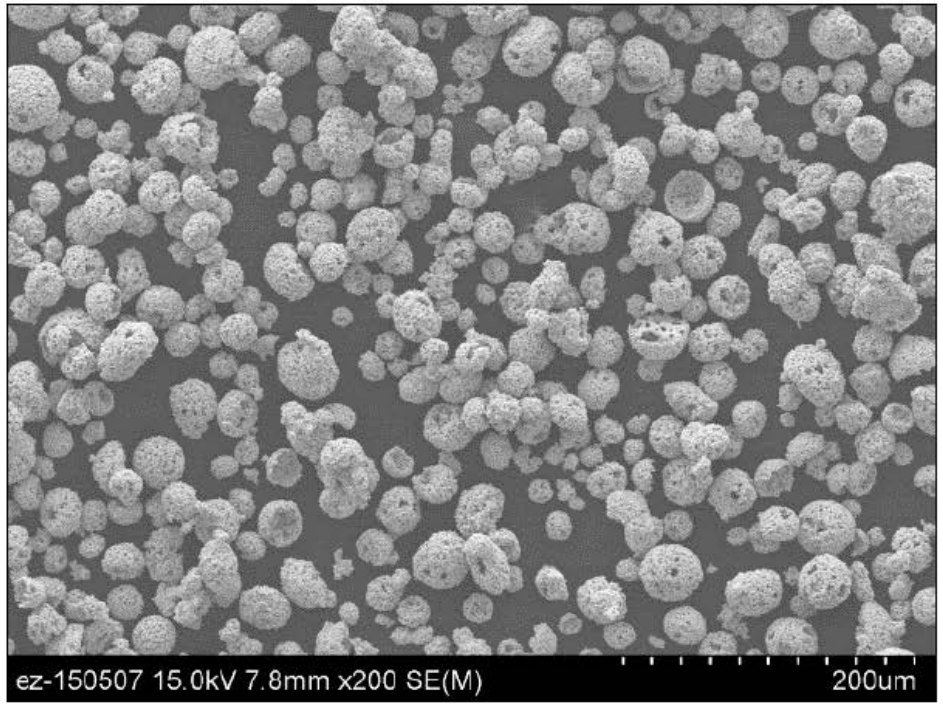


Laser-Melt Additive Manufacture

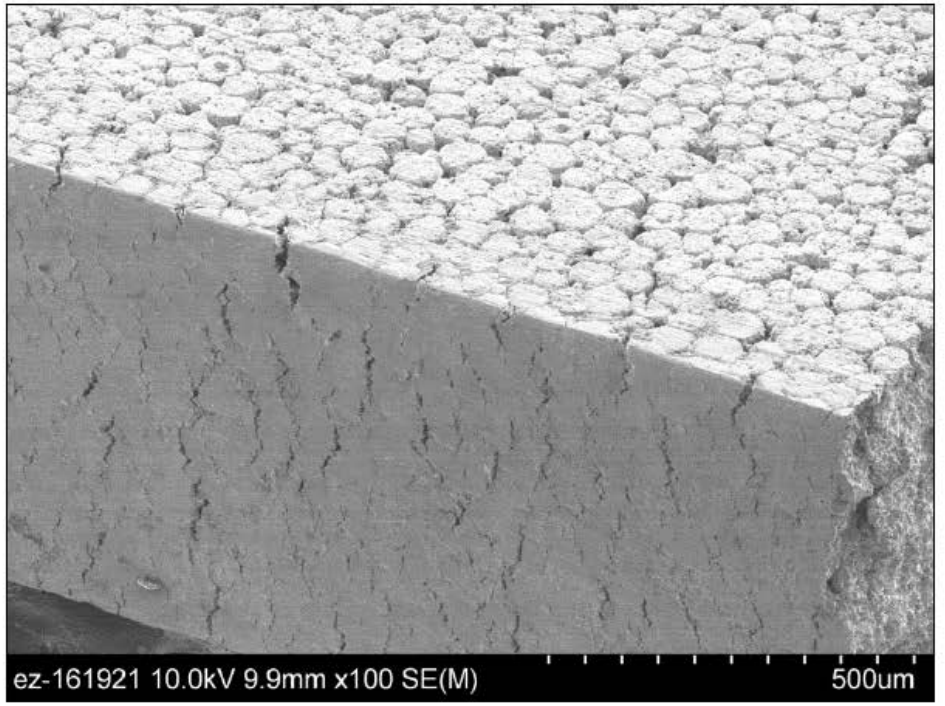
Reduced Powder Is Milled, Slurried and Spray-Dried to Produce "Flow-able" Feedstock



Targets are Fabricated by Compacting and Sintering the Spray-Dried Molybdenum Powder

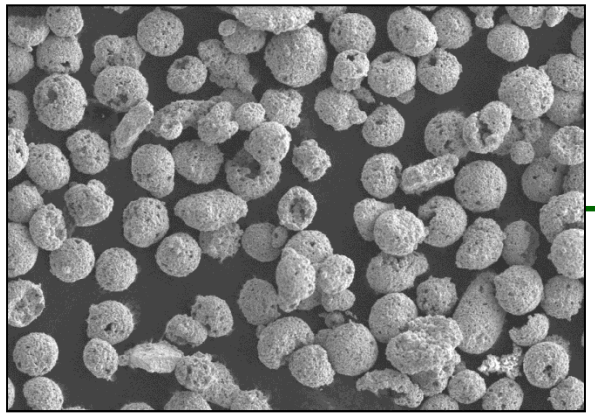


-100/+325 mesh (44 to 149 µm) Starck spray-dried molybdenum powder



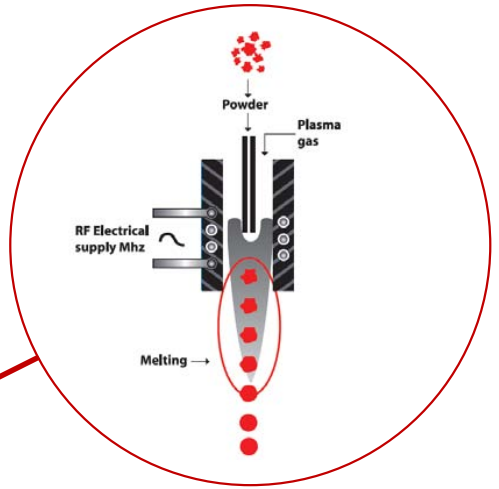
90% dense sintered molybdenum with approx. 5% open porosity

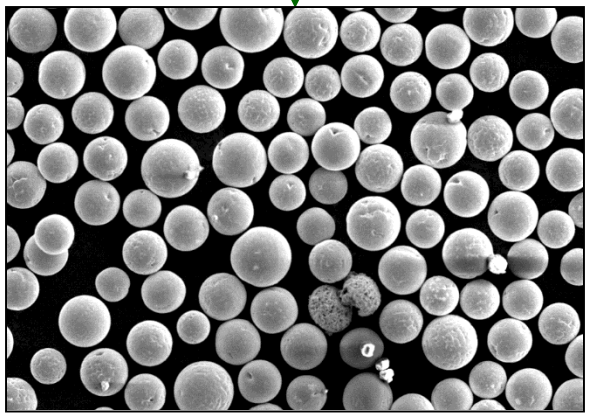
Spray-Dried Powders Can Also Be Spheroidized for Use in the Additive Manufacture of Targets



55 ± 13 um
(-200/+325 mesh)

TEKNA 15 kW Plasma Spheroidization System

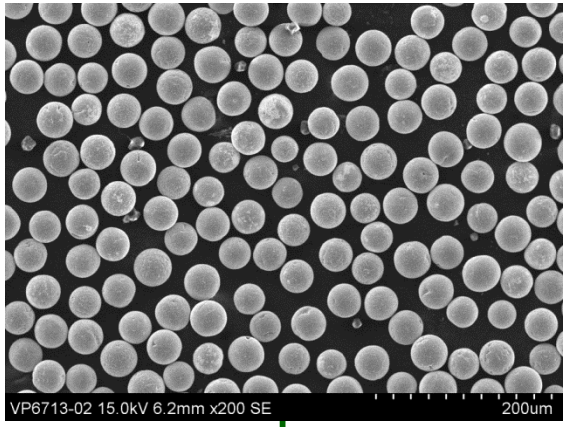


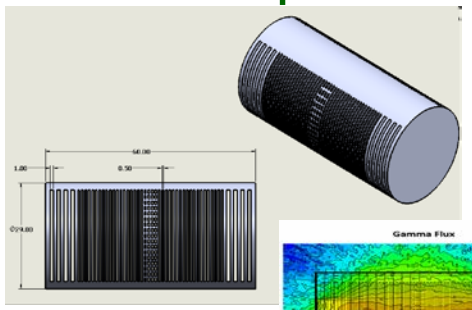


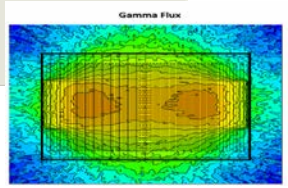
48 ± 9 um

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

Targets and Assemblies are Then Fabricated Employing a Selective Laser-Melt Additive Manufacturing Technique

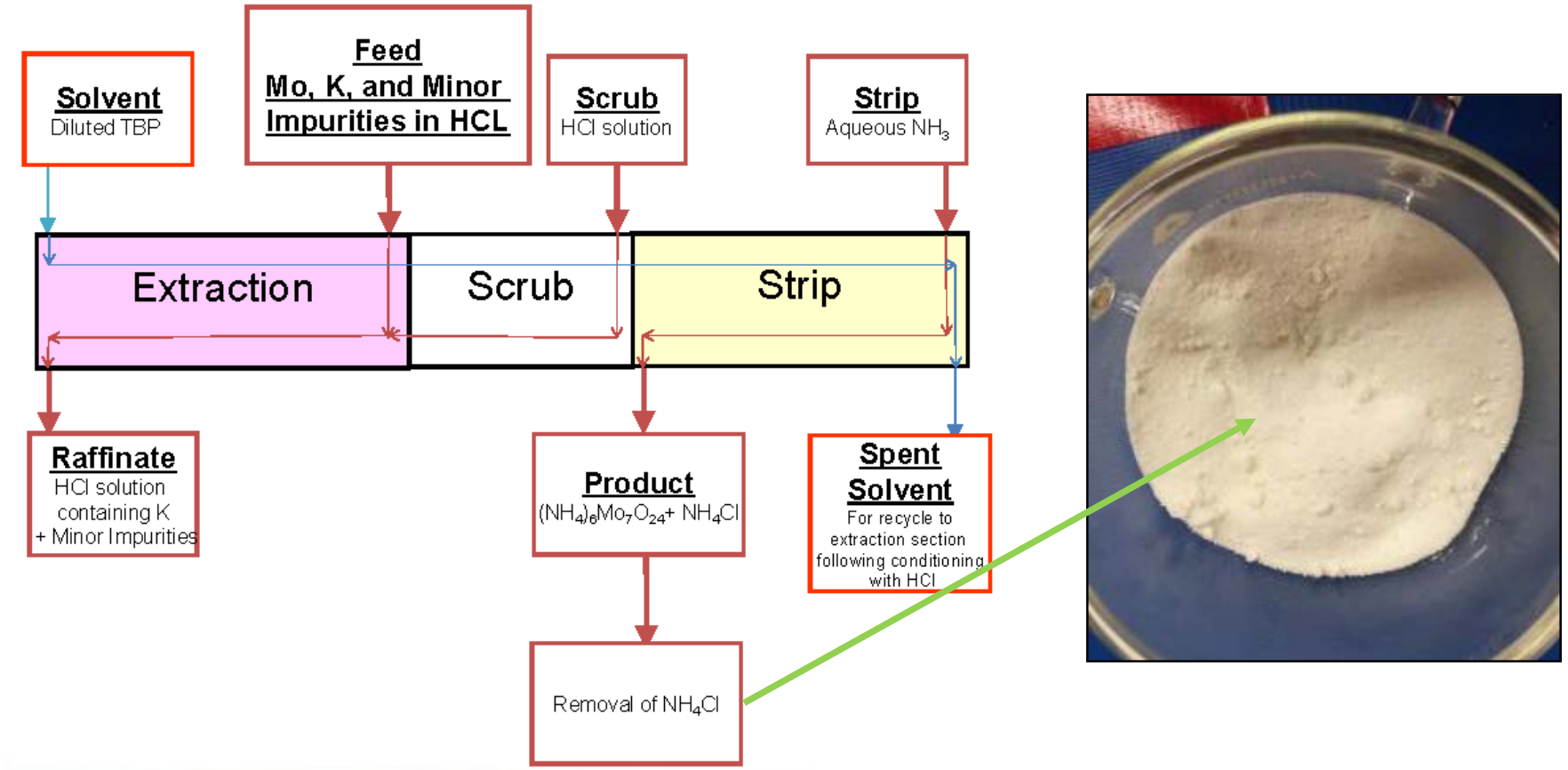






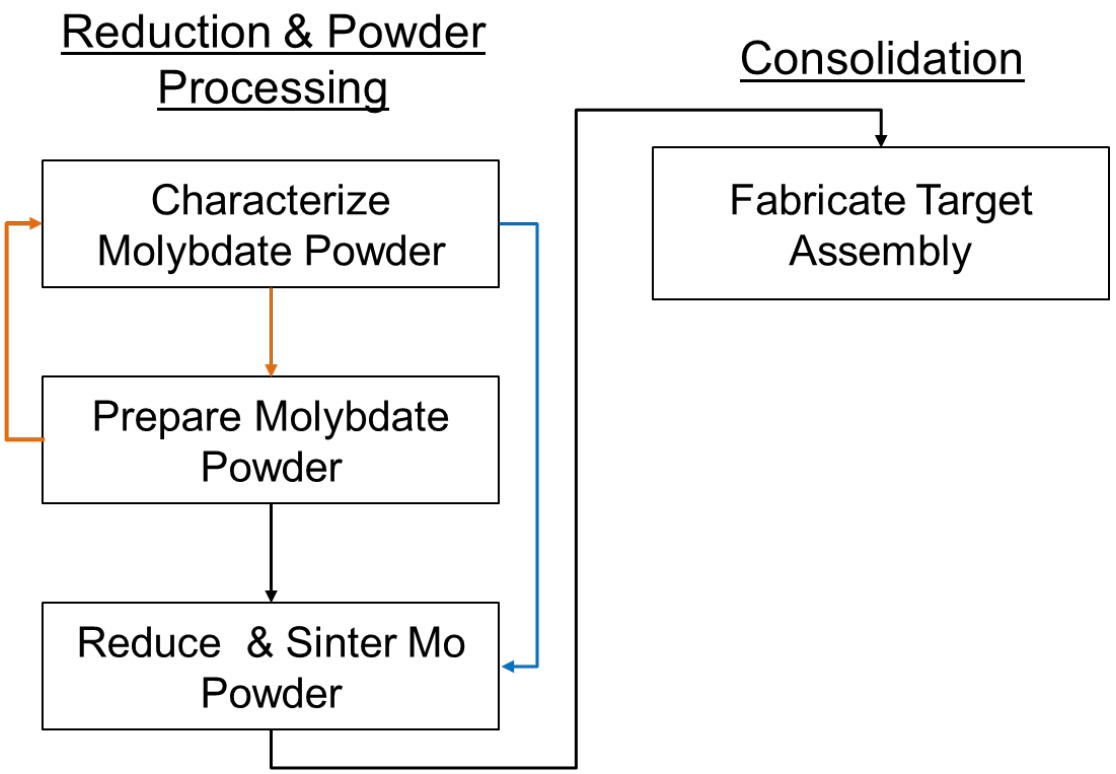
Notional 3D target design printed in stainless steel

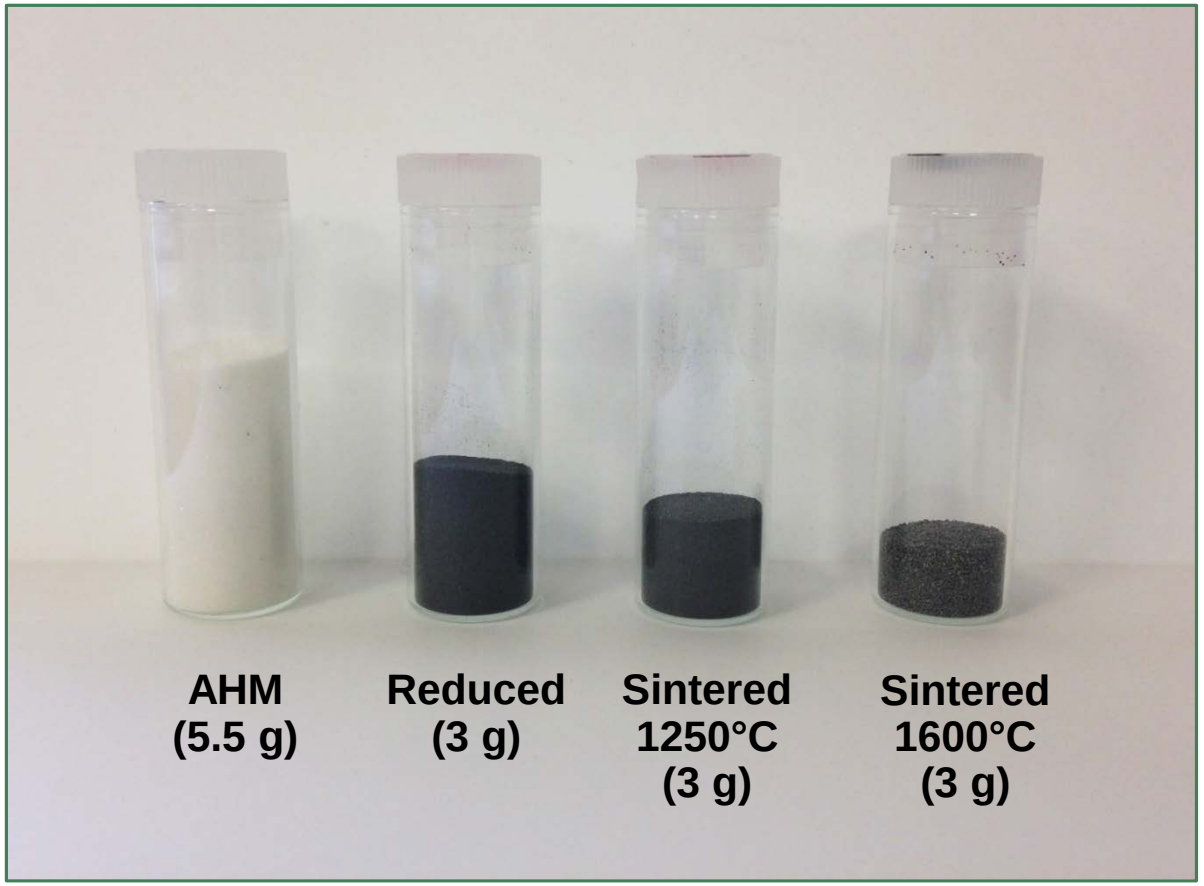
ANL is Developing a Solvent Extraction Process for the Recovery/Recycle of Enriched Molybdenum



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

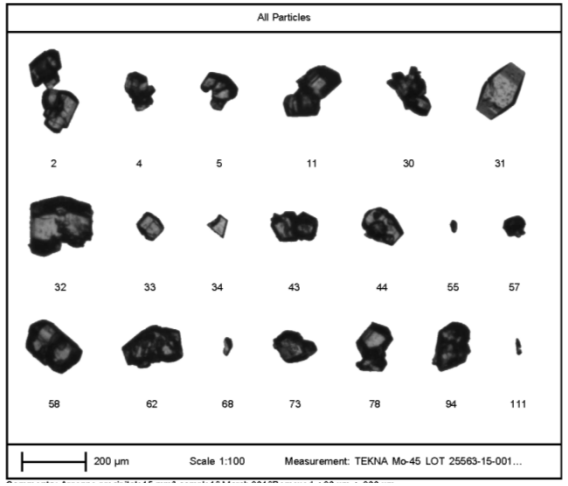
Feedstock Powders for Press-and-Sinter and Laser Melt AM Fabrication Approaches are Now Being Produced Directly from Ammonium Molybdate



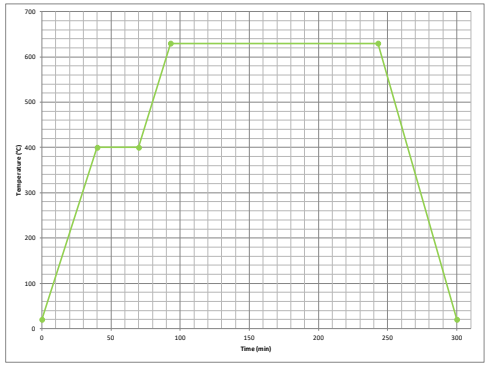


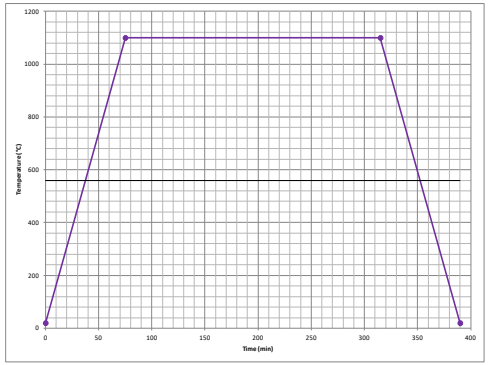
AHMTH is Reduced Using the Two-Stage Process Developed for Molybdenum Trioxide

Sieved



156 ± 62 um
(-100/+200 mesh)





Heat + Hydrogen

$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$

↓

MoO_3

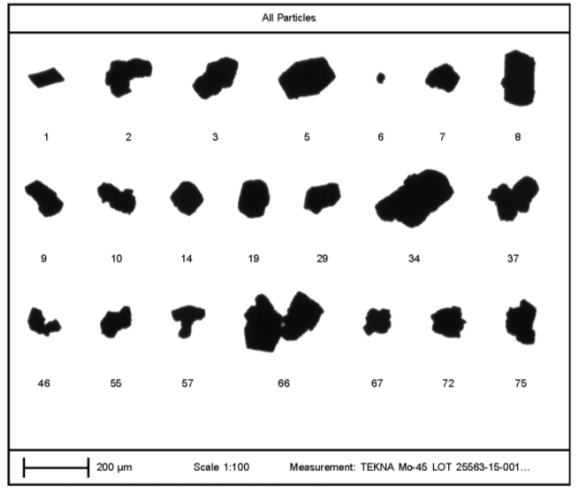
↓

MoO_2

↓

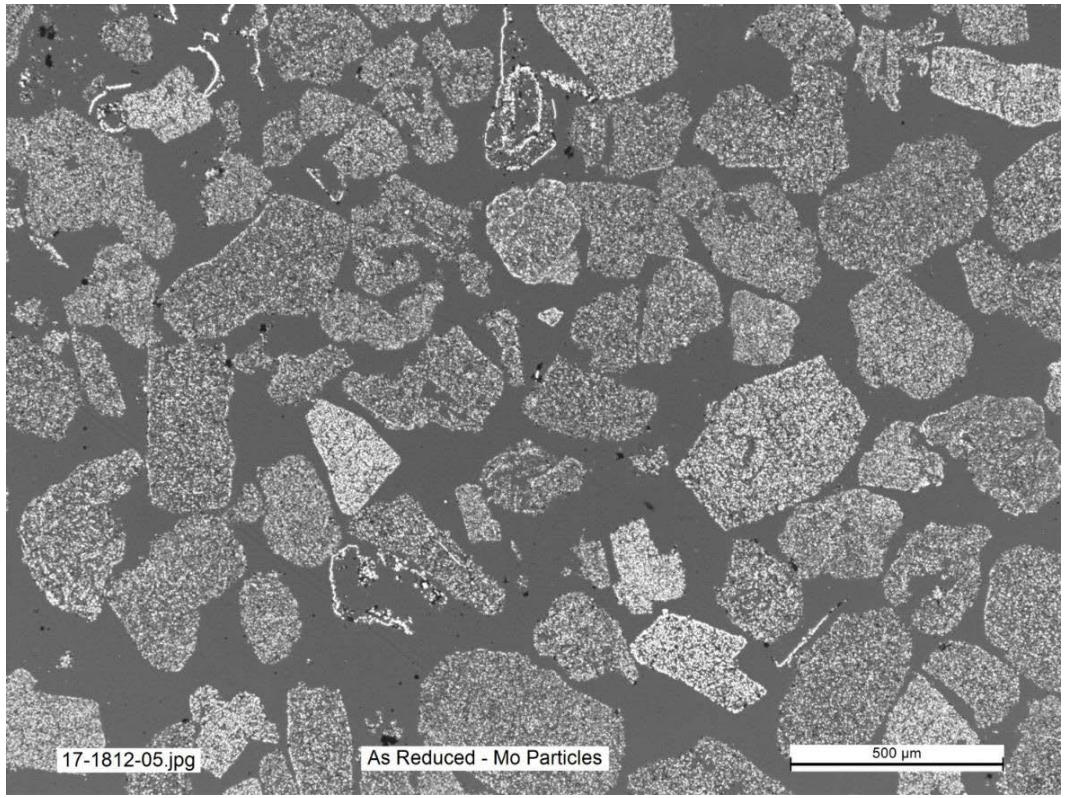
Mo

Reduced

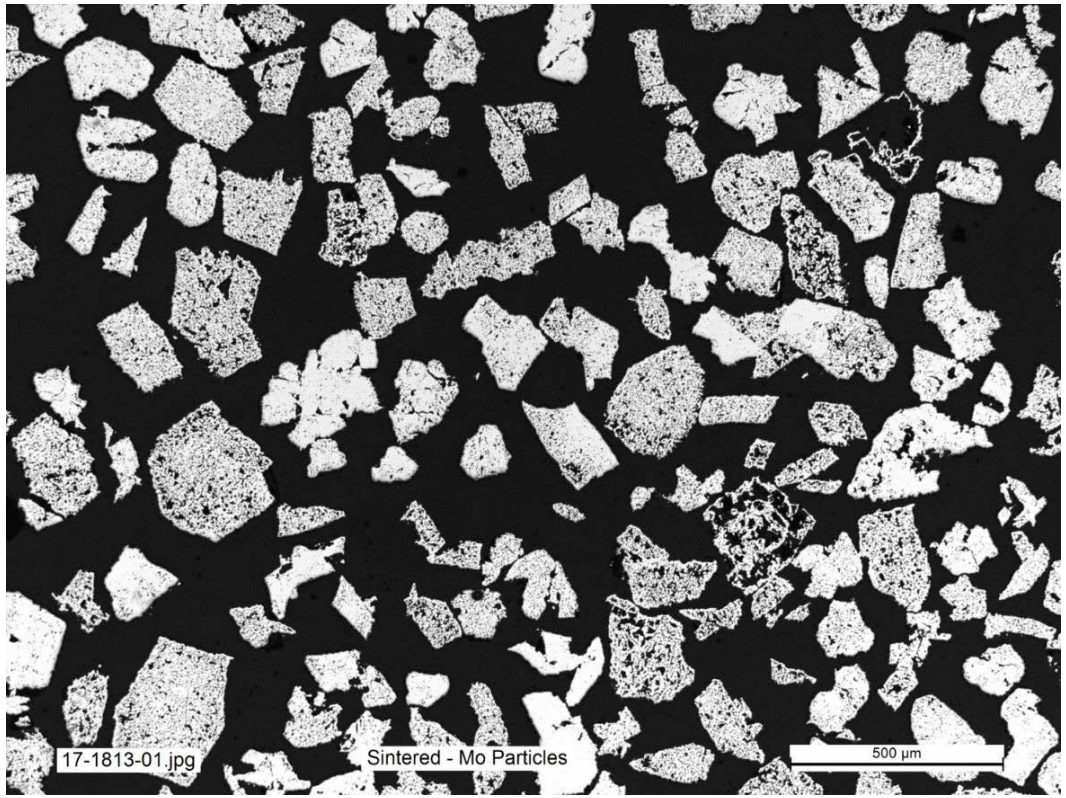


129 ± 41 um

Reduced Materials are Sintered to Produce Powder with the Desired Characteristics

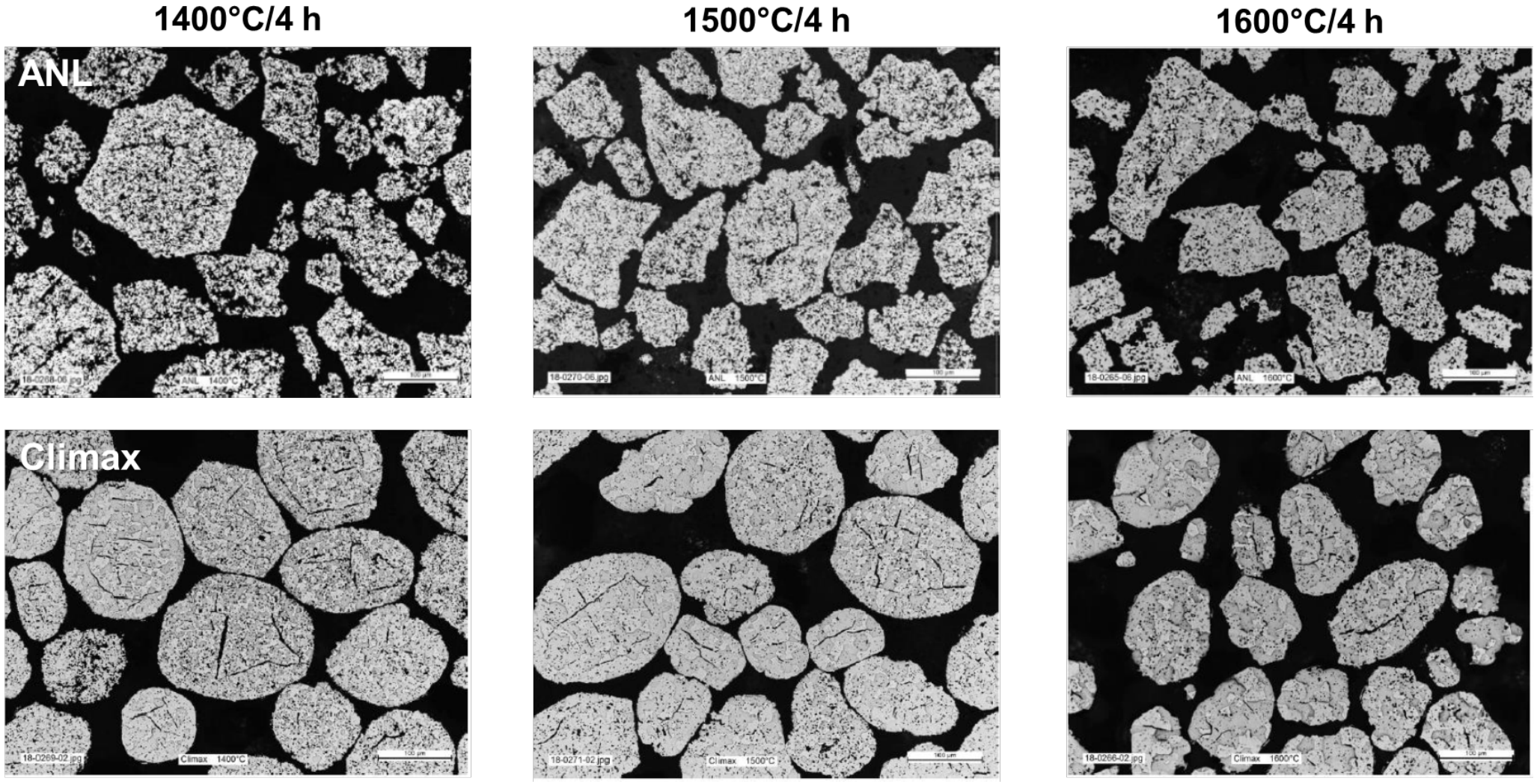


ANL AHM – As Reduced

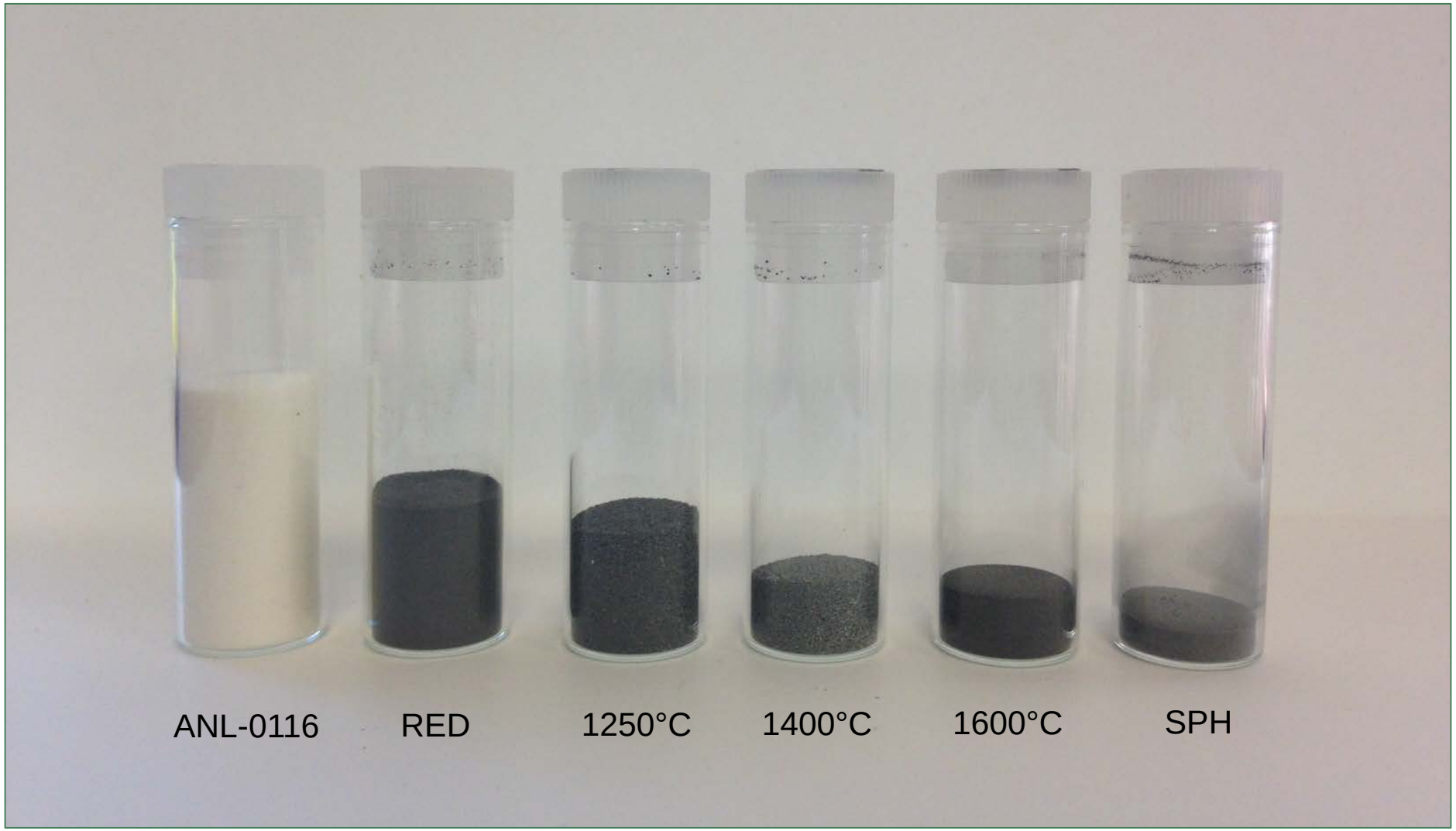


Sintered at 1250°C

Powder Properties are Controlled Via Gentle Milling, Sieving and Sintering



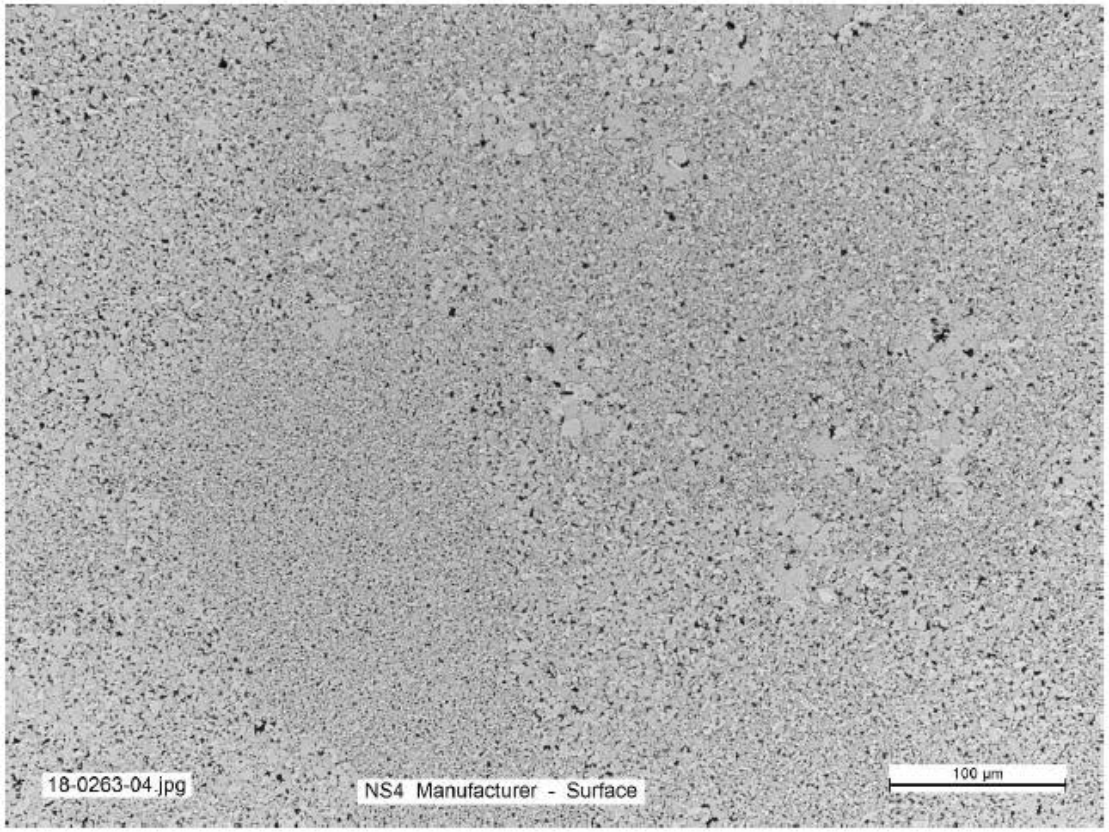
Volume Change is Quite Significant



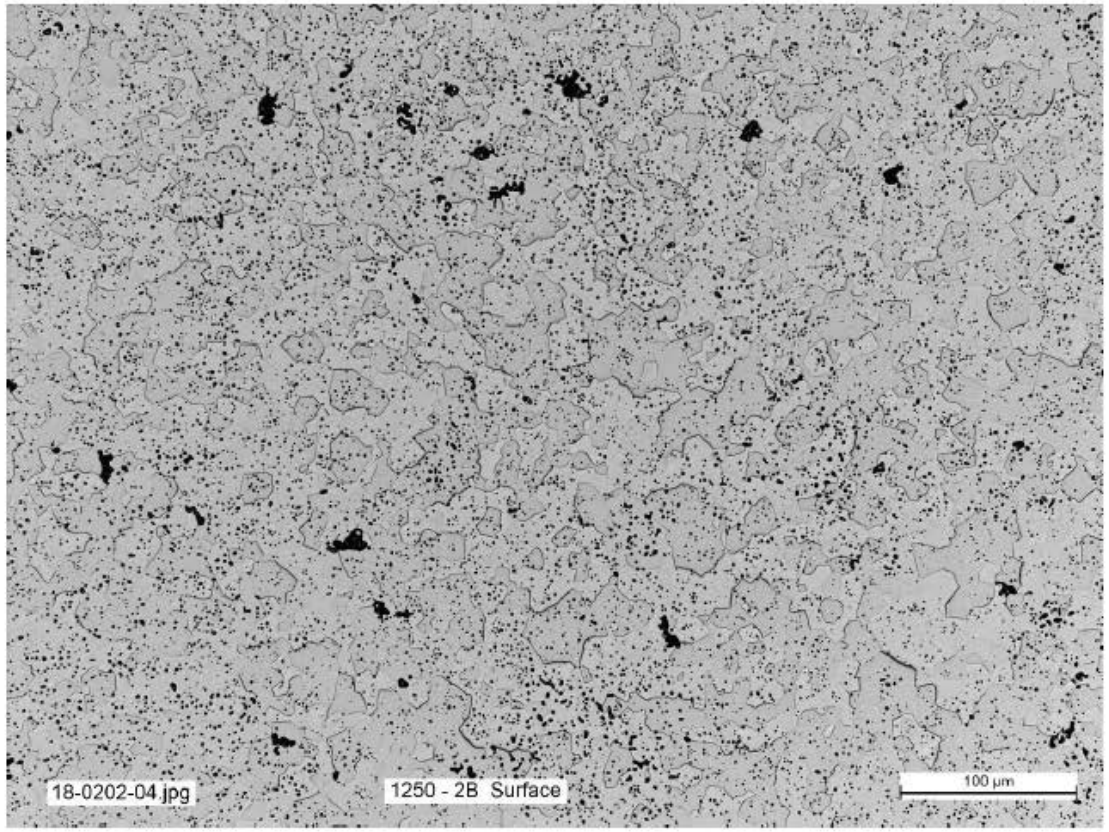
Material required for single target disk (3 g Mo)

ANL AHM was Used to Produce Powders that Mimicked the Characteristics of Spray-Dried Materials

Properties of Sintered Disks to be Used in the Capture Process are Being Optimized



Current NorthStar disk

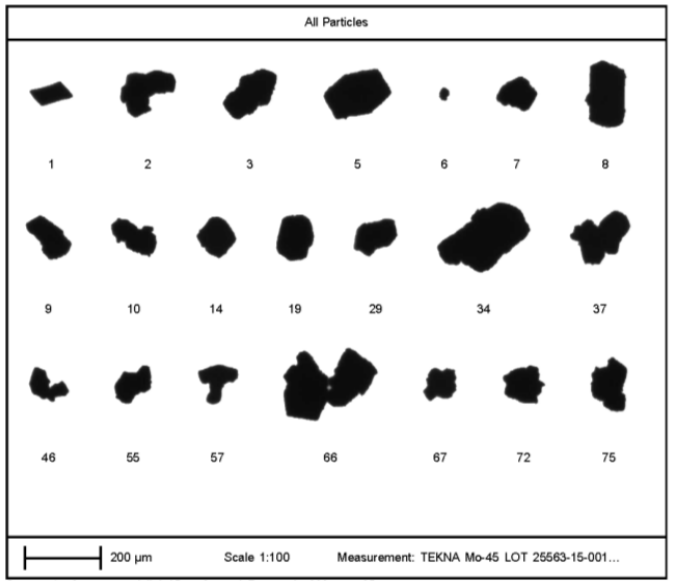


Disk fabricated using powder derived from ANL supplied molybdate

Goal: ~ 90% TD with 8 - 10% open porosity

Reduced AHM Can Also Be Spheroidized

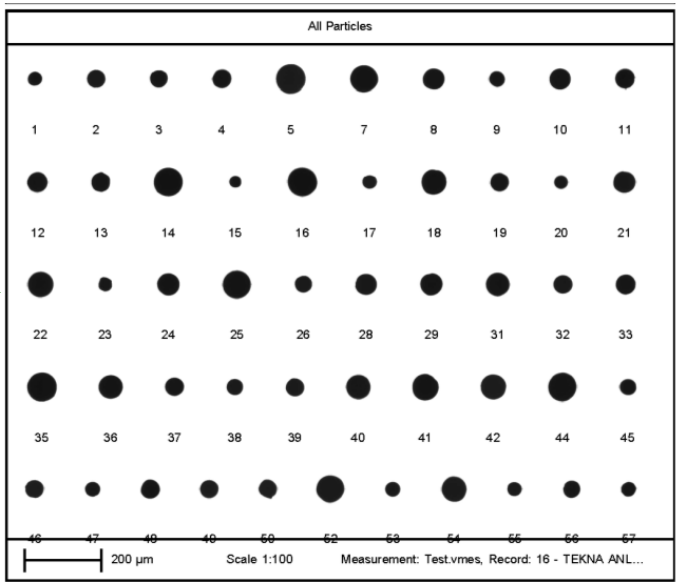
Reduced



129 ± 41 um

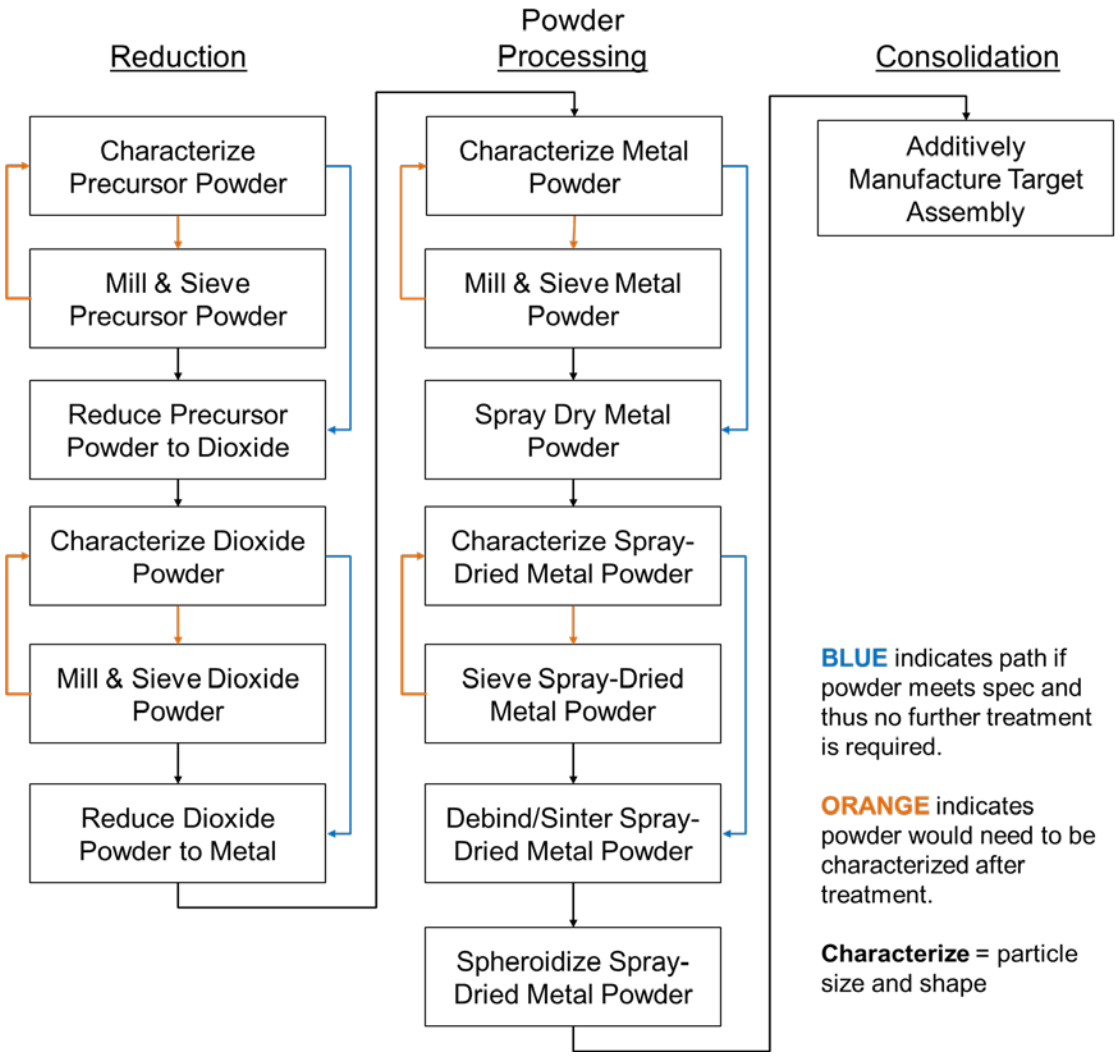
TEKNA 15 kW Plasma Spheroidization System

Spheroidized

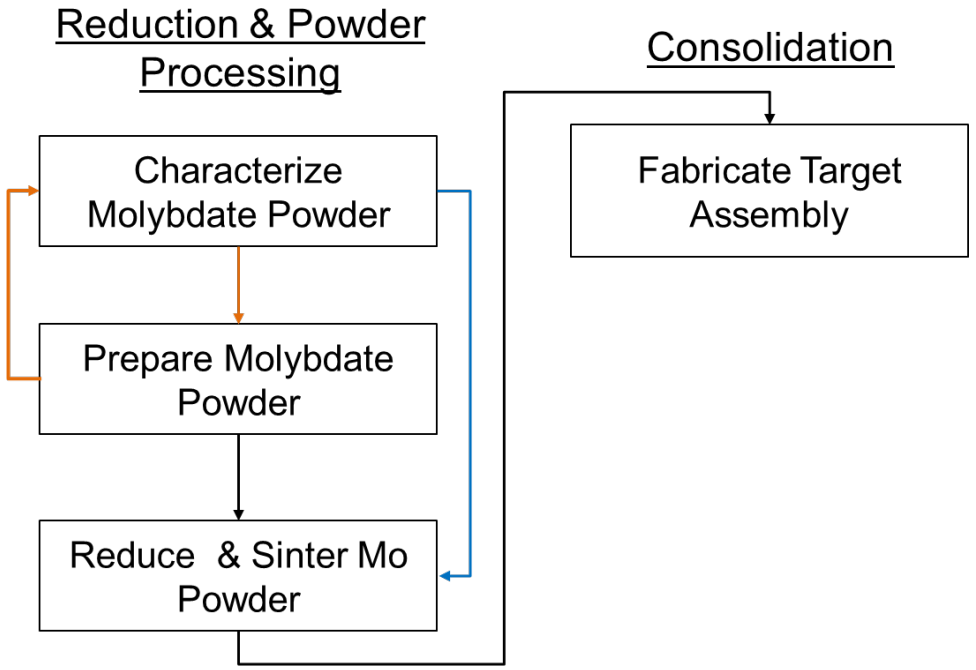


61 ± 14 um

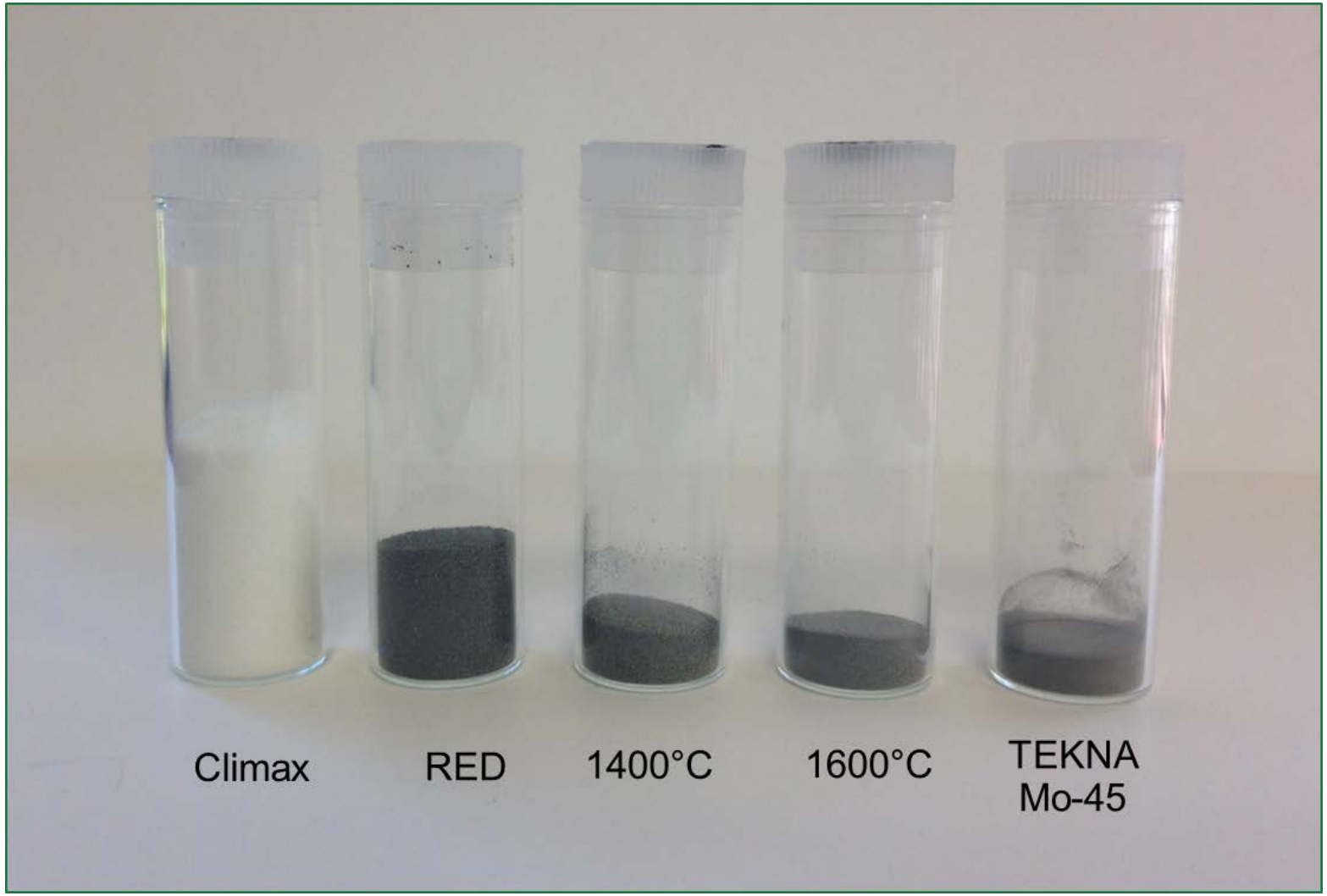
The Goal is to Eliminate Processing and Handling Steps!



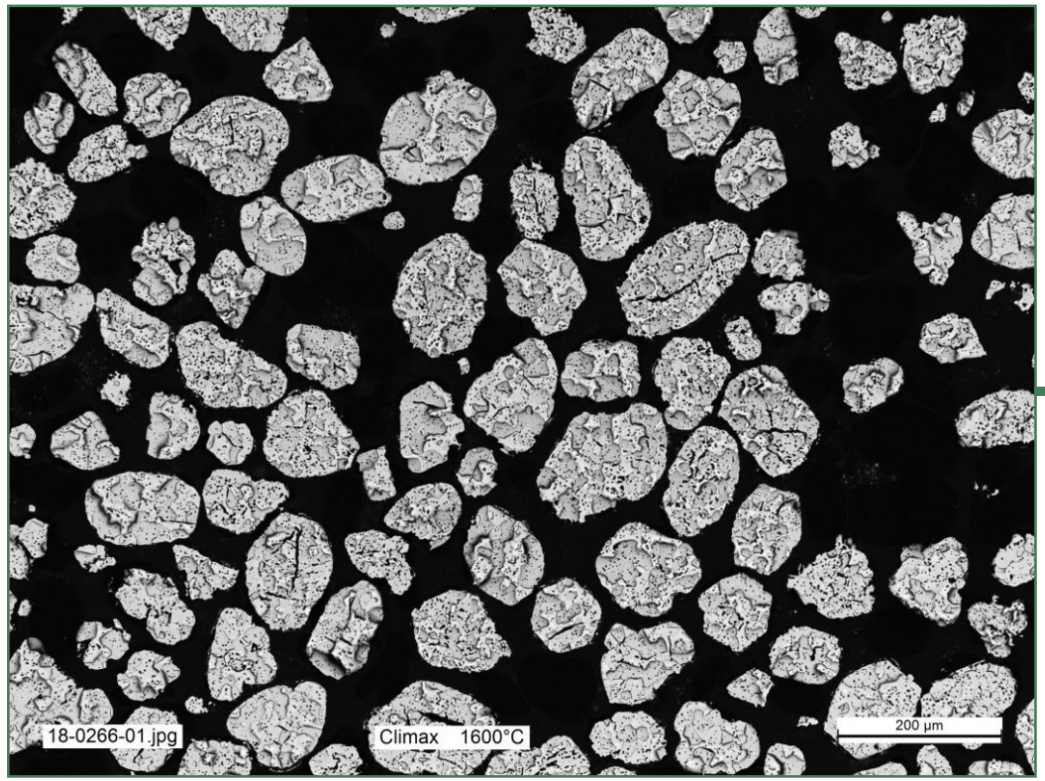
Laser-Melt Additive Manufacture

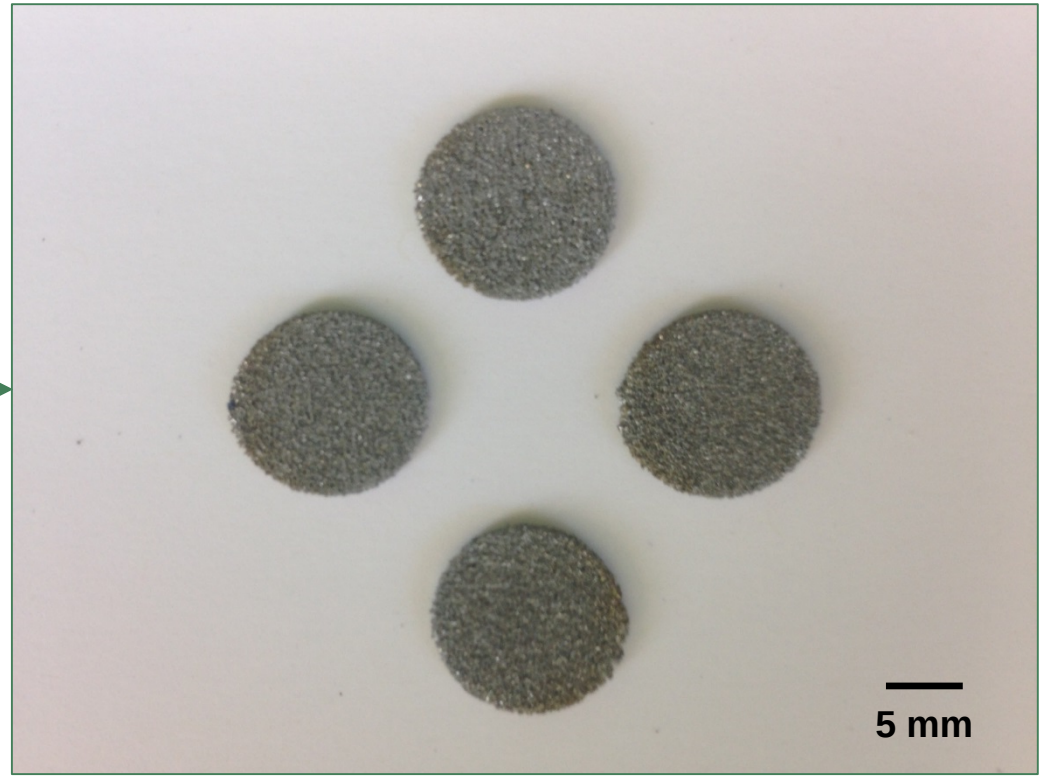


Commercially-Available AHM is Being Used to Produce Feedstock Powder for Laser-Melt Consolidation



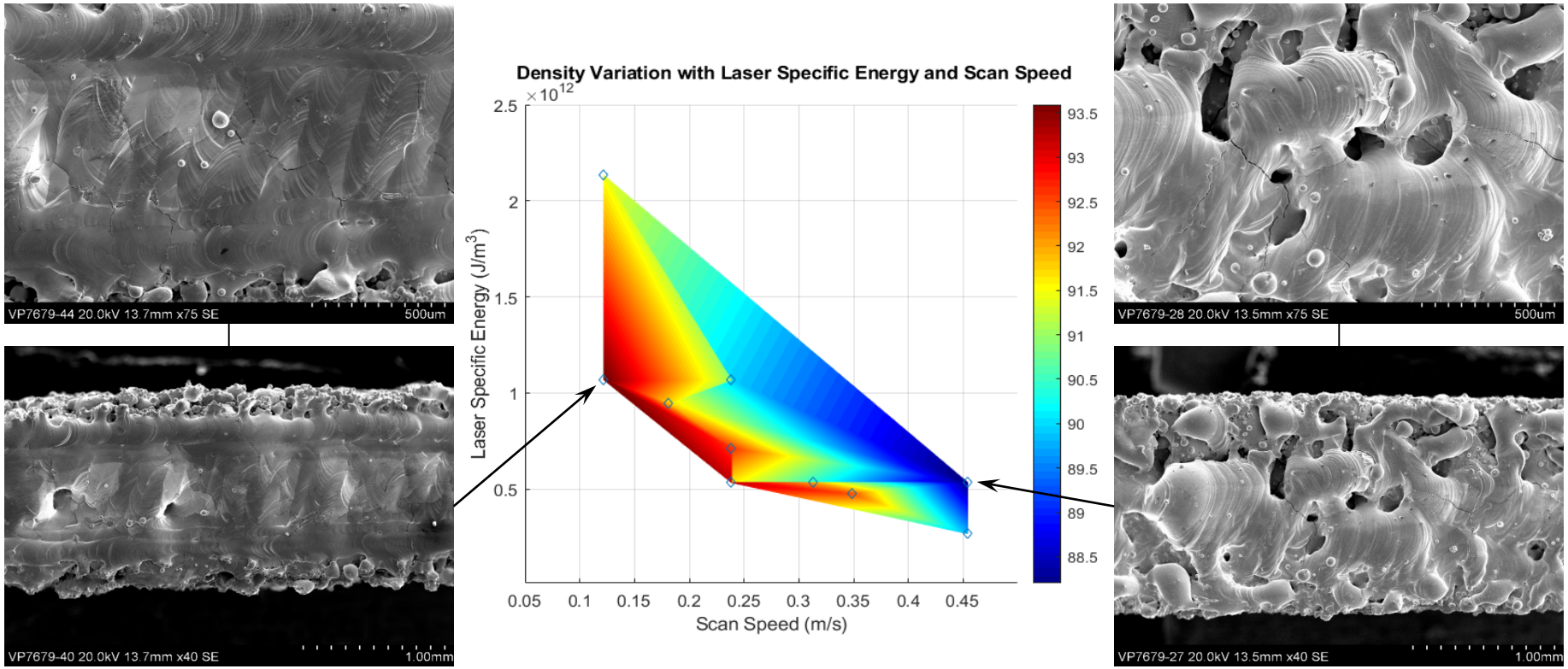
Powder Produced Directly from AHM has been Used to Fabricate Disks Using Laser-Melt AM





86% of TD with 14% open porosity

Dimensionless Analysis is Being Employed to Map and Optimize the Laser Melt Processing of Molybdenum

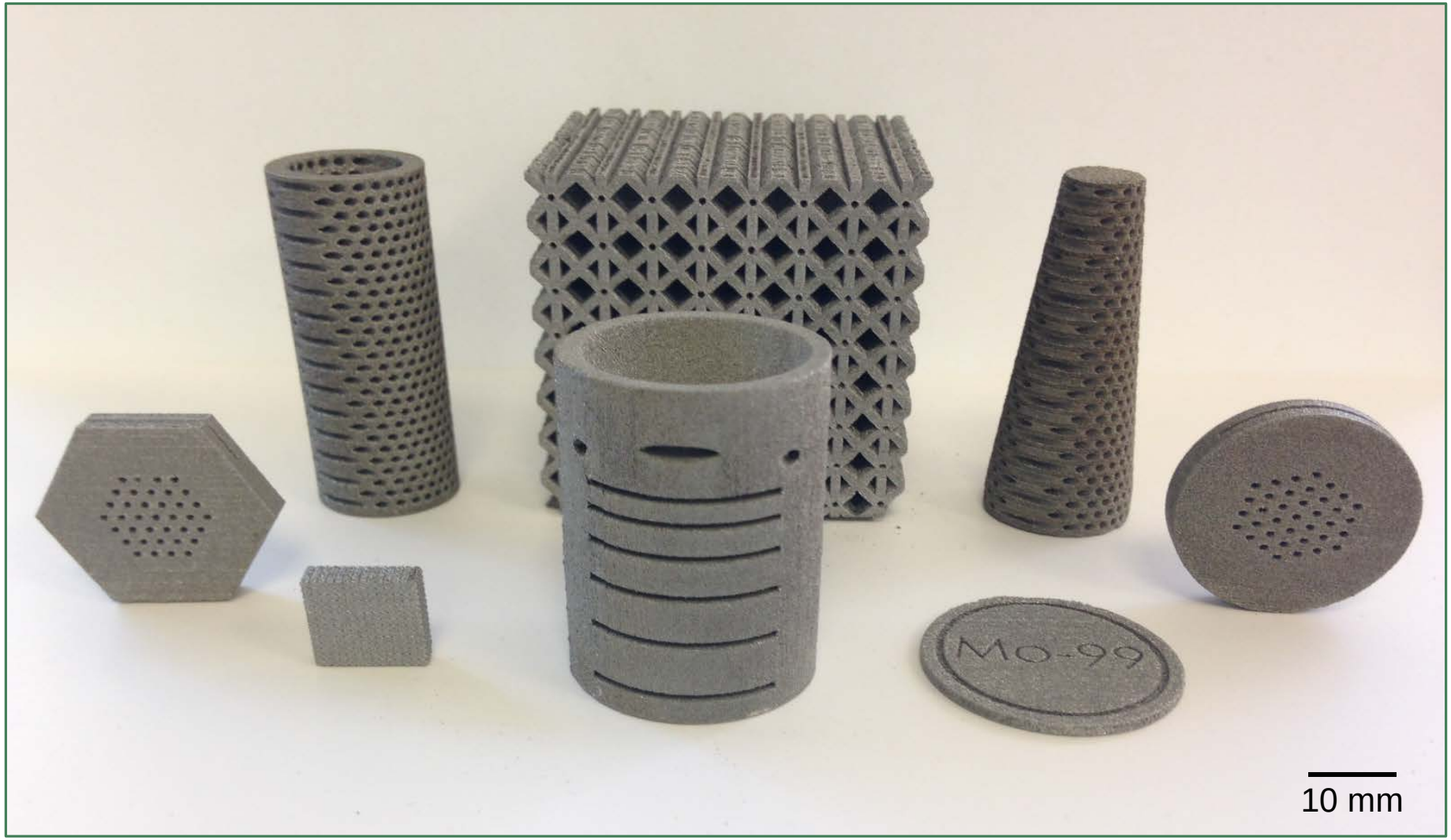


400 W Laser Power
400 µs Exposure Time
50 µm Point Distance
100 µm Hatch Spacing

400 W Laser Power
200 µs Exposure Time
100 µm Point Distance
50 µm Hatch Spacing

Build parameters are non-dimensionalized using material and process properties to enable the application of observations across a broad range of processing windows and component designs.

Practice Makes Perfect…



Summary

- “Targets” are being fabricated from commercially-available and recycled/recovered molybdenum powders employing traditional press-and-sinter and laser melt additive consolidation approaches.
- Feedstock powders are being produced directly from the ammonium molybdate compound being examined at ANL for the recovery of molybdenum from spent radiopharmaceutical solutions, eliminating numerous process steps and minimizing material losses.
- Feedstock powder properties can be varied and thus optimized for both consolidation techniques.
- “Dimensionless analysis”, an experiment-driven process modeling approach is being used to fully understand the effects of laser melt additive build parameters on important properties such as density, dimensional tolerances, and surface finish.