

# Introduction to: Cryogenic Milling & Spark Plasma Sintering



[www.calnanocorp.com](http://www.calnanocorp.com)

A photograph of a clean, modern laboratory. In the foreground, a person wearing a white lab coat and a blue hairnet is seated on a black office chair, facing away from the camera. In the background, another person in a white lab coat and blue hairnet is standing near a piece of equipment. The room has white walls, a tiled floor, and recessed ceiling lights.

# Cal Nano Mission

*Bringing next generation materials to market  
with cutting-edge technologies*

At Cal Nano, we envision a world in which our advanced technologies are used to help make the most innovative products on this planet and beyond.

We are trusted by global leaders to help push the boundaries of applied material science by utilizing our unique technical expertise and vision.



# Our History

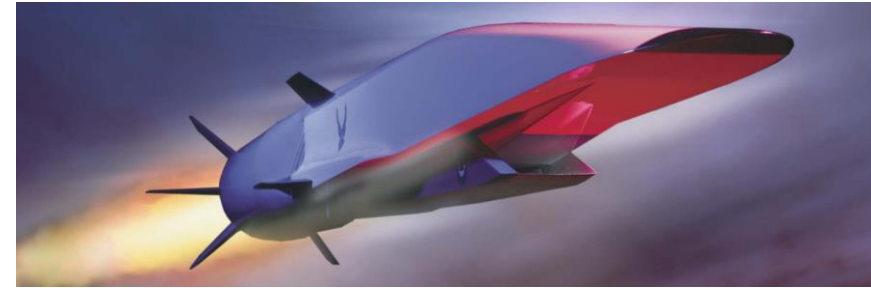
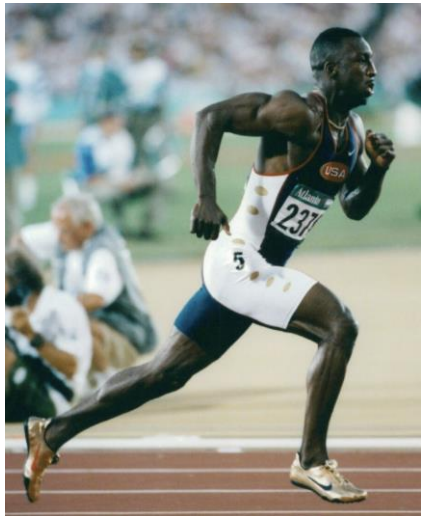
## From Olympic Shoes to Rocket Science



Veritek technologies + OML-EFORM +  
Due Diligence Cryogenics



**“The Golden Shoes”**  
worn by Michael Johnson to  
win several gold medals and  
set WRs at the 1996  
Olympics used special cold  
forged MMC (Metal Matrix  
Composite) track spikes made  
by Omni-Lite.



Cal Nano was founded with the focused goal of developing and implementing next generation materials into world changing products. Finding it's first commercial success implementing a nano reinforced MMC composites spikes used by adidas in the adizero Prime SP making it the new “Worlds lightest sprint shoe”

# What We Do

## R&D & Manufacturing Toll Services

Provide toll services to universities, national labs, start-ups, and large corporations that are in need of SPS and cryomilling  
Ranging from making a few samples to full-scale R&D & production programs

## Vertically Integrated materials R&D&T

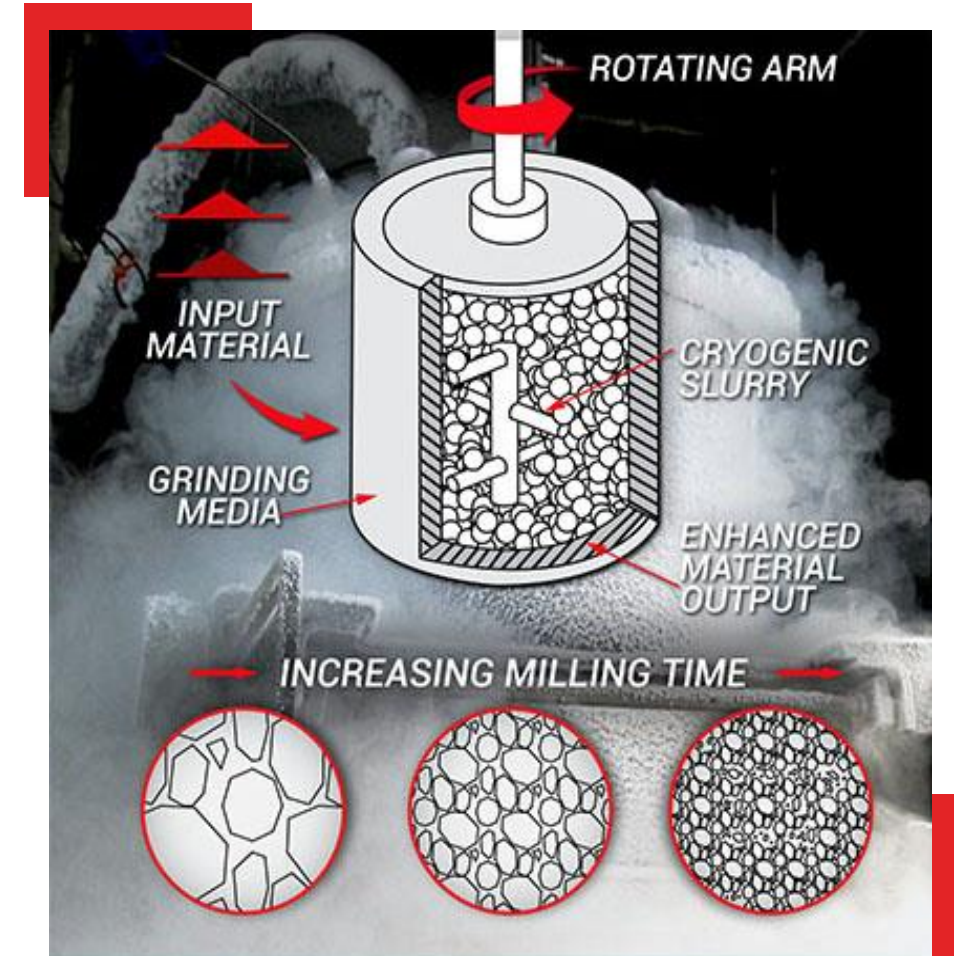
One-stop shop for all of our customers.  
We act as an extension of their R&D departments  
SPS Tooling & Sample Pre & post work  
- Lathe - Wire EDM - Planetary ball milling -  
Analysis/Testing - Tensile - Compression - Hardness  
- Micro Hardness - TRS - Surface Roughness -  
Particle Size Analysis  
**+ network of industry experts and resources**

# Cryogenic Milling

**Cryo-milling** is a specialized mixing / grinding process (attrition) conducted in an inert cryogenic environment ( $-190^{\circ}\text{C}$ )

## R&D and Production

- ✓ 0.5-1 kg capacity, R&D / pilot (~15kg+/week)
- ✓ 7-10 kg capacity, pilot production (~150kg+/week)
- ✓ 20-40 kg capacity coming soon (~600kg+/week)





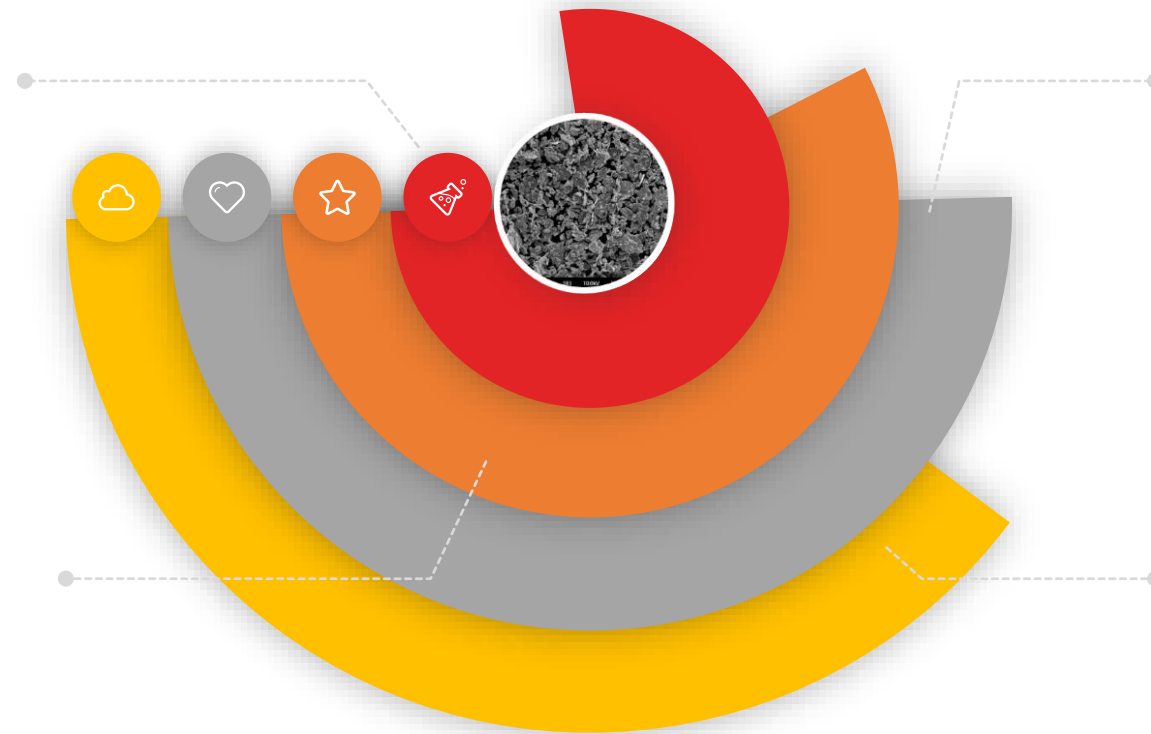
# Cryogenic Milling Benefits

## Particle Size Reduction

Rapidly reduces particle size in materials that otherwise smear (plastics, low density metals, water soluble etc.)

## Custom Alloys & MMCs

Cryo-milling produces uniform mechanical alloys in 1/5 the time of standard ball milling processes, while also preventing agglomeration and stratification of blended constituents or reinforcements of different sizes and densities (ODSs, MMCs, etc)



## Material Properties Improvement

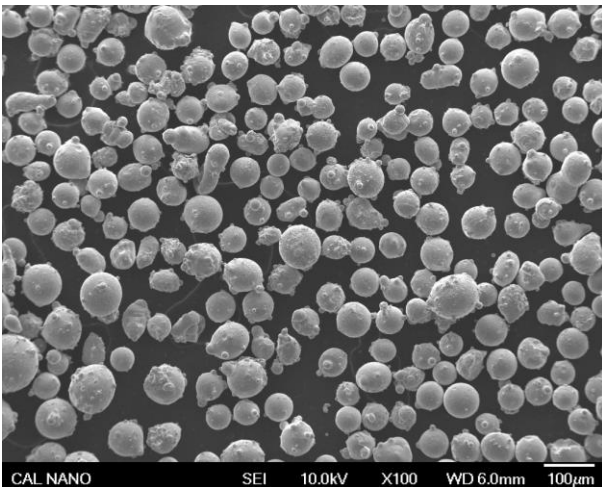
Cryo-milling can often double the strength of a given material system without changing its chemistry due to creating nanocrystalline grain structures.

## Moisture/Oxygen/Heat Sensitive Materials

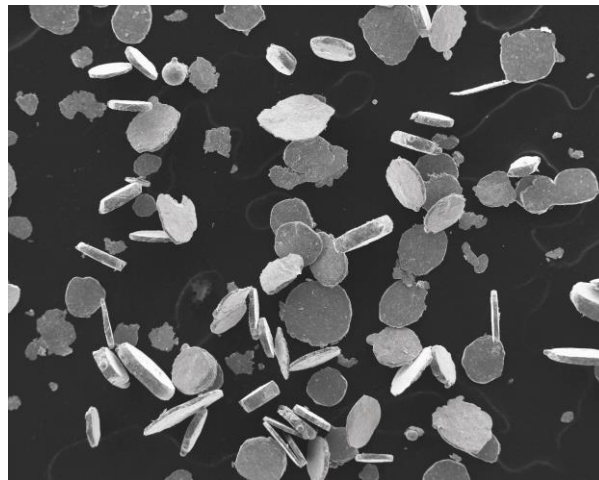
Materials remain fully protected from oxygen, moisture, or heat damage often experienced in other high energy powder processes.

# Powder Morphology

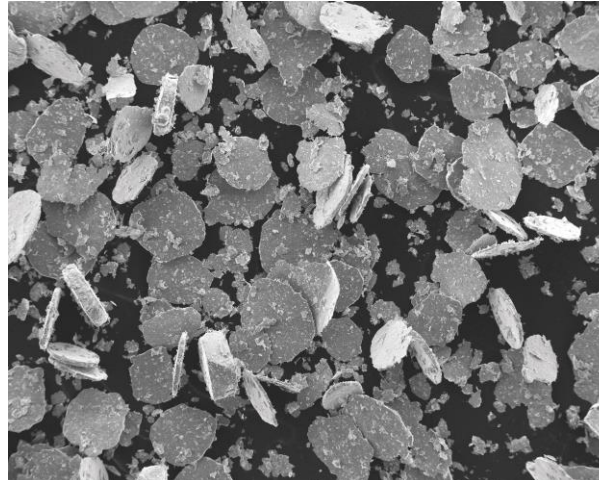
Metallic powders generally become flake like before layering, cold-welding and returning into more irregular low aspect ratio shapes. (Note, every material system will have a unique morphology)



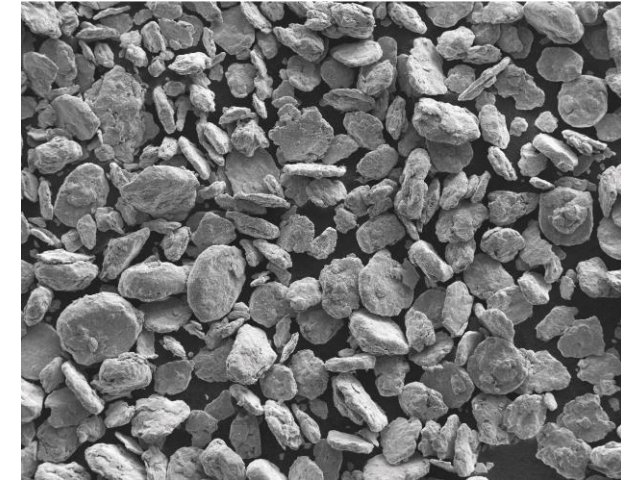
As received  
(Spherical GA powder)



Flattening  
(1-2hrs)



High Aspect Ratio Flake  
(3-7 hrs)

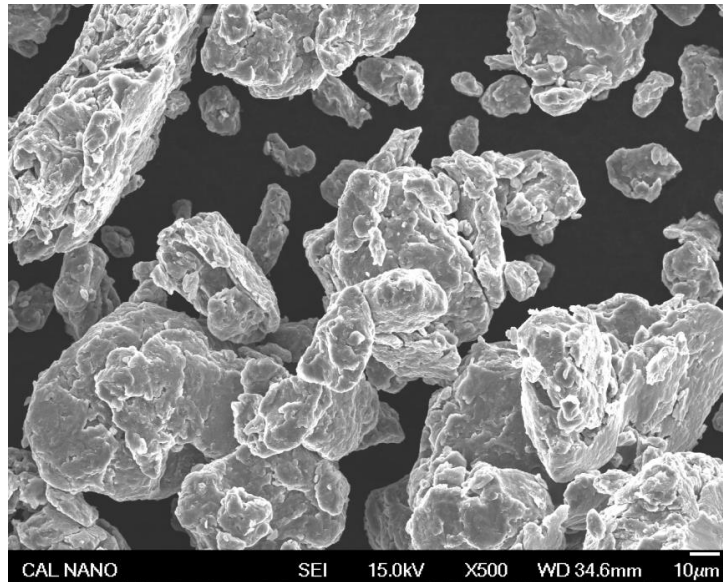


Agglomeration  
(8+ hrs)

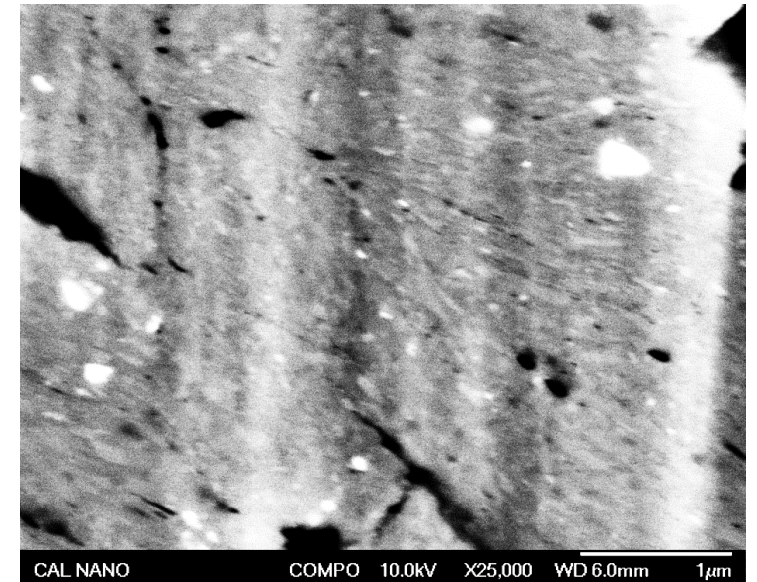
# Cryogenic Milling Post Processes

## Cryo-Milled Powders can be used for:

- HIP
- SPS
- Hot Press
- Press and Sinter
- CIP and sinter
- MIM
- Thermal Spray
- Cold Spray
- Pigments
- Catalysis
- Composite Fuels
- AM



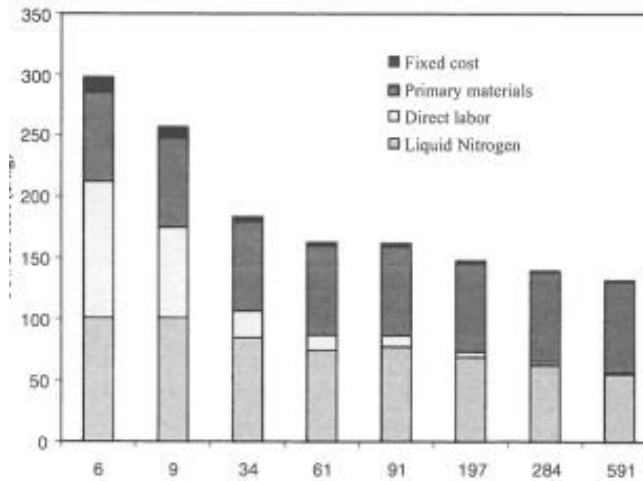
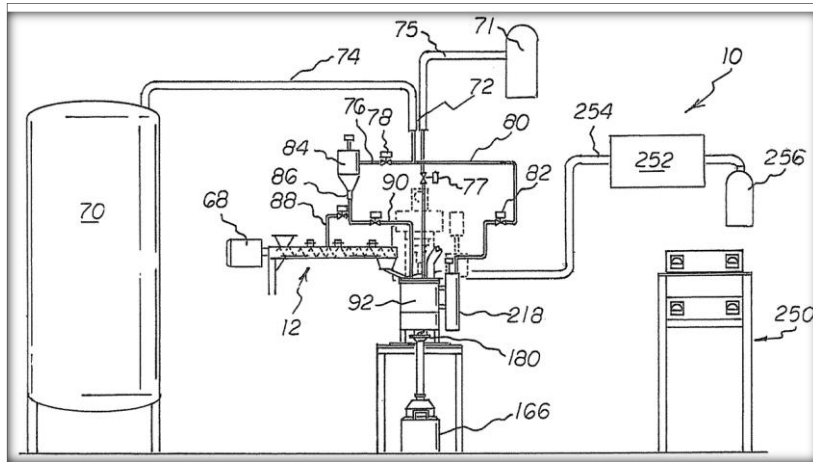
**Conventional powder sizes maintained**



**Nano Grains and homogeneous distribution of secondary phases imbedded throughout particles**



# Cryo-milling Scalability



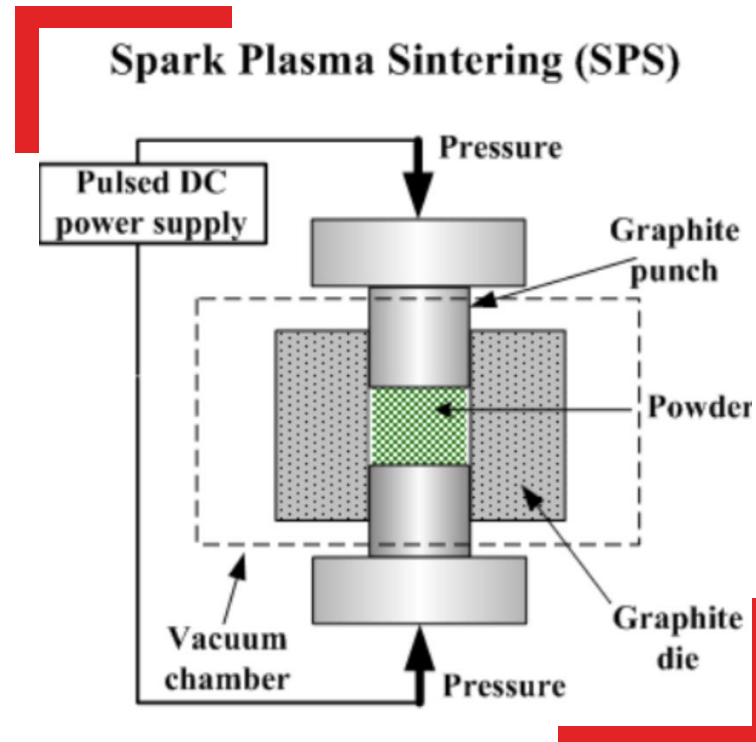
Example of large-scale batch attrition milling

**Cal Nano has a patent for high volume Cryo-milling of flammable or nitrogen sensitive metallic materials**

# SPARK PLASMA SINTERING "SPS"

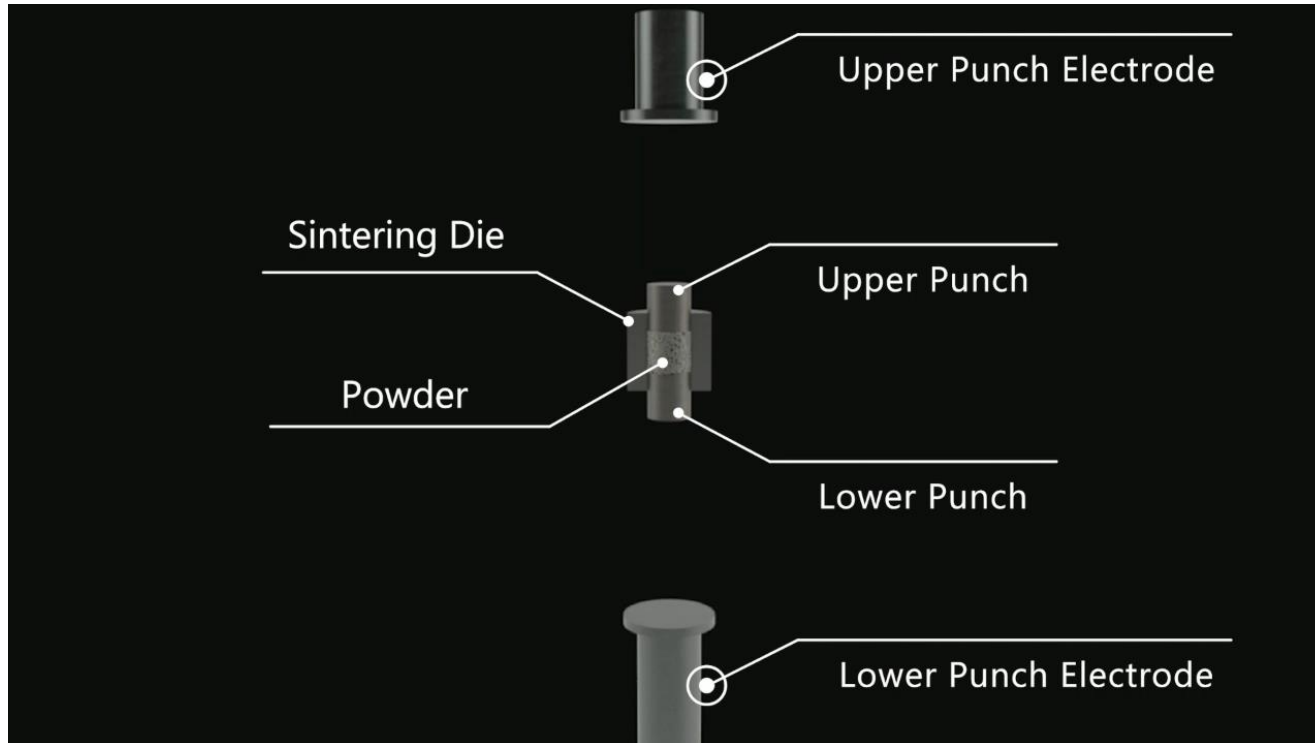
aka Field Assisted Sintering Technique "FAST"

A novel process that can rapidly create materials and components with unique properties that are not possible with traditional manufacturing techniques.



# SPARK PLASMA SINTERING "SPS"

aka Field Assisted Sintering Technique "FAST"



SPS presents many unique characteristics:

- ✓ Controlled atmosphere (vac, inert, forming)
- ✓ Precise application of pressure via direct axial loading resulting in controlled, repeatable density up to 100%
- ✓ Pulsed high current density discharged directly through the material and die assembly
- ✓ Enhanced diffusion bonding ideal for homogeneity, dissimilar material bonding, FGM and composites
- ✓ Minimum time at temp minimizing undesirable crystal & grain growth



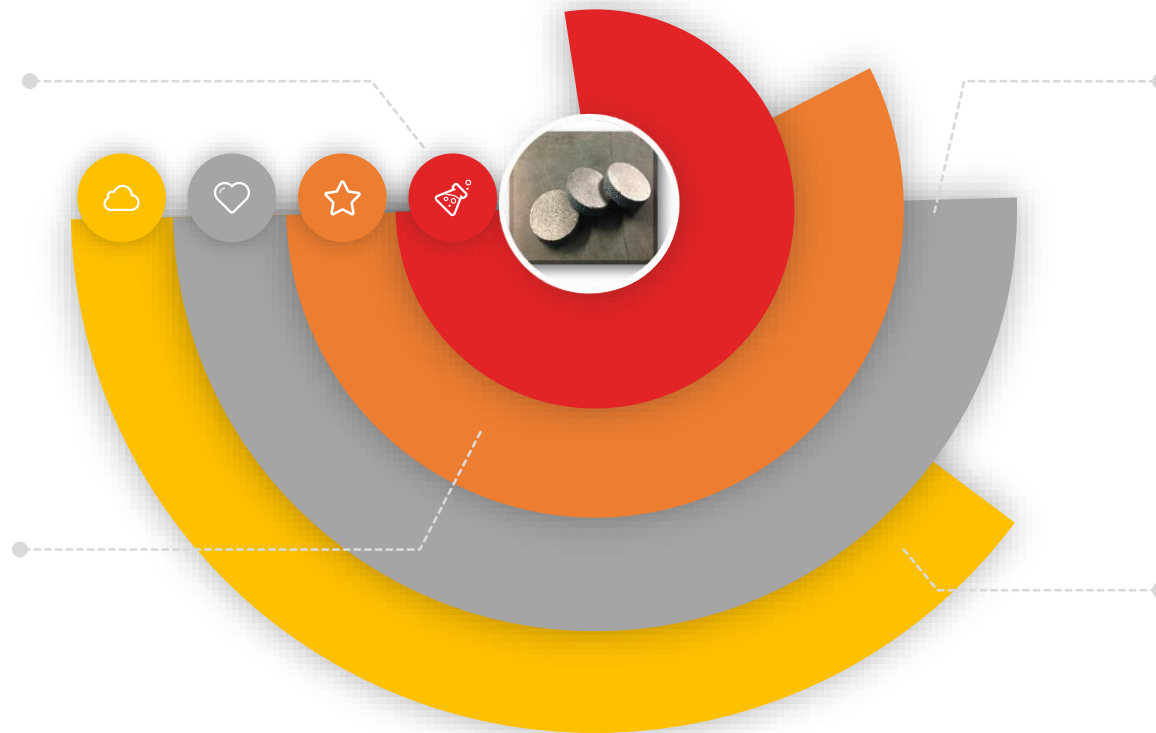
# SPS Benefits

## Extremely Versatile

Low to ultra high temperature capabilities (100-2400°C) make SPS ideal for all metallic and ceramic applications. Controlled porosity, from “flash sintering” up to 100% dense is possible

## Shorter Cycle Times

Rapid and repeatable sample production ensuring R&D efforts scale to commercially, 10-100x faster than standard furnace sintering, retain smaller grain size of materials



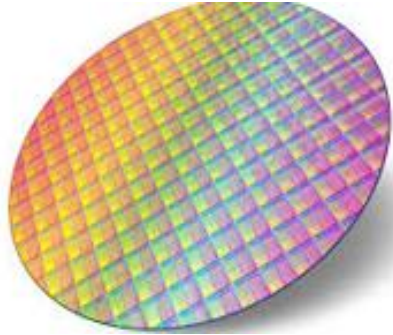
## Cost Effective

SPS is more cost effective than traditional sintering methods with energy savings exceeding 90% in some cases due to faster sintering rates. Near net shape sintering also reduces machining waste and costs.

## Bonding & Functionally Graded Materials

Diffusion bonding of dissimilar materials and the ability to create functionally graded materials adds to SPS capabilities. Also capable of bonding for repair operations.

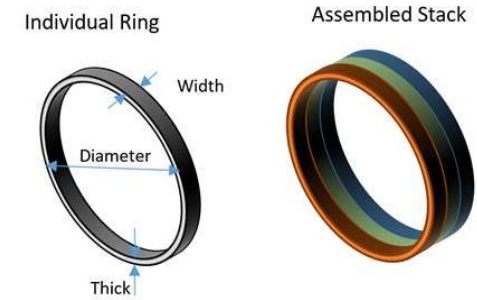
# Versatility of applications



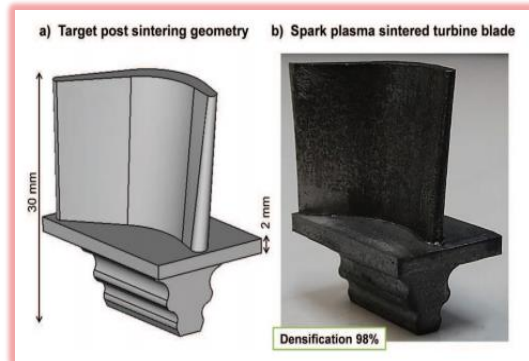
Wafers / Targets



Thermoelectrics



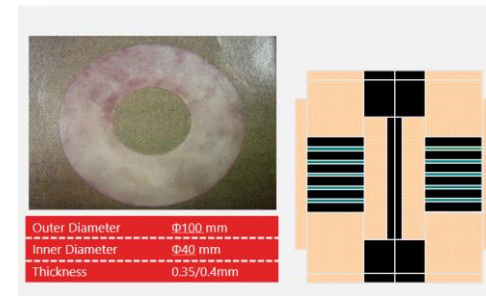
Bonded & FGMs



Near Net Shape



Ultra High Temp



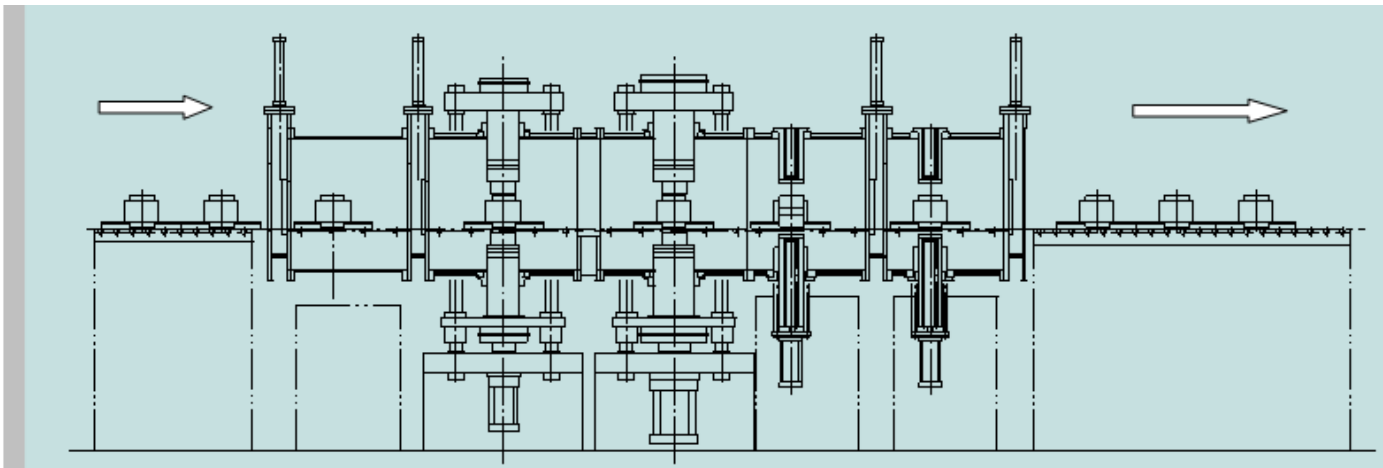
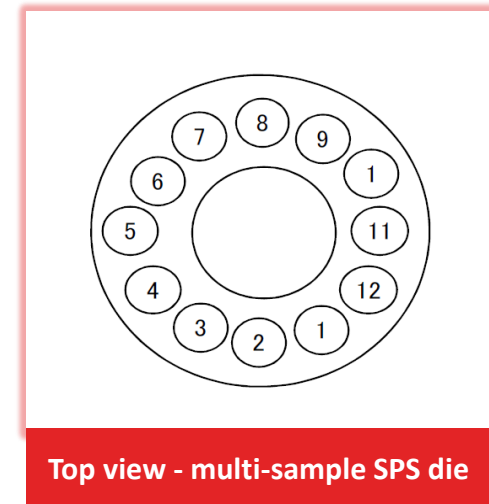
High Volume

# R&D to Production SPS Capabilities

Using an automated SPS tunnel-type system, SPS is capable of manufacturing high volumes of parts.

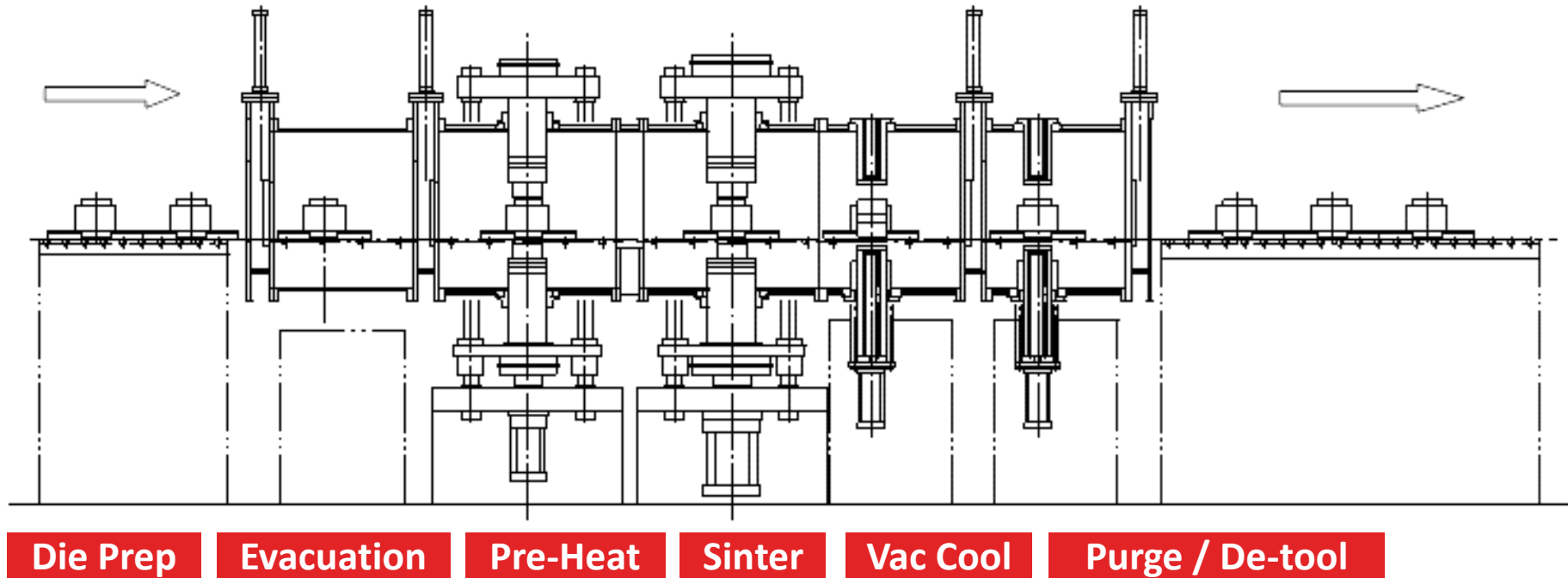
**Throughput Capability** – 15 min or less per cycle, 10s to 100s of parts per cycle depending on part size

**Cost Effective Manufacturing** – reduced energy consumption



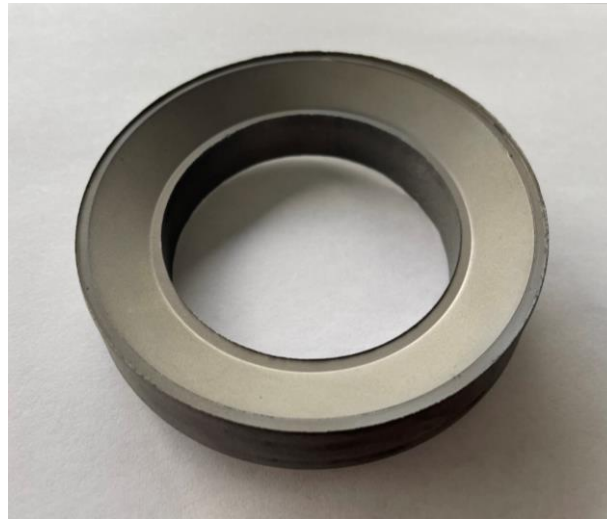
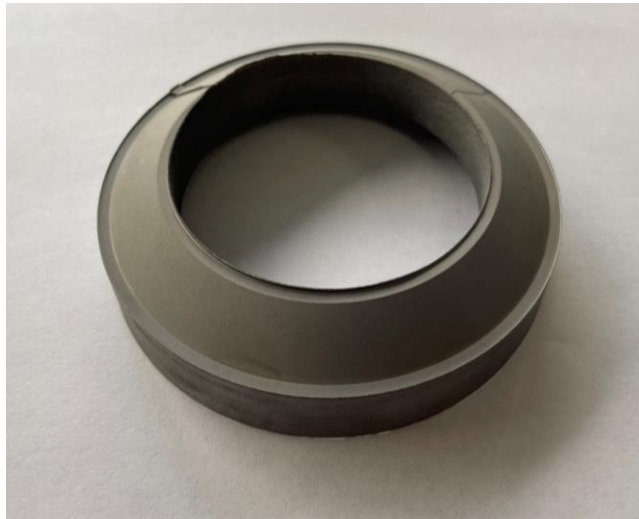


# SPS Tunnel Type Constraint Mitigation

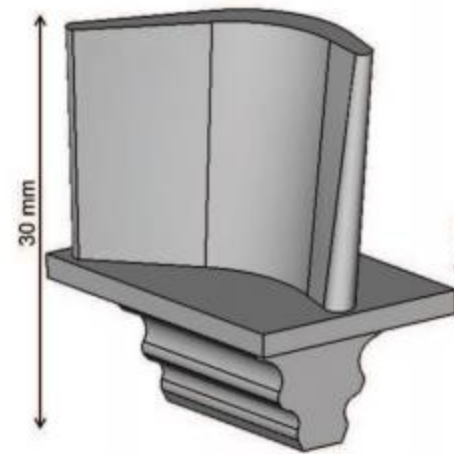


**Beyond automated loading and unloading of die sets, various process configurations and process techniques have been used to achieve many high-volume production applications.**

# Cost Effective Near Net Shapes



a) Target post sintering geometry      b) Spark plasma sintered turbine blade



Collaboration sintering for SDSU

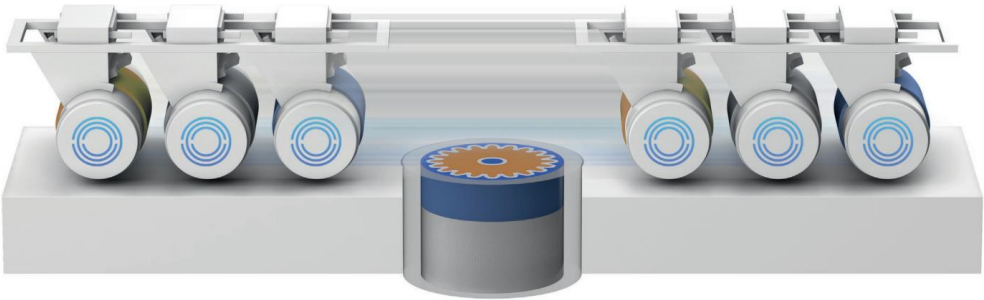
Large Scales and Near Net Shape Sintering is Achieved via Engineered Tooling or Selective Powder Deposition

[www.aerosint.com](http://www.aerosint.com)



**SPS Multi-Material Additive Manufacturing**

How Selective Powder Deposition technology is the key to multi-material additive manufacturing.



**Collaborations** SPS/FAST AM





# Collaborations SPS/FAST AM BJ

NORIMAT

Fully Dense Near Net Shape TiAl BLADES <1H

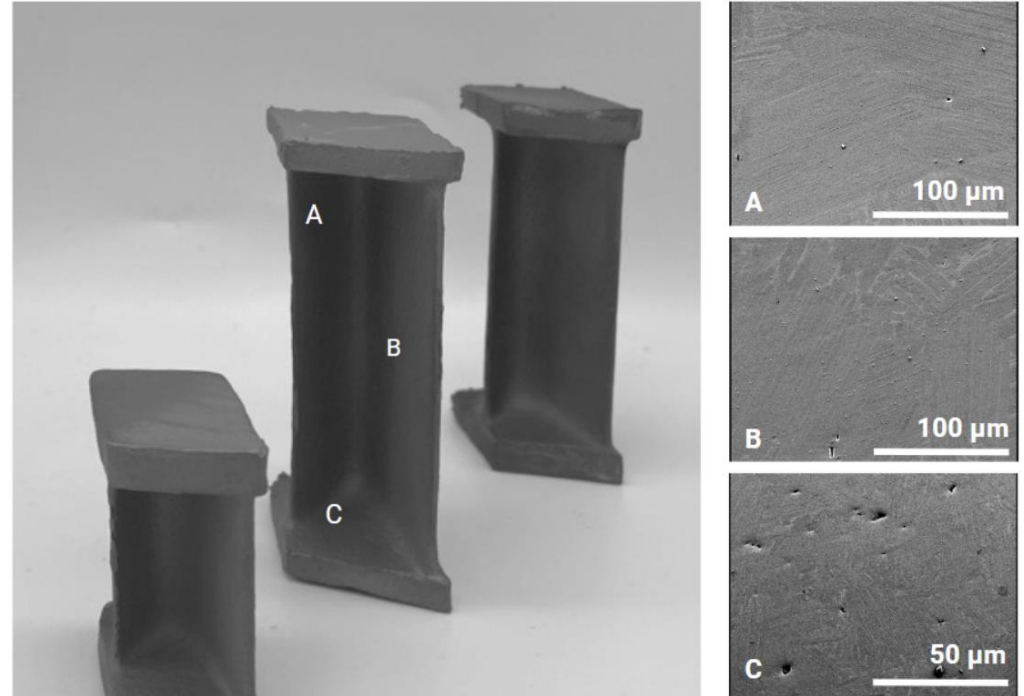
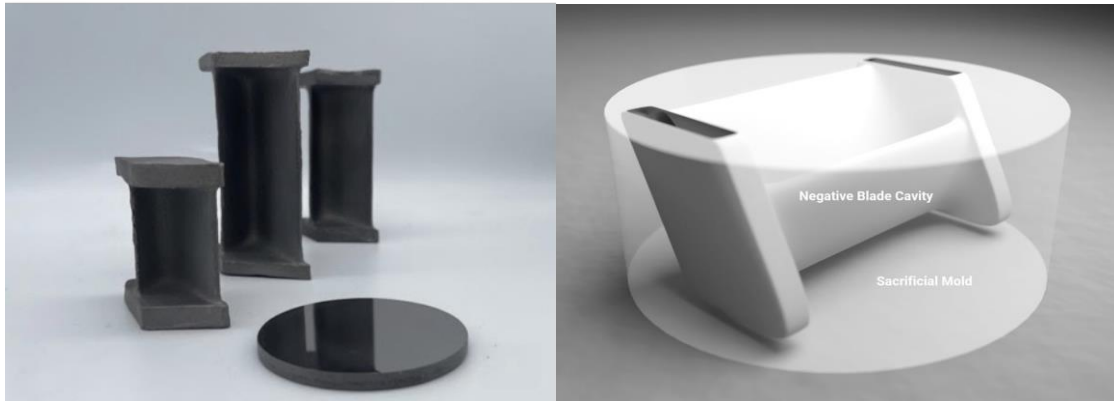
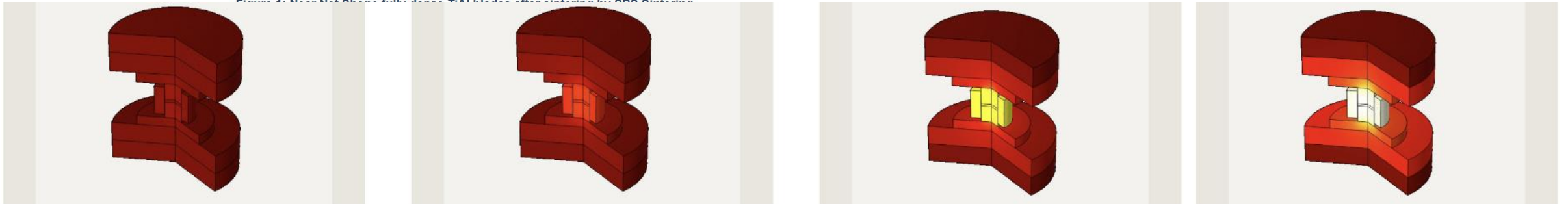


Figure 4. Near-Net-Shape Fully-Dense TiAl Blades Manufactured by SPS/FAST AM BJ



## WHAT WE DO SPS/FAST



SPS R&D Lab-scale system



SPS Graphite Tooling Set

### **Toll Sintering Services**

We perform toll sintering services for both production and research customers

### **Equipment Sales & Installation**

We sell, install, service and provide training for all SPS equipment, Partnered with SUGA Co. (AGUS Jp SPS equipment manufacturer) for the North American market

### **Consumables**

We supply customers with the highest quality SPS tooling and other consumables to meet their ongoing sintering needs

### **Technical Support**

We provide ongoing technical support for customers with SPS furnaces including upgrades, repairs and retraining as needed.

# Spark Plasma Sintering Capabilities

## SPS-515

Specs – 1 500 A/50 kN

- Max size- ~ 1" OD
- Max Temp ~ 2400 C
- Has been used for R&D at Cal Nano for 10+ years
- Over 10,000 SPS trials performed
- Vacuum/N/Ar atmosphere

## SPS-2000

Specs – 3000 A/70 kN

- Max size- ~ 3" OD
- Max Temp ~ 2400 C
- Installation Q1 2025
- Vacuum/N/Ar atmosphere

## SPS 7.40 Mk IV

Specs – 10 000 A/1000 kN

- Max size ~ 7" OD
- Max Temp – 2400 C
- Installed in Fall 2017
- Over 3 000 SPS trials performed
- Vacuum/N/Ar atmosphere

## SPS MSP5

Specs – 85 500 A/5000 kN

- Max size ~ 17.5" OD
- Max Temp – 2400 C
- installation Q2 2024
- Vacuum/N/Ar atmosphere

**Inert Glove Box Handling Available For Materials That Require It (Ar/N)**

# Cal Nano Post Sinter Capabilities



Multiple Lathes & Mills capable of machining up to ~10" OD parts



Wire EDM - precision cuts on pieces over 24" L x 24" W and ~8" Tall

Cal Nano offers a variety of post-SPS processing capabilities including machining & Wire EDM. We will often cut out specimens for customers after SPS for testing such as tensile, fatigue, compression, gleeble, CTE, and much more



# Testing & Analysis Labs

Instron Unit - Tensile & Compression

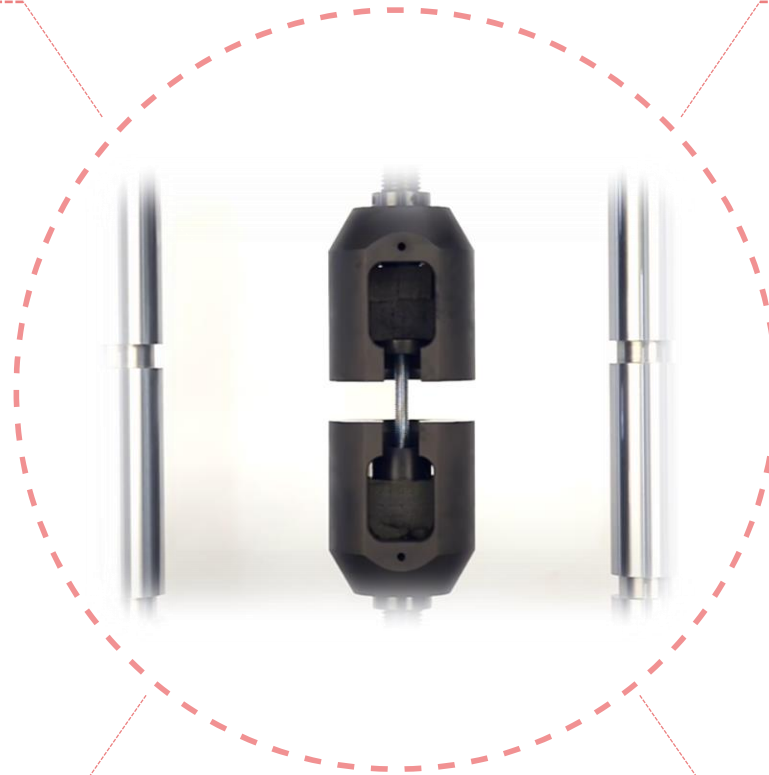
Planetary Ball Milling

Standard Hardness & Micro Hardness



Profilometer – Surface Roughness

Laser Diffraction Particle Size Analyzer  
- 0.01 um- 3800 um range



# Applications/Customers


- ✓ **Energy** – high performance thermoelectric materials, nuclear reactor components
- ✓ **Aerospace/Space** – ultra-high temp ceramics for shielding/engine components/hypersonics
- ✓ **Defense** – transparent ceramics like Spinel for high temperature applications, Ultra-hard Ballistic Armor
- ✓ **Next-gen Batteries** - Cathode Materials, Nd-Fe-B, Soft Magnetic Materials
- ✓ **Automotive** – High volume disc brakes, Cu-based & composites, brake rotors – specialty applications
- ✓ **Specialized Parts Repair via Bonding**





Stay in Touch !

For further information,  
please contact us!

 (562)-991-5211

 [info@calnanocorp.com](mailto:info@calnanocorp.com)

 [www.calnanocorp.com](http://www.calnanocorp.com)