Worcester Polytechnic Institute

U.S. Army Research Laboratory
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# Advanced Powder Characterization & Modeling


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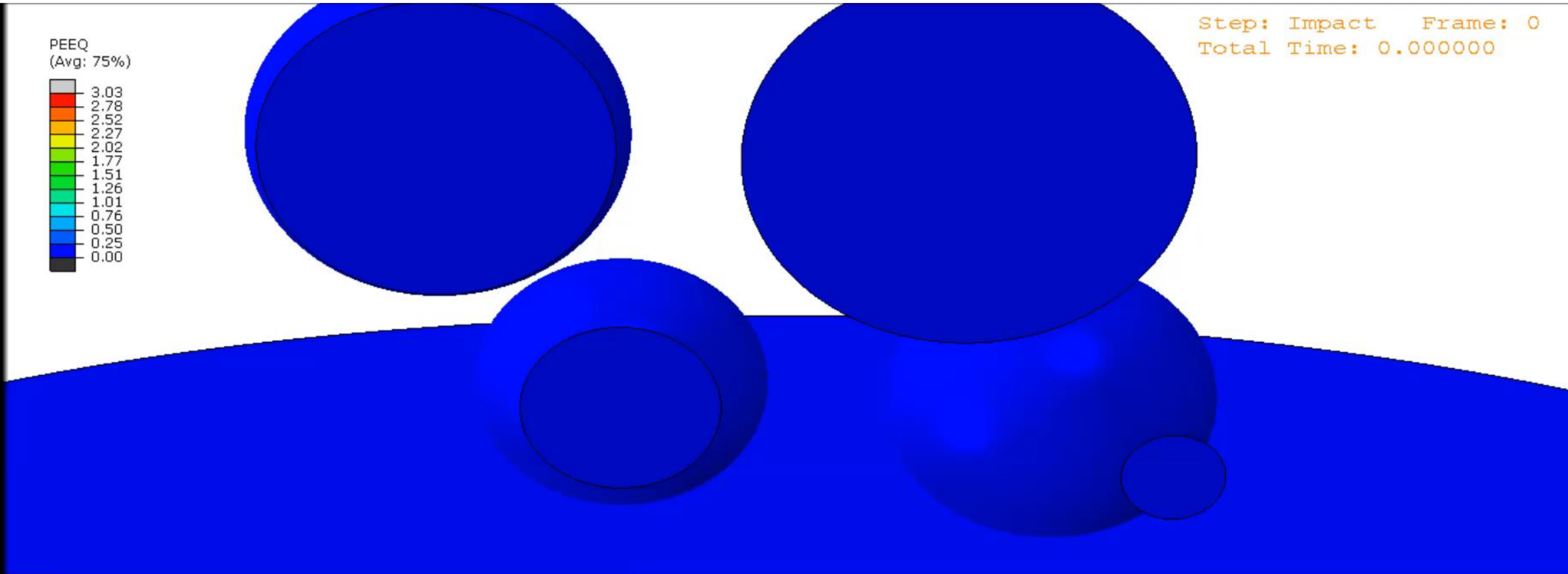
# Cold Spray Impact Model



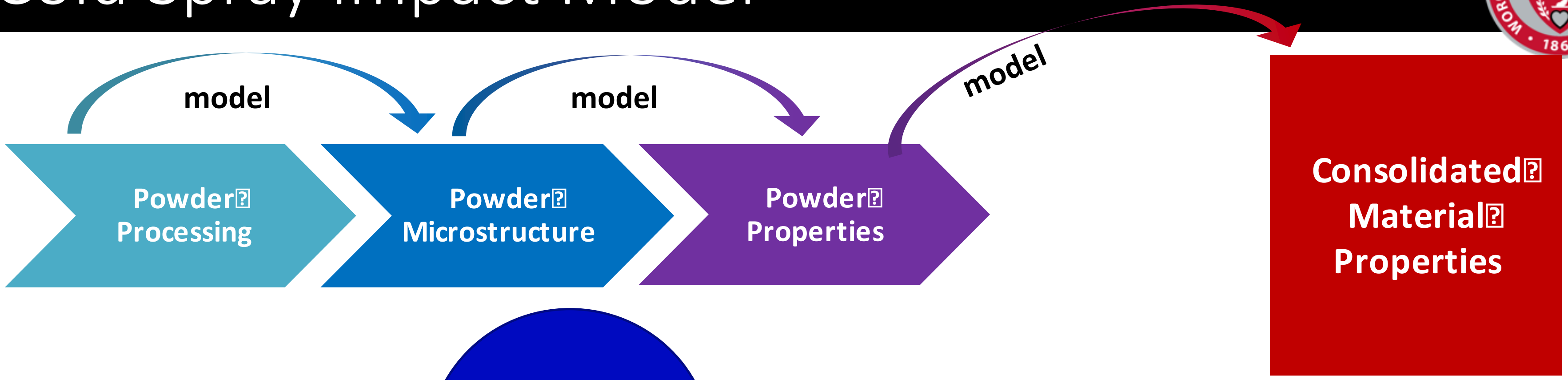

## Johnson-Cook Model

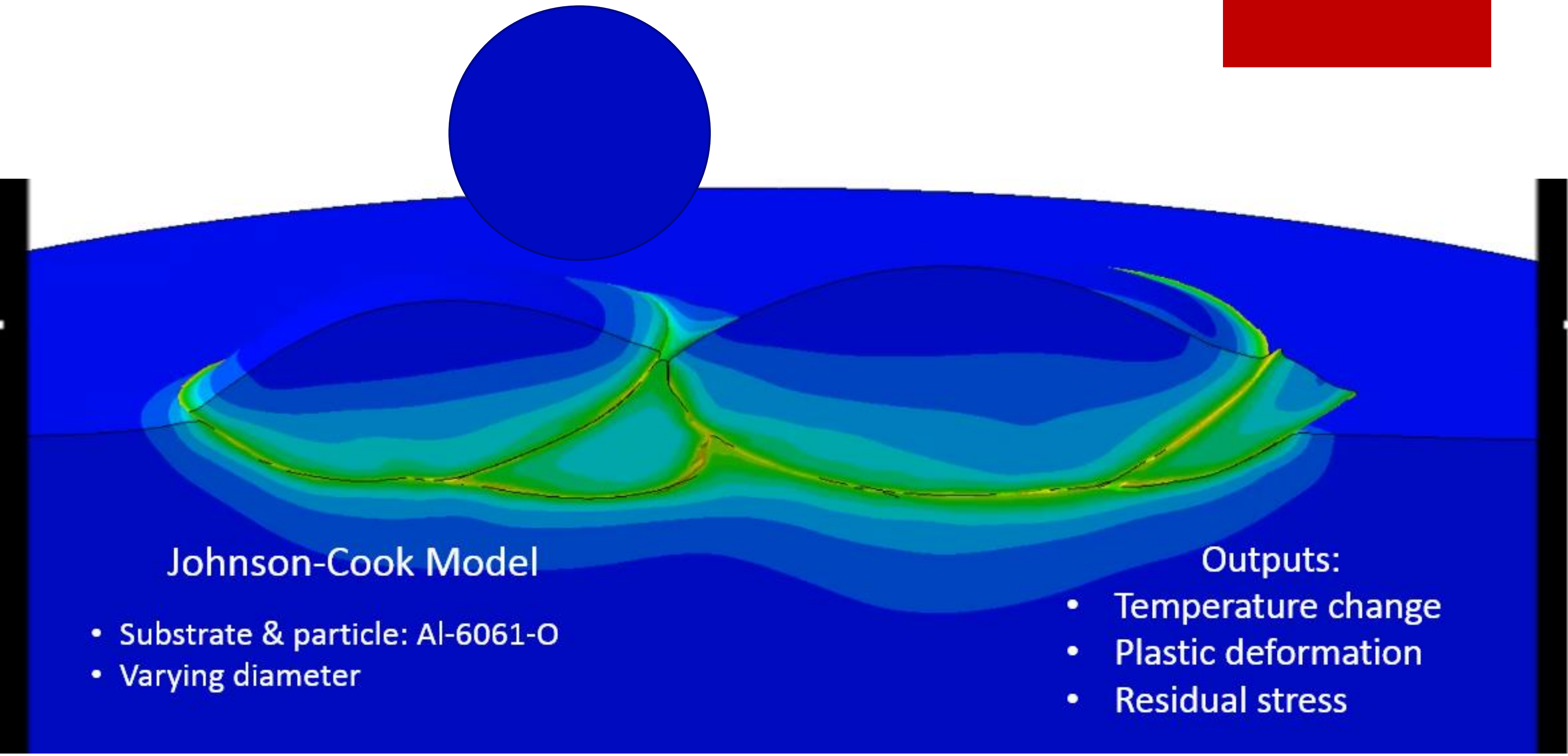
- Substrate & particle: Al-6061-O
- Varying diameter

## Outputs:

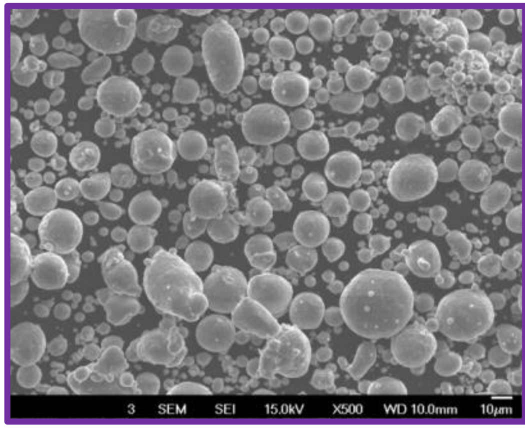
- Temperature change
- Plastic deformation
- Residual stress

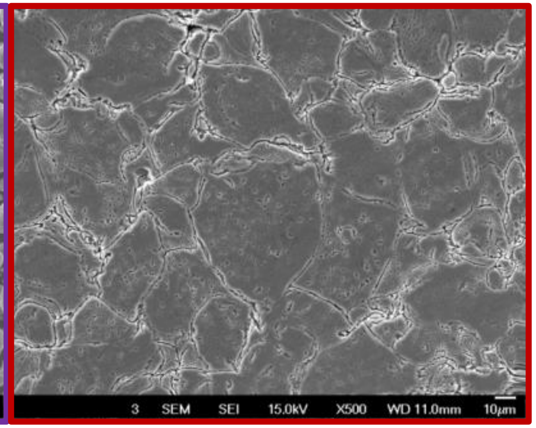
# Cold Spray Impact Model



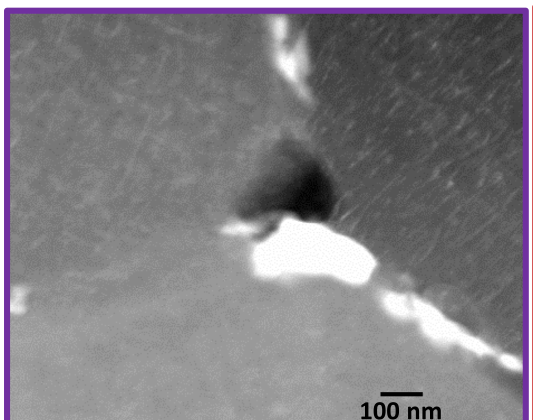



# Powder Significance

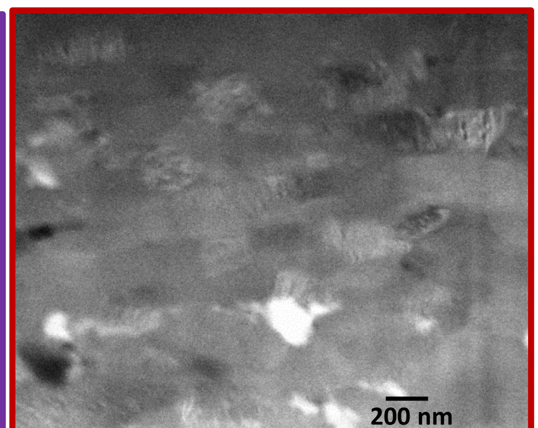


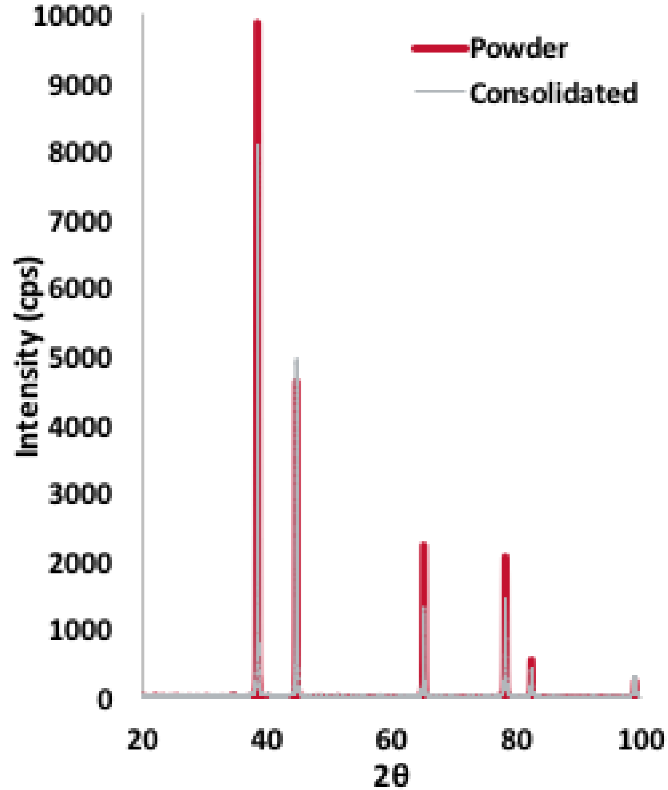

**Powder** → **Consolidated**



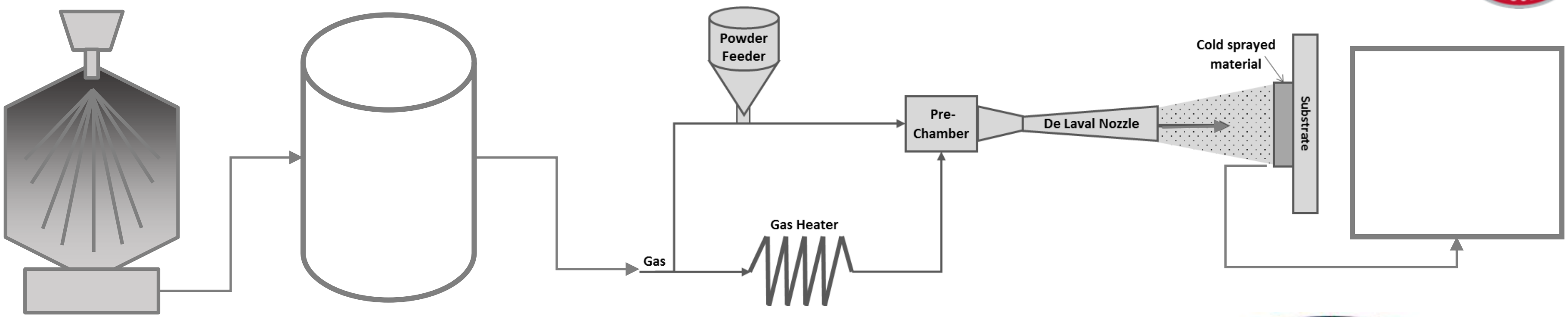

STEM Dark Field

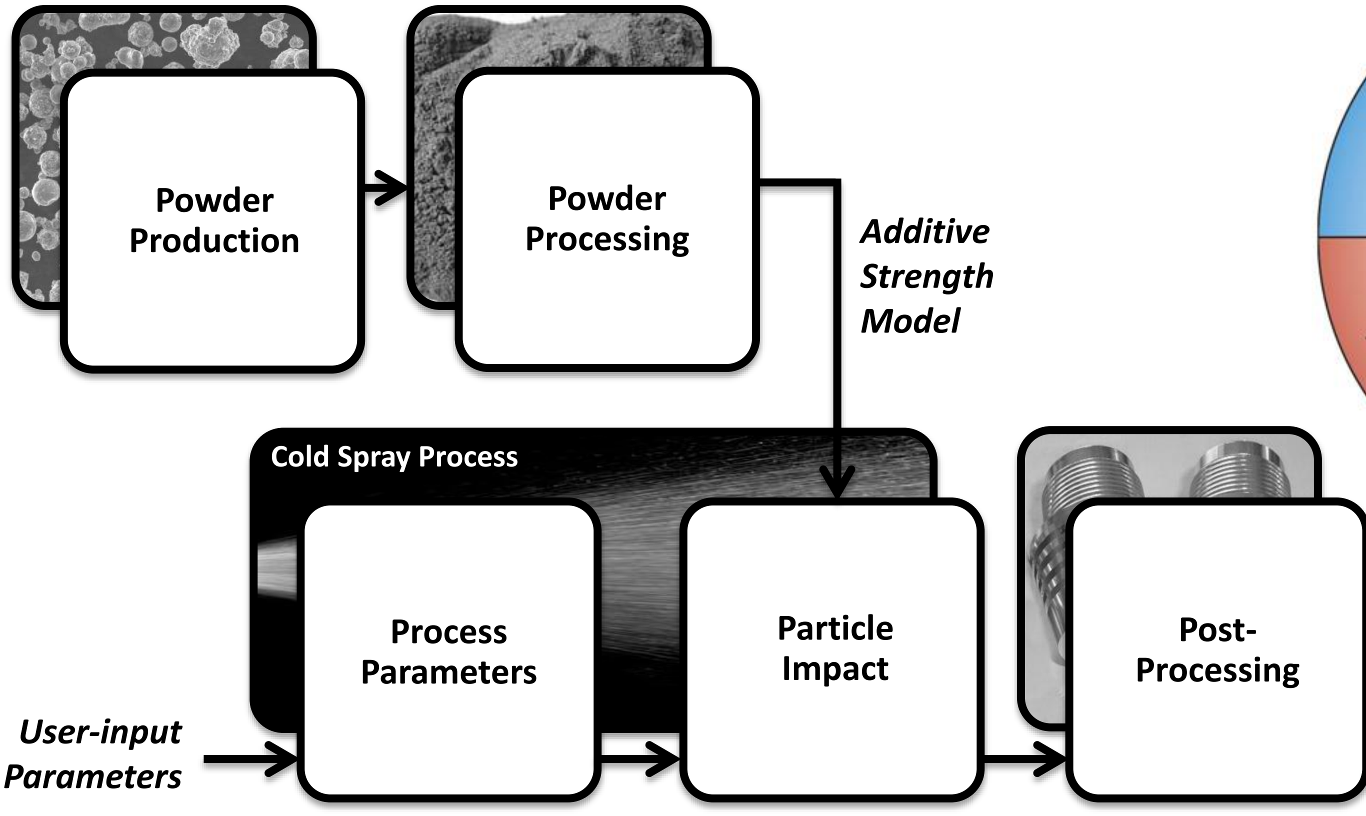


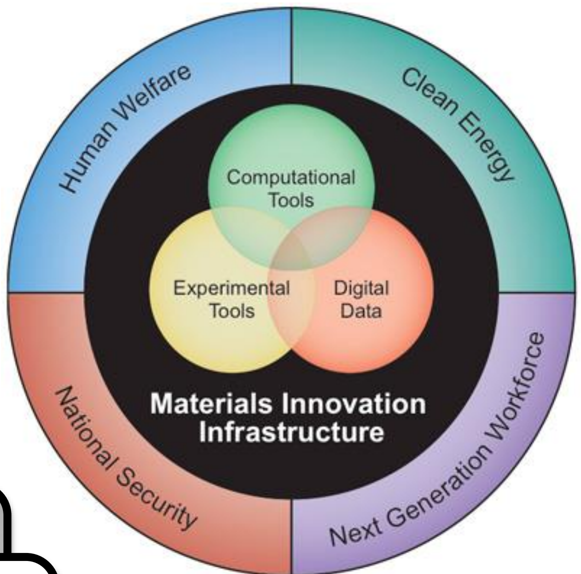

STEM Dark Field



# Through-Process Model



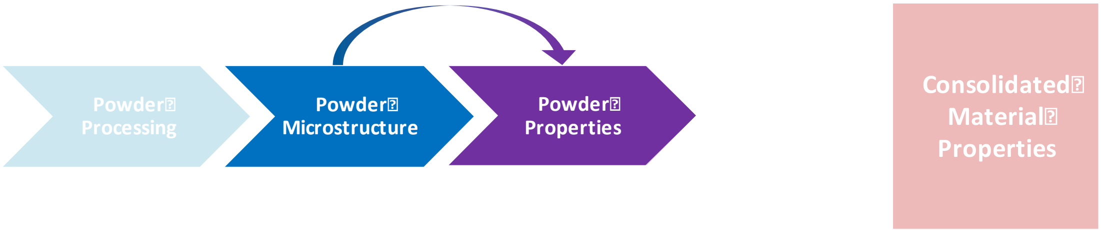




Council, N.S.a.T., *Materials Genome Initiative for Global Competitiveness*. 2011.

# Quantification of Powder Properties

**Function of:**
- **Alloy composition,**
- **Powder particle size,**
- **T, t of heat treatment**



# Modeling Approach

$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^{m}(d,t,T) + \Delta\sigma_{mic}^{n}(d,t,T) + \Delta\sigma_{ppt}^{p}(d,t,T)$$

$f(C_i, f_i, r_i, L_i, d, t, T)$

**Predict properties of powders**

**Chemical Composition**

PANDAT

JMatPro

$C_i$

**Solidification Properties**

JMatPro

$d$

**Thermal Treatment**

JMatPro

$f_i, r_i, L_i, d$

**Thermodynamic, Kinetic, and Solidification Models**

# Experimental Approach

**Function of:**
- **Alloy composition,**
- **Powder particle size,**
- **T, t of heat treatment**

$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^{m}(d,t,T) + \Delta\sigma_{mic}^{n}(d,t,T) + \Delta\sigma_{ppt}^{p}(d,t,T)$$

**Solid Solution Strengthening**

$$\Delta\sigma_{ss}(d,t,T) = \sum_i (G\varepsilon_s^{3/2} c^{1/2})/700$$

Substitutional Solid-Solution

→ Solidification Model
Composition

**Grain Size/Microstructural Influence**

$$\Delta\sigma_{mic}(d,t,T) = \frac{k_{gs}}{\sqrt{d}}$$

Hall-Petch Behavior

→ Solidification Model
Particle & Grain Size

**Precipitation Strengthening**

$$\Delta\sigma_{ppt}(d,t,T) = \sum_i [\sigma_{Coherency,\,i} + \sigma_{Modulus,\,i} + \sigma_{Chemical,\,i} + \sigma_{incoh,\,i}]$$

$$\Delta\sigma_{Coherency,i} = 7\varepsilon_{coh,i}^{3/2} G \left(\frac{rf}{b}\right)^{1/2}$$

$$\Delta\sigma_{Modulus,i} = 0.01\varepsilon_{Gp,i}^{3/2} G \left(\frac{rf}{b}\right)^{1/2}$$

$$\Delta\sigma_{Chemical,i} = 2\varepsilon_{Ch,i}^{3/2} G \left(\frac{rf}{b}\right)^{1/2}$$

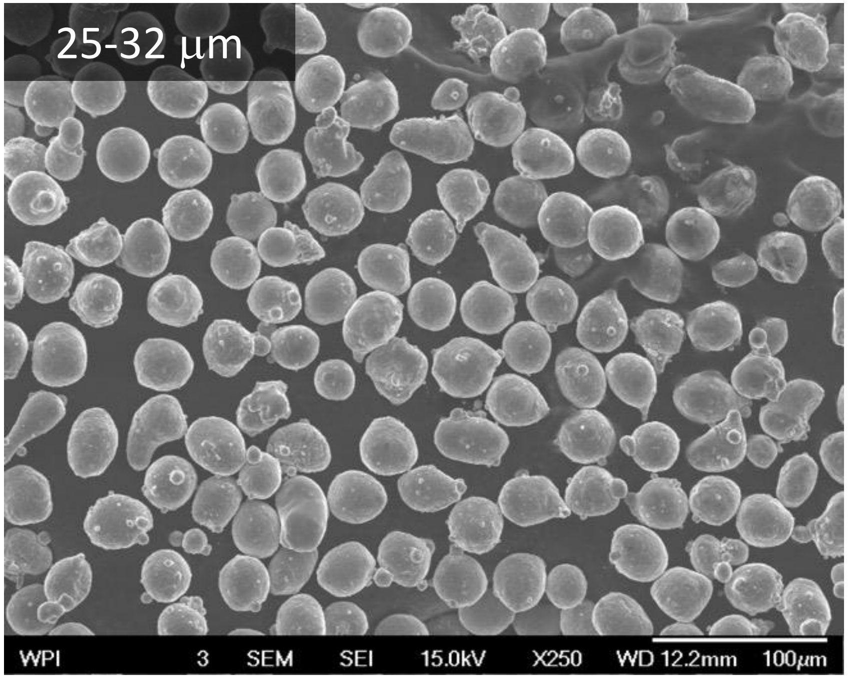
$$\Delta\sigma_{Incoh} = \mathrm{Gb}/(\mathrm{L} - 2\mathrm{r})$$

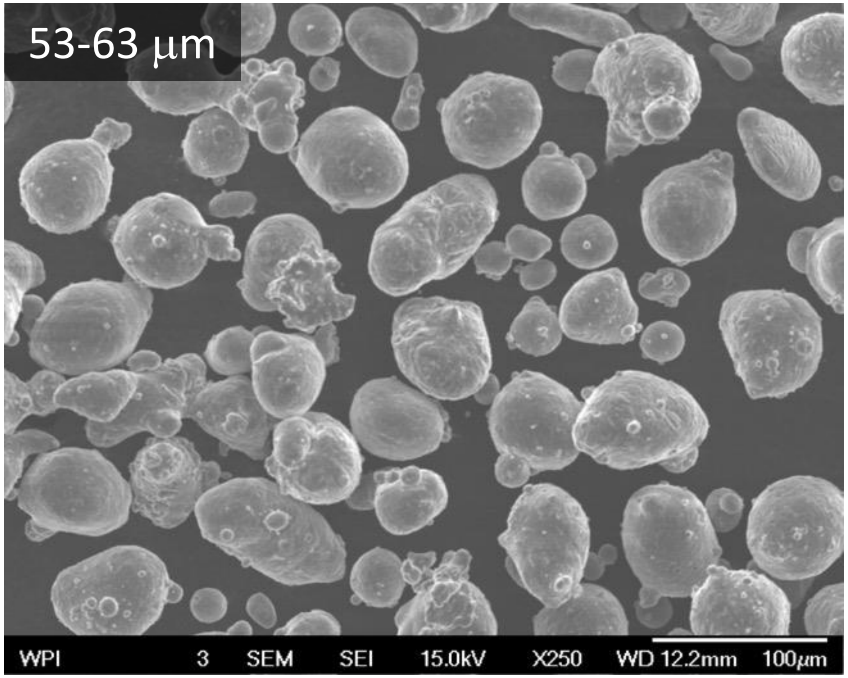
→ Thermodynamic & Solidification Models
Phase Identification
Phase Morphology (Radius & Fraction)

- X-Ray Diffraction
- Optical Microscopy
- Scanning Electron Microscopy
- Transmission Electron Microscopy
- Nanoindentation

# Sieved Powder – Al 7075

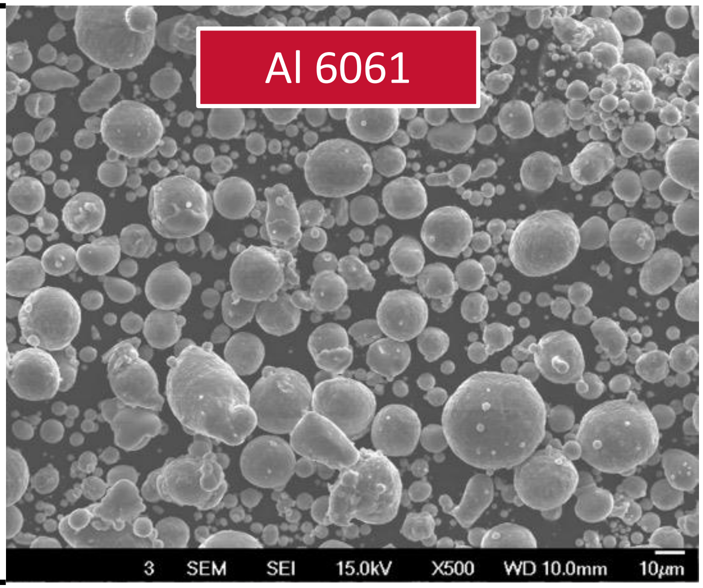
1. Remove fine particles < 20 μm
2. Sieve into 5 size ranges with stainless steel mess

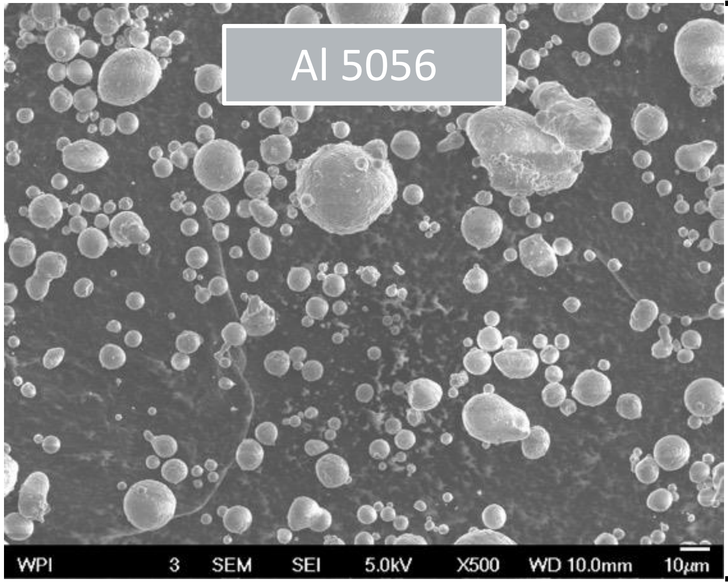


# Alloys Studied – Compositions [Wt%]

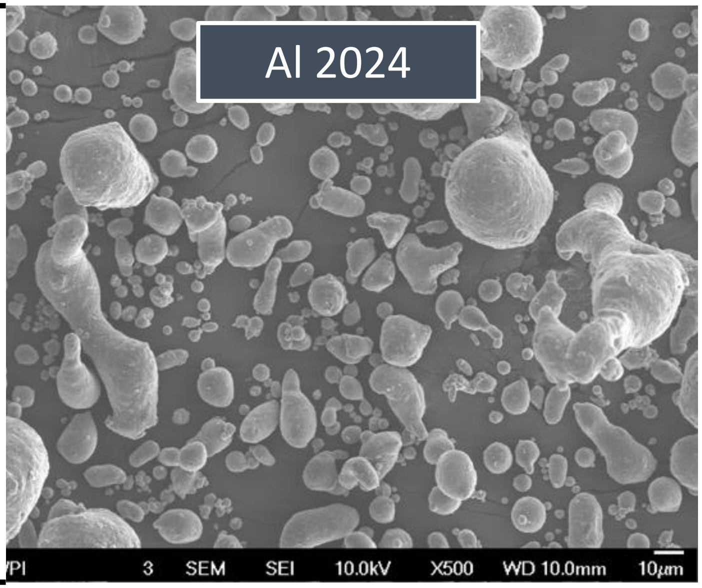
| | |
|---|---|
| Cr | 0.110 |
| Cu | 0.260 |
| Fe | 0.280 |
| Mg | 0.922 |
| Mn | 0.078 |
| Ti | 0.024 |
| Zn | 0.020 |
| Si | 0.591 |
| Al | 97.7 |

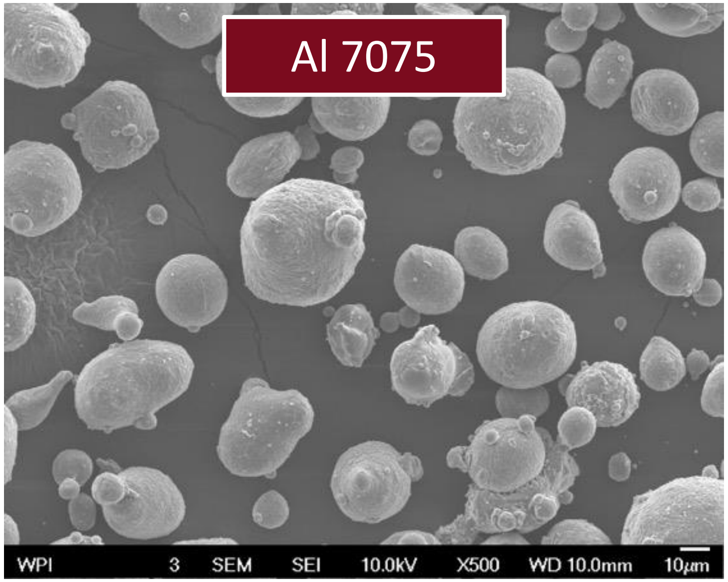


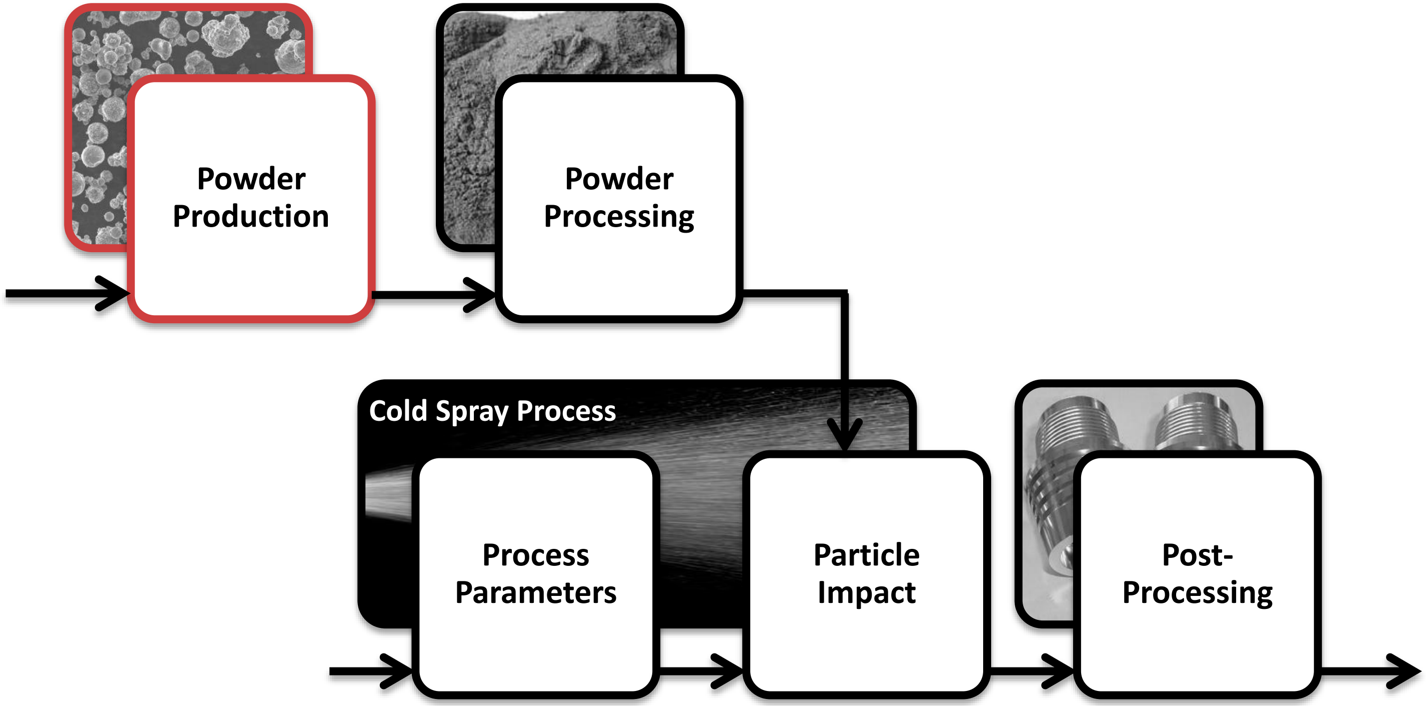

| | |
|---|---|
| Cr | 0.160 |
| Cu | 0.006 |
| Fe | 0.110 |
| Mg | 5.300 |
| Mn | 0.150 |
| Ti | 0.000 |
| Zn | 0.006 |
| Si | 0.050 |
| Al | 94.2 |

| | |
|---|---|
| Cr | 0.006 |
| Cu | 4.000 |
| Fe | 0.078 |
| Mg | 1.500 |
| Mn | 0.610 |
| Ti | 0.100 |
| Zn | 0.014 |
| Si | 0.130 |
| Al | 93.6 |




| | |
|---|---|
| Cr | 0.210 |
| Cu | 1.700 |
| Fe | 0.180 |
| Mg | 2.300 |
| Mn | 0.031 |
| Ti | 0.031 |
| Zn | 5.600 |
| Si | 0.110 |
| Al | 89.8 |




# Powder Production

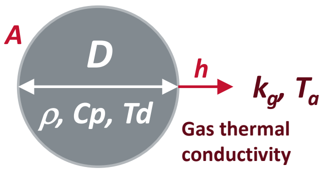
Solidification, Thermodynamic & Segregation Models

Characterization: Grain Size & Phase Identification

# Solidification Model


| Thermophysical Properties of Al 2024[1] | | | | |
|---|---|---|---|---|
| Ar Atomizing Gas | | Molten Droplet | | |
| $T_f$ | $K_g$ | ρ | $C_p$ | $T_d$ |
| [K] | [W/(mK)] | [kg/m³] | [J/(kgK)] | [K] |
| 300 | 1.79E-02 | 2270 | 1140 | 1473 |

## Heat Transfer Model Gas Atomization Process




**Gas Atomization: Newtonian Heat Flow**

- Heat transfer from droplet: forced convection
- Radiational cooling: neglected
- Heat conduction (w/in droplet): neglected (small size – Biot number < 0.1)

**Heat balance,**

$$-V\cdot\rho\cdot C_p\cdot\frac{dT_d}{dt}=h\cdot A\cdot(T_d-T_f)+\sigma\varepsilon\left(T_d^4-T_f^4\right)$$

$$h=\frac{k_g}{d}\left(2.0+0.6\sqrt{R_e}\cdot\sqrt[3]{P_r}\right)$$ [2]

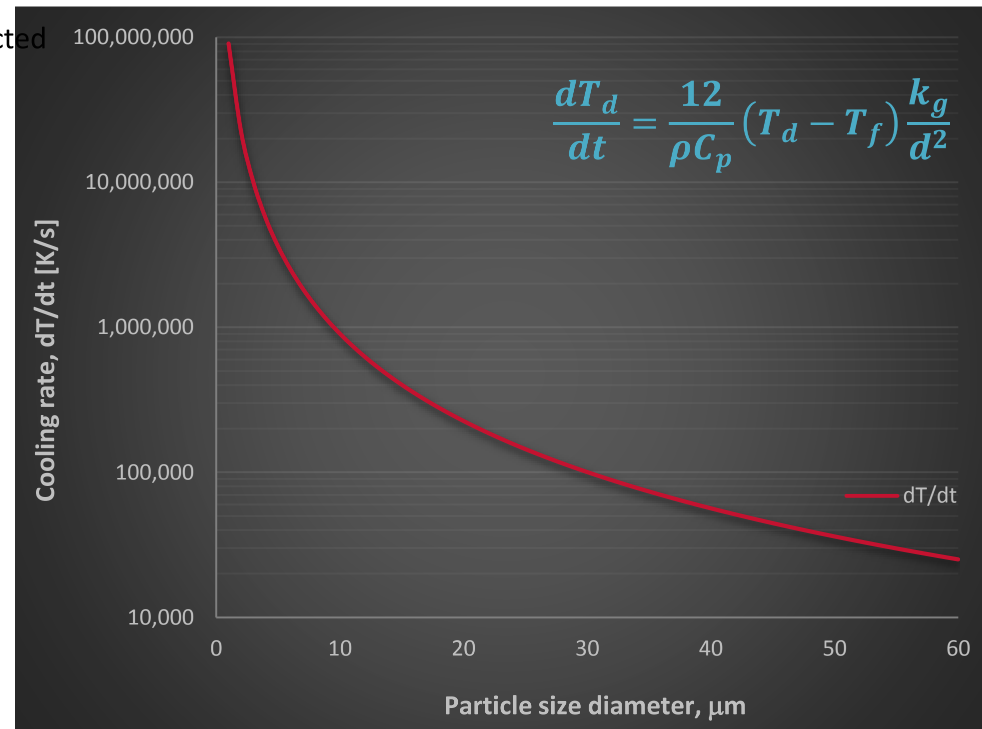
$$R_e=\frac{U\cdot\rho_g\cdot d}{\mu_g}$$

$$Pr=(C_g\mu_g/k_g)$$

$$\left|\frac{dT_d}{dt}\right|=\frac{6}{\rho\cdot C_p}\cdot(T_d-T_f)\cdot\left(\frac{2.0k_g}{d^2}+0.6\frac{k_g}{d}\sqrt{\frac{\rho_g U}{\mu_g d}}\cdot\sqrt[3]{P_r}\right)$$

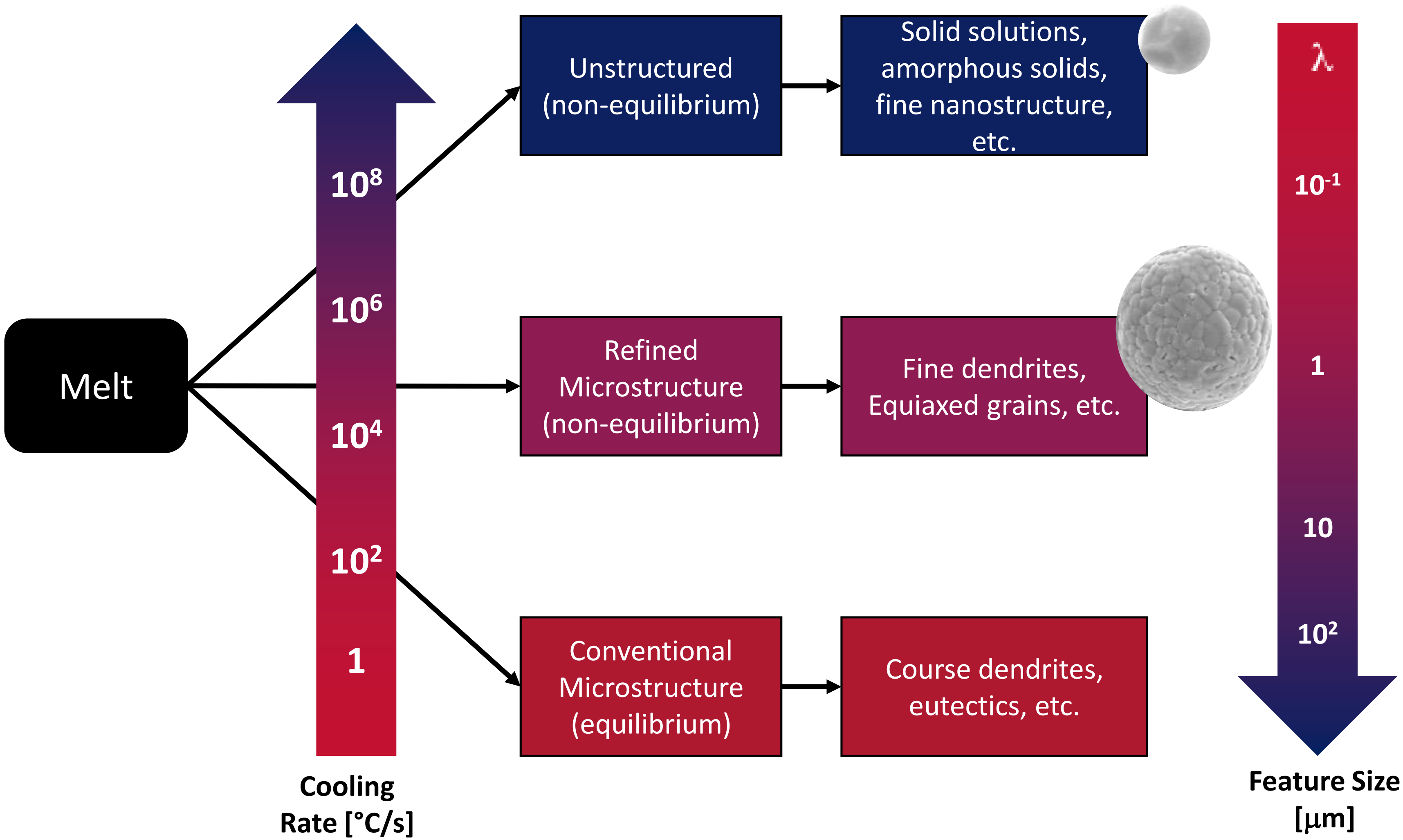
0

$$\left|\frac{dT_d}{dt}\right|=\frac{12}{\rho\cdot C_p}\cdot(T_d-T_f)\cdot\frac{k_g}{d^2}$$ [3]




[1]Property Data Calculated using JMatPro
[2]Ranz, Marshall (1952)
[3]C He, S.W., Y. Liu, S. Guo (2009)

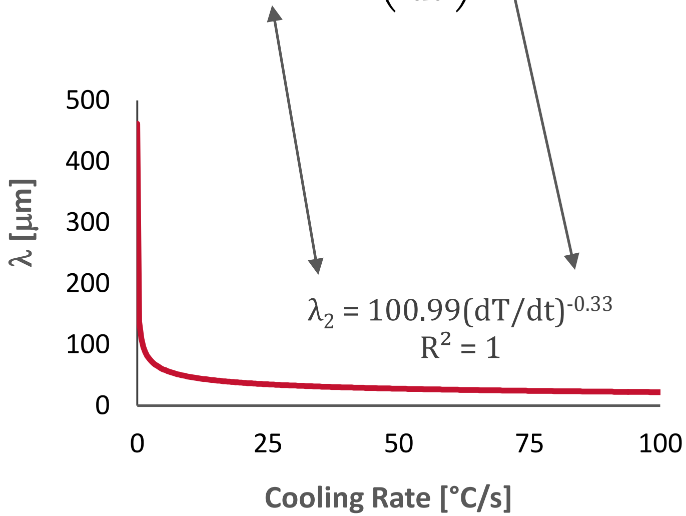
# Microstructural Dependence on Solidification Rate



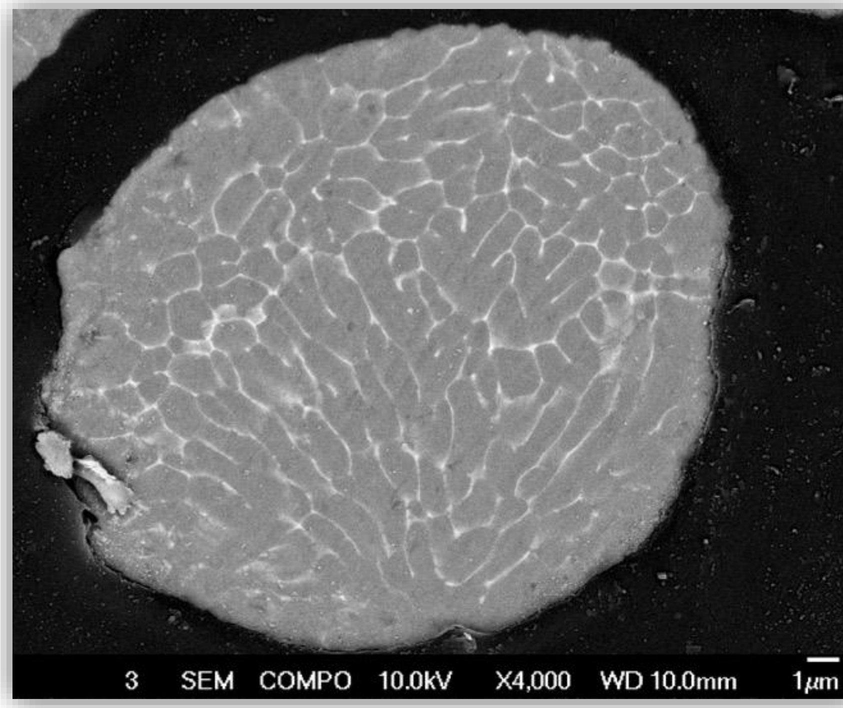
# Solidification Model

## SDAS (feature size) and Cooling Rate:

$$\lambda_2 = \lambda_0 \left(\frac{dT_d}{dt}\right)^{-n}$$




Model Data from JMatPro

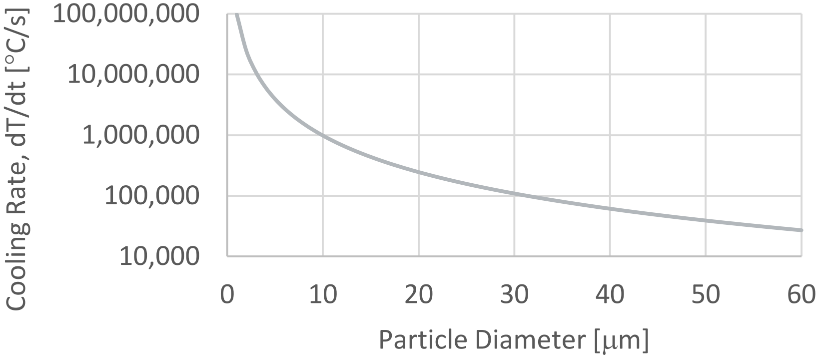


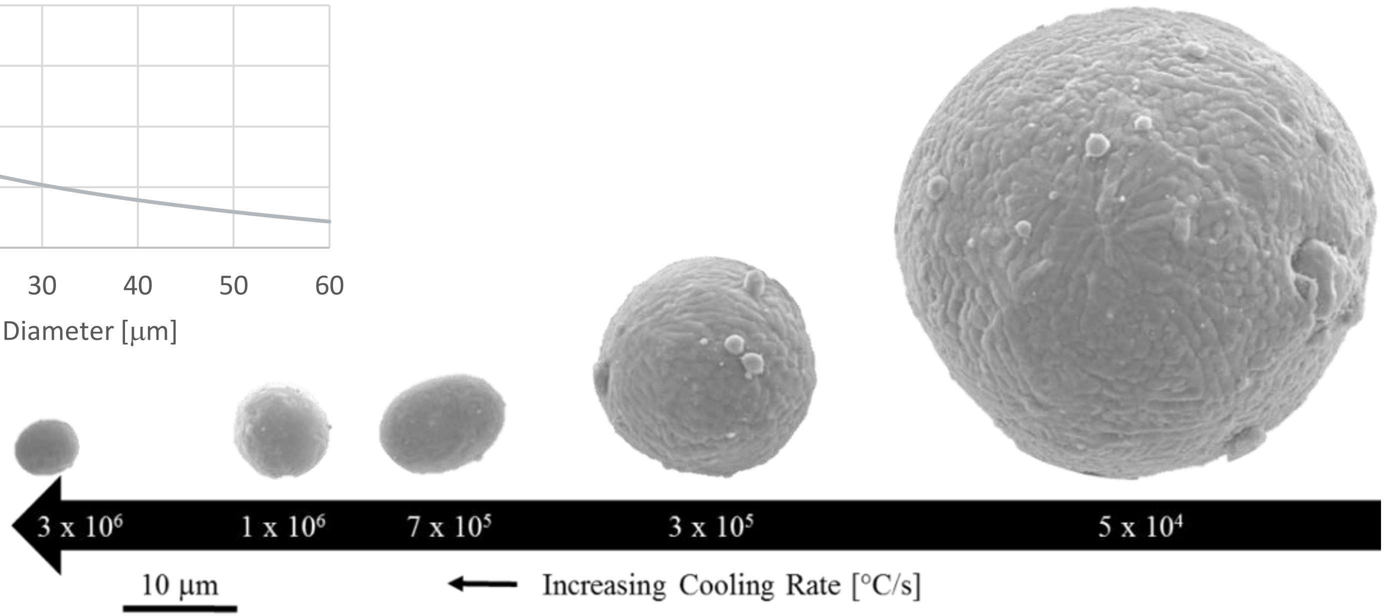

Relationship between feature size and particle diameter

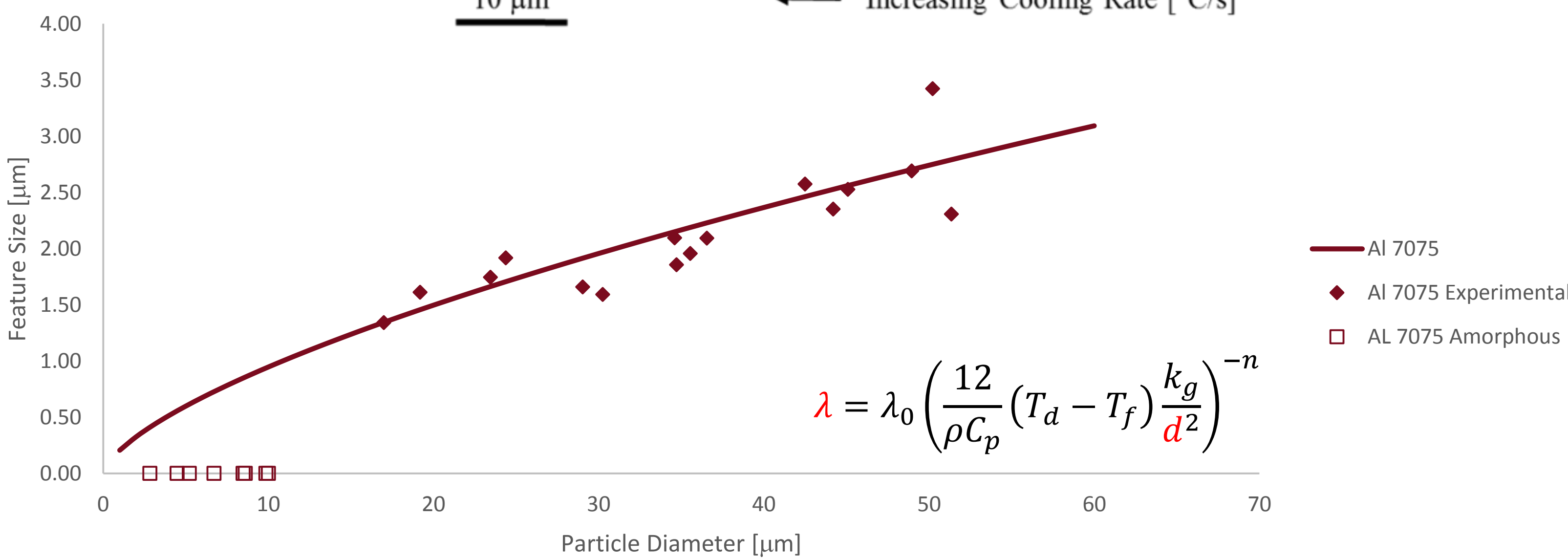
$$\lambda = \lambda_0 \left(\frac{12}{\rho C_p}(T_d - T_f)\frac{k_g}{d^2}\right)^{-n}$$

$$\Delta\sigma_{mic}(d,t,T) = \frac{k_{gs}}{\sqrt{\lambda}}$$

# Solidification – Grain Size

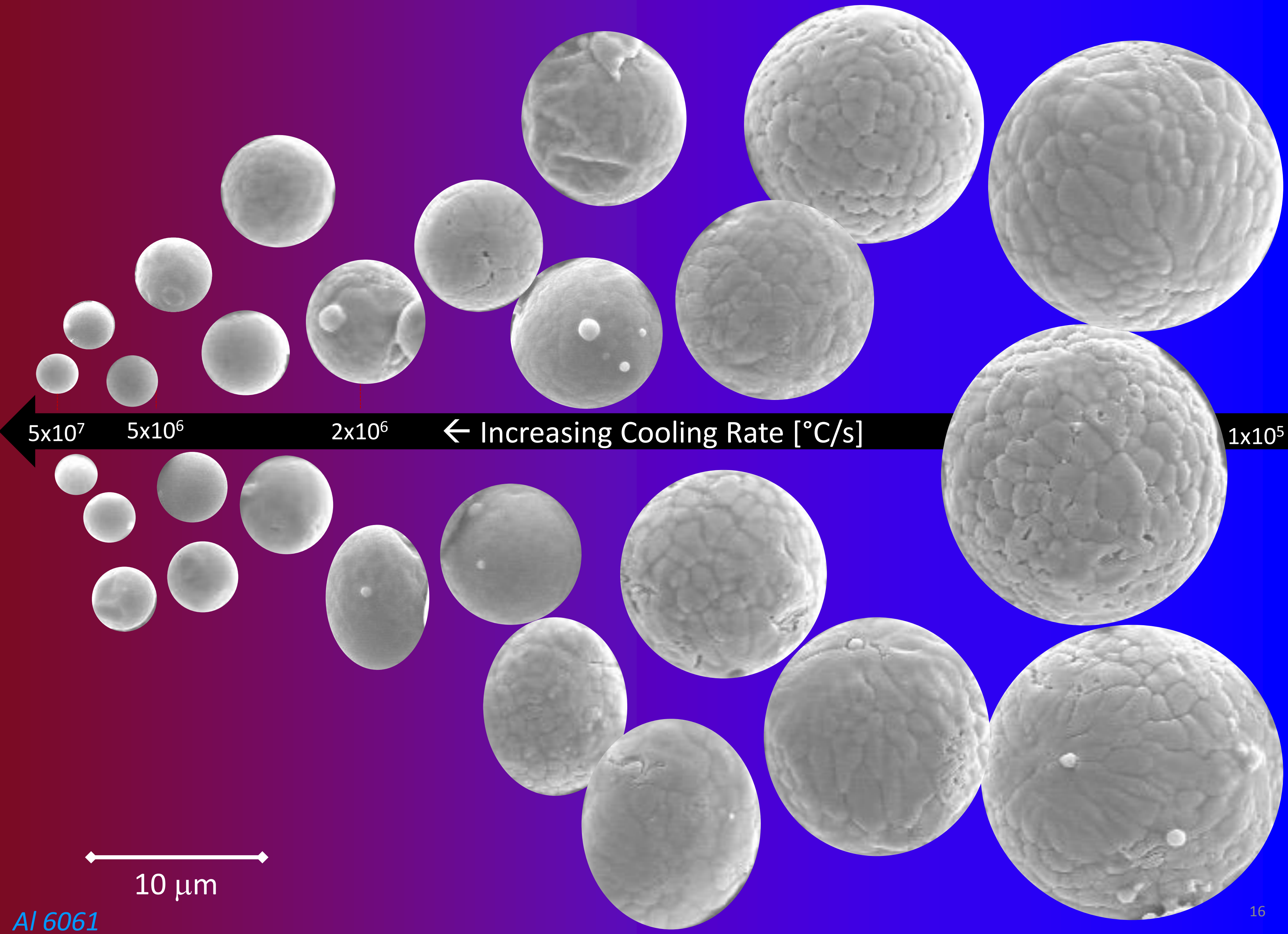


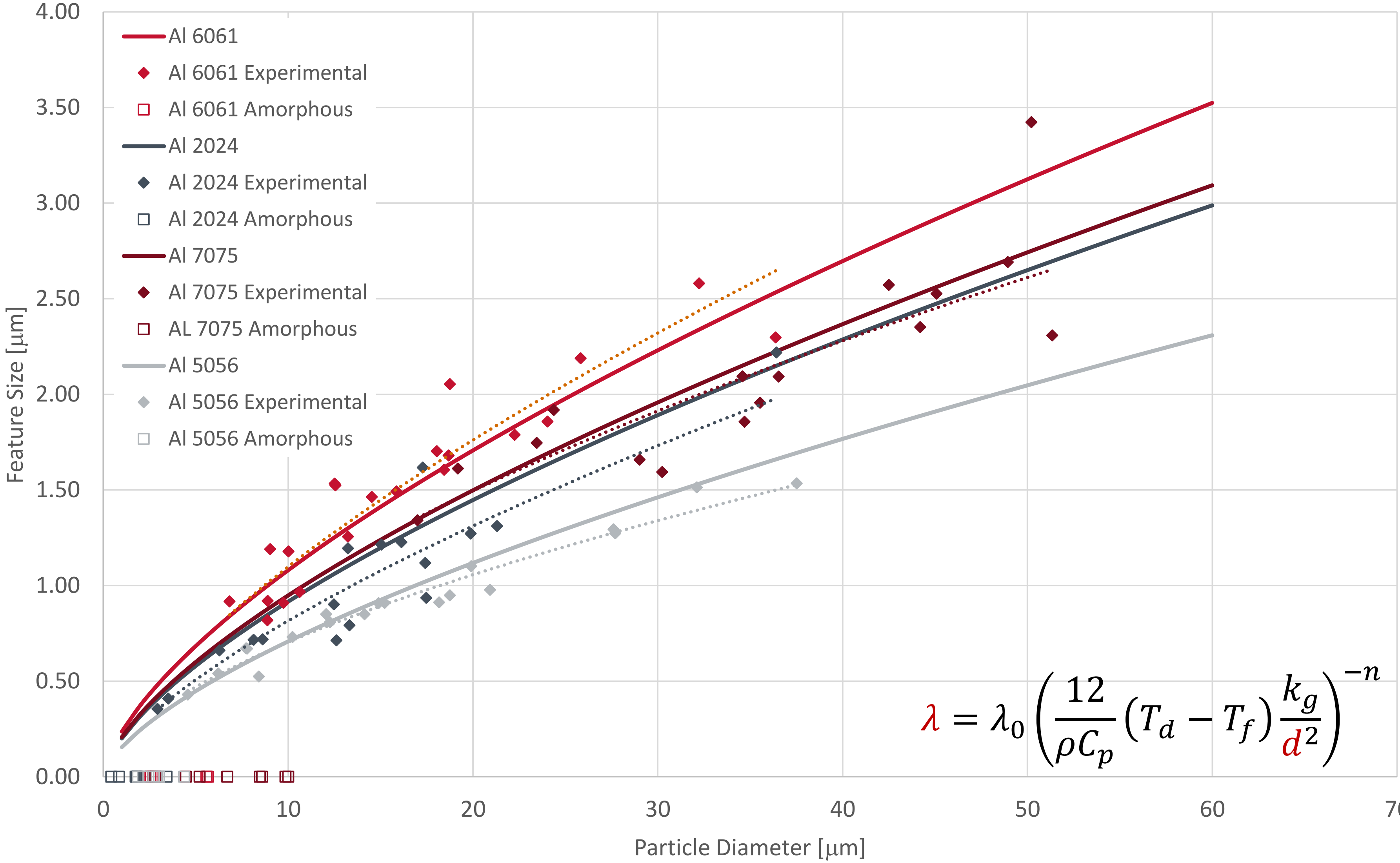




*Al 7075*

$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^{m}(d,t,T) + \Delta\sigma_{mic}^{n}(d,t,T) + \Delta\sigma_{ppt}^{p}(d,t,T)$$


5x10^7
5x10^6
2x10^6
← Increasing Cooling Rate [°C/s]
1x10^5
10 μm
Al 6061

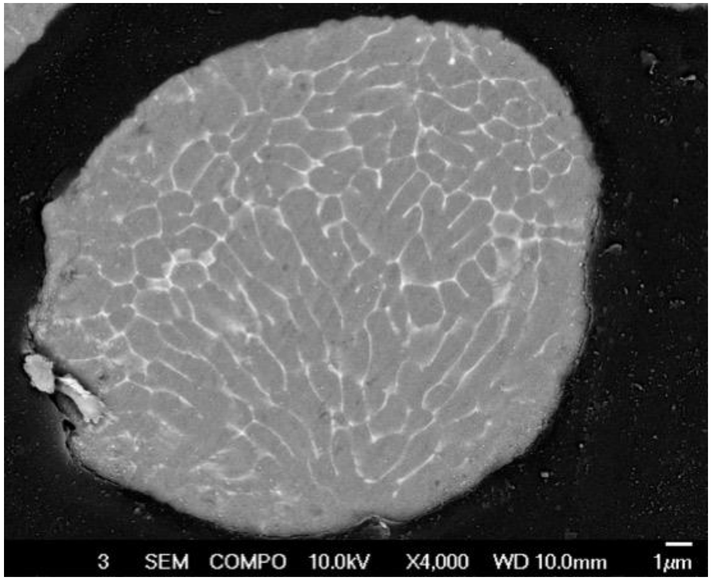
# Grain Size Variation with Alloy




$$\lambda = \lambda_0 \left( \frac{12}{\rho C_p} \left(T_d - T_f\right) \frac{k_g}{d^2} \right)^{-n}$$

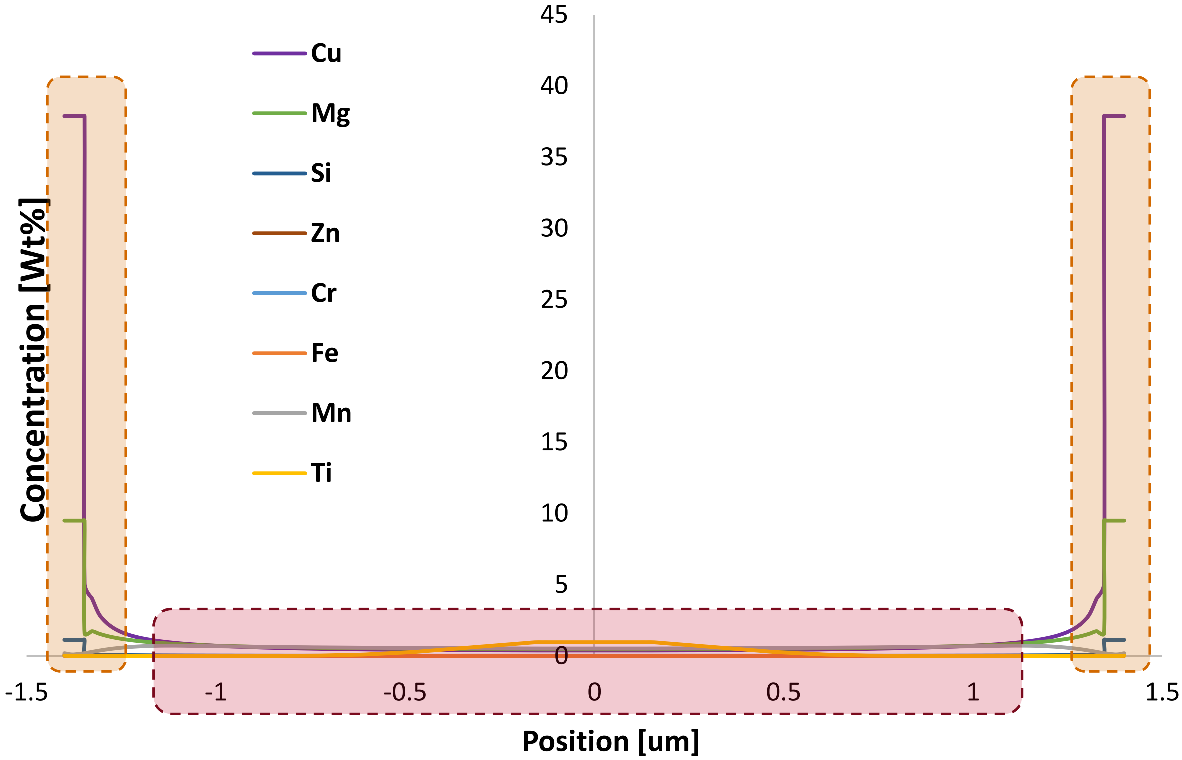
$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^{m}(d,t,T) + \Delta\sigma_{mic}^{n}(d,t,T) + \Delta\sigma_{ppt}^{p}(d,t,T)$$

# Solid Solution Strengthening



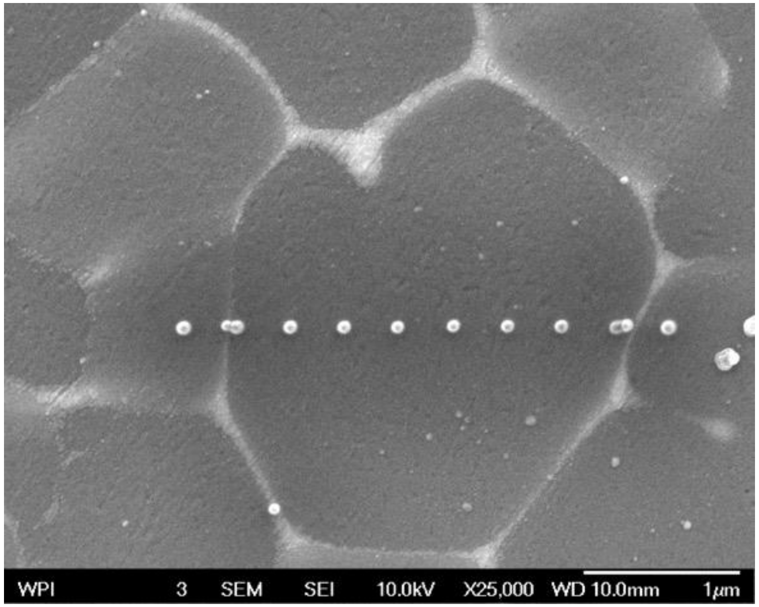

$$\Delta\sigma_{ppt}(d,t,T) = \sum_i [\sigma_{Co,i} + \sigma_{Mo,i} + \sigma_{Ch,i} + \sigma_{in,i}]$$

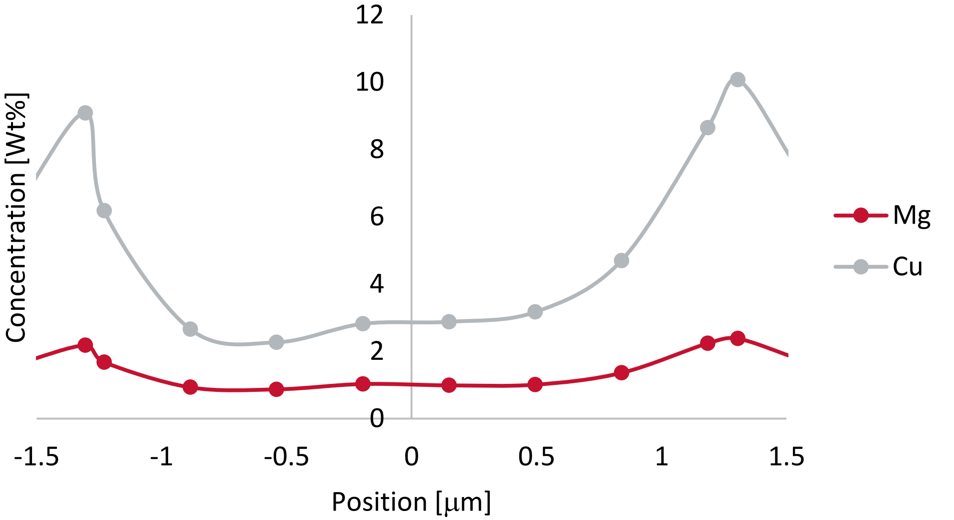
$$\Delta\sigma_{ss}(d, t=0, T) = \sum_i (G\varepsilon_s^{3/2} c^{1/2})/700$$

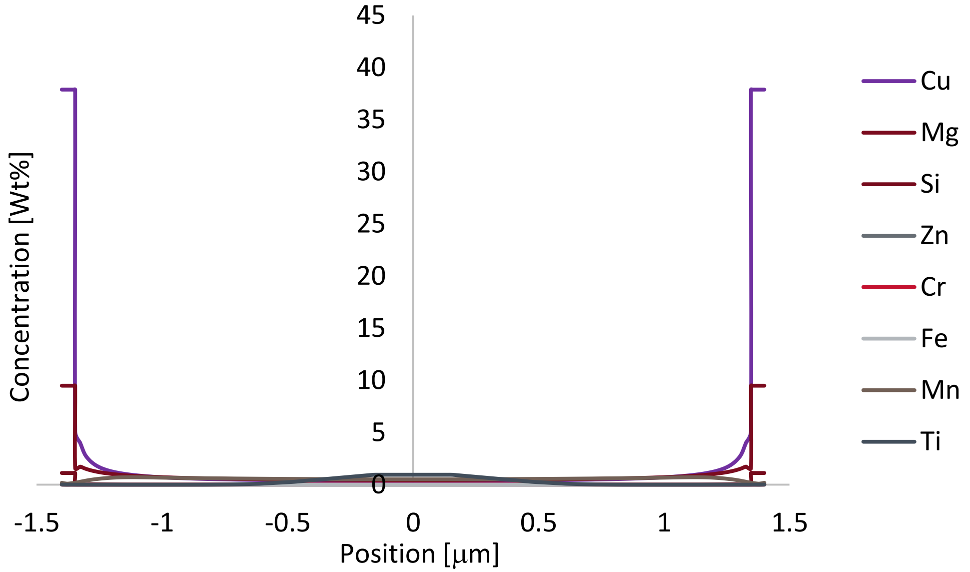



$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^m(d,t,T) + \Delta\sigma_{mic}^n(d,t,T) + \Delta\sigma_{ppt}^p(d,t,T)$$

# Segregation – Al 2024




Particle Diameter: 45 μm
Grain Diameter: 2.8 μm diameter
Cooling Rate: 5x10[4] °C/s

**Phases at Grain Boundary**

| Phase | Wt% |
|---|---|
| $Al_2Cu$ | 48.58 |
| T (Al,Cu,Mg) | 32.01 |
| FCC Al | 14.99 |
| $Mg_2Si$ | 3.06 |
| $Al_{12}Mn$ | 1.32 |
| $Al_{15}Fe_3Si_2$ | 0.02 |
| $Al_3Ti$ | 0.01 |

$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^{m}(d,t,T) + \Delta\sigma_{mic}^{n}(d,t,T) + \Delta\sigma_{ppt}^{p}(d,t,T)$$

# Phase Identification – Al 6061

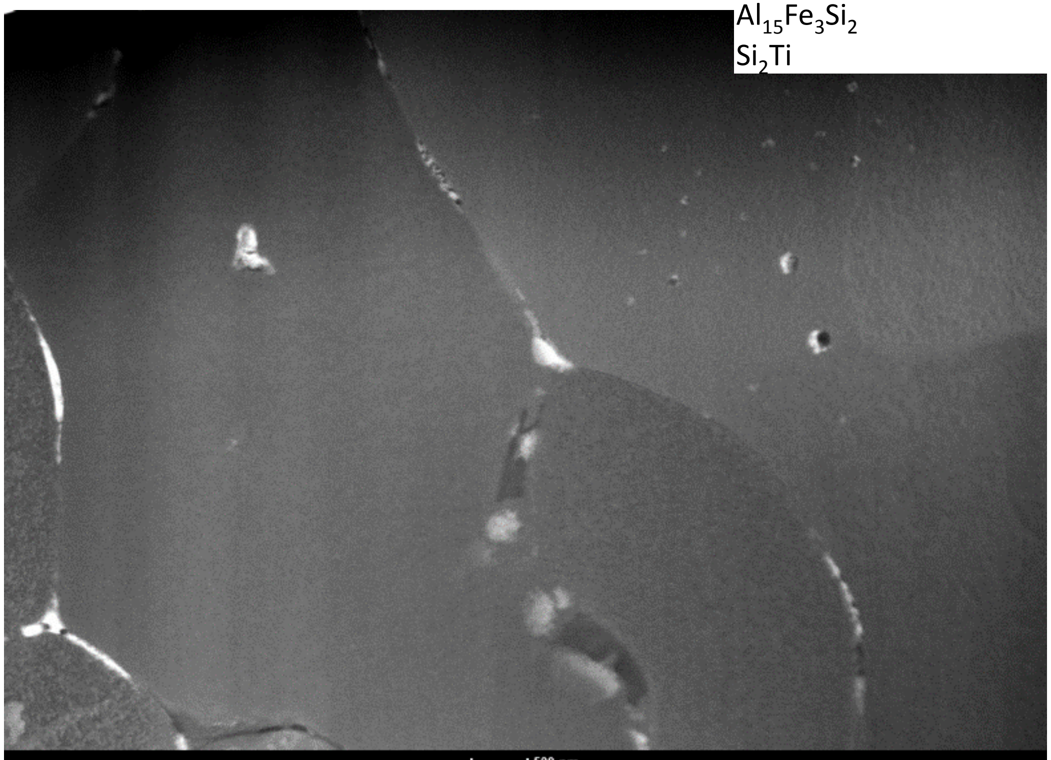
Particle Diameter: 28 μm
Grain Diameter: 2.15 μm diameter
Cooling Rate: 1.16x10$^4$ °C/s

**Phases at Grain Boundary**

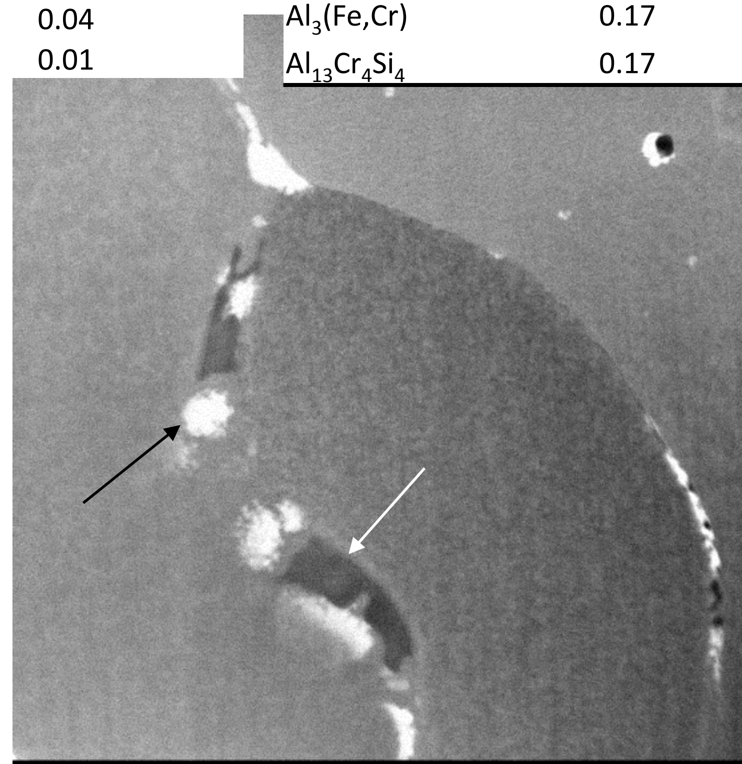
| Phase | Wt% |
|---|---|
| FCC Al | 62.08 |
| $Al_2Cu$ | 20.12 |
| Q ($Al_5Cu_2Mg_8Si_6$) | 12.79 |
| Si (Diamond) | 4.84 |
| $Al_{15}Si_2(Fe,Mn)_4$ | 0.07 |
| $Al_{13}Cr_4Si_4$ | 0.06 |
| $Al_{15}Fe_3Si_2$ | 0.04 |
| $Si_2Ti$ | 0.01 |

**Equilibrium Phases**

| Phase | Wt% |
|---|---|
| FCC Al | 96.5 |
| $Mg_2Si$ | 1.41 |
| $Al_7Cu_2M$ | 0.76 |
| α ($Al_{47}(Fe,Mn,Cr)_{11}Si_5$) | 0.65 |
| E ($Al_{18}Cr_2Mg_3$) | 0.23 |
| $Al_3(Fe,Cr)$ | 0.17 |
| $Al_{13}Cr_4Si_4$ | 0.17 |



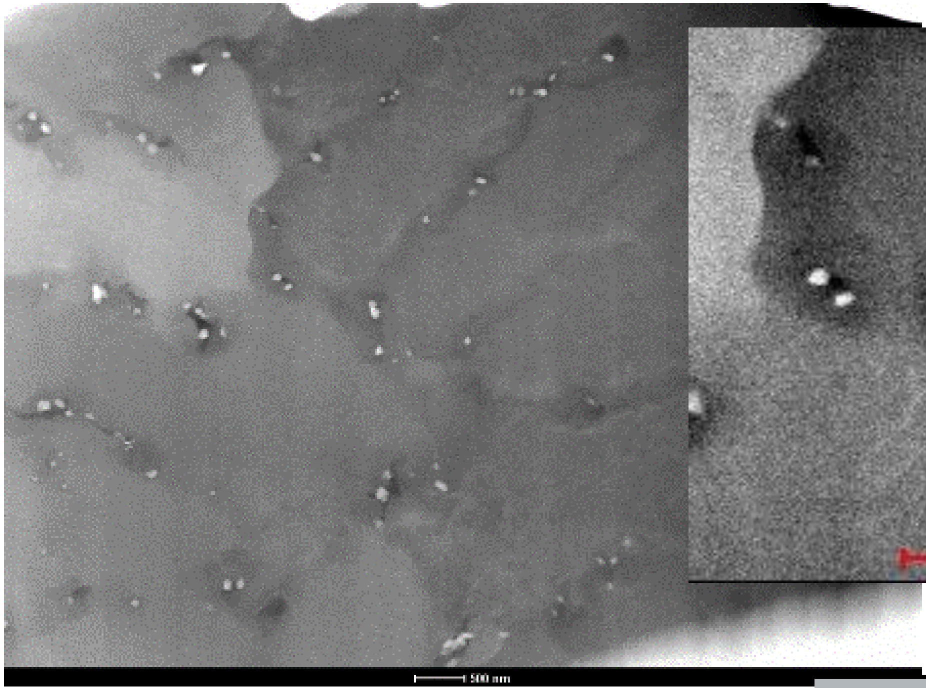

Scale bar: 500 nm

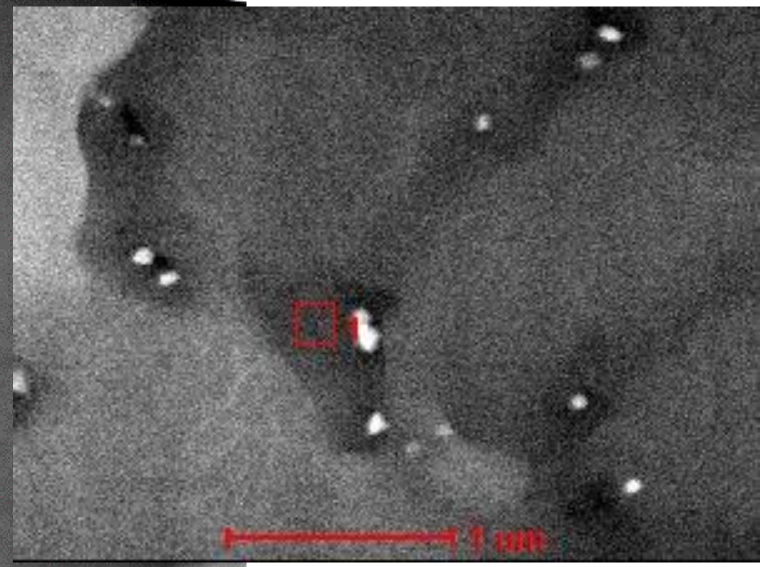


Scale bar: 500 nm

$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^{m}(d,t,T) + \Delta\sigma_{mic}^{n}(d,t,T) + \Delta\sigma_{ppt}^{p}(d,t,T)$$

# Phase Identification – Al 5056




| | Average Weight % | |
|---|---|---|
| | White | Gray |
| Mg | 9.60 | 8.72 |
| Al | 66.95 | 85.63 |
| Si | 1.05 | 0.37 |
| Cr | 0.34 | 0.21 |
| Mn | 1.46 | 0.12 |
| Fe | 14.02 | 0.11 |
| Cu | 6.52 | 4.83 |
| Zn | 0.08 | 0.00 |

Particle Diameter: 22 μm
Grain Diameter: 1.2 μm diameter
Cooling Rate: 2.04x$10^4$ °C/s

**Phases at Grain Boundary**

| Phase | Wt% |
|---|---|
| FCC Al | 83.45 |
| β ($Al_3Mg_2$) | 15.58 |
| $Al_{12}Mn$ | 0.83 |
| T ($Al_{29}Mg_{20}(Cu,Zn)_2$) | 0.06 |
| $Al_{13}Fe_4$ | 0.04 |
| $Al_{45}Cr_7$ | 0.03 |
| $Mg_2Si$ | 0.02 |

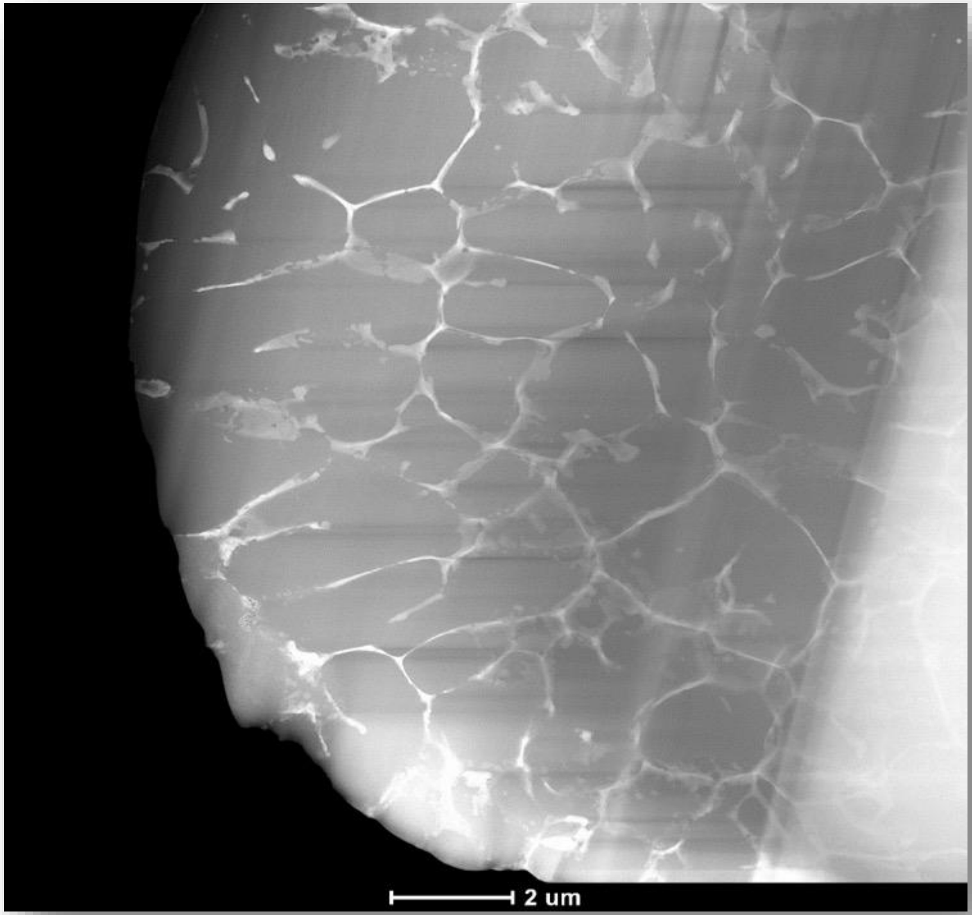
**Equilibrium Phases**

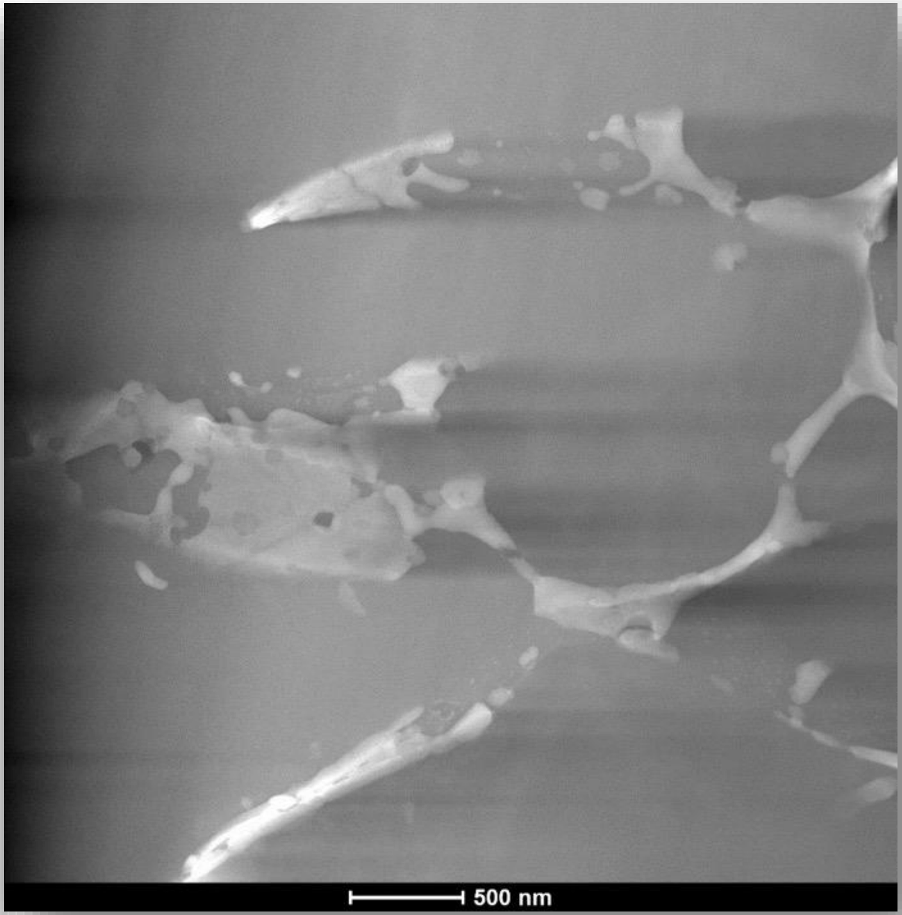
| Phase | Wt% |
|---|---|
| FCC Al | 84.8 |
| β $Al_3Mg_2$ | 13.3 |
| $Al_{45}Cr_7$ | 0.7 |
| $Al_{12}Mn$ | 0.6 |
| $Al_{13}Fe_4$ | 0.5 |
| T | 0.1 |
| $Mg_2Si$ | 0.1 |

$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^m(d,t,T) + \Delta\sigma_{mic}^n(d,t,T) + \Delta\sigma_{ppt}^p(d,t,T)$$

# Phase Identification – Al 7075

HAADF STEM Al 7075 (25-32 um)

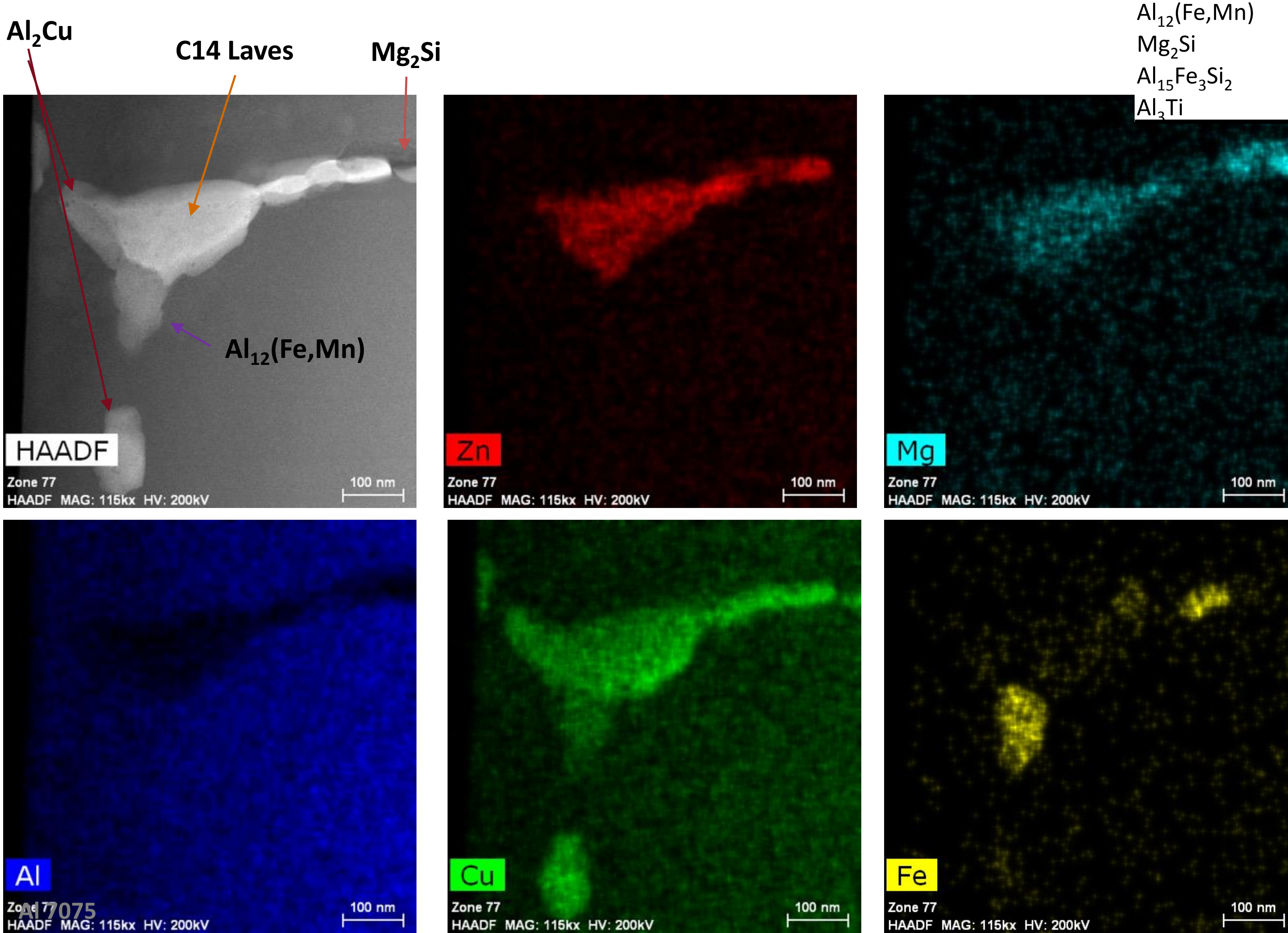


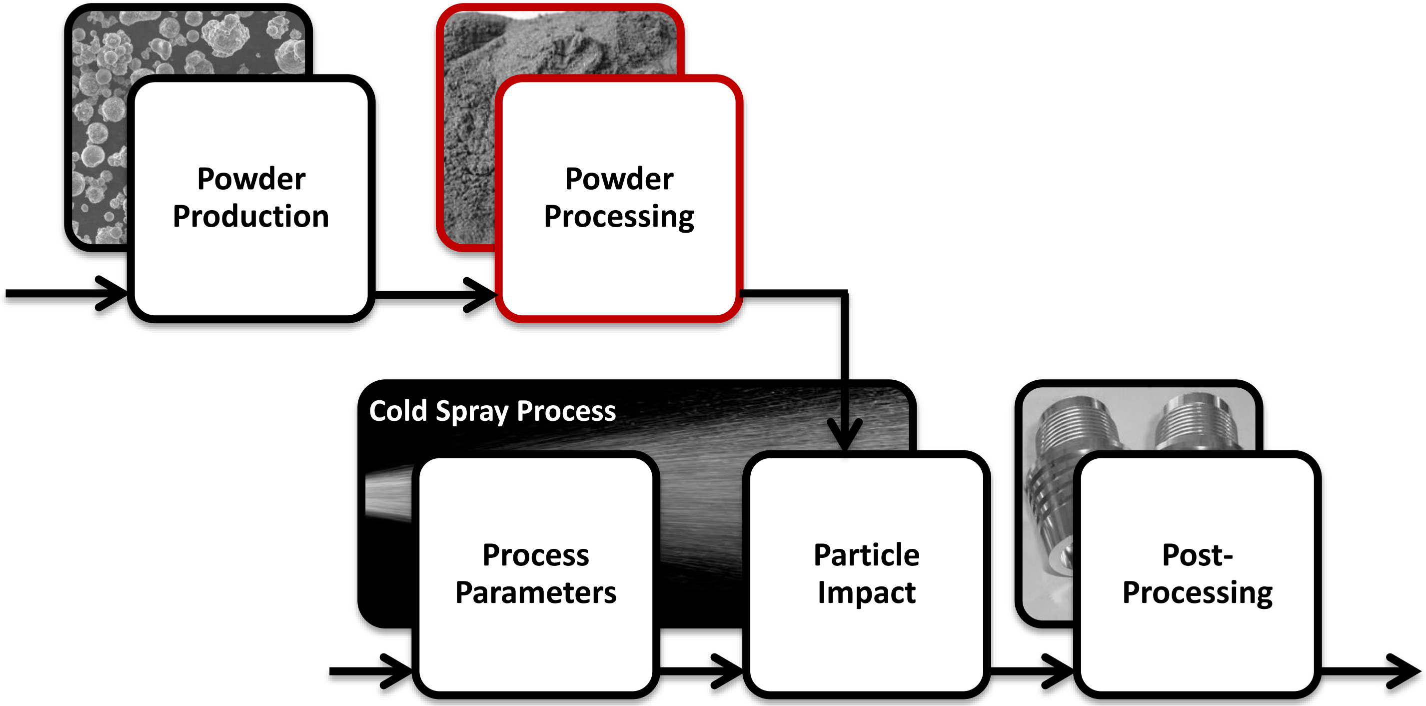



# Phase Identification – Al 7075

| Grain Boundary Phase | Wt% |
|---|---|
| FCC Al | 85.91 |
| C14 Laves ($Mg(Zn,Al,Cu)_2$) | 11.16 |
| T (Al,Cu,Mg) | 1.76 |
| $Al_2Cu$ | 0.77 |
| $Al_{45}Cr_7$ | 0.13 |
| $Al_{12}(Fe,Mn)$ | 0.14 |
| $Mg_2Si$ | 0.08 |
| $Al_{15}Fe_3Si_2$ | 0.04 |
| $Al_3Ti$ | 0.03 |

$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^{m}(d,t,T) + \Delta\sigma_{mic}^{n}(d,t,T) + \Delta\sigma_{ppt}^{p}(d,t,T)$$






# Powder Processing

## Kinetic Modeling

# Thermal Treatment Effects

$$\Delta\sigma_{ppt}(d,t,T) = \sum_i [\sigma_{Co,i} + \sigma_{Mo,i} + \sigma_{Ch,i} + \sigma_{in,i}]$$

$$\Delta\sigma_{Coherency,i} = 7\varepsilon_{coh,i}^{3/2} G \left(\frac{rf}{b}\right)^{1/2}$$

$$\Delta\sigma_{Modulus,i} = 0.01\varepsilon_{Gp,i}^{3/2} G \left(\frac{rf}{b}\right)^{1/2}$$

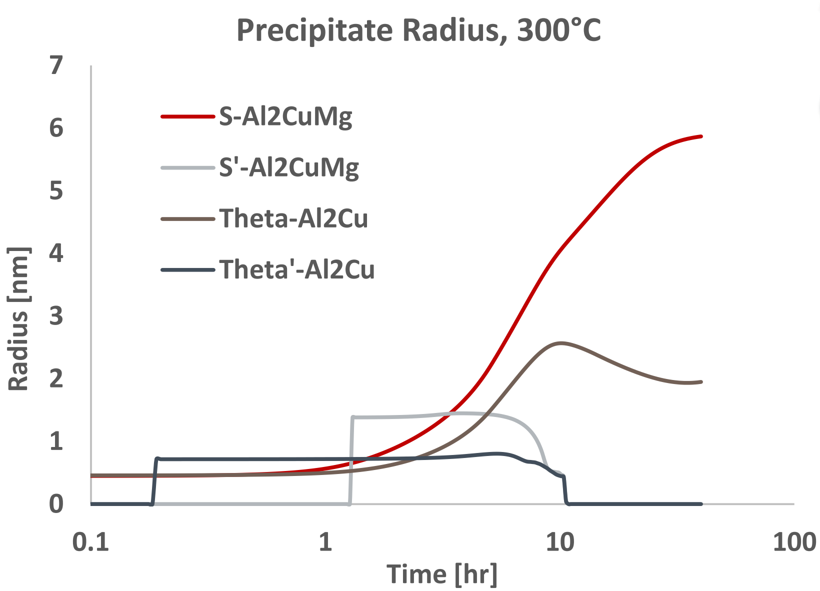
$$\Delta\sigma_{Chemical,i} = 2\varepsilon_{Ch,i}^{3/2} G \left(\frac{rf}{b}\right)^{1/2}$$

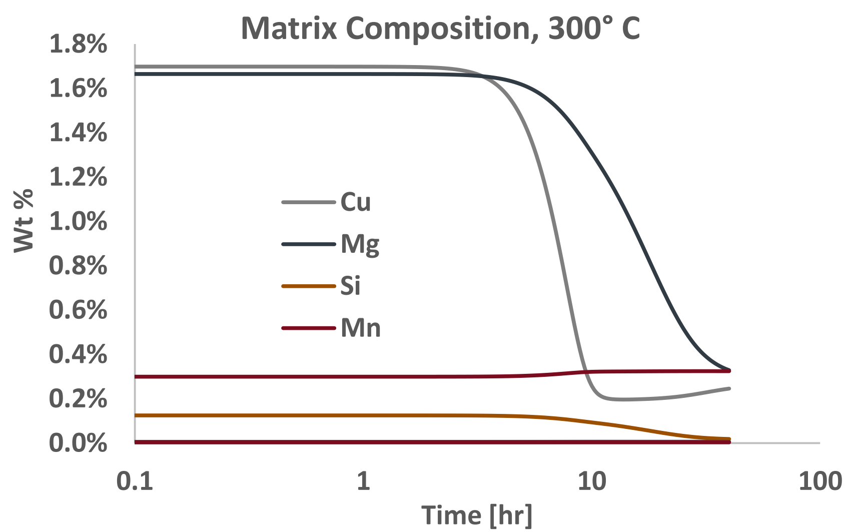
$$\Delta\sigma_{Incoh} = \mathrm{Gb}/(\mathrm{L} - 2\mathrm{r})$$

$$\Delta\sigma_{ss}(d, t=0, T) = \sum_i (G\varepsilon_s^{3/2} c^{1/2})/700$$

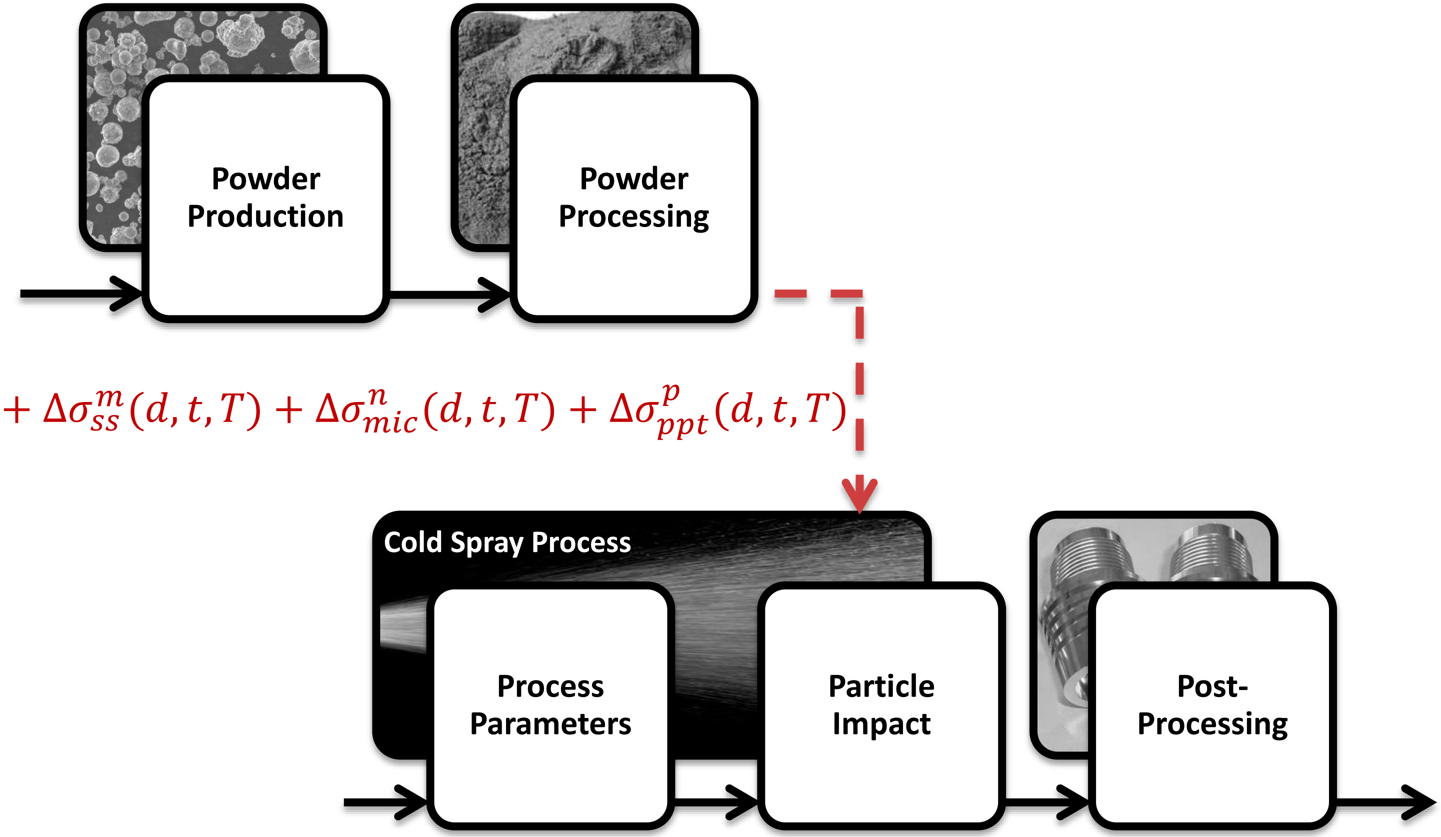
$$\Delta\sigma_{mic}(d,t,T) = \frac{k_{gs}}{\sqrt{\lambda}}$$

$$d^n - d_0^n = kt$$




$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^m(d,t,T) + \Delta\sigma_{mic}^n(d,t,T) + \Delta\sigma_{ppt}^p(d,t,T)$$



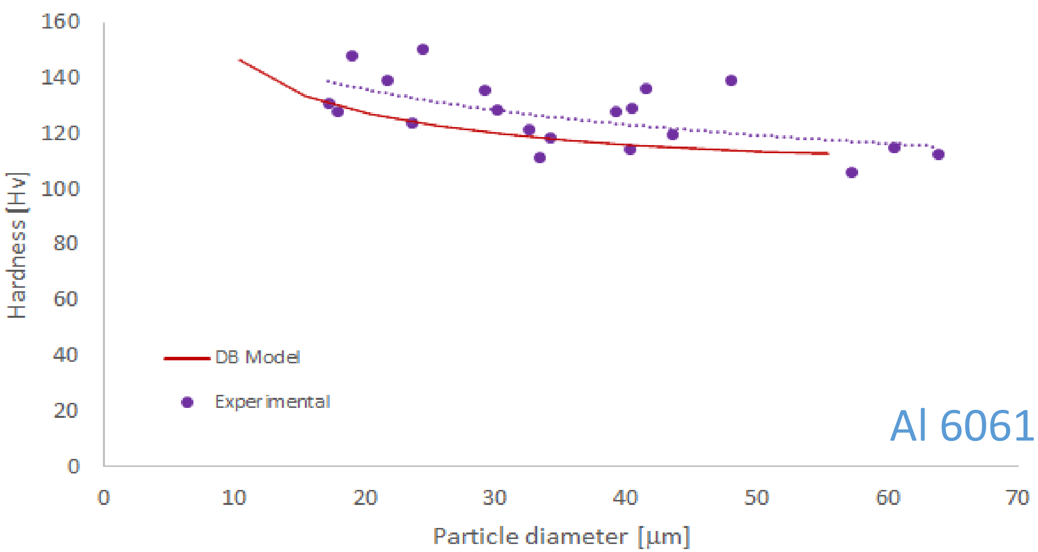

$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^{m}(d,t,T) + \Delta\sigma_{mic}^{n}(d,t,T) + \Delta\sigma_{ppt}^{p}(d,t,T)$$

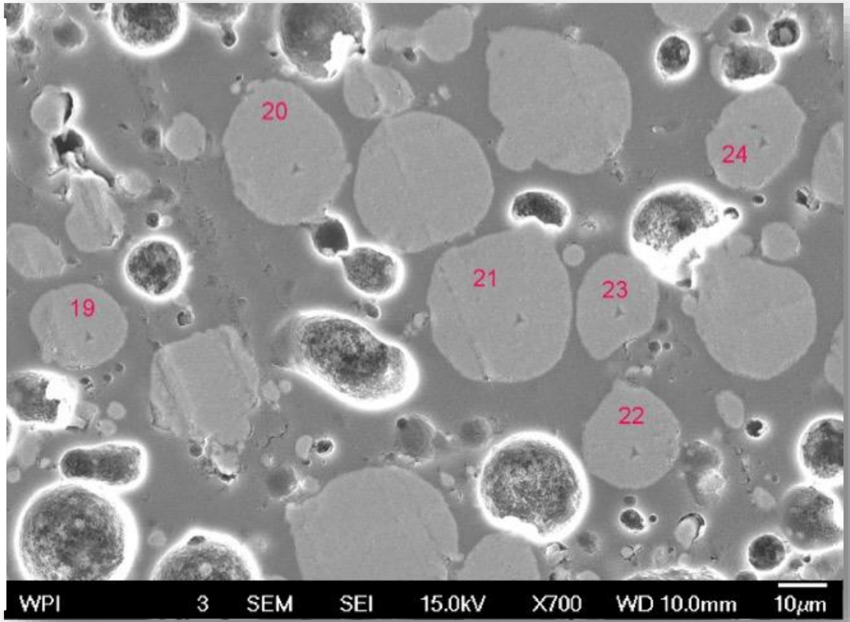
# Additive Yield Strength Model

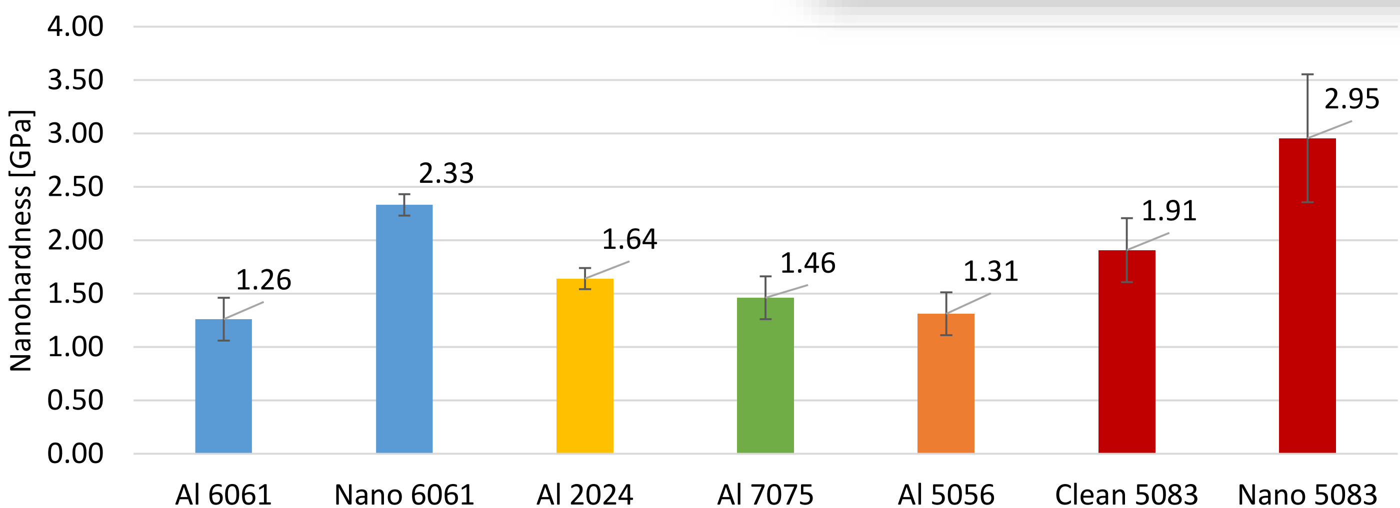
## Characterization: Nanohardness

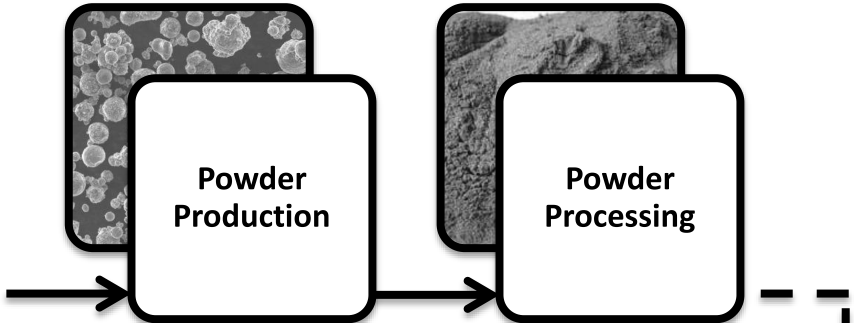
# Additive Hardness Model – Al 6061

$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^{m}(d,t,T) + \Delta\sigma_{mic}^{n}(d,t,T) + \Delta\sigma_{ppt}^{p}(d,t,T)$$

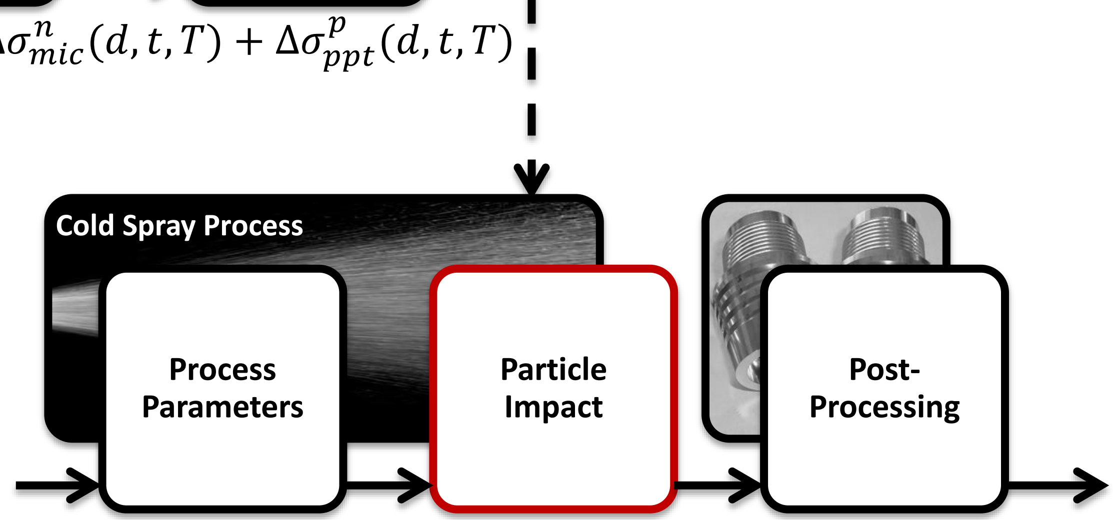





$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^{m}(d,t,T) + \Delta\sigma_{mic}^{n}(d,t,T) + \Delta\sigma_{ppt}^{p}(d,t,T)$$

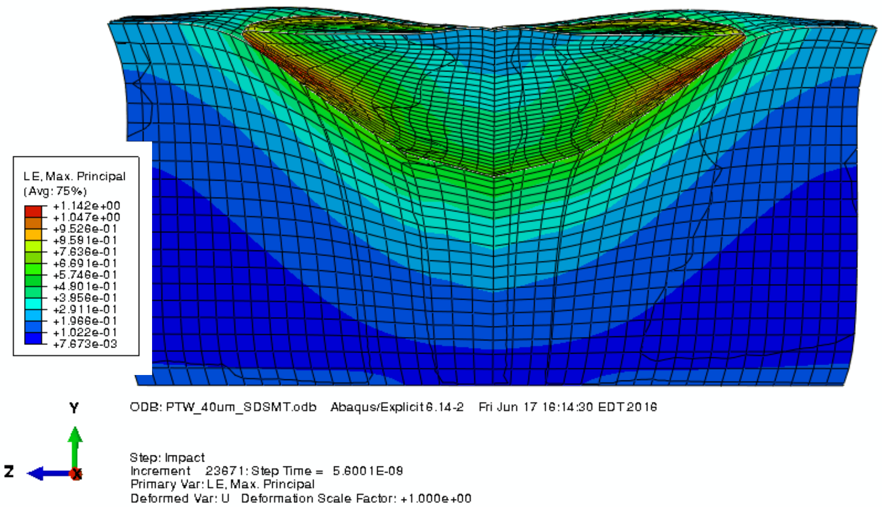


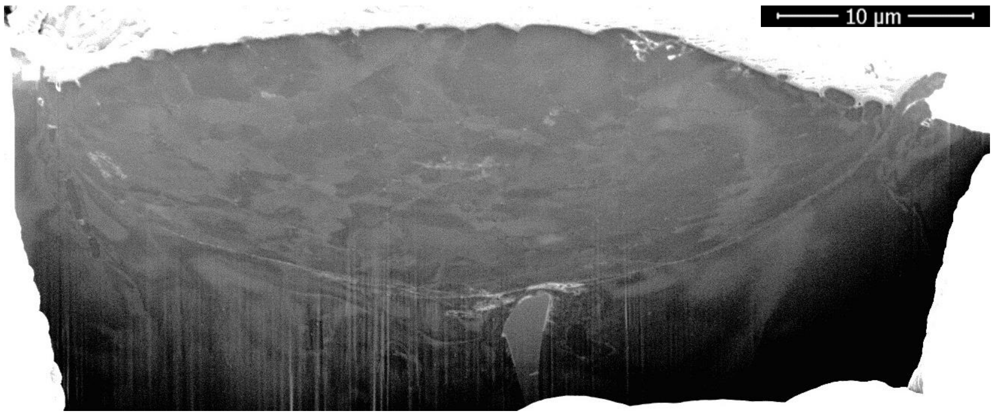

# Particle Impact

Impact Model

Characterization of Deposit

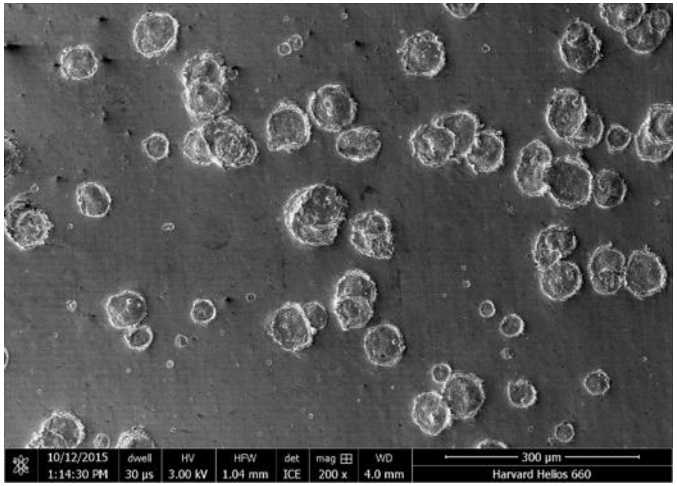
# Particle Impact Model and Experimental

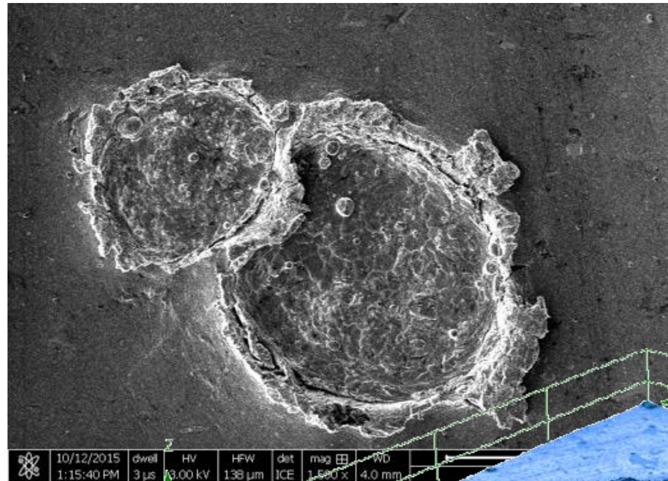


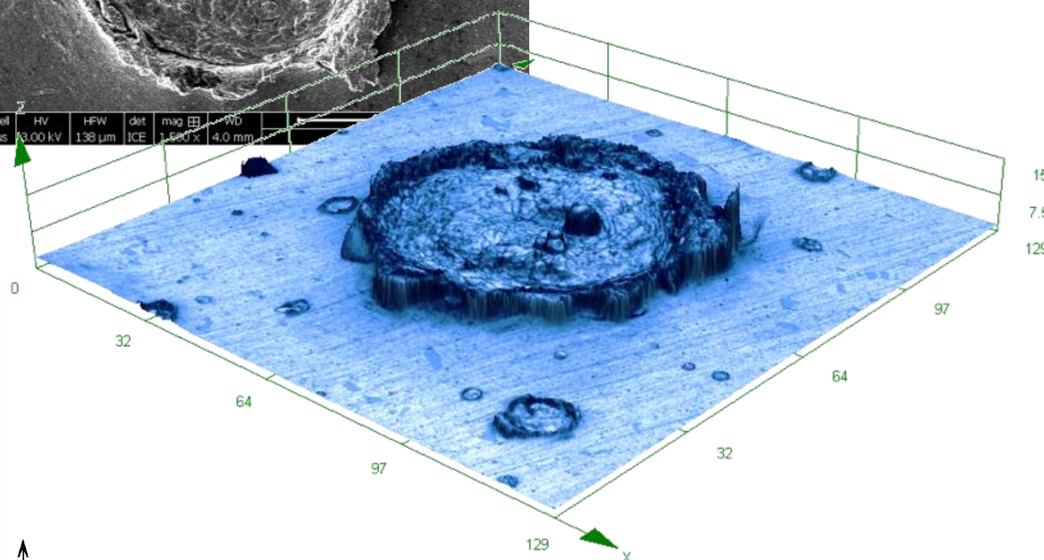




- Substrate: Al 6061 T6
- Particle: Al 6061 HT 230°C for 1 hour
- 40 μm diameter
- Output: Strain

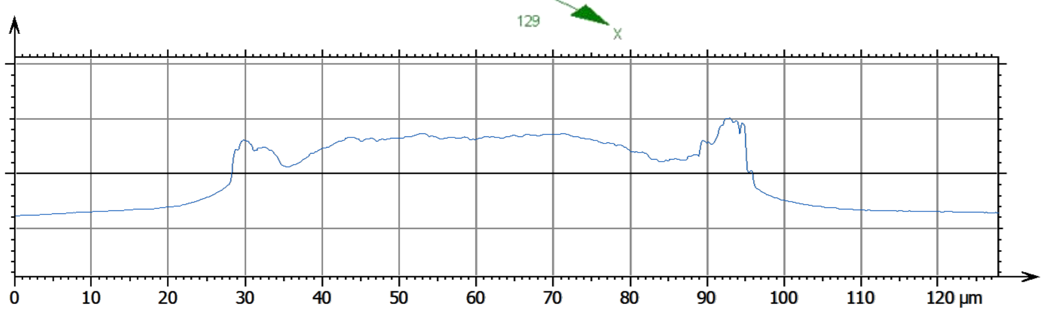
# Single Particle Splats

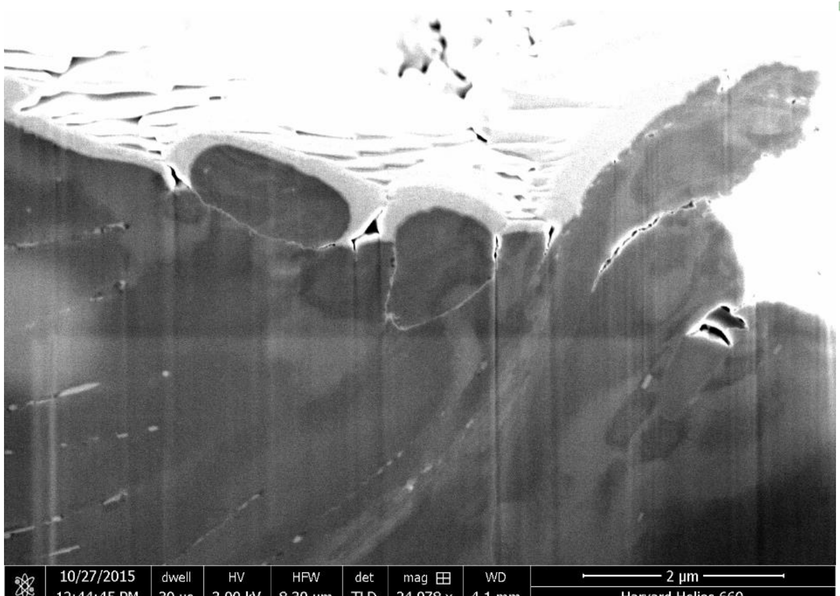



SEM Images

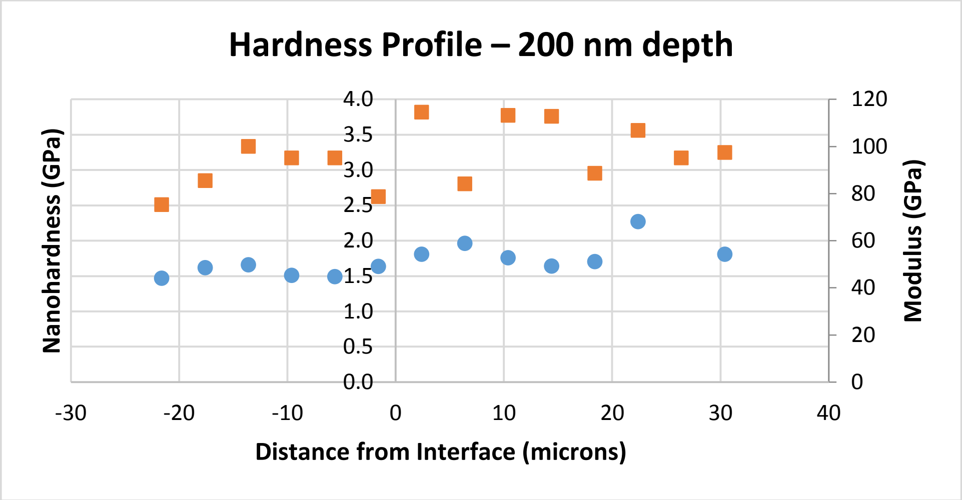


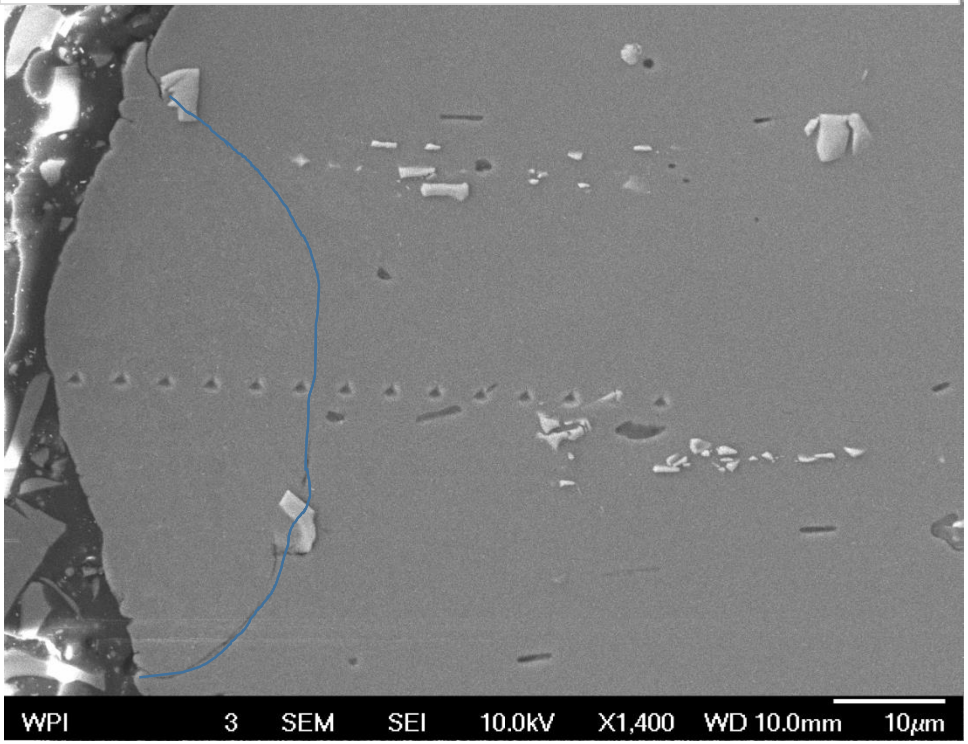




Center for Nanoscale Systems (CNS)

Laser Scanning Confocal Microscope

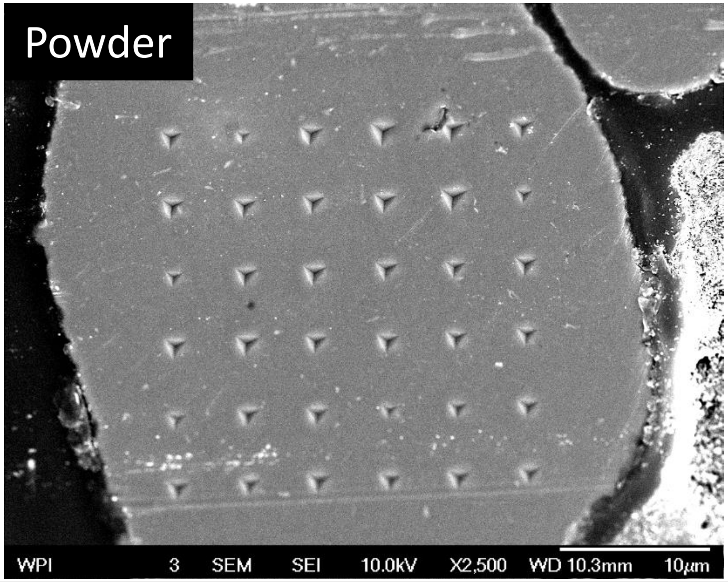
# Single Impact Nanohardness

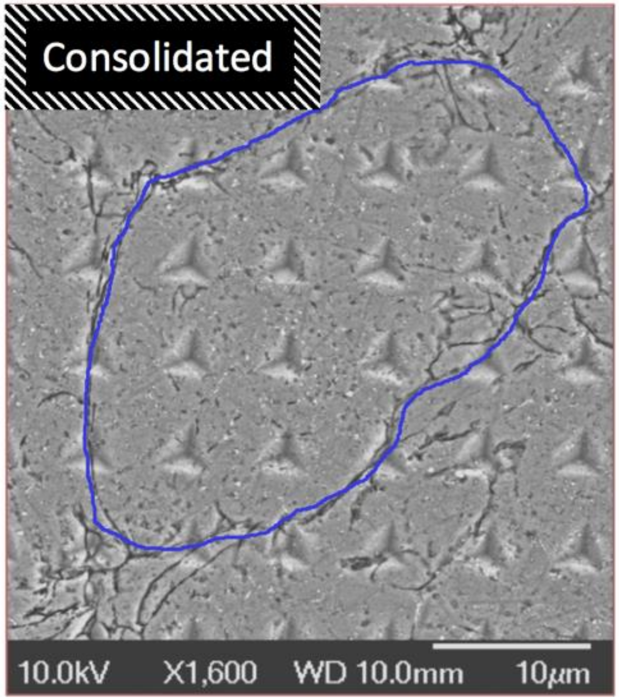


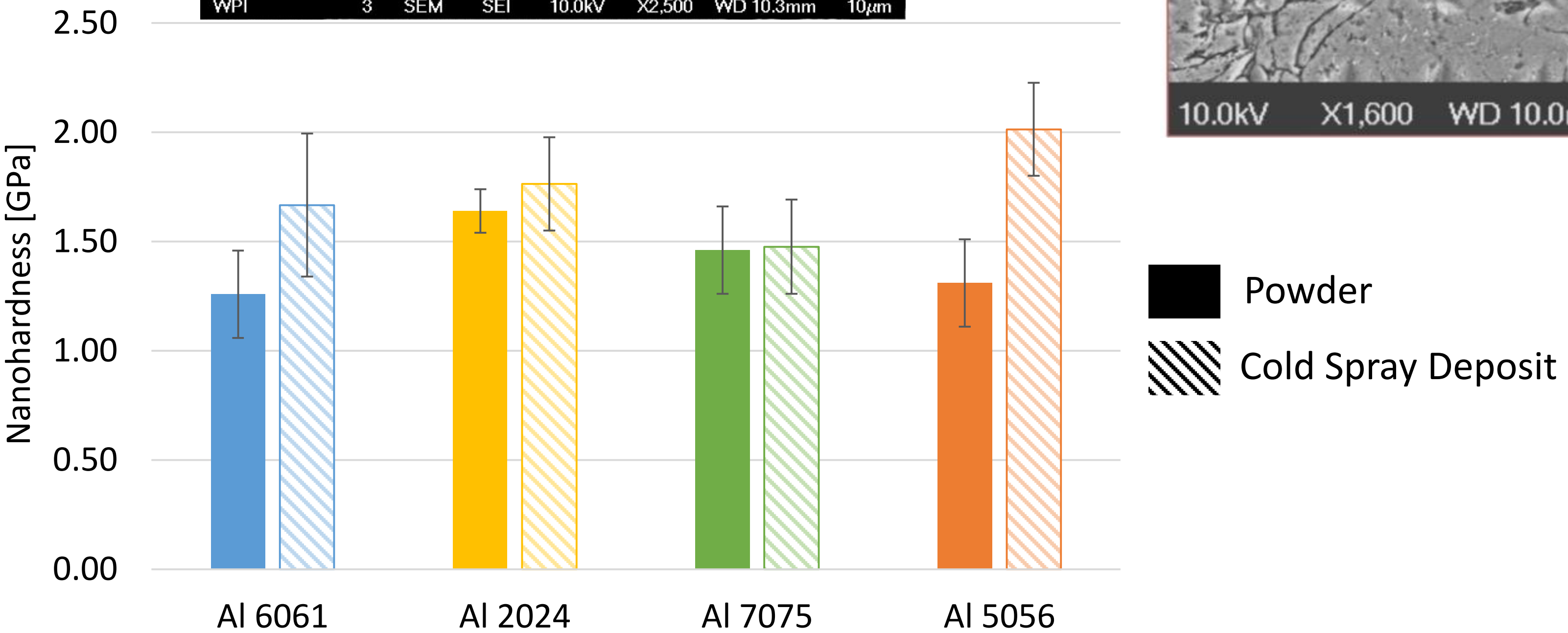



# Cold Spray Deposit Properties

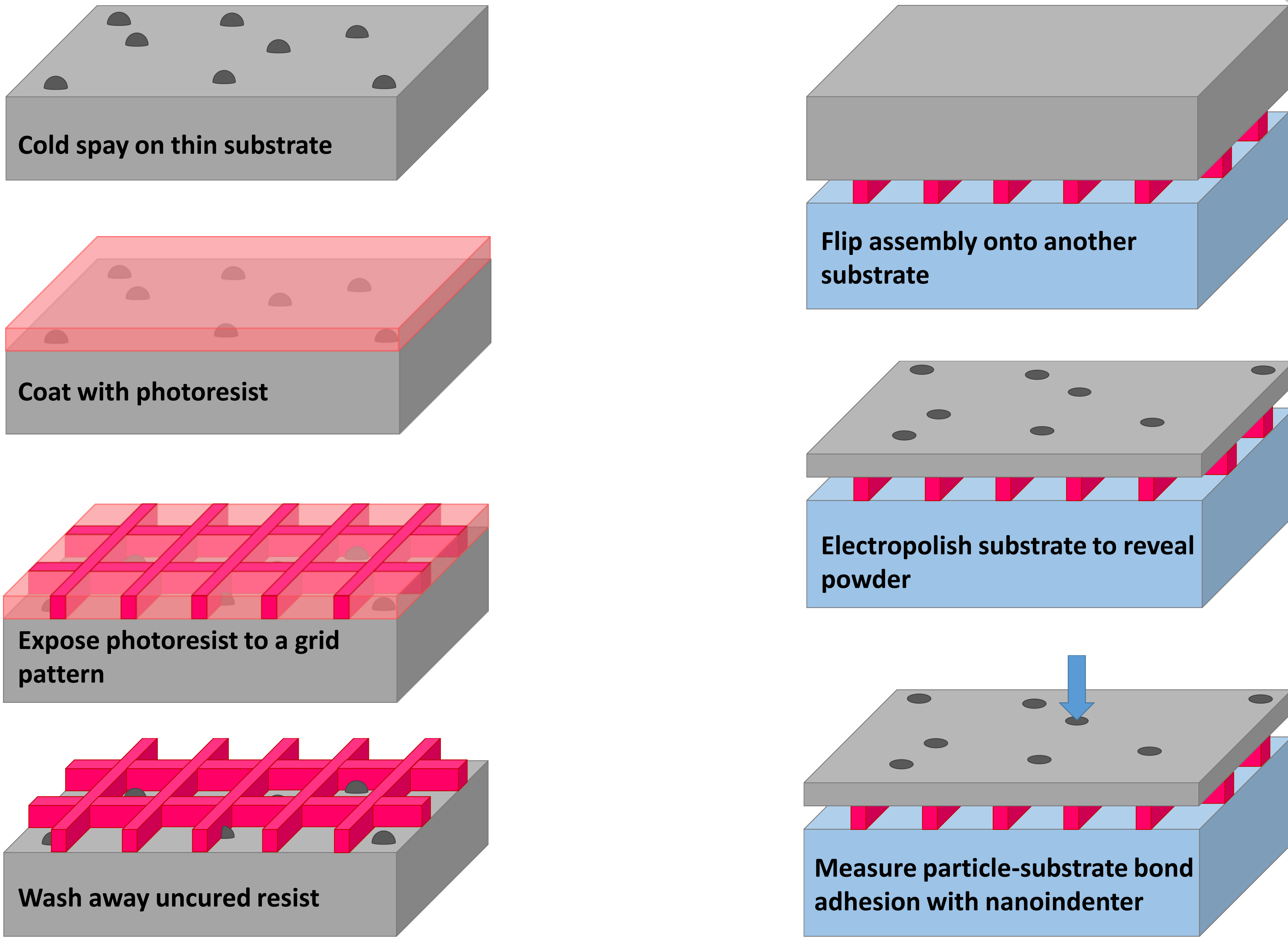
Powder & Cold Spray Nanohardness



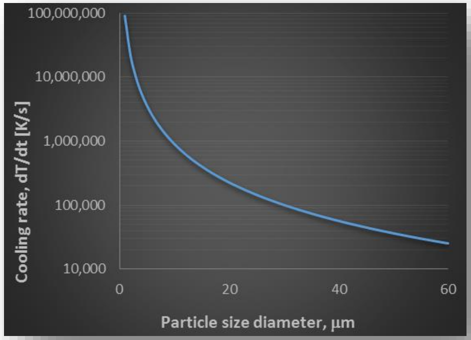



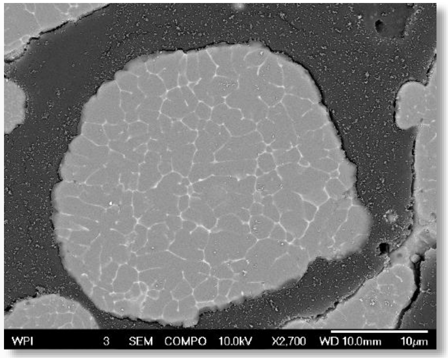



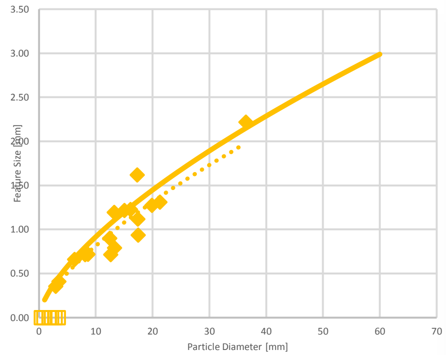
# Particle-Substrate Adhesion Test

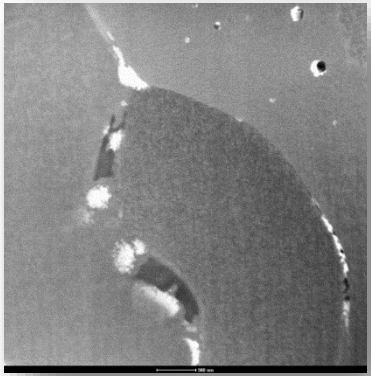


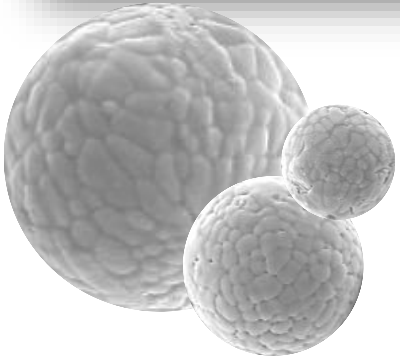
# TPM Overview

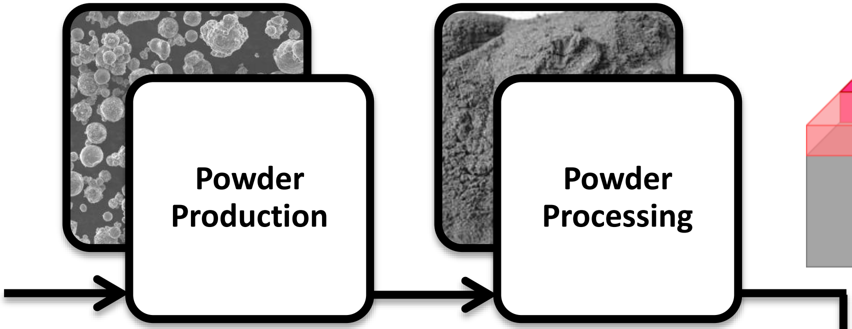


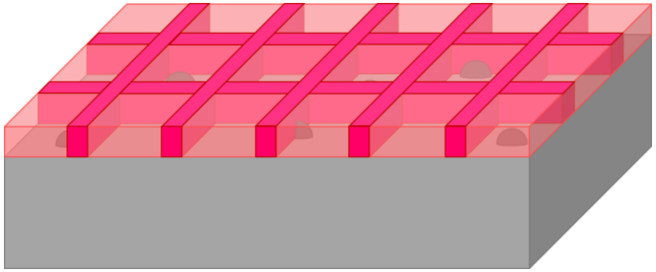



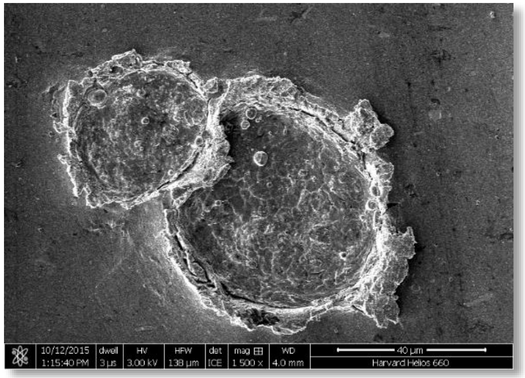



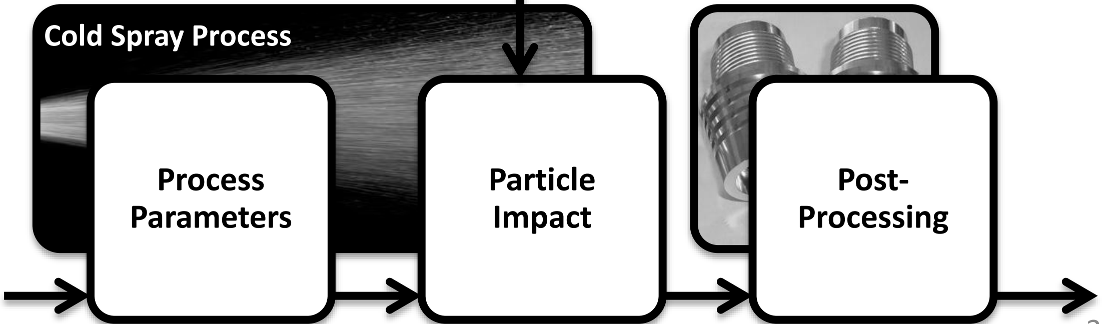




$$\sigma_{YS}(d,t,T) = \sigma_o + \Delta\sigma_{ss}^{m}(d,t,T) + \Delta\sigma_{mic}^{n}(d,t,T) + \Delta\sigma_{ppt}^{p}(d,t,T)$$



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PENNSTATE ARL
**Applied Research Laboratory**

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