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Lessons Learned from Heat Treating N_2-sprayed Al 6061 Coatings

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https://coldspray.coe.neu.edu

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Acknowledgment
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Differences between He and N_2 sprayed Al 6061

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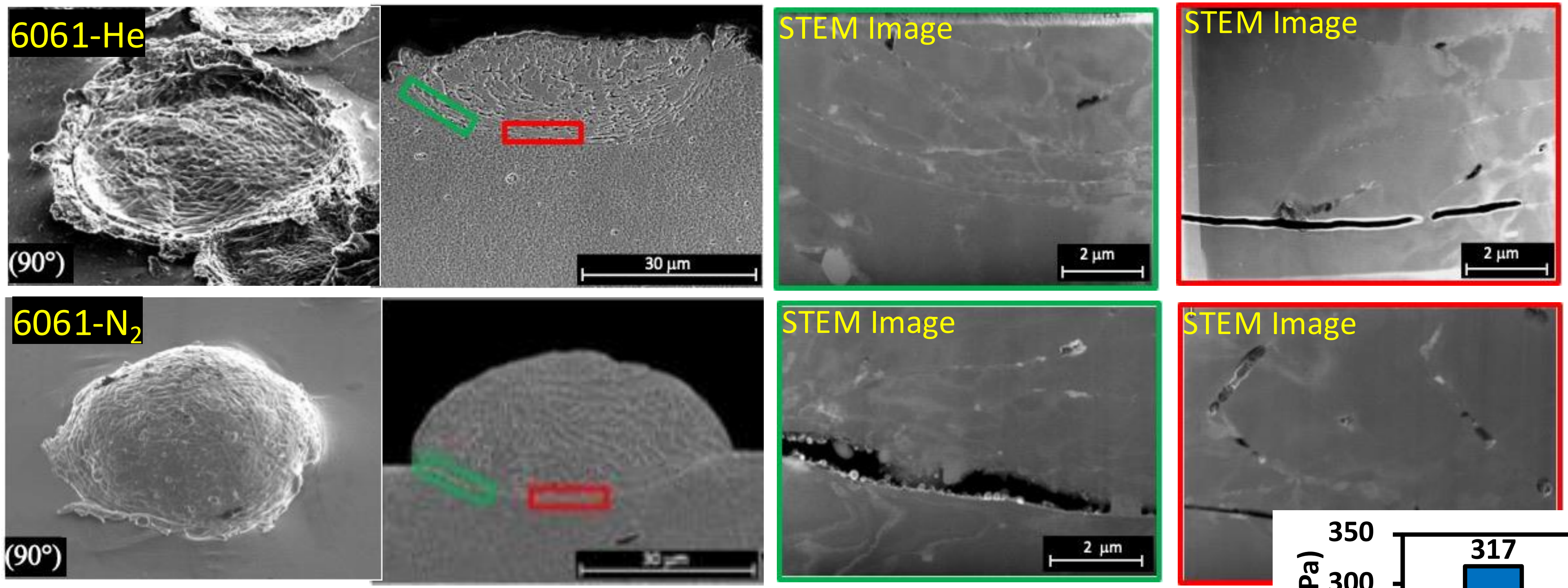
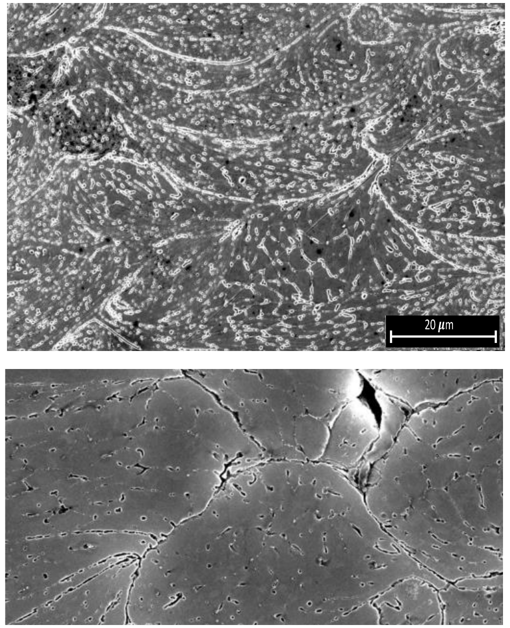
Single particle deposits (AL-6061)

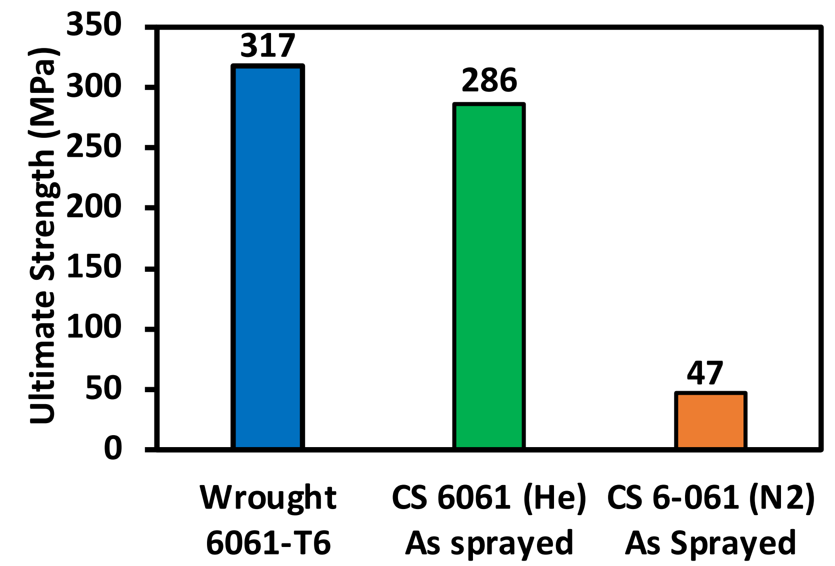


Multi-layer deposits



Bonding quality in different deposits

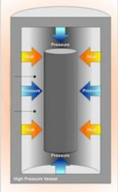
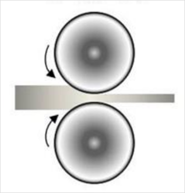
	Peripheral region	Polar region
He-Sprayed deposits	perfect bonding	partial bonding
N2-Sprayed deposits	Partial bonding	no bonding



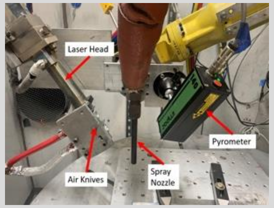
A, Nourian, S. Müftü, Surface & Coatings Technology, 2022, 444, 128676
A, Nourian, T, Schwartz, S. Müftü, J Therm Spray Tech, 2022, 31:2517–2536

in-situ, pre- and post-spray heat treatments

- Post spray treatment of deposits
  - Regular heat treatment
  - Hot isostatic press (HIP) treatment
  - Thermomechanical treatment

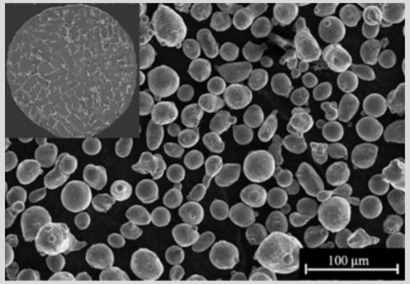




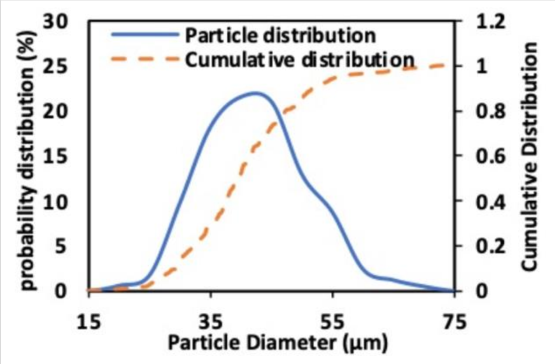
- Laser assisted cold spray (LACS)



Tests
- Deposition efficiency
- Porosity
- Tensile properties
- Fatigue properties

Spray conditions





Parameters	Al6061-N_2	Al6061-He
Carrier gas	N_2	He
Gas pressure (bar)	65	40
Gas Temperature (°C)	455	425
Standoff distance (mm)	25	25
Nozzle material	PBI	PBI
Nozzle length (mm)	170	170
Nozzle throat dia. (mm)	2.0	1.5
Nozzle exit dia. (mm)	6.0	6.0
Powder feed rate (rpm)	6.0	6.0

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Post-spray heat treatment of Al-6061

Reminder

Sintering: T_S = 585 - 652°C Heat above the solidus temperature. Coalesce particles.

Solution HT: T_{SH} = 516 - 579°C Distribute alloying elements within the microstructure.

Annealing: T_{Ann} = 415°C. Heat above the recrystallization temperature. Increases ductility and reduce hardness

Aging: T_{Age} = 171-183°C Provide precipitation hardening. form new (Mg_2Si) precipitates

Post-spray heat treatments in this work

Conventional heat treatments, CHT

Annealing
Annealing+Solution HT + Aging
High temp (HT)-annealing (sintering)
High temp (HT)-annealing (sintering) + Solution HT + Aging

Hot Isostatic Press, HIP, (160 MPa) at HT conditions

Annealing
Annealing+Solution HT + Aging
High temp (HT)-annealing (sintering)
High temp (HT)-annealing (sintering) + Solution HT + Aging

Group	Designation	Post-spray treatment	Procedure
Conventional Heat Treatment	CHT-Ann	Annealing	413°C x 2hr
	CHT-SA	Anneal + Solution HT + Aging	(413°C x 2hr) + (530°C x 1.5hr) + (175°C x 8hr)
	CHT-H-Ann	HT-Anneal (Sintering)	(600°C x 2hr)
	CHT-H-SA	HT-Anneal + Solution HT + Aging	(600°C x 2hr) + (530°C x 1.5hr) + (175°C x 8hr)
Hot Isostatic Pressing	HIP-Ann	(HIP & Solution HT) + Anneal	(160 MPa & 530°C x 3hr) + (413°C x 1hr)
	HIP-SA	(HIP & Solution HT) + Aging	(160 MPa & 530°C x 3hr) + (175°C x 8hr)
	HIP-H-SA	(HIP & HT-Anneal) + Anneal	(160 MPa & 600°C x 3hr) + (413°C x 1hr)
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Microstructure after different spray and post-spray conditions

Group	Designation	Post-spray treatment	Procedure
Conventional Heat Treatment	CHT-Ann	Annealing	413°C x 2hr
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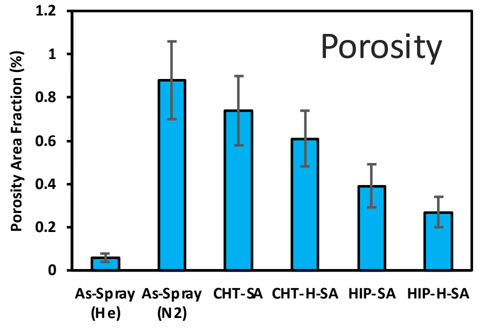
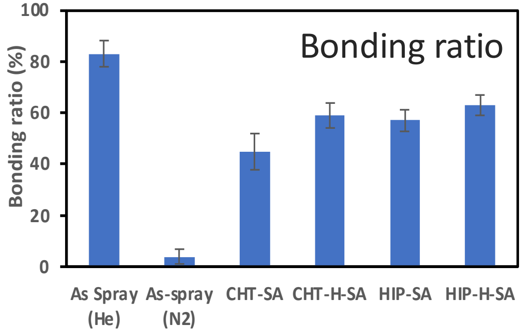
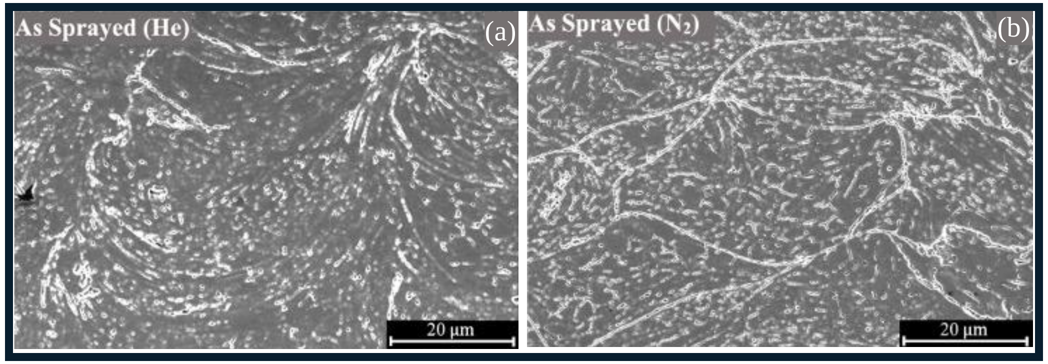
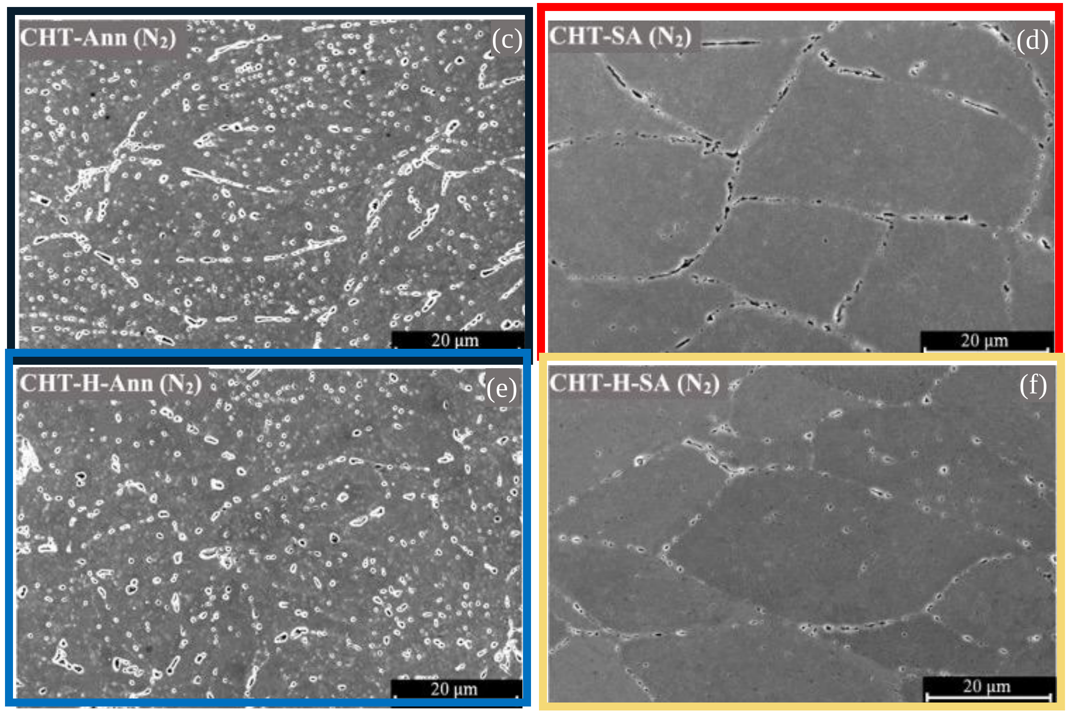




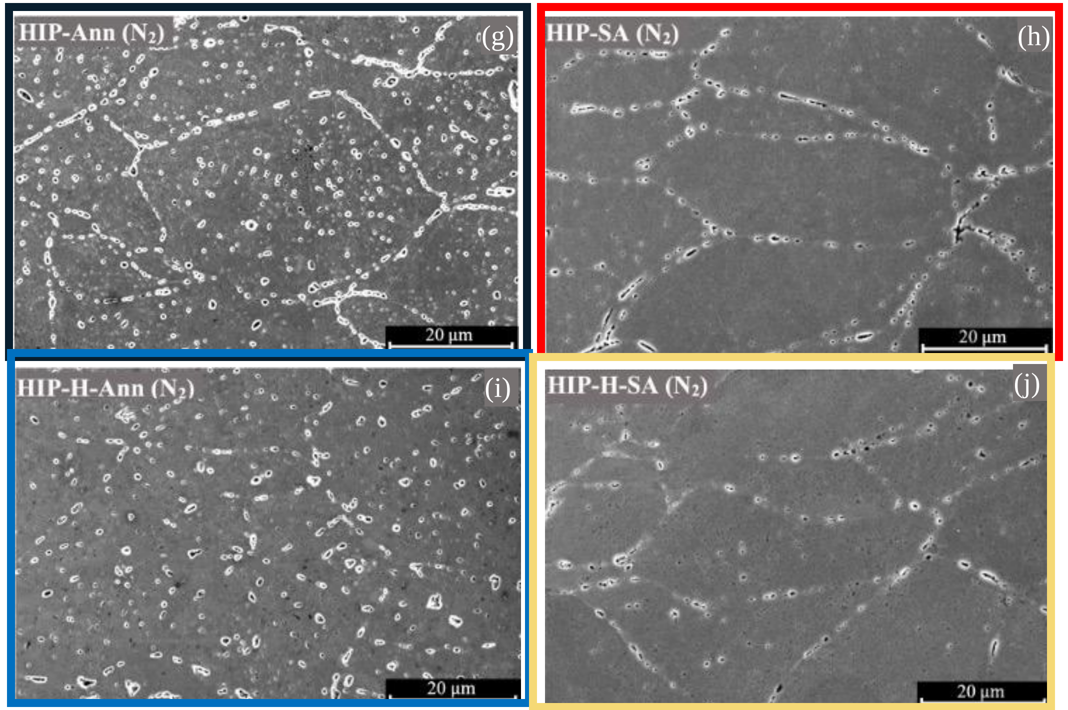
As sprayed



Conventional heat treatment(s)



Hot isostatic press treatments



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Tensile properties after different spray and post-spray conditions

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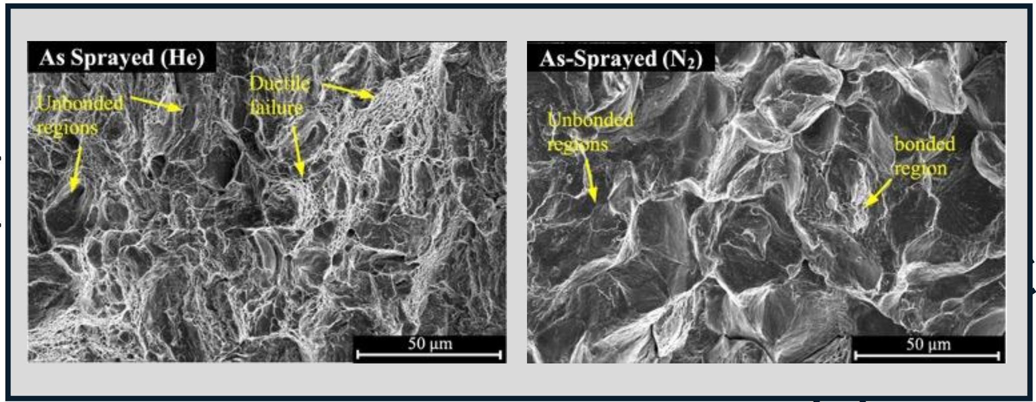
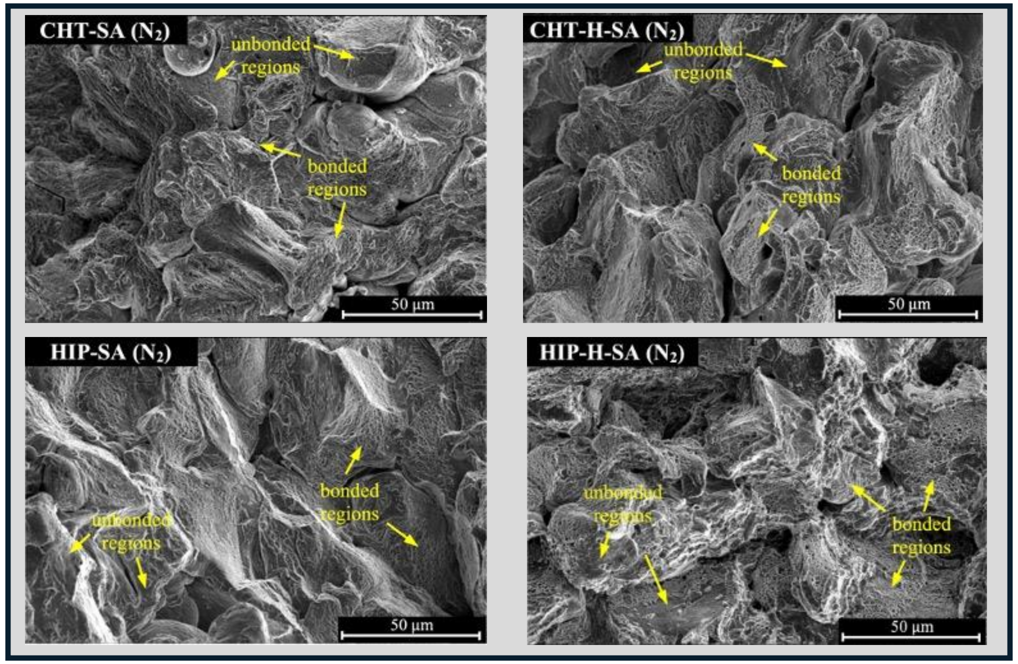
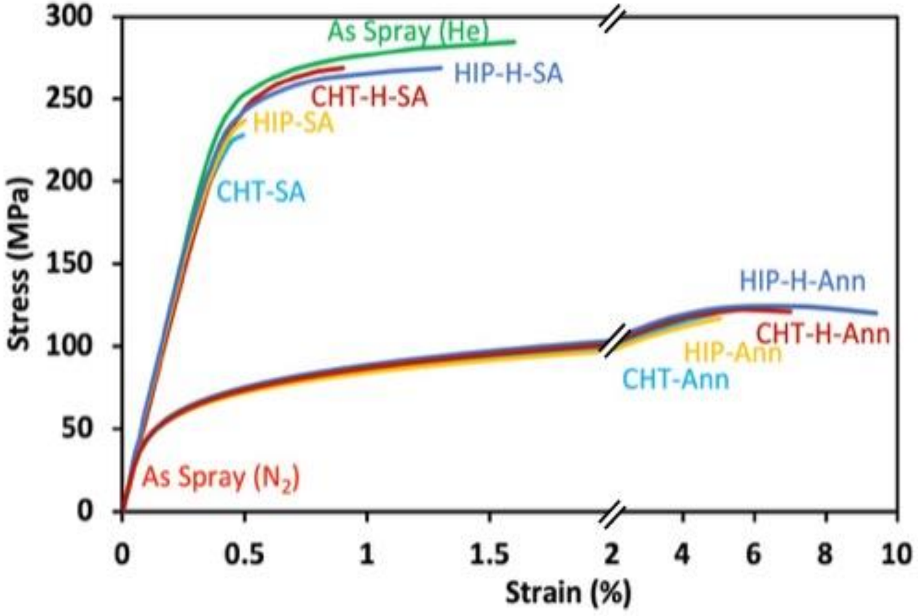
As sprayed

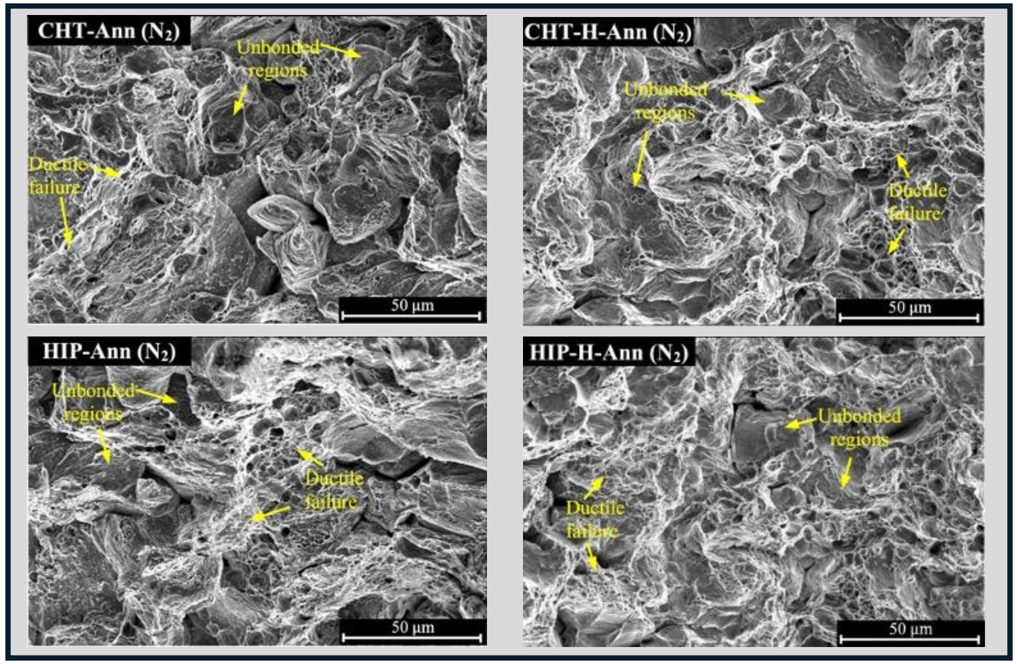


Solutionized





Annealed



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Fatigue properties after different spray and post-spray conditions

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Group	Designation	Post-spray treatment	Procedure
Conventional Heat Treatment	CHT-Ann	Annealing	413°C x 2hr
	CHT-SA	Anneal + Solution HT + Aging	(413°C x 2hr) + (530°C x 1.5hr) + (175°C x 8hr)
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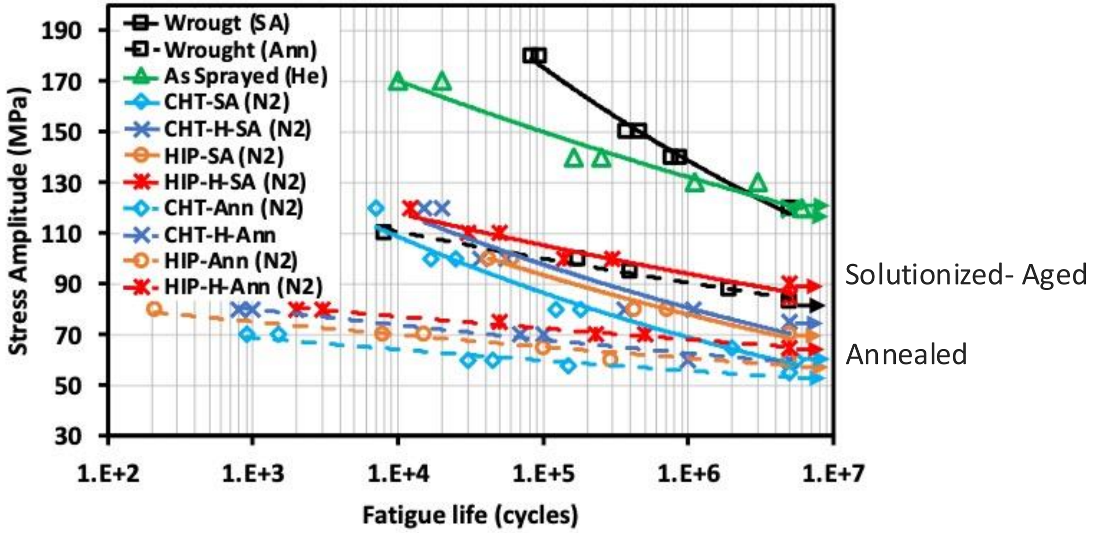
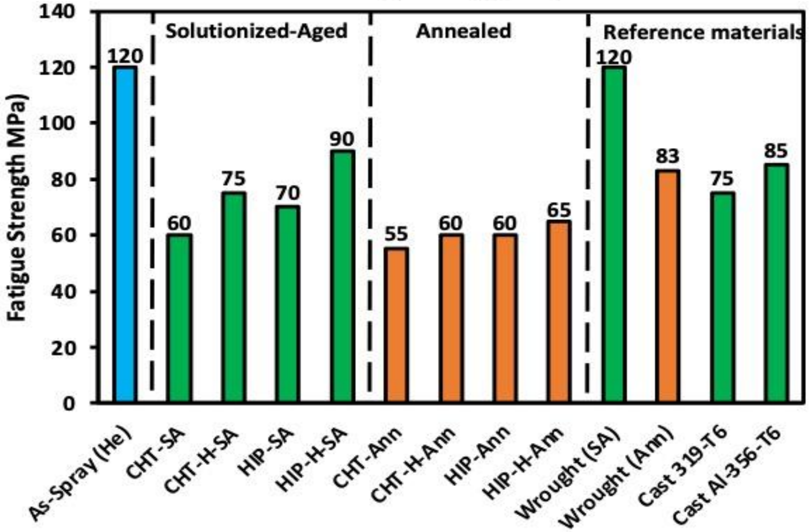
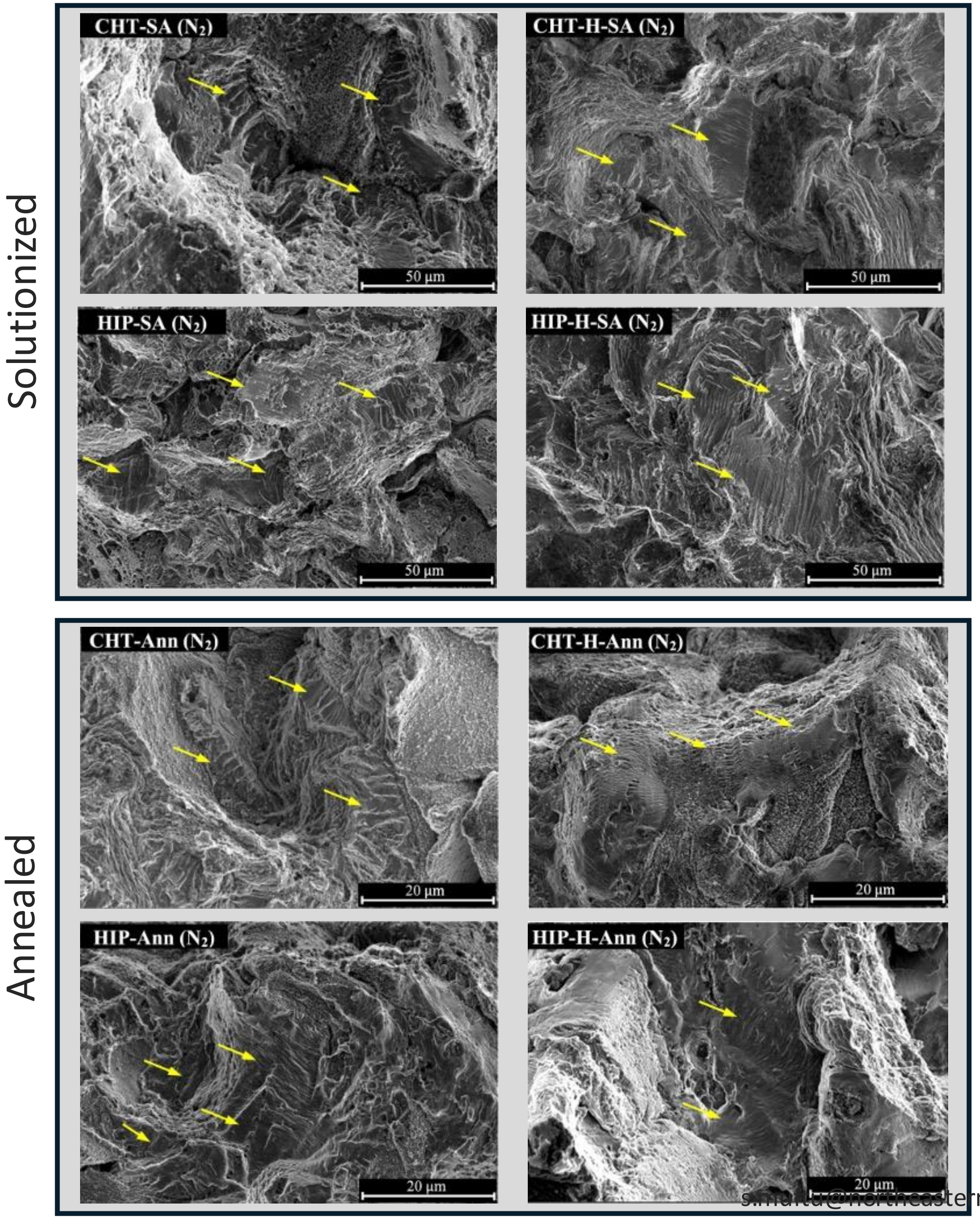
	HIP-H-Ann	(HIP & HT-Anneal) + Aging	(160 MPa & 600°C x 3hr) + (175°C x 8hr)

Load, control (R=-1, 15 Hz) ASTM E466–15. Fatigue life of 5×10^6 cycles.







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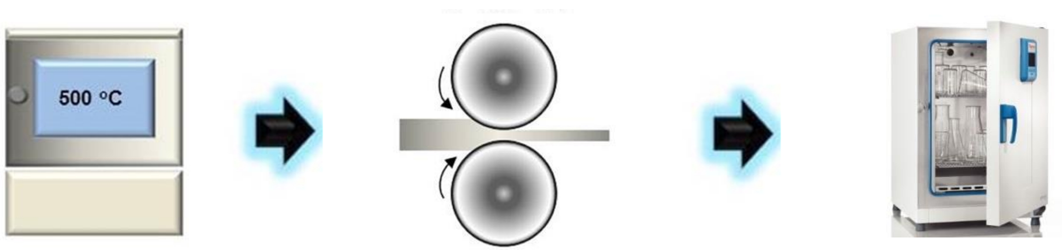
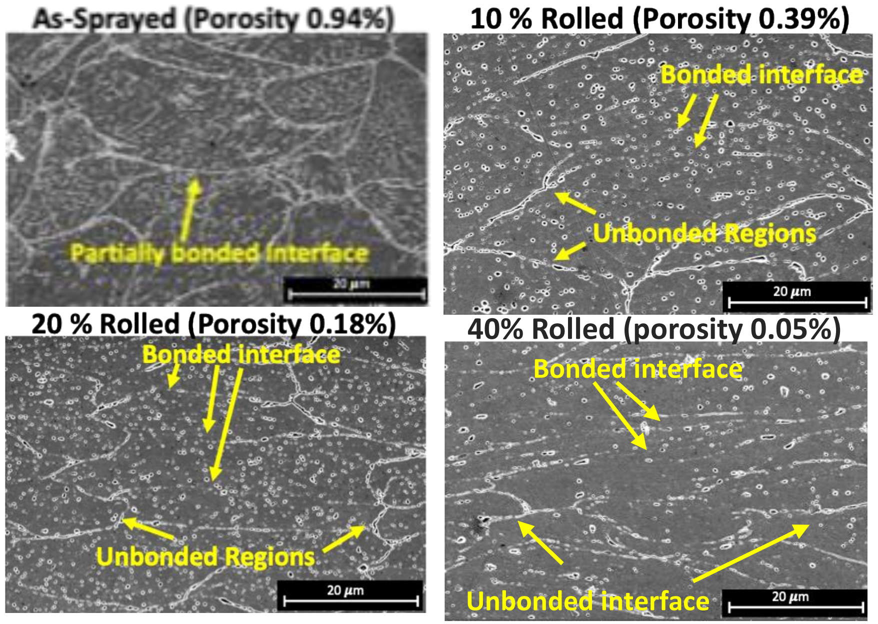
Post-spray thermomechanical treatment (warm rolling)

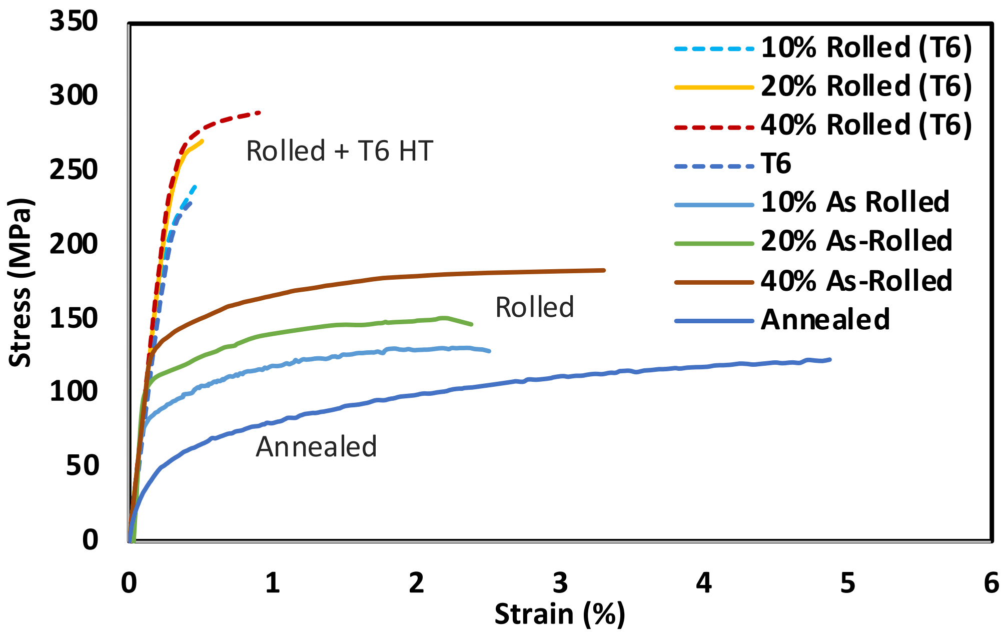
Treatment	Conditions
Hot Rolling	500°C (2 hr) + Rolling 10% (one pass) / 20% (2 passes)

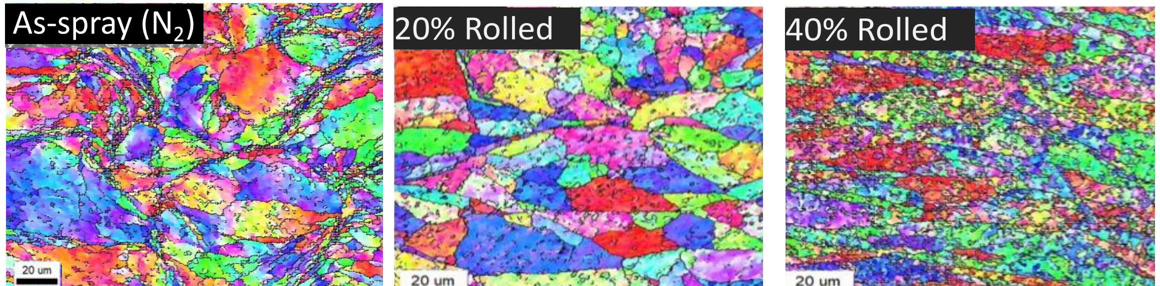
Followed by **HT**: i) none; ii) T-6; iii) Annealing

Treatment	Conditions
Solution and aging (T6)	530 °C (2 hr) / 180 °C (12 hr)
Annealing	413 °C (2 hr)









Thermo-mechanical treatment improves ductility & strength

- T6-treatment: Improves strength but low ductility
- Annealing: Improves ductility low strength
- Hot-roll+anneal: Improves both (in the middle)

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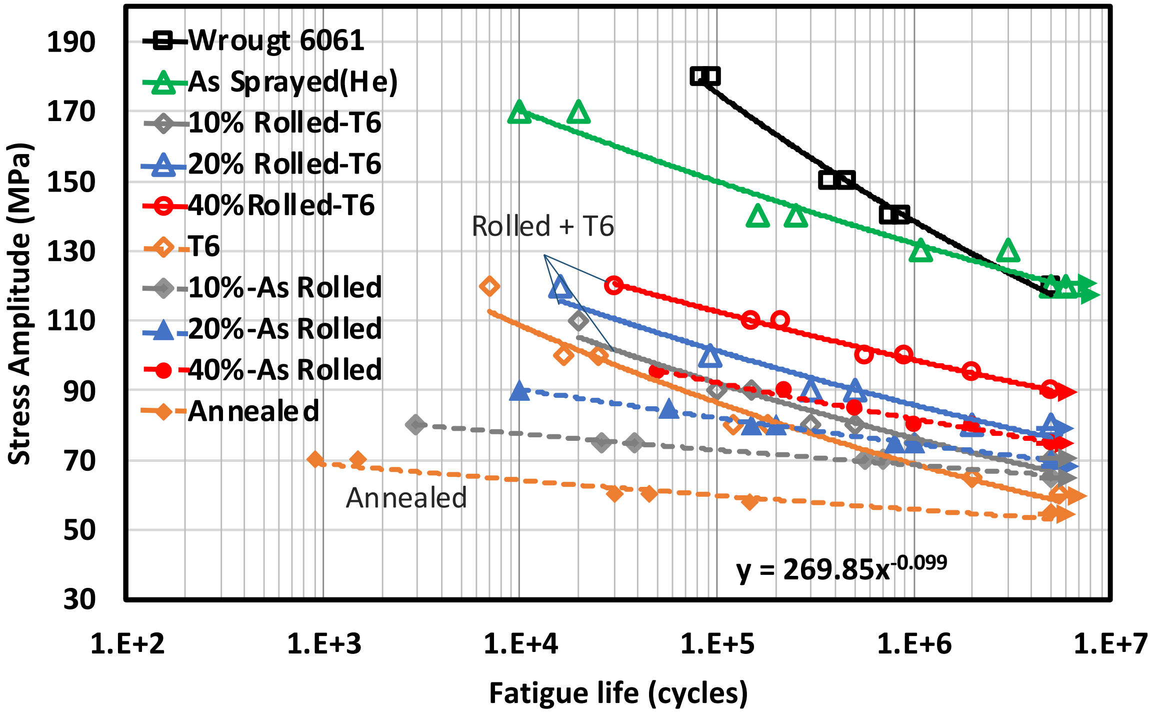
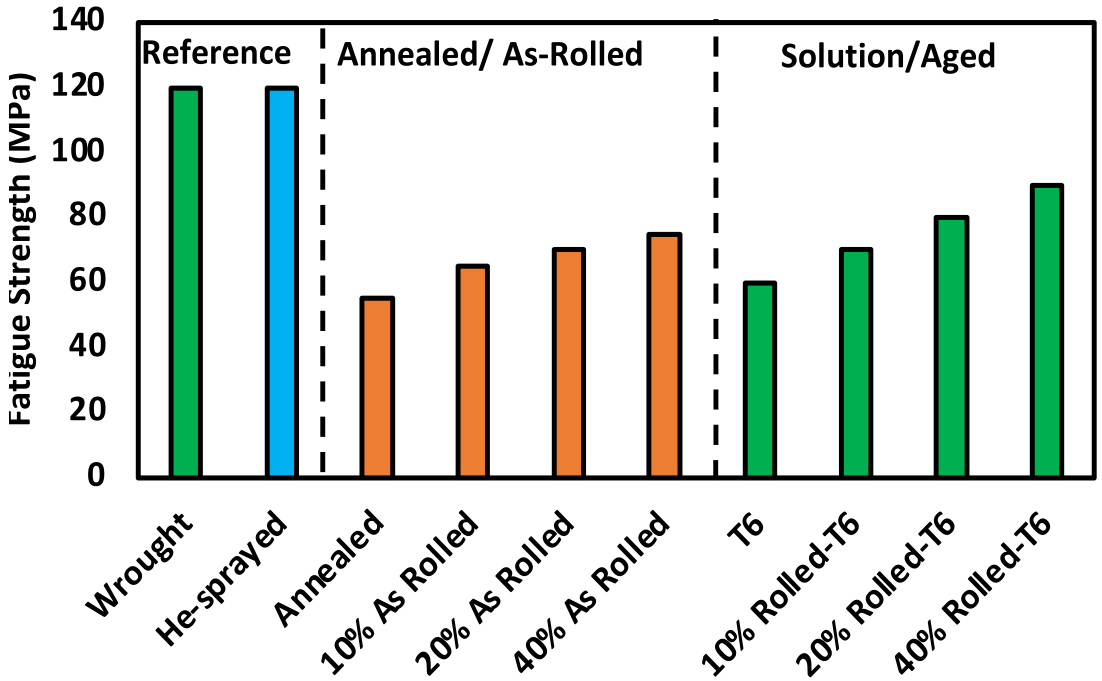
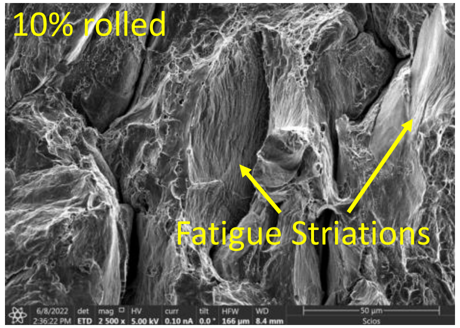
Post-spray thermomechanical treatment (warm rolling)

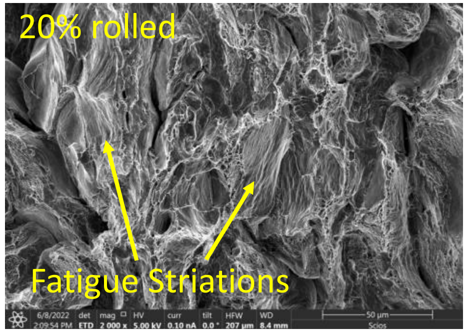




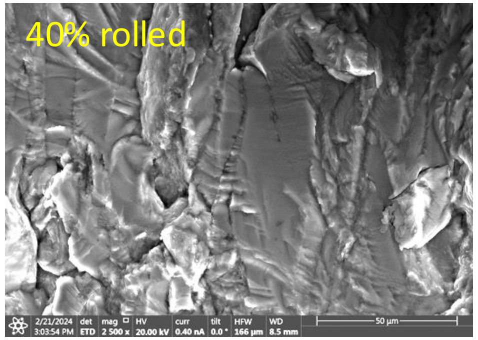
Inter-particular crack growth

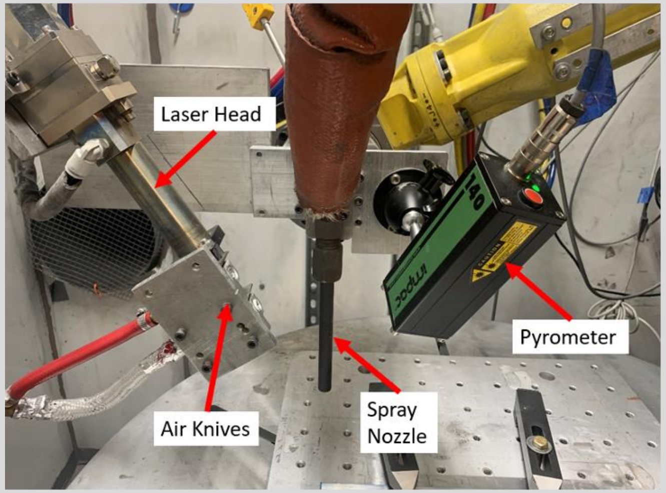


inter- and trans-particular crack growth



trans-particular crack growth

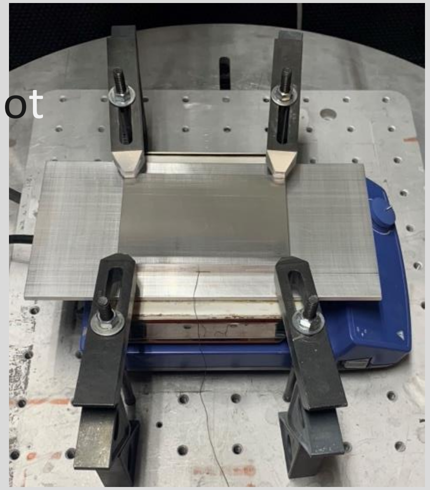




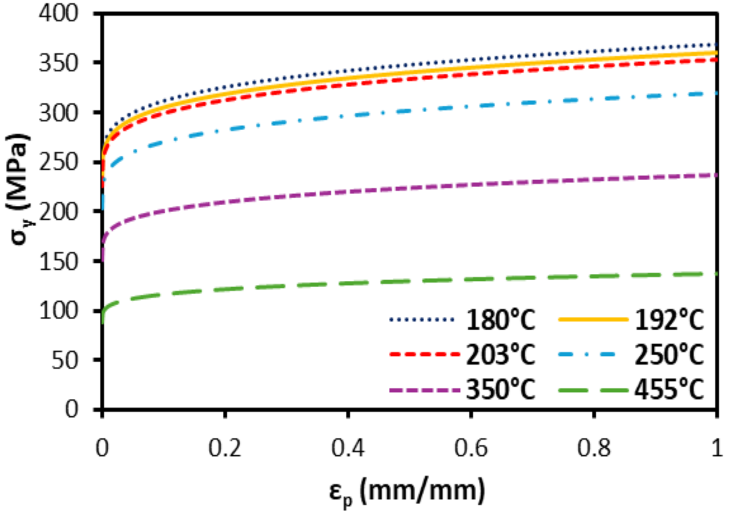
LACS setup

Apply a 1 cm dia., 6 kW laser* overlapping the CS deposition spot
Underlying material is softened to reduce the critical velocity
Deposition efficiency and deposit quality enhanced
Thermal stresses are induced in the substrate and deposit
Hot plate used to prevent delaminations

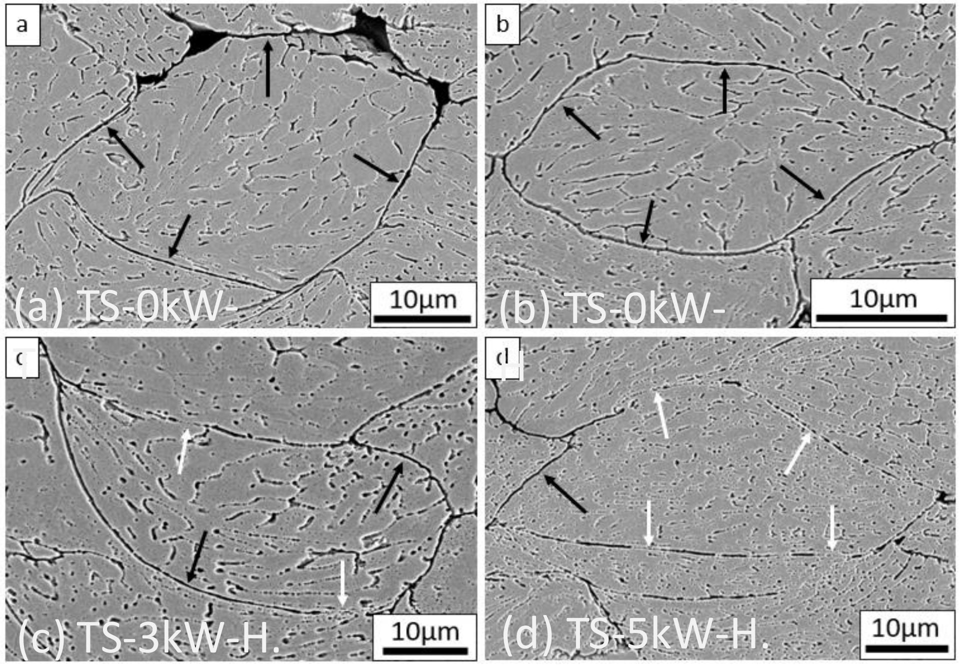
*IPG Photonics, YLS-6000-CUT-Y17 laser system



Effect of temperature on flow stress
Johnson-Cook model



Laser power kW	T^*_{surf}, °C
0	180
1	250
3	350
5	455
*Pyrometer measured, PID controlled T_{surf}	



S. Boese, A. Sevinsky, A. Nourian, O. Özdemir, S. Müftü, 2024, *in review, Additive Manufacturing.*

Laser-Assisted Cold Spray (LACS)

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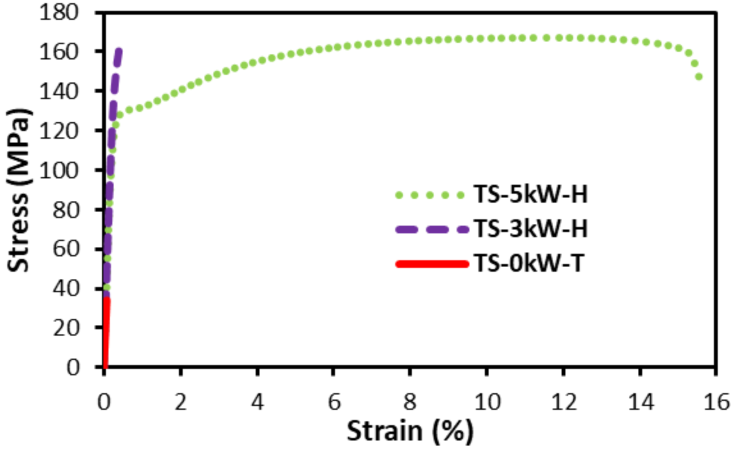

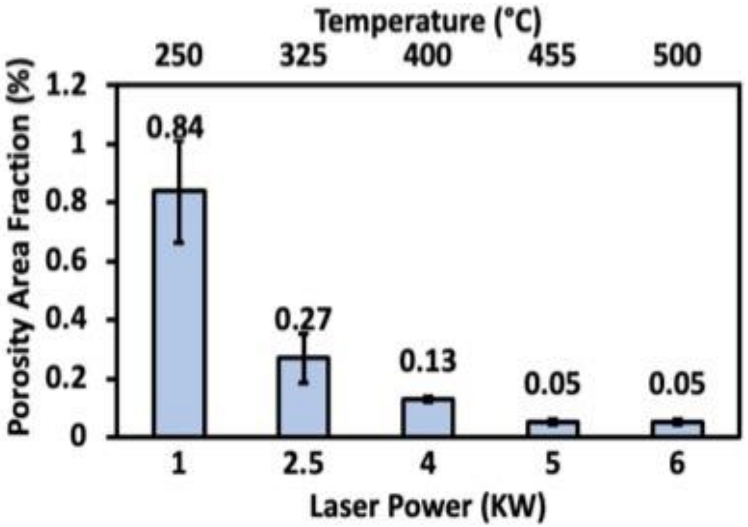

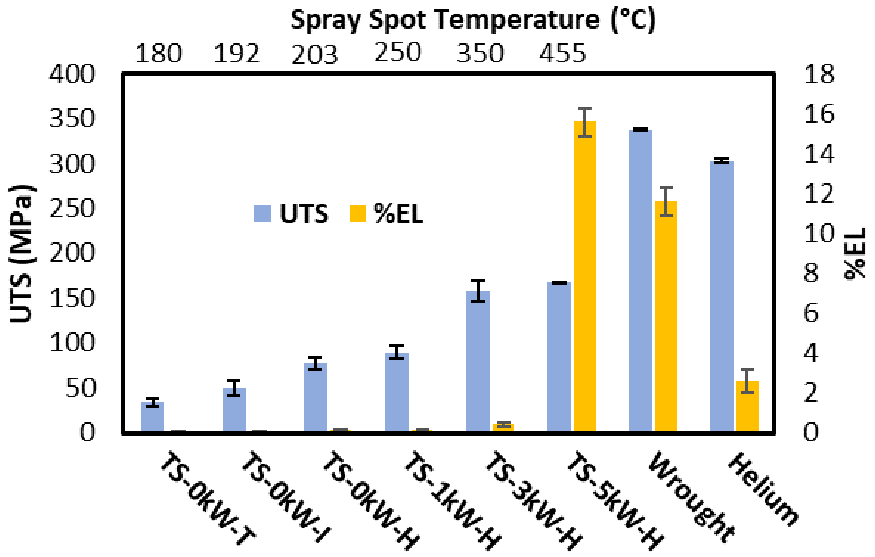

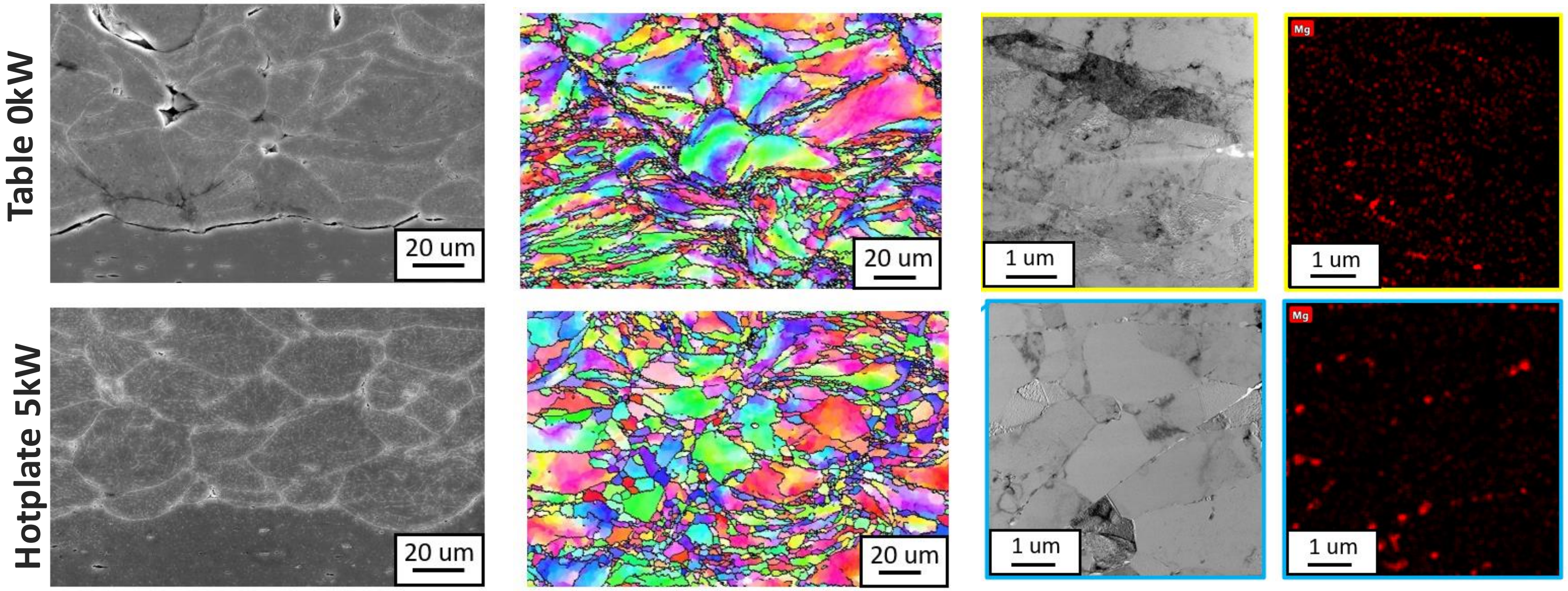

- LACS results in decreased porosity, and improved bonding
- LACS results in recrystallization, reduced dislocation density, and precipitation growth
- All combined contribute to ductility and strengthening of the material

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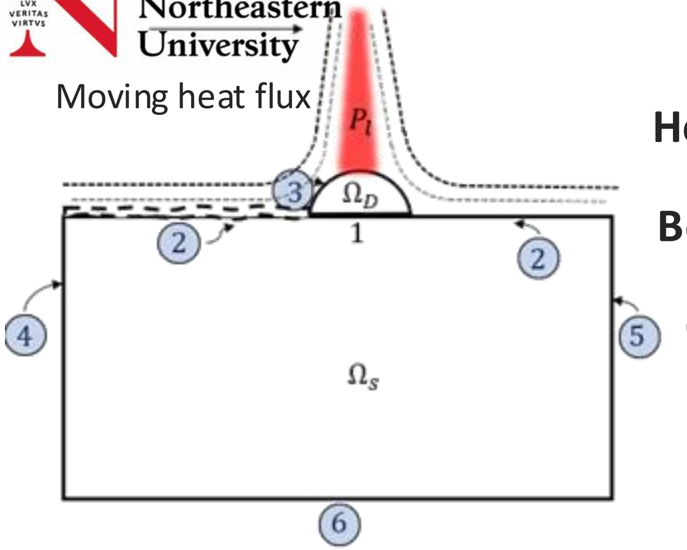
Powder deposition model (for LACS)

Moving heat flux



Heat conduction equation $\nabla \cdot \left(k_i \nabla T \right) = \rho_i c_i \dot{T}$

Boundary Conditions

4 5 6

$$k\frac{\partial T}{\partial n} + h_c(T - T_0) + \sigma\varepsilon(T^4 - T_0^4) - \dot{q} = 0$$

($\dot{q}$ → 0)

2

$$k\frac{\partial T}{\partial n} + h_c(T - T_0) + \sigma\varepsilon(T^4 - T_0^4) - \dot{q} = 0$$

($h_c(T - T_0)$ → 0, $\sigma\varepsilon(T^4 - T_0^4)$ → 0)

$$\dot{q} = h_g(T_g - T) + \frac{\beta P_l}{\pi r^2}$$

$$T_g = 0.0453r^2 - 8.8625r + 836.49$$

$$Nu = -109.4ln(r) + 494.02$$

Cooling

$$k\frac{\partial T}{\partial n} + h_c(T - T_0) + \sigma\varepsilon(T^4 - T_0^4) - \dot{q} = 0$$

($\dot{q}$ → 0)

- Moving heat flux due to sprayed gas
- Particles gain heat in the hot gas.
- Elements are activated at particle temp.
- Heat input due to plastic dissipation
- Convection and radiation on the boundaries

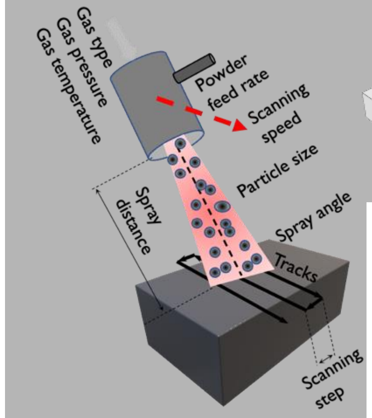
Equation of equilibrium

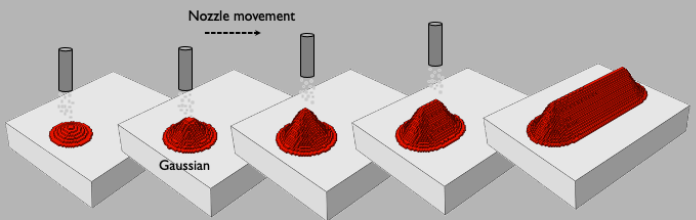
$$\vec{\nabla} \cdot \boldsymbol{\sigma} + \rho_i \boldsymbol{b} - \frac{\alpha_i E_i}{1 - 2\nu_i} \nabla T = \rho_i \ddot{\mathbf{u}}$$

- Material (E, mu, rho, alpha) properties are temperature dependent
- Plastic deformation is included in analysis
- Flow stress is computed by using Johnson-Cook model
- Thermal problem is solved transient domain
- Solid mechanics problem is solved quasistatically

Mesh: min ~80µm
Heat transfer: DC3D8,
Stress: C3D8R

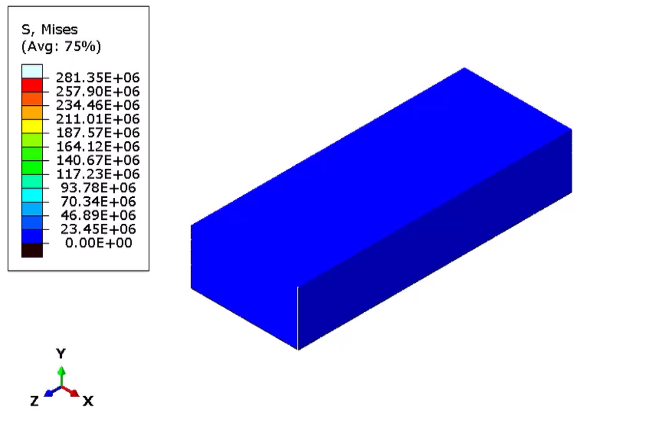
Powder deposition model



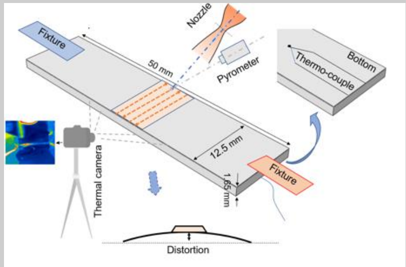


Process parameters

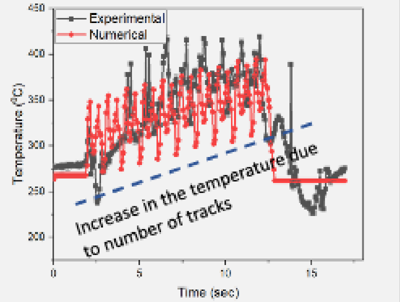
Gas: gas type; pressure; temperature.
Nozzle: geometry; distance; angle
Robot: speed; step distance
Powder: material; size distribution; feed rate



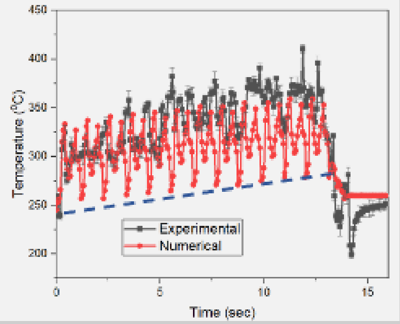
Temperature verification



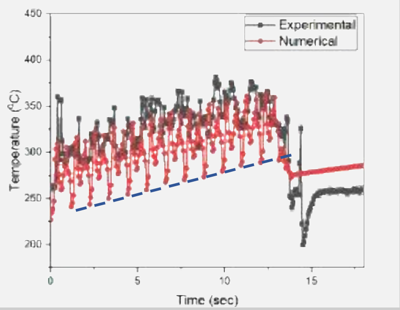
Step=0.8mm, velocity =50 mm/sec



Step=1mm, velocity =50 mm/sec

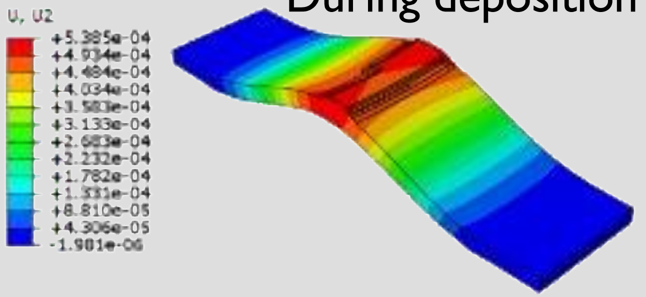


Step=1mm, velocity =100 mm/sec

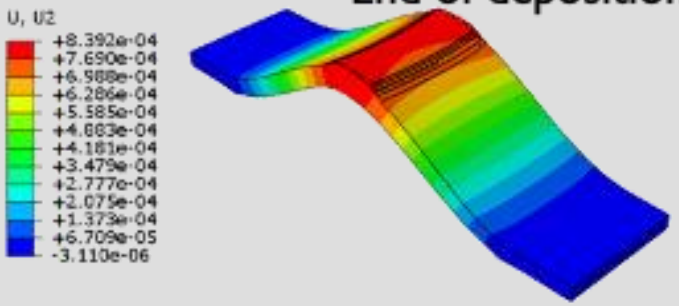


Distortion

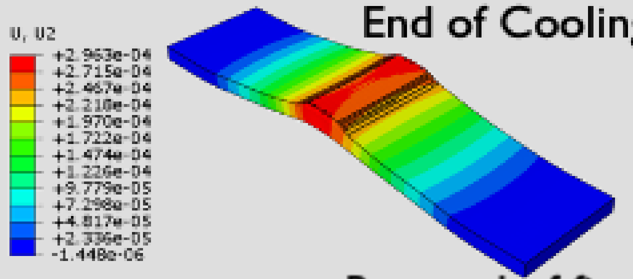
During deposition



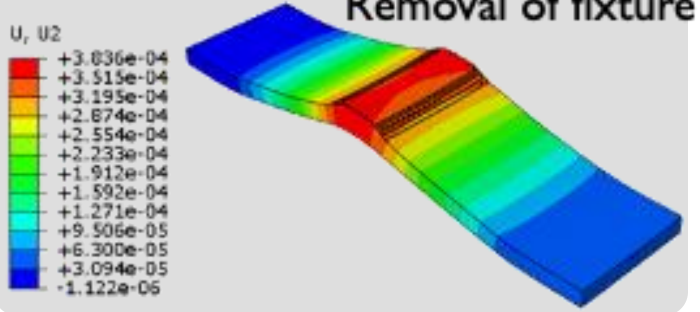
End of deposition



End of Cooling



Removal of fixture

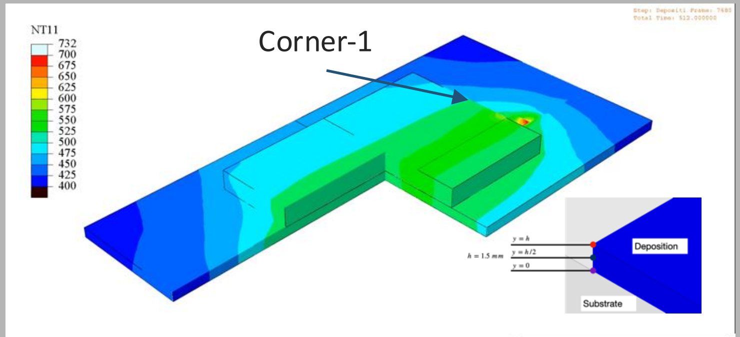


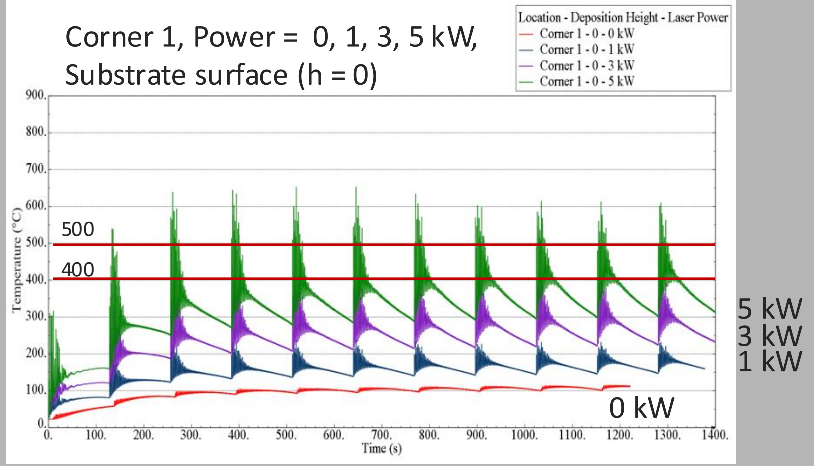
Simulation of the LACS process

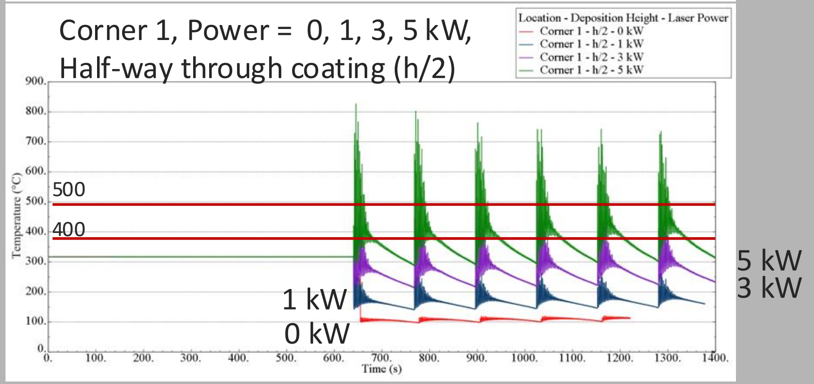
Northeastern University

https://coldspray.coe.neu.edu

LACS temperature simulations

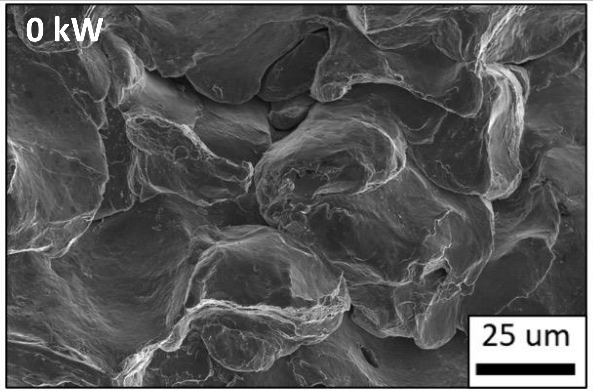




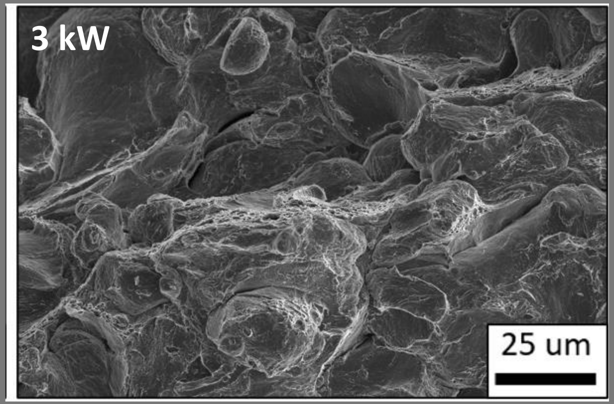


Heat treatment temperatures for Al-6061

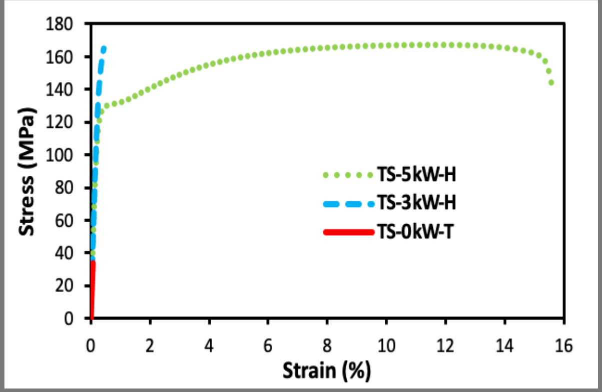
- Sintering: T_S = 585 - 652 °C (sol - liq)
- Solutionizing: T_{SH} = 516 - 579 °C
- Annealing: T_{Ann} = 415 °C (typical)
- Aging: T_{Age} = 171-183 °C

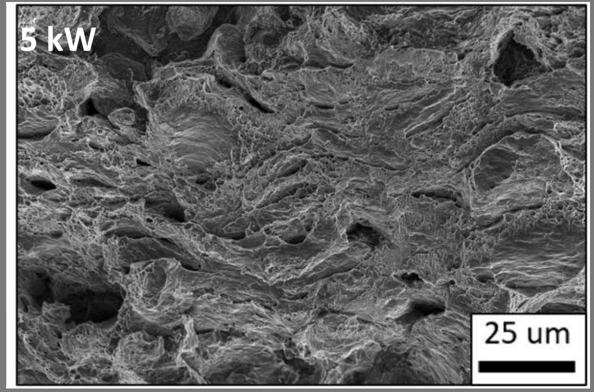


Inter-splat fracture



Inter-splat fracture





Trans-splat fracture

Simulations: temps reach into 600 - 700 °C range and average around 450 °C for the 5 kW laser power.

Experiments: show evidence of annealing and partial sintering

Summary (1/2)

https://coldspray.coe.neu.edu

Alloy condition	Fatigue str. (MPa)	YS (MPa)	UTS (MPa)	Elongation (%)	Porosity (%)
Wrought material					
Wrought 6061-T6	120	289	318	13.1	
Wrought 6061-Ann	83	57	126	22.3	
Cast aluminum					
Cast 319-T6	75				
Cast-356-T6	85				
As-sprayed condition					
As-sprayed (He)	120	259	282	1.6	0.05
As-sprayed (N2)	N/A	N/A	49	0.1	0.87
Conventional heat treatment					
CHT-SA	60	-	223	0.48	0.75
CHT-H-SA	75	261	269	0.9	0.6
CHT-Ann	55	56	120	4.9	
CHT-H-Ann	60	57	122	7.4	
Hot isostatic pressing					
HIP-SA	70	-	235	0.5	0.4
HIP-H-SA	90	257	268	1.3	0.28
HIP-Ann	60	57	123	5.1	
HIP-H-Ann	65	57	123	9.4	
Hot rolling					
10% as-rolled	65	95	129	2.46	
20% as rolled	70	114	146	2.38	
40% as rolled	75	145	183	3.3	
Solution aged					
T6	60	-	223	0.48	
10% T6	70	-	241	0.49	
20% T6	80	264	271	0.53	
40% T6	90	280	292	0.92	
Laser assisted cold spray					
0 kW (203 deg C)	-	-	77	0.15	
1 kW (250 deg C)	-	-	90	0.14	0.84
3 kW (350 deg C)	60	-	158	0.4	0.2
5 kW (455 deg C)	68	128	167	15.2	0.05

HT-annealing/sintering + SA helps close pores with high static and fatigue props

Annealing helps ductility

HIP: HT-annealing/sintering + SA helps close pores with high static and fatigue pro

HIP: Annealing helps ductility

Increasing compression in rolling helps reduce porosity and improve fatigue str.

Applying SA (T6) treatment to rolled material improves fatigue to cast-Al level

LACS can provide properties close to Al-6061 (T0) in as sprayed condition.

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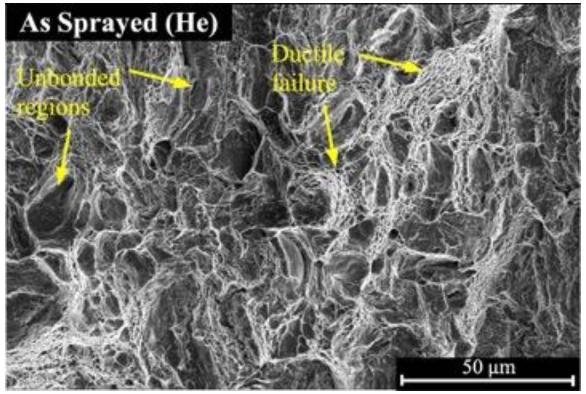
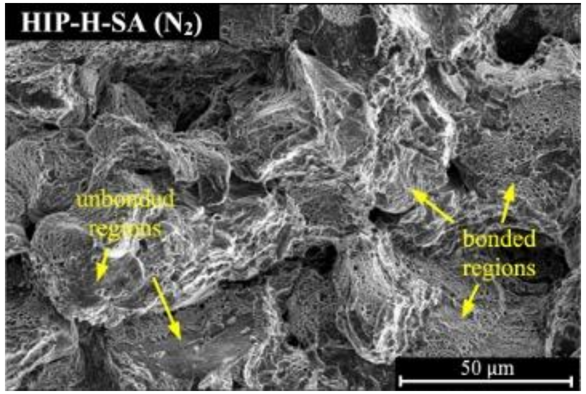
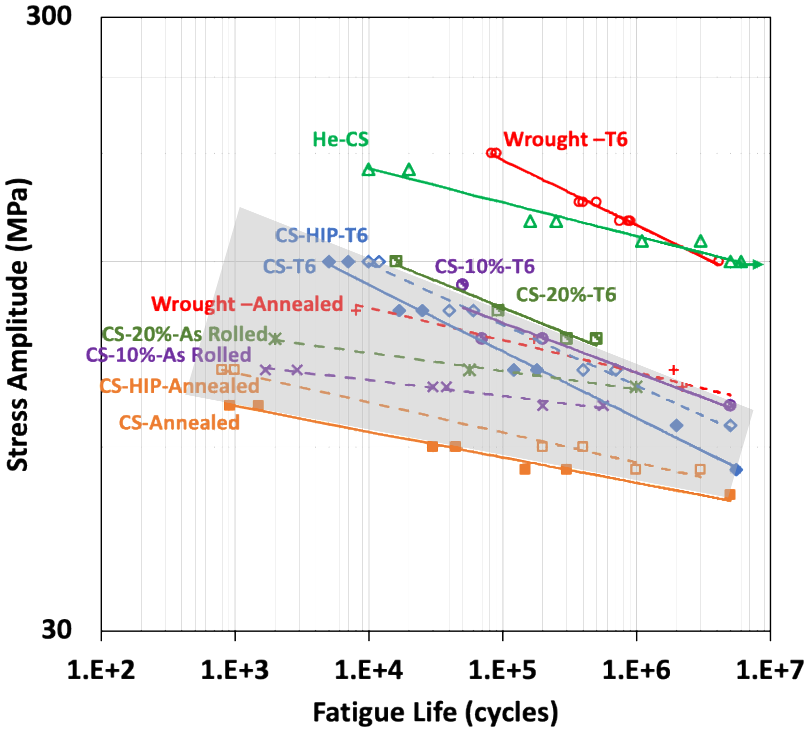
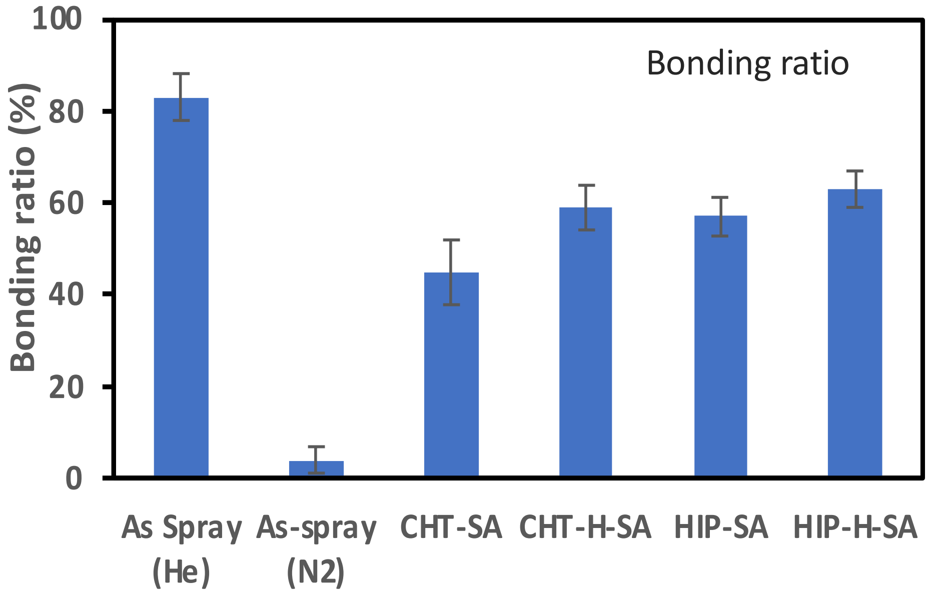
Summary and conclusions (2/2)









Summary and conclusions

- N2-sprayed deposits become load-bearing by applying post-spray or in-situ heat treatments.
- A combination of **reduction in porosity due to sintering** and metallurgical changes contribute to this enhancement
- The **tensile strength** of N_2-sprayed 6061 deposits are enhanced up to 80% of wrought 6061
- **The long-life fatigue strength** of CS 6061 was enhanced up to ~70% of wrought material.
- Failure mechanism moves from inter-particular to trans-particular with HT.
- Large-scale **porosity** and smaller-scale **particle bonding ratio** can be decreased with sintering and rolling **but there appears to be a limit in particle bonding ratio**

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Thank you