NATIONAL ENERGY TECHNOLOGY LABORATORY

University Turbine Systems Research

Project Title: Integrated Transpiration and Lattice Cooling Systems developed by Additive Manufacturing with Oxide-Dispersion-Strengthened Alloys

Project No.: FE0031277, 10/1/2017 -9/30/2022 (w no cost extension)

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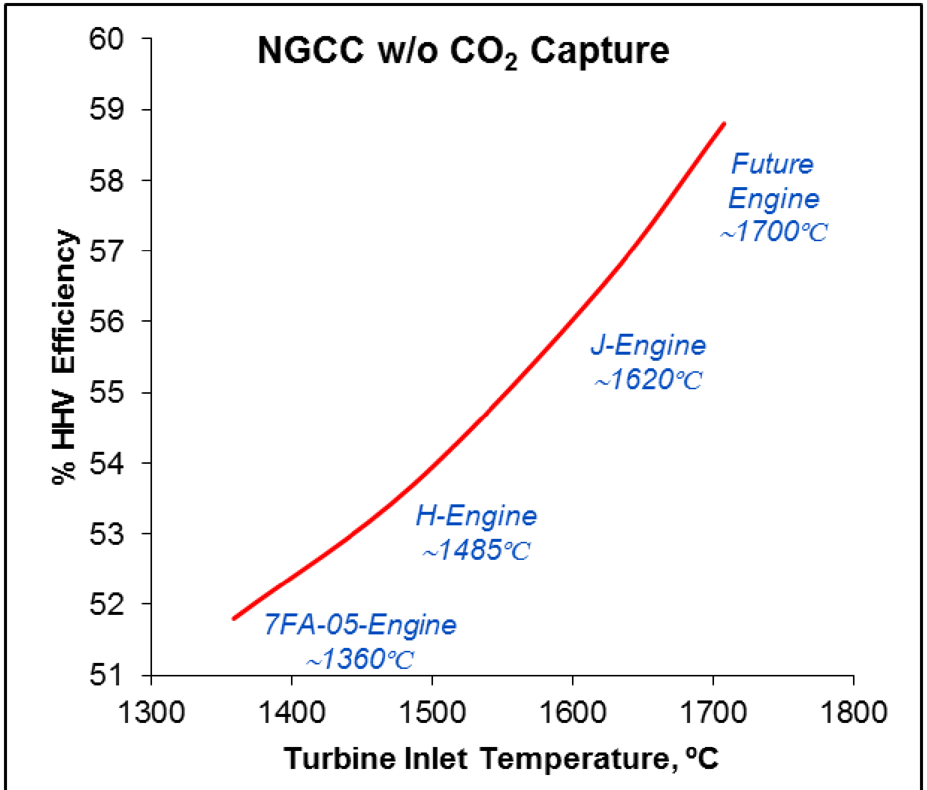
DEPARTMENT OF ENERGY • UNITED STATES OF AMERICA

Outlines

- ***Background, Challenges, Objectives, Benefits of Technology, Research Task Plans***
- *Additive Manufacturing Processes*
- *Heat Transfer Results: Transpiration Cooling*
- *Heat Transfer Results: Lattice Cooling*
- *Heat Transfer Results: Integrated Cooling*
- *ODS Powder Development*

Need for Turbine Cooling

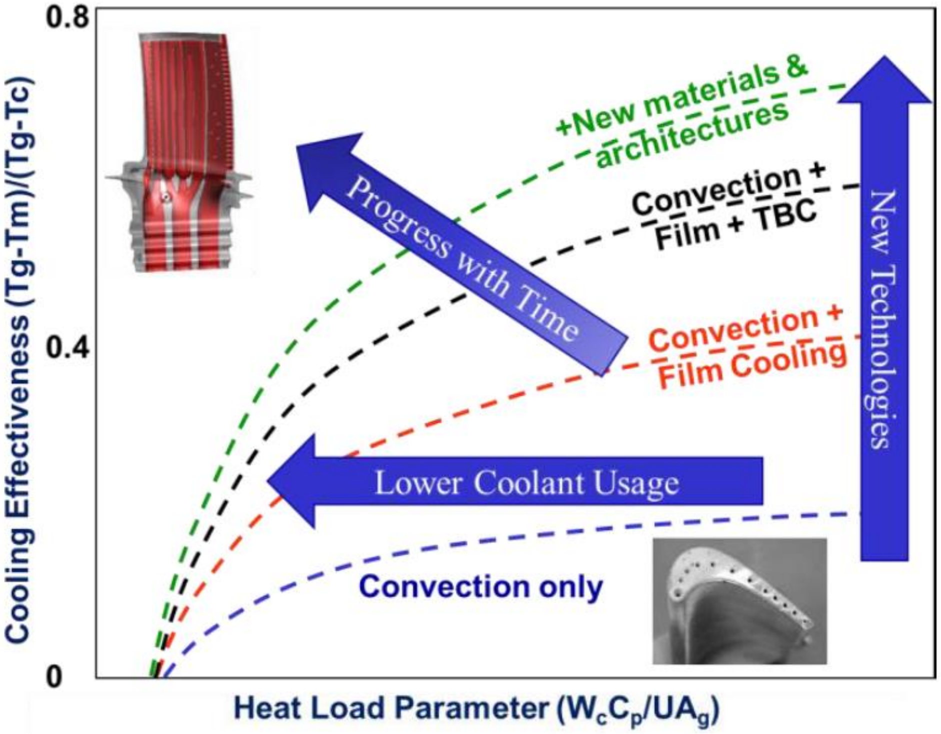


– W.Shelton, NETL Communication December 2011 –

Airfoil metal temperature distributions (in K)
h_c=3000W/m²-K

- ***Gas temperature: Hydrogen-fired turbine (~1430°C)***

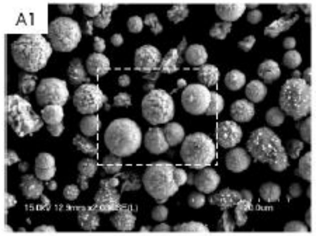
Near surface 'skin cooling' or 'double-wall' internal cooling **arrangement leads to a significant reduction of metal surface temperature, ~50 – 100°C, compared to conventional serpentine cooling designs**



Bunker RS. Evolution of Turbine Cooling. ASME. Turbo Expo: Power for Land, Sea, and Air, Volume 1: Aircraft Engine; Fans and Blowers; Marine; Honors and Awards ():V001T51A001. doi:10.1115/GT2017-63205.

One key will be the marriage of design and manufacturing to bring about the concurrent use of engineered micro cooling or transpiration, with the ability of additive manufacturing. If successful, this combination could see a further 50% reduction in coolant usage for turbines.

Proposed Technologies

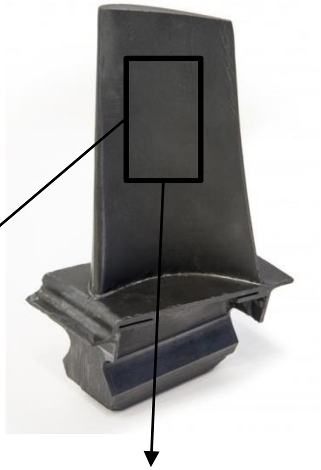


ODS

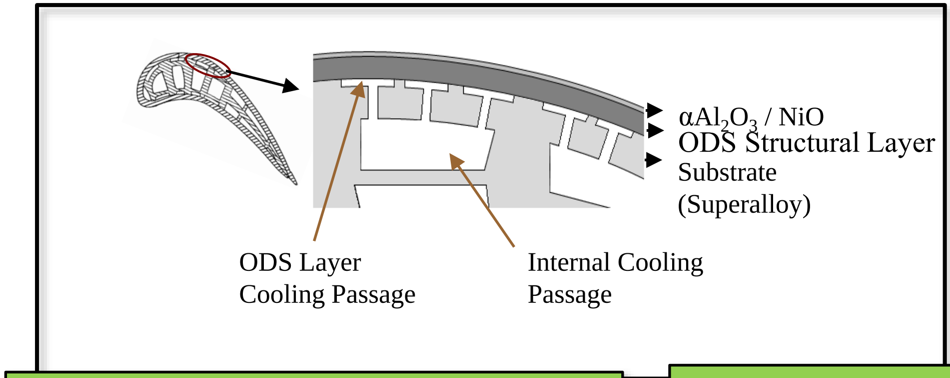
Enhanced oxidation resistance and high temperature strength

Advanced Additive Manufacturing

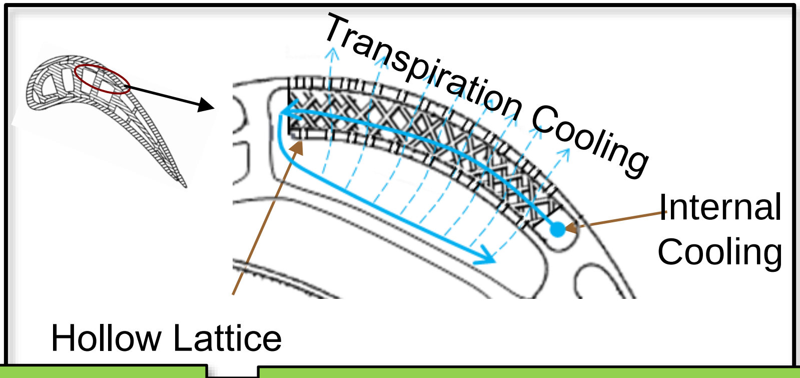
Intricate Heat Transfer Enhancement Features



Near Surface Embedded Cooling Channel (NSECC)



Integrated Transpiration and Lattice Cooling Systems



ODS with AM

- *Both internal and external protection.*
- *Oxidation Resistance*
- *Realized with additive manufacturing*

Novel ODS Surface Coating

- *Ultra-High Temperature (1200℃) Strength: thinner outer wall for better cooling*
- *Oxidation Resistance*
- *Higher turbine inlet temperatures*

Enhanced Heat Removal

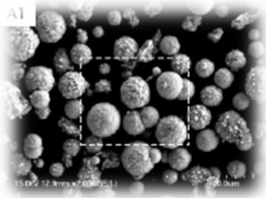
- *NSECC: high internal heat transfer and higher coolant flow near hot end wall.*
- *Lattice and transpiration combined conjugate cooling: ~2x of state-of-the-art film cooling.*

Project Work Breakdown Structure

Goal: Enhanced Oxidation Resistance and Thermal Protection of Turbine Blades

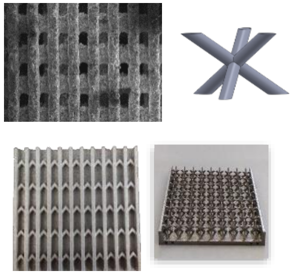
ODS Powder Fabrication and Characterization

- *MCB/BM for powders fabrication*
- *Characterization*



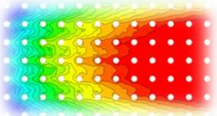
Cooling Geometry Design

- *Transpiration and Lattice Geometry development and optimization*



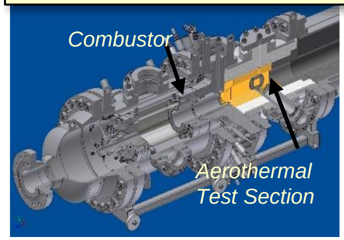
Lab-Scale Heat Transfer Characterization

- *CFD modeling & steady state and scaled TLC testing*
- *Mini/micro scale cellular units*



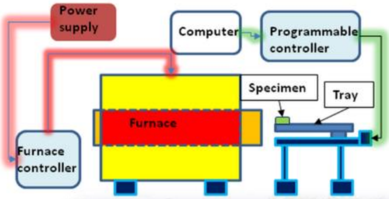
High Temperature Heat Transfer Characterization

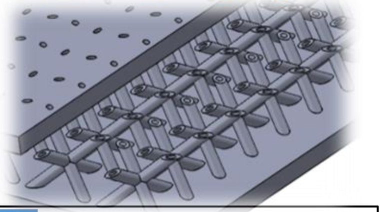
High Temperature, Pressurized Testing (NETL)

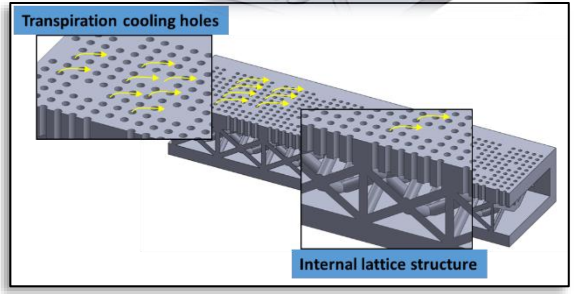


Thermal Cyclic Testing

- *In-situ non-destructive micro indentation facility*
- *Thermal Cyclic Tests, Micro-hardness*







Process Optimization to Fabricate ODS Transpiration and Lattice Structures

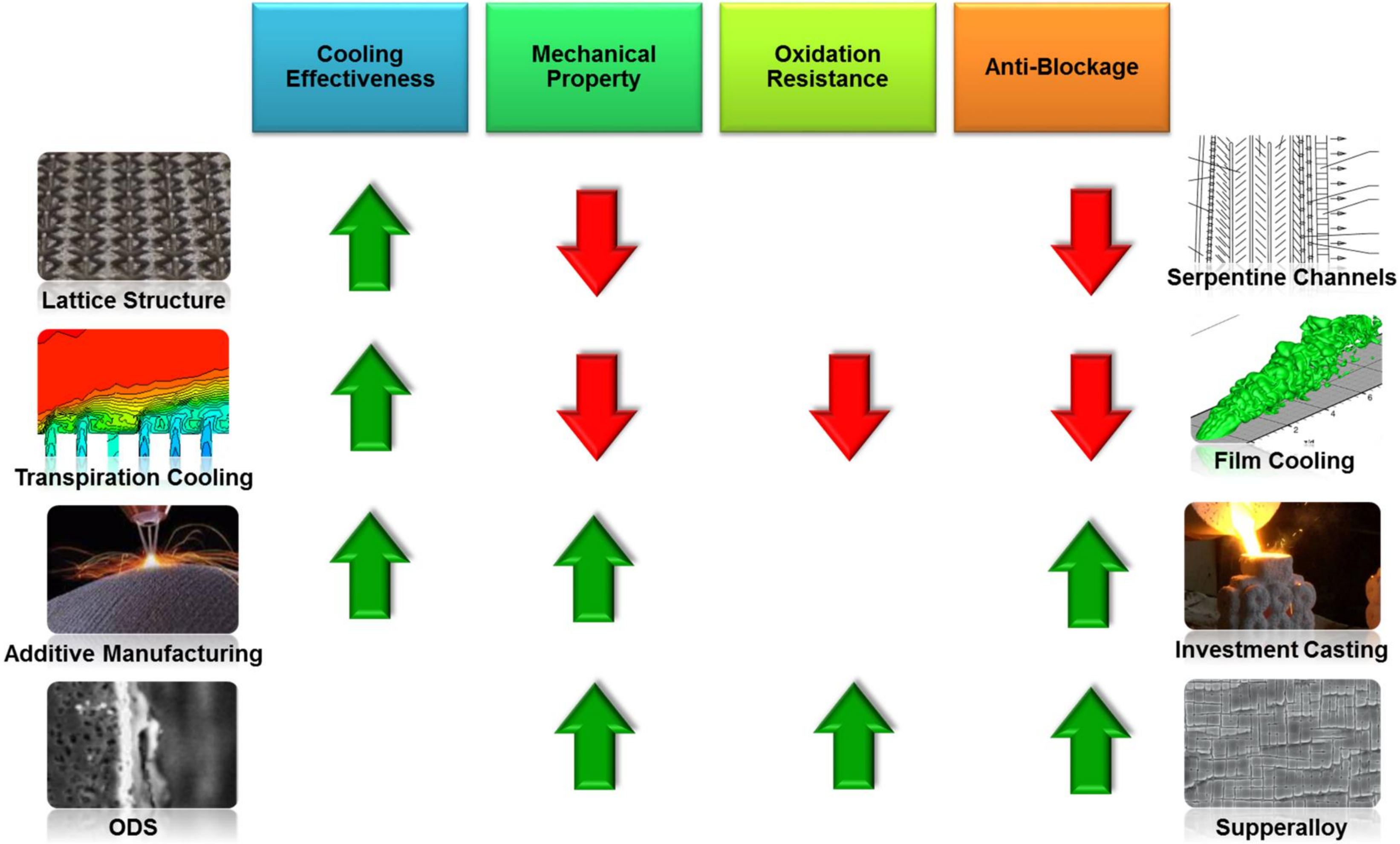
- *DMLS, BJT printing parameters*
- *Posting printing characterization*
- *OM, SEM*

Milestones

- All completed milestones achieved on schedule
- Milestones E moved to 2022 due to delays caused by COVID-19 shutdowns

Milestone Title	Planned Date	Verification Method
A - Identify prototypes for integrated transpiration and internal cooling	6/30/2018	Prototypes geometries will be generated through the original optimization algorithm, and CFD simulation should be conducted for each geometry.
B - Identify optimal configurations for integrated transpiration and internal cooling	9/30/2019	Systematic experimental tests should be conducted to proof the cooling effectiveness of the optimal geometry.
C - Integrate new unit types into the optimization algorithm for ODS lattice structure	12/30/2019	Mechanical and heat transfer property of new lattice unit type should be obtained and input into the optimization algorithm. Several optimization cases should be conducted to proof the reliability of the algorithm.
D - Identify the capability of AM equipment to print ODS Structure	9/30/2018	The capability of printing ODS structures should be identified for the AM equipment at Pitt. Decision should be made which AM equipment is the most suitable to fabricate ODS lattice structure.
E - Develop successful approach to make ODS Structure for integrated transpiration and internal cooling	5/31/2022	Complete the fabrication of ODS structures with complex lattice geometry. Microstructure inspection should be conducted using OM and SEM.
(Descoped) F - Complete high temperature experiments for integrated cooling structures made from ODS	9/30/2021	Heat transfer analysis will be conducted for the data obtained under high temperature. SEM characterization should also be included to exam the microstructure of ODS after high temperature operation.
G - Develop successful approach to produce ODS powder suitable for additive manufacturing and lattice structures	9/30/2019	SEM and TEM characterization should be conducted to identify the sphericity, microstructure and size distribution of ODS powder.
H - Complete thermal cyclic loading tests	5/31/2021	Optical micrographs, SEM and nondestructive micro-indentation tests should be conducted to proof the long term stability of ODS material.

Benefits of Technology
to the DOE Turbine Program



	Cooling Effectiveness	Mechanical Property	Oxidation Resistance	Anti-Blockage	
Lattice Structure	↑	↓		↓	Serpentine Channels
Transpiration Cooling	↑	↓	↓	↓	Film Cooling
Additive Manufacturing	↑	↑		↑	Investment Casting
ODS		↑	↑	↑	Supperalloy

☐ **Technical Advancement** ☐ **Turbine industry** ☐ **Knowledge Base** ☐ **Additive manufacturing promotion**

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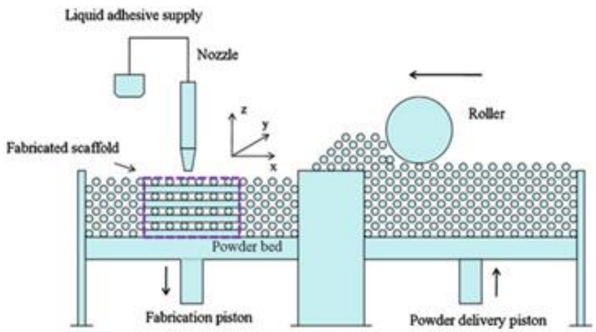
AM Processes

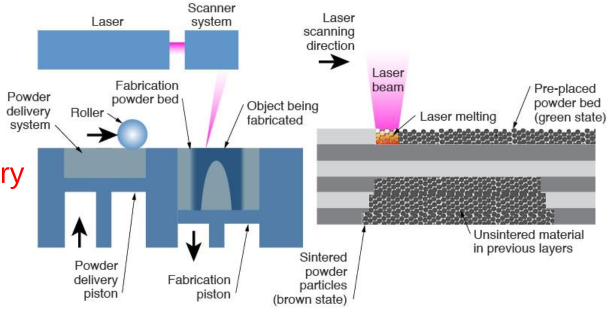
Bind Jetting (EXOne)

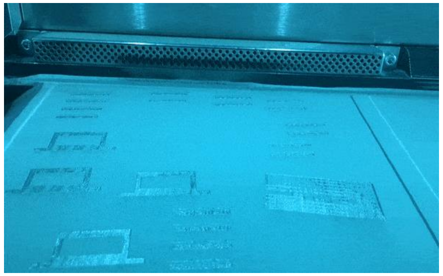
Pros: geometry free
cons: high porosity



Powder Bed Fusion (EOS)

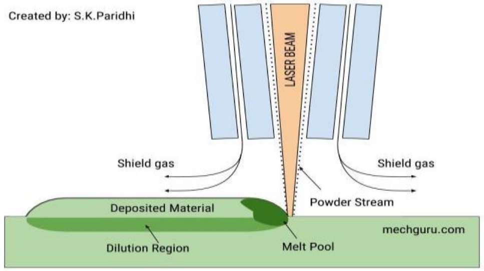
Pros: complex geometry
cons: single material

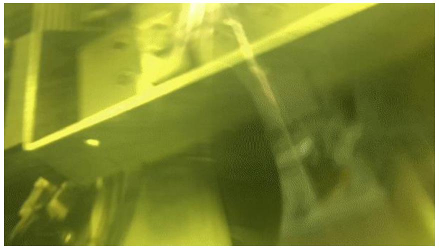




Direct Energy Deposition (LENS)

Pros: multiple materials
cons: no overhangs or unsupported structure

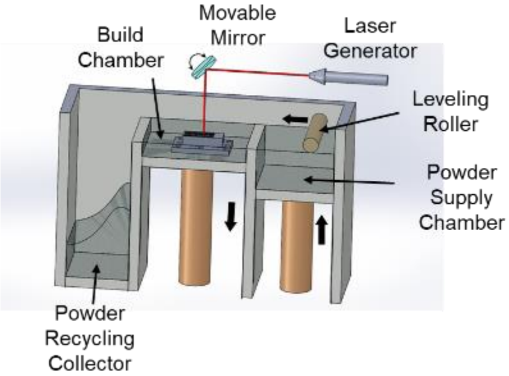




As metallic additive manufacturing technologies advance significantly over the recent past, complex metal products, such as turbine components, can be manufactured by this innovative technology.

ODS Coupons using DMLS

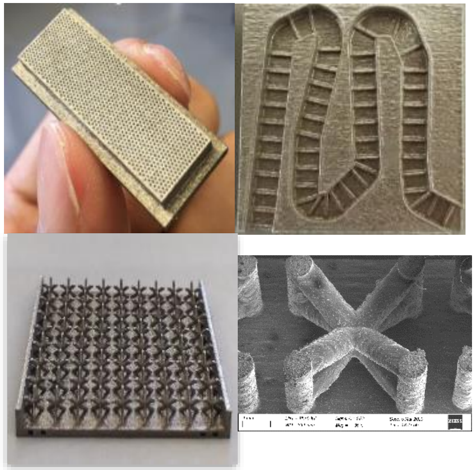
- ➢ Powder bed fusion process using laser
- ➢ Well developed process parameters for Inconel superalloys
- ➢ Challenges regarding ODS powder process development to be mitigated by systematic study

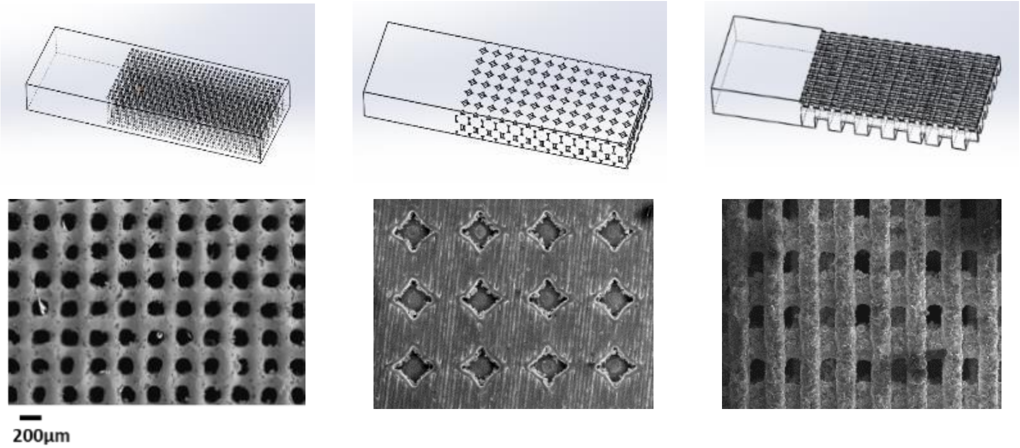


EOS M290

- **Laser Power**: 400W
- **Laser Focal Diameter**: 100μm
- **Scan Speed:** up to 7m/s
- **Printing Material:** Inconel 718 （similar composition to ODS)

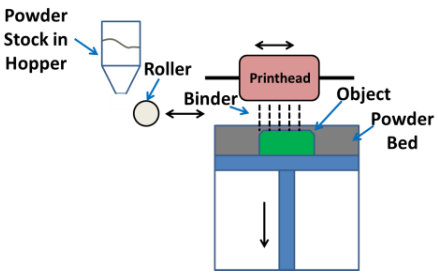
- ➢ Fabricated coupons (In718)





ODS Coupons using Binder Jetting

- Powder bed fusion process using binder
- Close flowability and particle size to IN718
- Printing parameters:
  - Hopper frequency: ~2100 Hz
  - Coating layer speed: ~9 mm/s
  - Layer thickness: 100-120 µm
  - Layer curing time



Process overview

These steps are repeated until full part is printed

- **Layer Deposition**
  - Powder from hopper deposited with desired layer thickness
- **Binder Jetting**
  - Printhead sprays fine jets of binder to join powder
- **Layer Curing**
  - Layer is heated (~100 C) to cure binder
- **Presintering**
  - 8 hrs @ 200 C to remove binder
- **Full sintering**
  - High temperature sintering for full densification

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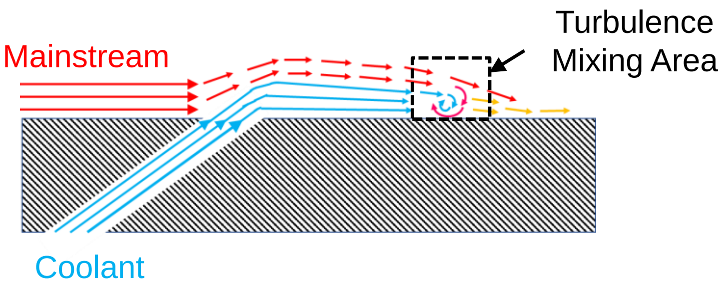
Transpiration Cooling – Introduction

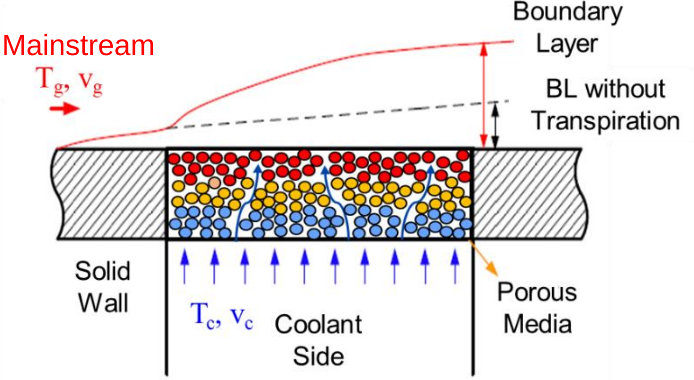
- ➢ Concept: The coolant was forced through a porous wall or multiple micro-cooling channels to form an insulating layer of coolant film between the outer wall surface and hot stream.

Conventional Film Cooling

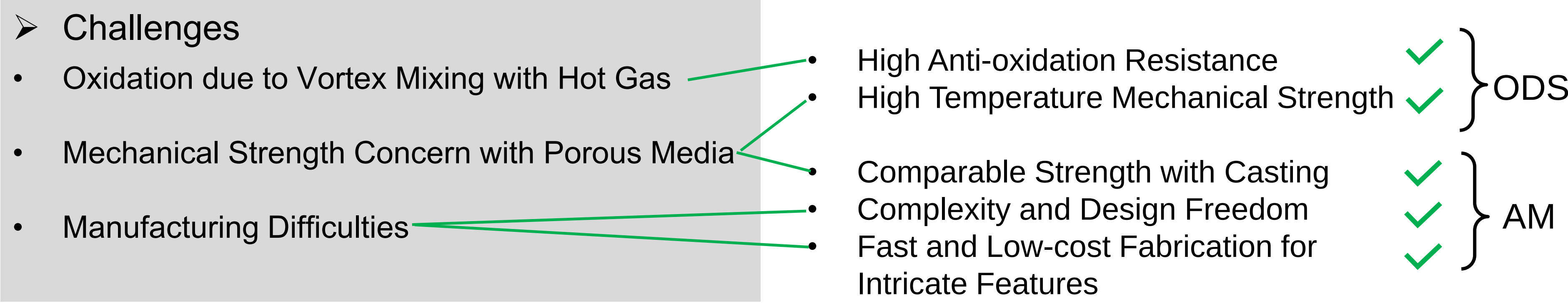


- "Lift-off" of the film due to jet momentum at the outlet
- Film protection weakened by turbulence mixing

Transpiration Cooling



- Uniform coolant film coverage
- Enhanced heat removal ability due to higher inner-wall heat transfer



- ➢ Challenges
- Oxidation due to Vortex Mixing with Hot Gas
- Mechanical Strength Concern with Porous Media
- Manufacturing Difficulties

- High Anti-oxidation Resistance ✓
- High Temperature Mechanical Strength ✓

} ODS

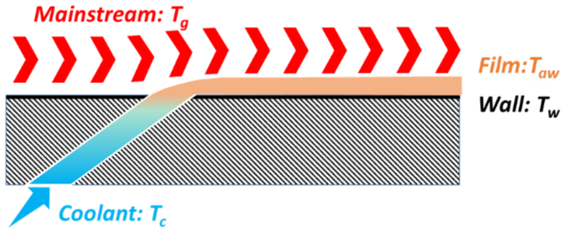
- Comparable Strength with Casting ✓
- Complexity and Design Freedom ✓
- Fast and Low-cost Fabrication for Intricate Features ✓

} AM

Thermo-fluid investigations

➢ Thermo-fluid investigation – film cooling

- Flat surface without coolant protection: $q_0 = h_0(T_{ref} - T_w), \quad T_{ref} = T_g$
- Film covered surface: $q = h_f(T_{ref} - T_w), \quad T_{ref} = T_{aw}$



$$Net\ heat\ flux\ reduction\ (NHFR) = 1 - \frac{q}{q_0} = 1 - \frac{h_f}{h_0}\frac{(T_{aw} - T_w)}{(T_g - T_w)} = 1 - \frac{h_f}{h_0}(1 - \eta/\varphi)$$

- Unknowns: $T_w, T_{aw}, \frac{h_f}{h_0}$
- In film cooling:

$\eta = \frac{T_g - T_{aw}}{T_g - T_c}$: Adiabatic cooling effectiveness

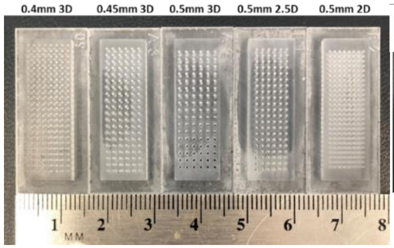
$\frac{h_f}{h_0}$: Heat transfer coefficient ratio

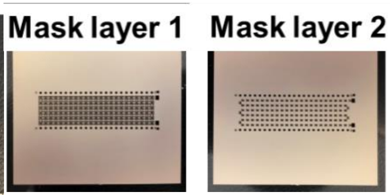
$\}$ ➡ Obtained from polymer coupons with low thermal conductivity

$\varphi = \frac{T_g - T_w}{T_g - T_c}$: Overall cooling effectiveness

Ranged from 0.5 – 0.7 in real engine conditions

Transpiration Cooling – Surface Heater

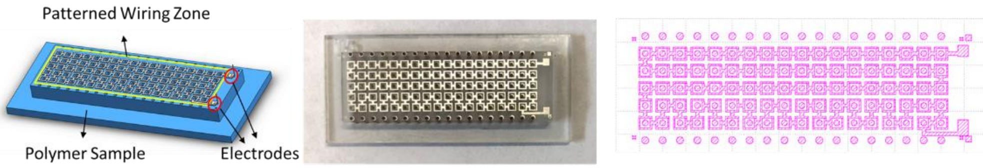


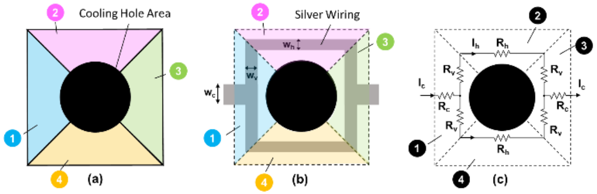


- *Photo Sciences, Inc.*

➢ Surface heater fabrication

1. SLA printed resin samples with low thermal conductivity
   - Reduction of conductive heat loss
2. Micro-lithography fabrication for surface heater
   - Direct deposition of silver coil onto the target surface
   - No blockage/plugging of the outlets



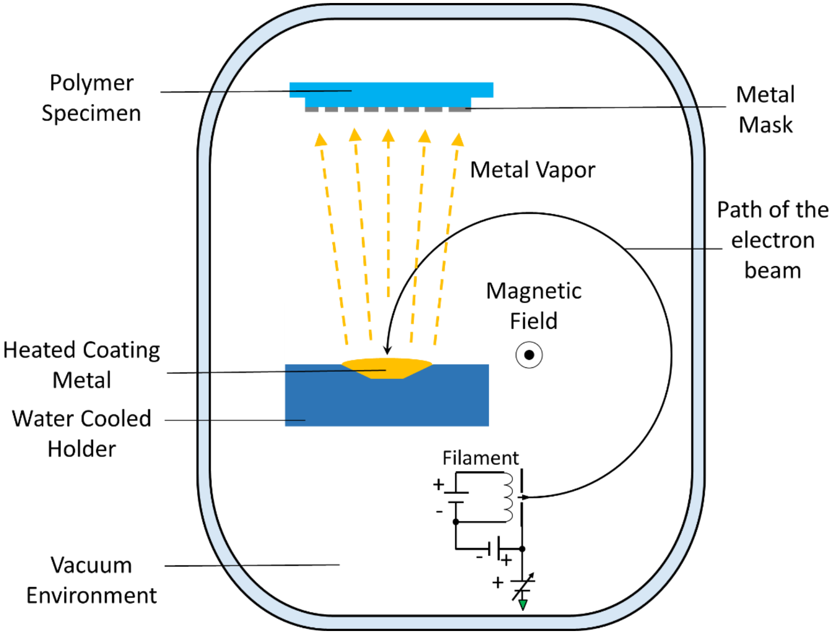


$$P_1 = I_c^2 R_c + 2I_h^2 R_v \quad = \quad P_2 = I_h^2 R_h$$

$$R_c = \rho \frac{L_c}{w_c t}$$

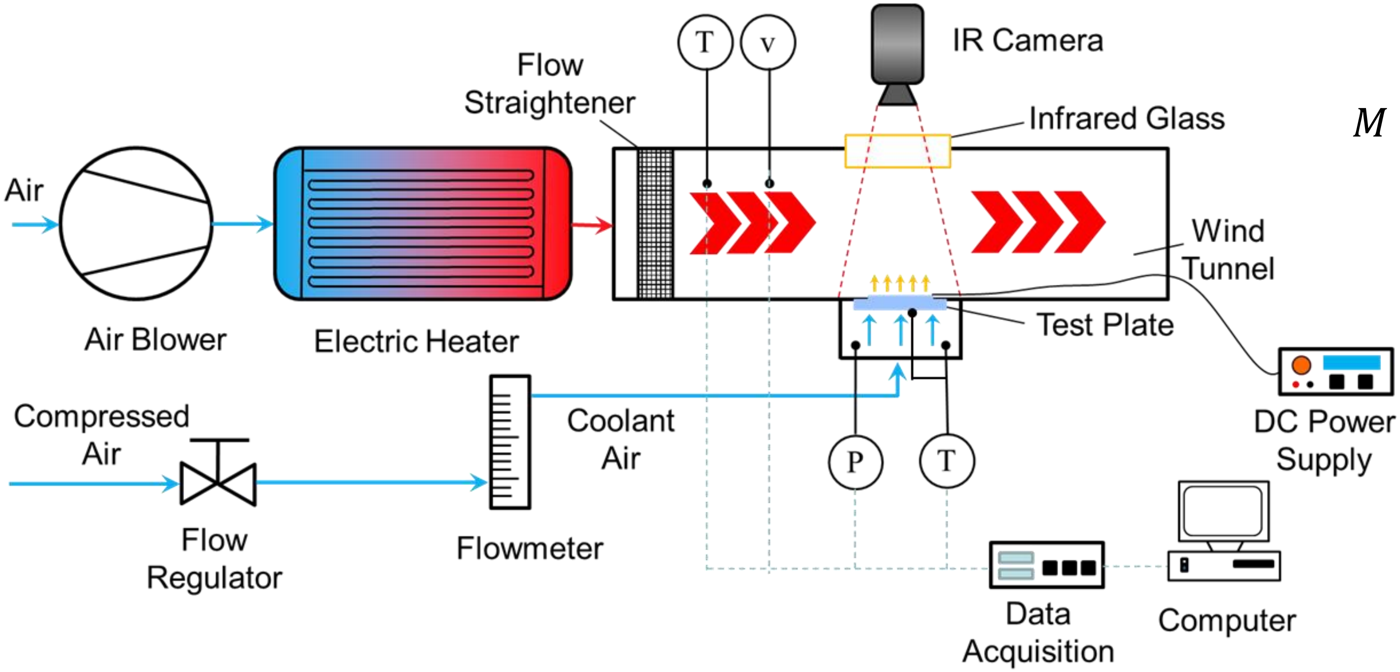
$$R_v = \rho \frac{L_v}{w_v t}$$

$$R_h = \rho \frac{L_h}{w_h t}$$



- *Plassys Electron Beam Evaporation System*

Transpiration Cooling – Wind Tunnel Test



$$M = \frac{\rho_c u_c}{\rho_g v_g}$$

- **Heat transfer coefficient test:**

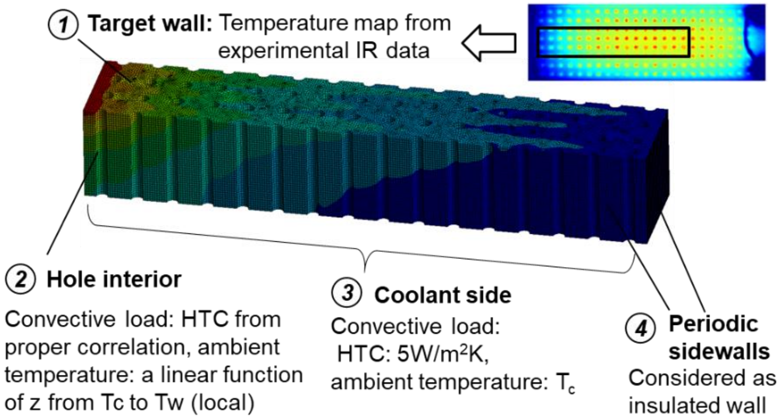
1. Blowing Ratio: M = 0.125, 0.25, 0.5
2. Coolant Temperature: $T_c = 35$ ℃
3. Mainstream Temperature: $T_g = 35$ ℃
4. Mainstream Velocity: $v_g = 11$m/s (Re_g=98,000)
5. Heater power on for h_f: 0.2W
6. No coolant injection to obtain h_0

- **Adiabatic cooling effectiveness test:**

1. Blowing Ratio: M = 0.125, 0.25, 0.5
2. Coolant Temperature: $T_c = 21$ ℃
3. Mainstream Temperature: $T_g = 50$ ℃
4. Mainstream Velocity: $v_g = 11$m/s (Re_g=98,000)
5. Heater power off for T_{aw}

Conduction Loss Evaluation

➢ 3D finite element method (FEM)



① **Target wall:** Temperature map from experimental IR data

② **Hole interior**

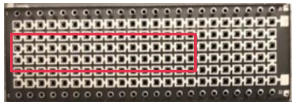
Convective load: HTC from proper correlation, ambient temperature: a linear function of z from Tc to Tw (local)

③ **Coolant side**

Convective load: HTC: 5W/m²K, ambient temperature: T_c

④ **Periodic sidewalls**

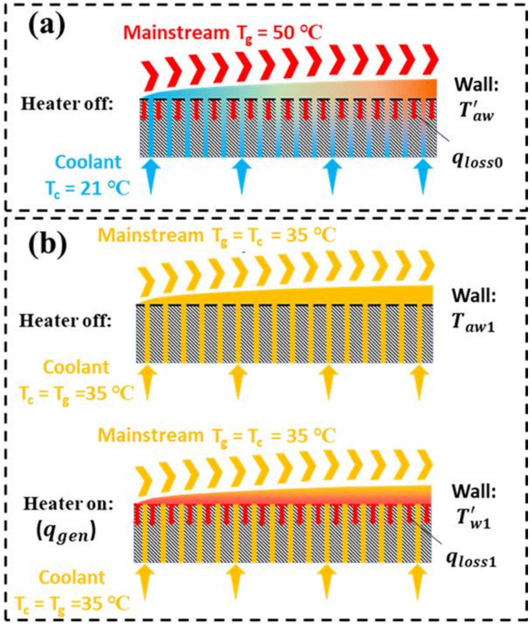
Considered as insulated wall



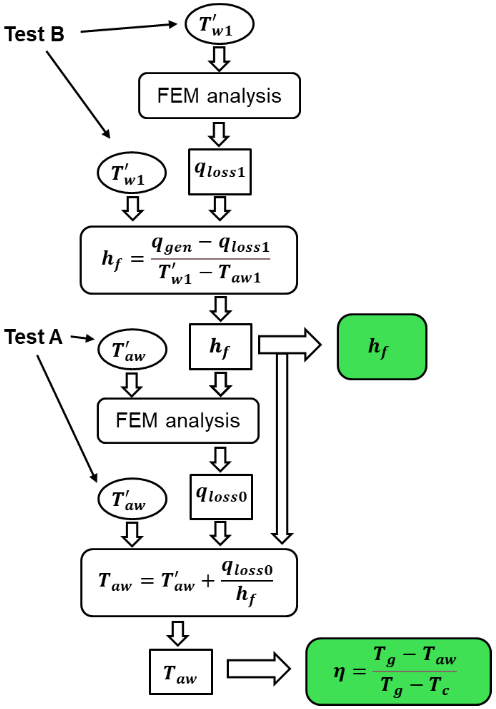
- Periodic section (16 x 4 holes)
- 400,000 – 700,000 elements

ANSYS 2019 Steady-state Thermal

Objective: to evaluate conduction loss q_{loss} at the target surface



$$T_{aw} = T'_{aw} + \frac{q_{loss0}}{h_f} \qquad h_f = \frac{q_{gen} - q_{loss1}}{T_{w1} - T_{aw1}}$$



Test B → T'_{w1} → FEM analysis → q_{loss1}

Test B → T'_{w1}

$$h_f = \frac{q_{gen} - q_{loss1}}{T'_{w1} - T_{aw1}}$$

→ h_f → h_f

Test A → T'_{aw} → FEM analysis → q_{loss0}

Test A → T'_{aw}

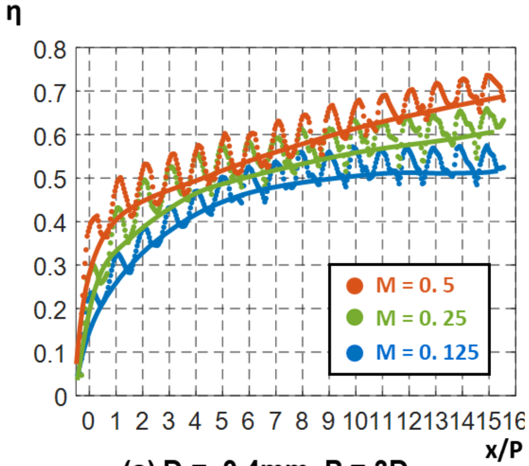
$$T_{aw} = T'_{aw} + \frac{q_{loss0}}{h_f}$$

→ T_{aw} →

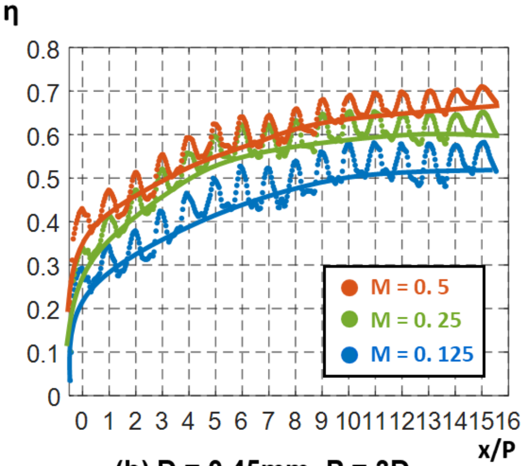
$$\eta = \frac{T_g - T_{aw}}{T_g - T_c}$$

Transpiration Cooling – Adiabatic Cooling Effectiveness

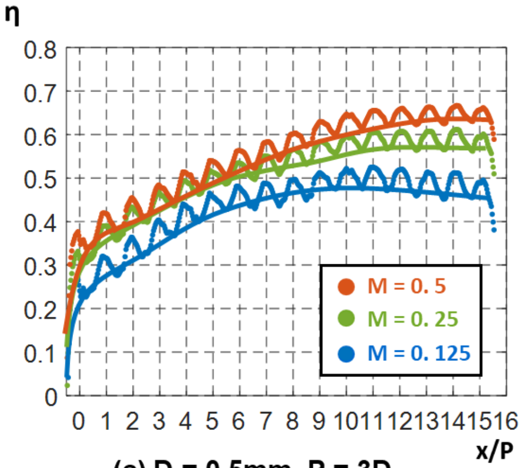
Hole size variation:



(a) D = 0.4mm, P = 3D

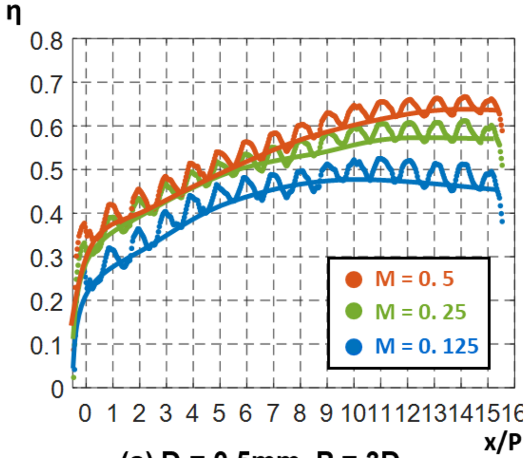


(b) D = 0.45mm, P = 3D

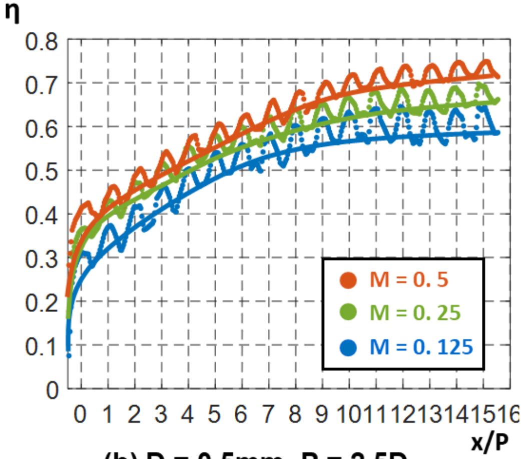


(c) D = 0.5mm, P = 3D

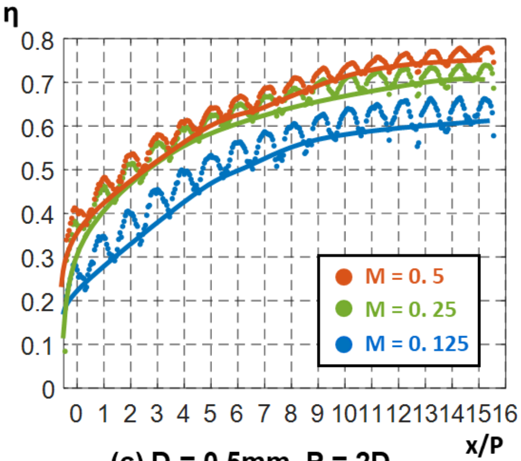
Hole pitch variation:



(a) D = 0.5mm, P = 3D



(b) D = 0.5mm, P = 2.5D

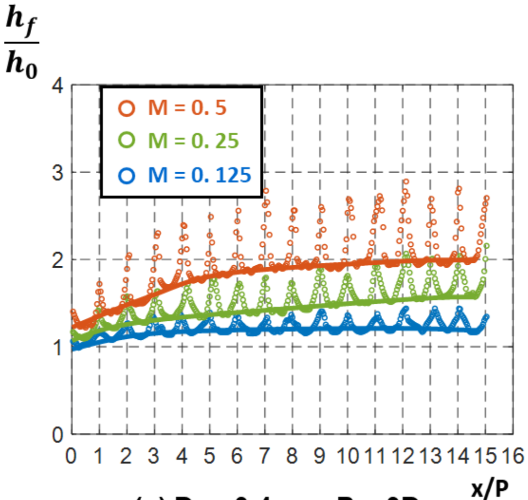


(c) D = 0.5mm, P = 2D

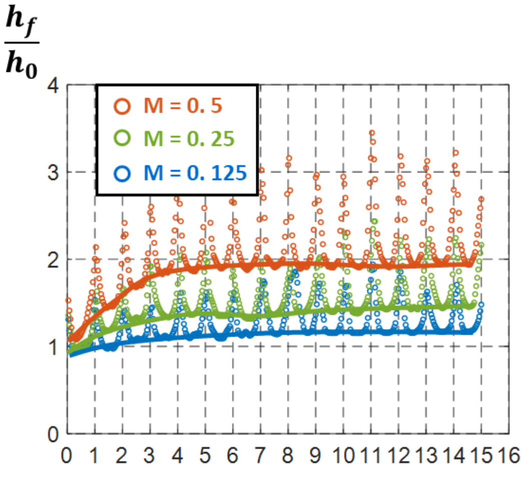
- Smaller hole size or smaller hole pitch present better performance
- The impact of increasing blowing ratio from 0.25 to 0.5 is not as significant as the increase from 0.125 to 0.25

Transpiration Cooling – HTC

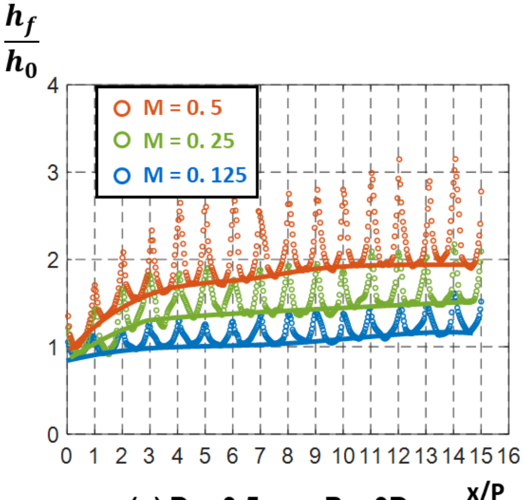
Hole size variation:



(a) D = 0.4mm, P = 3D

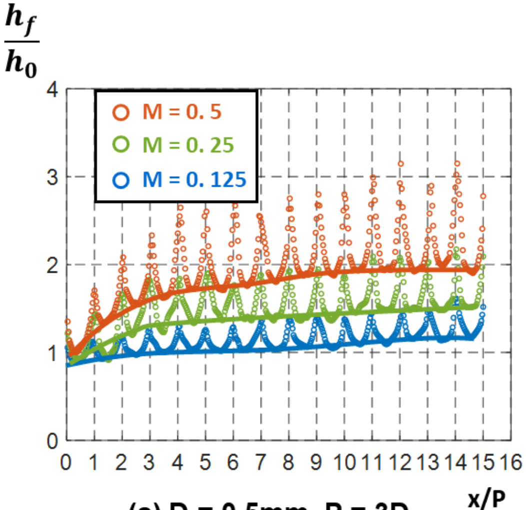


(b) D = 0.45mm, P = 3D

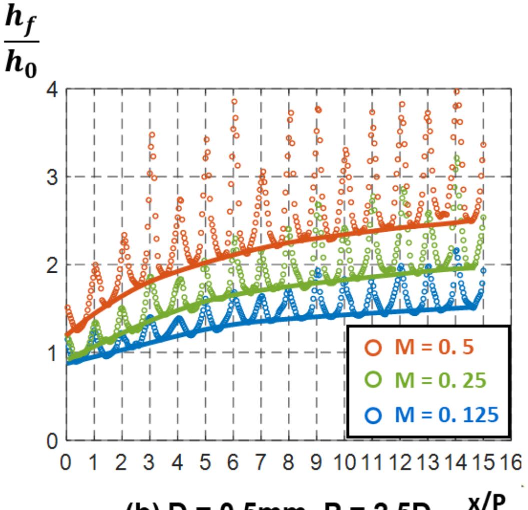


(c) D = 0.5mm, P = 3D

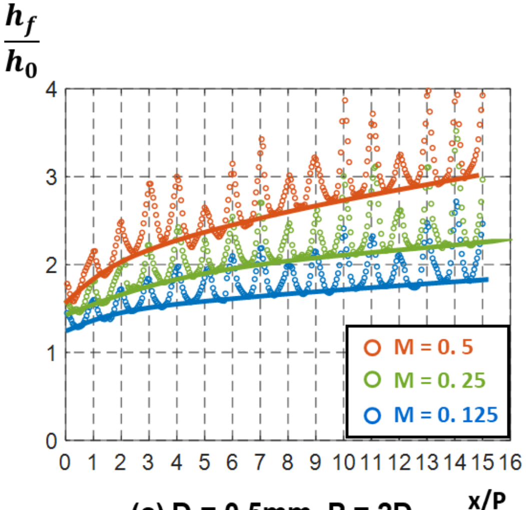
Hole pitch variation:



(a) D = 0.5mm, P = 3D



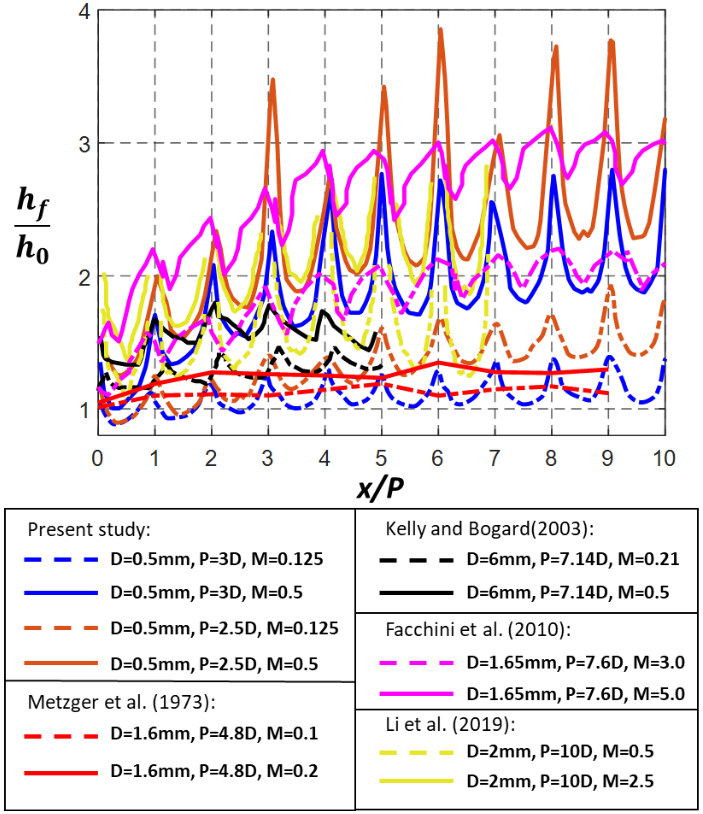
(b) D = 0.5mm, P = 2.5D

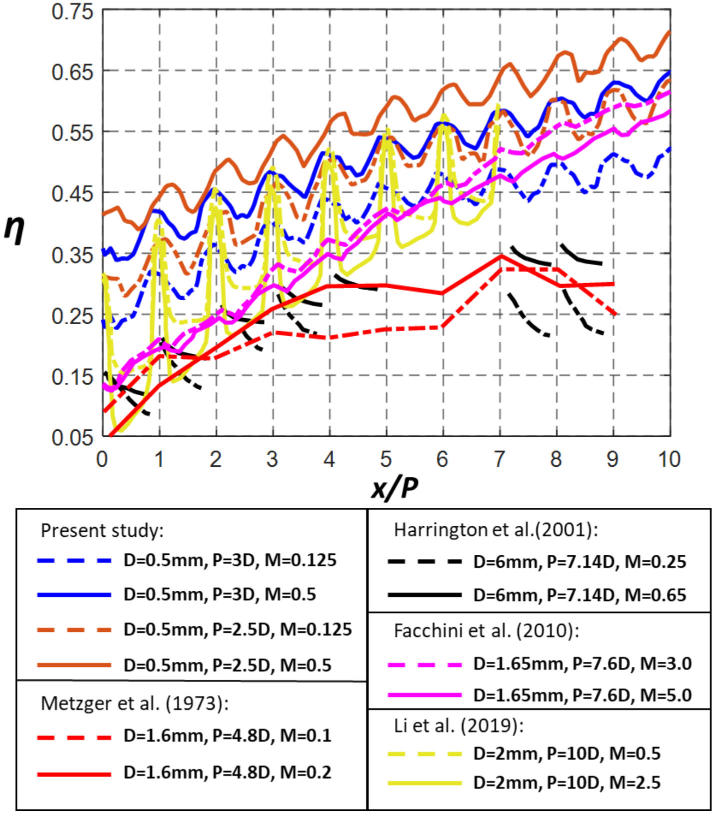


(c) D = 0.5mm, P = 2D

- HTC sensitive to blowing ratio and pitch-to-diameter ratio; but less sensitive to hole size
- Smaller pitch leads to higher HTC, due possibly to interactions between closely adjacent coolant discharge

Comparison with Film Cooling



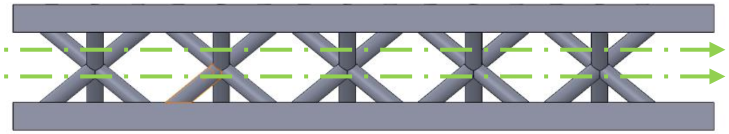


Min, Z, Parbat, S, Wang, Q, & Chyu, MK. "Surface Heater Fabrication Using Micro-Lithography for Transpiration Cooling Heat Transfer Coefficient Measurements." *Proceedings of the ASME Turbo Expo 2021: Turbomachinery Technical Conference and Exposition*. *Volume 5B: Heat Transfer — General Interest; Internal Air Systems; Internal Cooling*. Virtual, Online. June 7–11, 2021. V05BT13A007. ASME. https://doi.org/10.1115/GT2021-59275

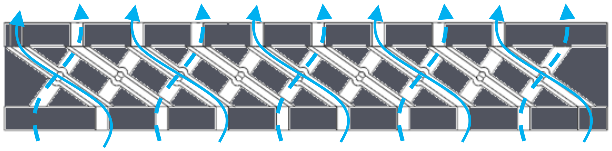
Outlines

- *Background, Challenges, Objectives, Benefits of Technology, Research Task Plans*
- *Additive Manufacturing Processes*
- *Heat Transfer Results: Transpiration Cooling*
- ***Heat Transfer Results: Lattice Cooling***
- *Heat Transfer Results: Integrated Cooling*
- *ODS Powder Development*

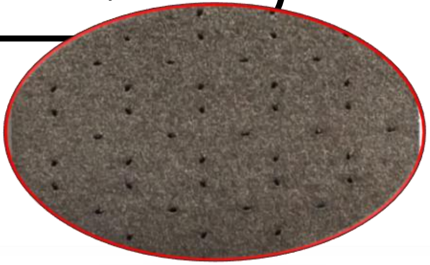
Approaches to Fabricate ODS Lattice Structures

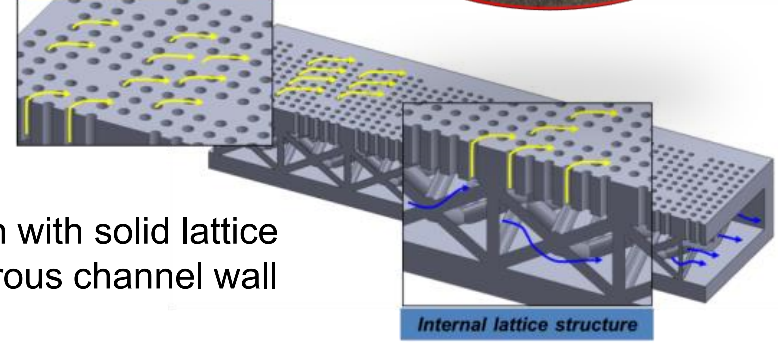


Internal Cooling with Macro Scale Lattice (Phase 1)

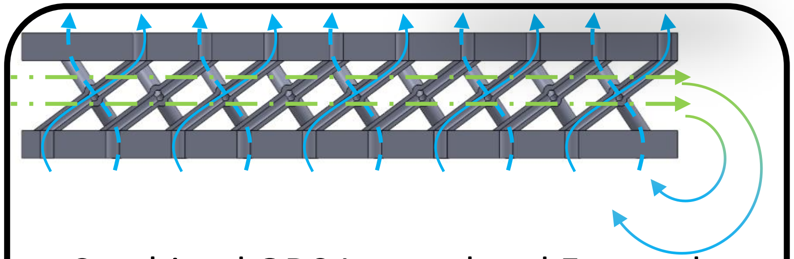


Transpiration Cooling with Micro Scale Lattice (Phase 2)

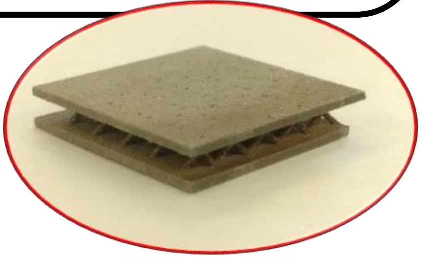




Coupon with solid lattice and porous channel wall



Combined ODS Internal and External Cooling with Multi-scale Lattice (Phase 3)



Challenges:

- AM process parameters for ODS
- Controlling dimension change and deformation
- Identify minimum limitation of pore diameters

Lattice Heat Transfer Characterization

Objective: Design highly efficient and manufacturable integrated transpiration and internal cooling which has an overall averaged cooling efficiency of more than 0.6.

- Internal Cooling with Macro Scale Lattice (Phase 1)
- Transpiration Cooling with Micro Scale Lattice (Phase 2)

Approach

- Identification of candidate transpiration and lattice geometries
  - Transpiration: Candidate geometries from transpiration experiments
  - Lattice: Unit cell geometries, porosity, unit cell arrangement
- Experimental and numerical iteration
  - Experimental studies on cooling effectiveness
  - CFD to obtain flow fields
- Integrated design
  - Combining transpiration and lattice designs together to get high cooling effectiveness of more than 0.6
- Lattice optimization for improved thermal fluid characteristics

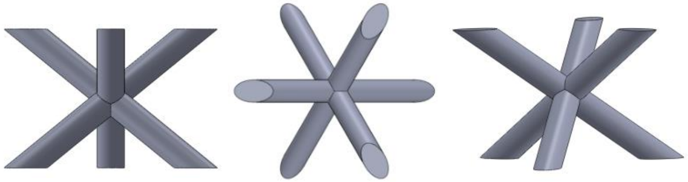
Unit Cells

Overview of unit cells under investigation:

Advantages

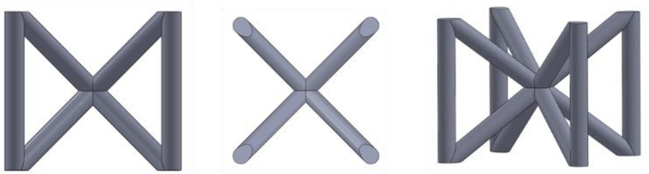
- High heat transfer surface area
- Promote turbulence in the bulk flow
- Provide structural rigidity

Kagome



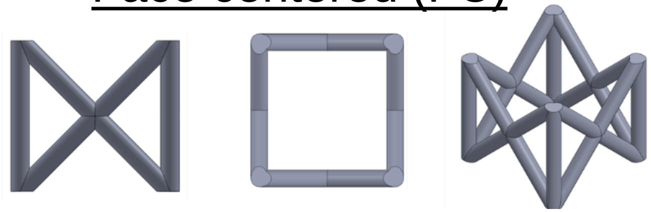
Front View Top view Perspective

Body centered (BC)



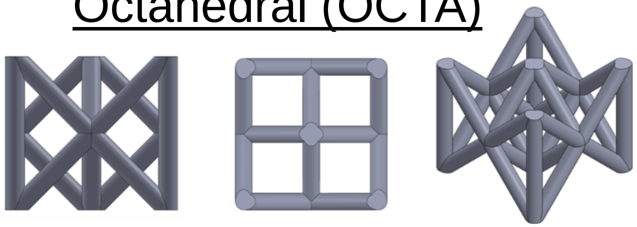
Front View Top view Perspective

Face centered (FC)



Front View Top view Perspective

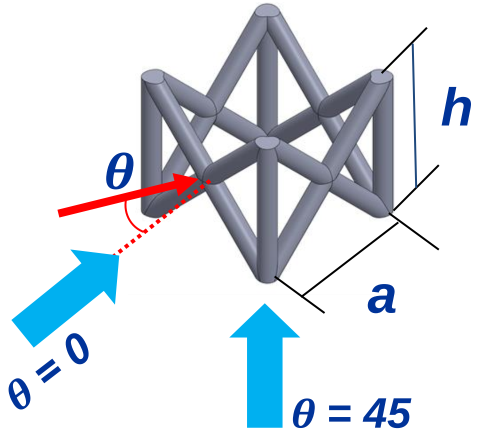
Octahedral (OCTA)



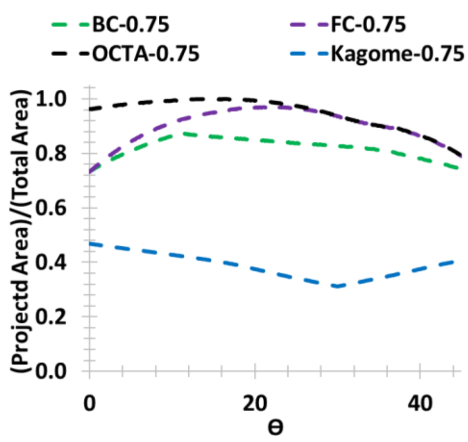
Front View Top view Perspective

Parameters

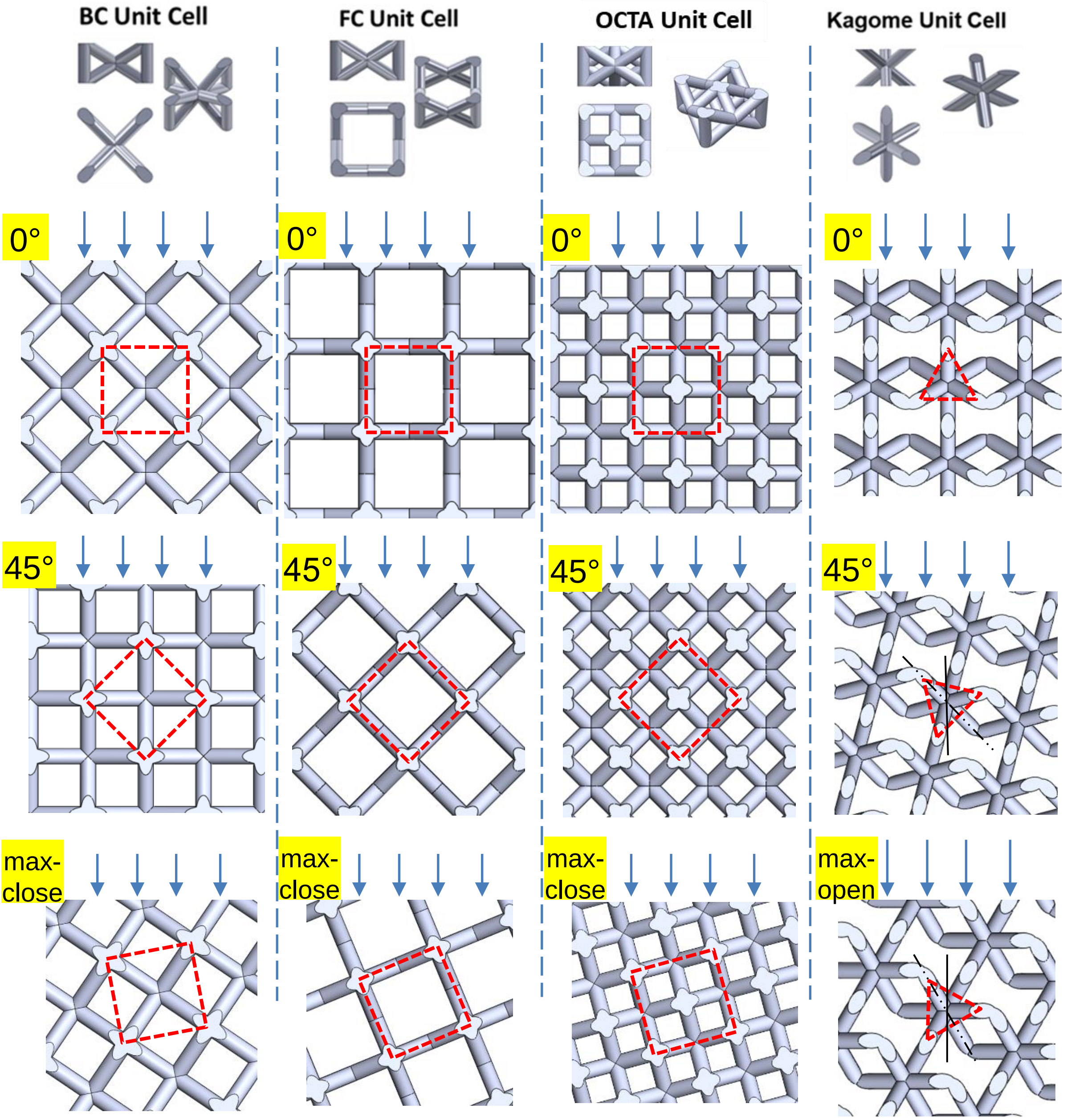
Ligament diameter:	d
Unit cell base:	a
Unit cell height:	h
Porosity:	p
Total surface area:	$A_{lattice}$
Footprint area:	$A_{footprint}$
Orientation with flow:	θ

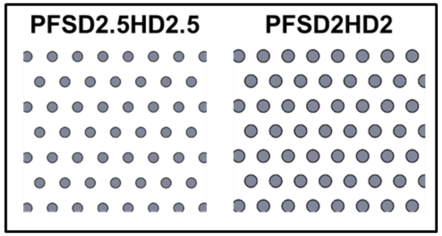


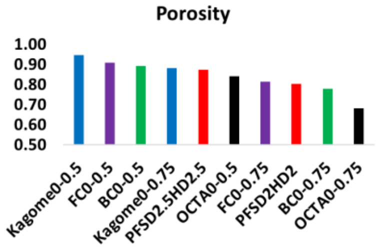
Effect of rotation on projected area



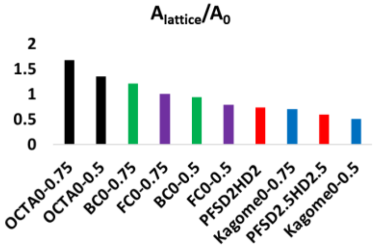
Lattices from Unit Cells

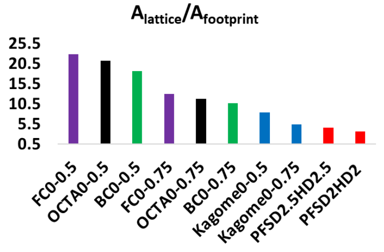


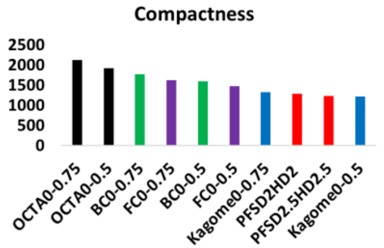




$$Porosity = 1 - \frac{V_{solid}}{V_0}$$





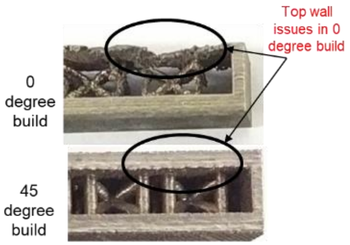


$$Compactness = \frac{A_{wetted}}{V_0}$$

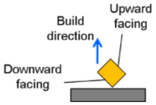
True –Scale Geometries

- True scale geometries fabricated using DMLS additive manufacturing process
  - varying diameter and build orientation

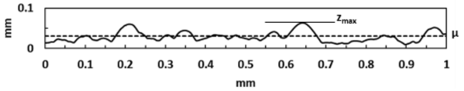
Overhang issues



Roughness



$$Ra = \frac{1}{n}\sum_{i=1}^{i=n}|z_i - \mu|$$

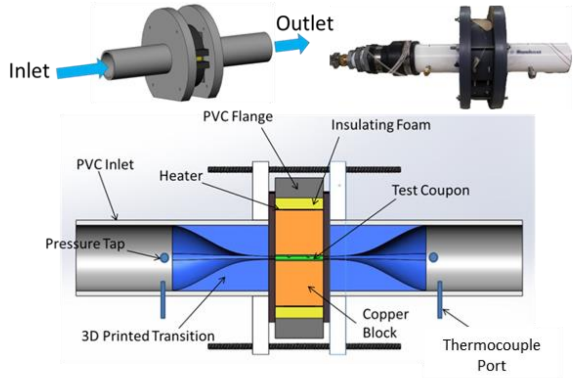


Surface	Ra(μm)	Ra/D_{ch}
Upward	11.9	0.0047
Downward	22.2	0.0088
Vertical	12.7	0.005

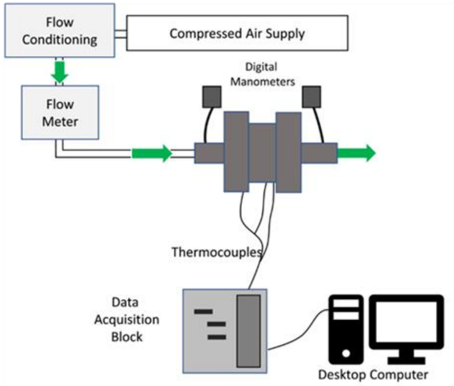
45° build orientation selected for fabrication

- Conjugate heat transfer tests

Test Rig



Test Setup Layout

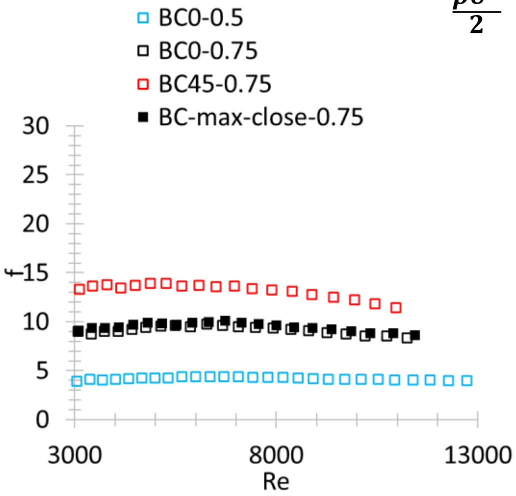


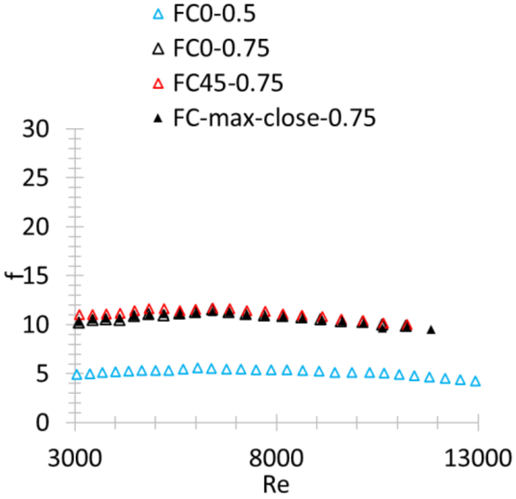
- True-scale test coupons for conjugate heat transfer analysis
- Constant wall temperature boundary condition
- Obtain overall heat transfer
- Re_{ch} : ~3000 to ~13,000

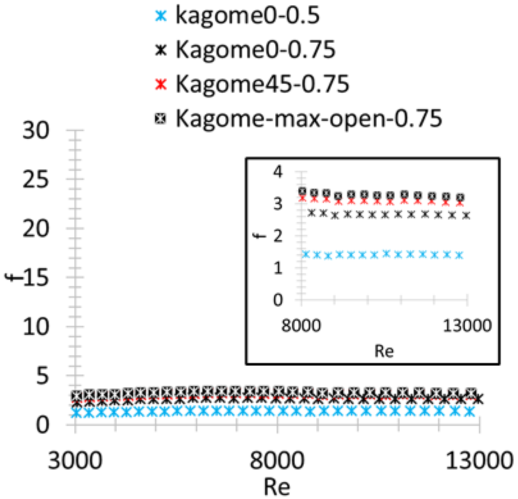
Parbat, S., Min, Z., Yang, L., and Chyu, M. (May 12, 2020). "Experimental and Numerical Analysis of Additively Manufactured Inconel 718 Coupons With Lattice Structure." ASME. *J. Turbomach*. June 2020; 142(6): 061004. https://doi.org/10.1115/1.4046527

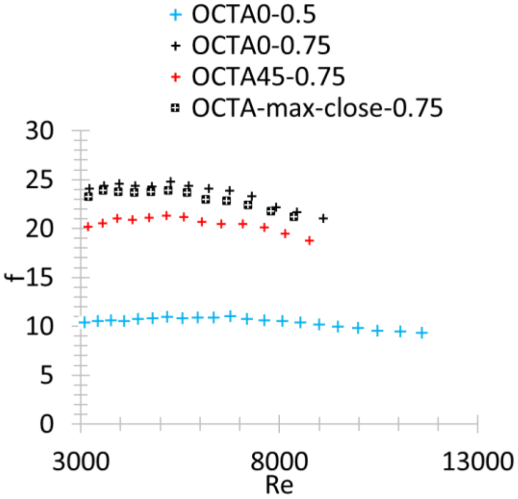
Pressure Drop

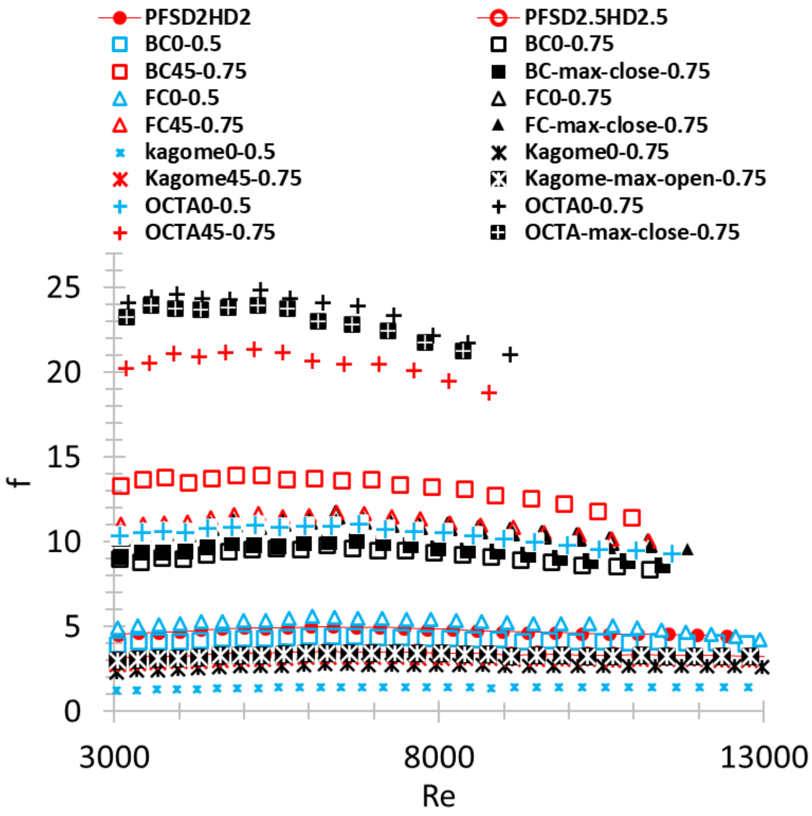
$$f = \frac{\left(\frac{\Delta p}{\Delta x}\right) D_{ch}}{\frac{\rho U^2}{2}}$$











Heat Transfer

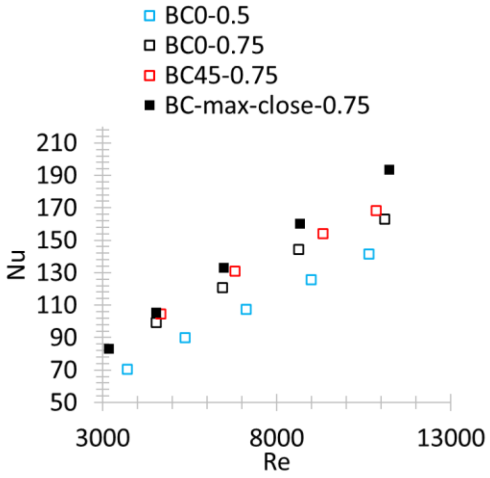
$$LMTD = \frac{(T_{wall} - T_{in}) - (T_{wall} - T_{out})}{ln\frac{(T_{wall} - T_{in})}{(T_{wall} - T_{out})}}$$

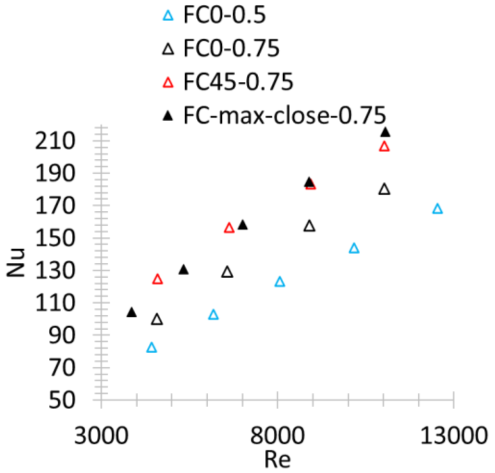
$$q" = V.\frac{I}{A_{wetted}}$$

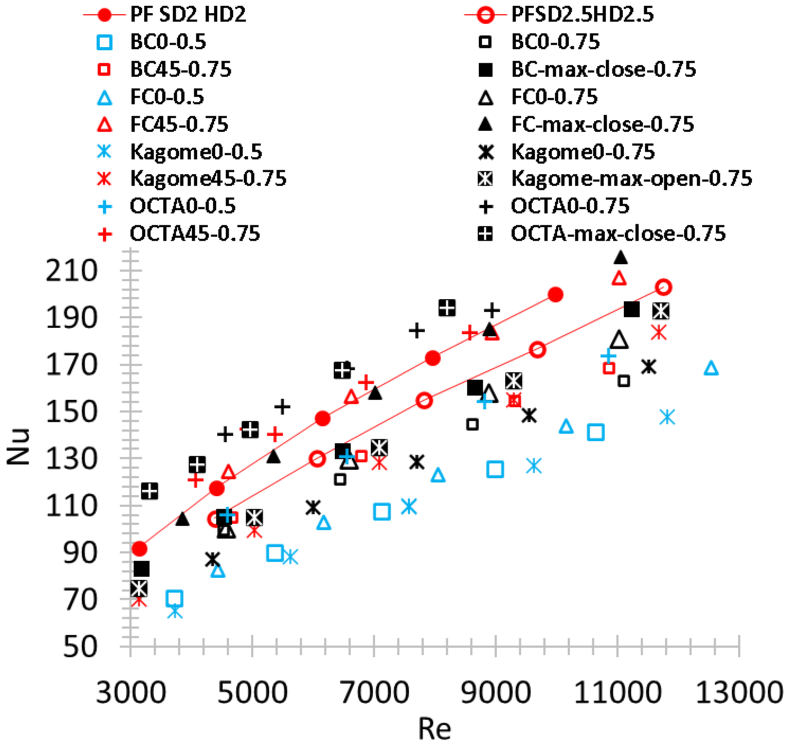
Awetted obtained from CAD

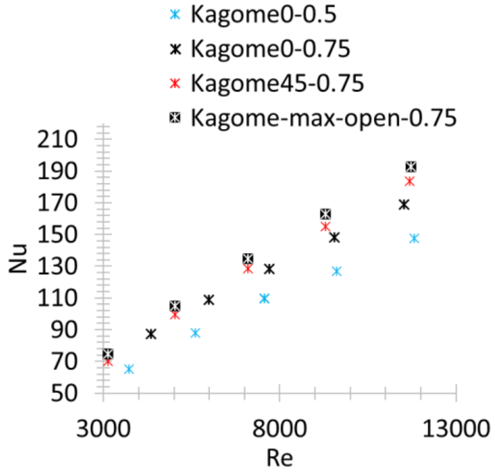
$$HTC = \frac{q" - q"_{loss}}{LMTD}$$

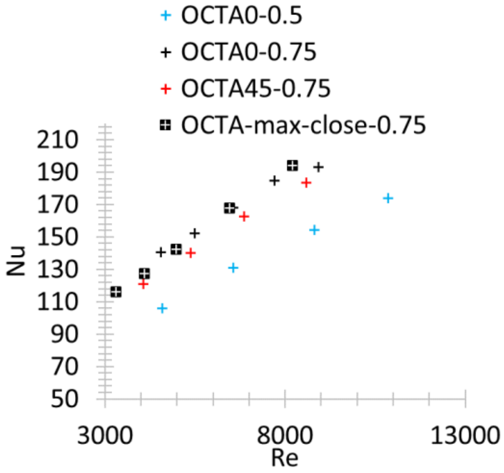
$$Nu = \frac{HTC\ D_{ch}}{k}$$







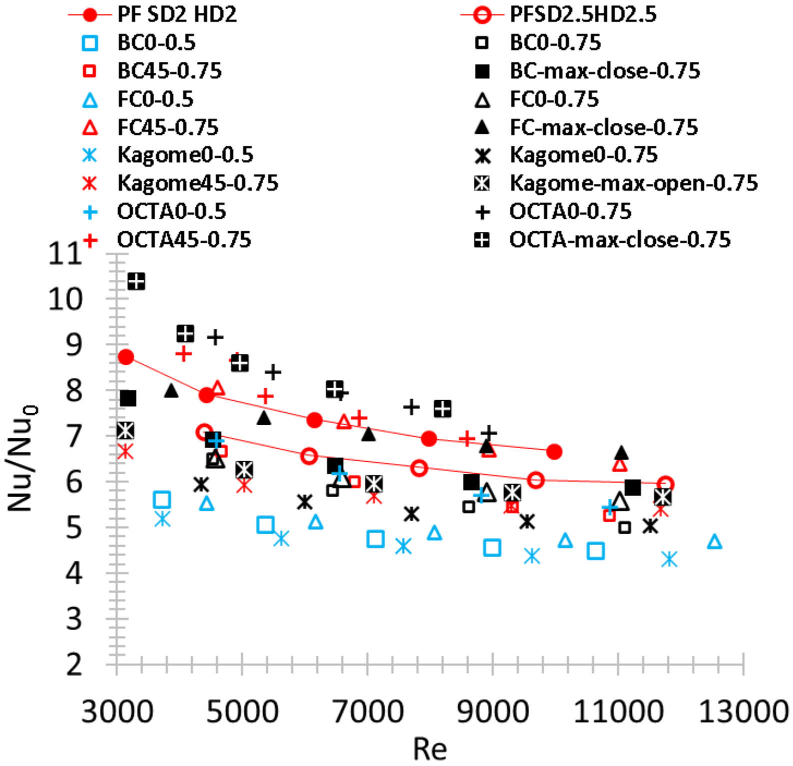


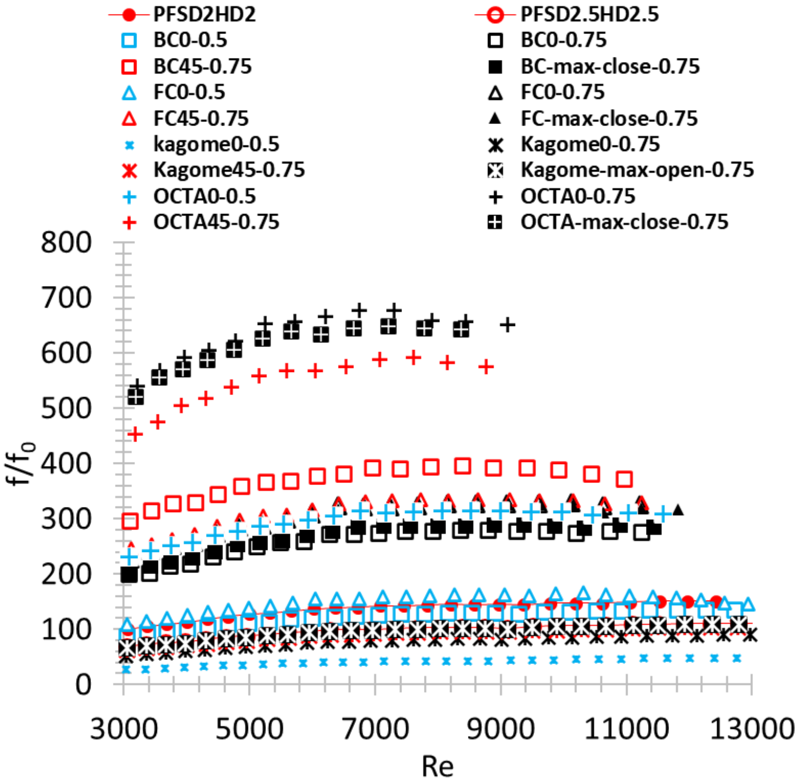


Nu and f Augmentation

$$Nu_0 = \frac{\frac{f_0}{8}(Re - 1000)Pr}{1 + 12.7\left(\frac{f_o}{8}\right)^{\frac{1}{2}}\left(Pr^{\frac{2}{3}} - 1\right)}$$

$$f_o = (0.790 \ln Re - 1.64)^{-2}$$
$$3000 \leq Re \leq 5\,x10^6$$





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- *Heat Transfer Results: Lattice Cooling*
- ***Heat Transfer Results: Integrated Cooling***
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Integrated Cooling with Micro Scale Lattice (Phase 2)

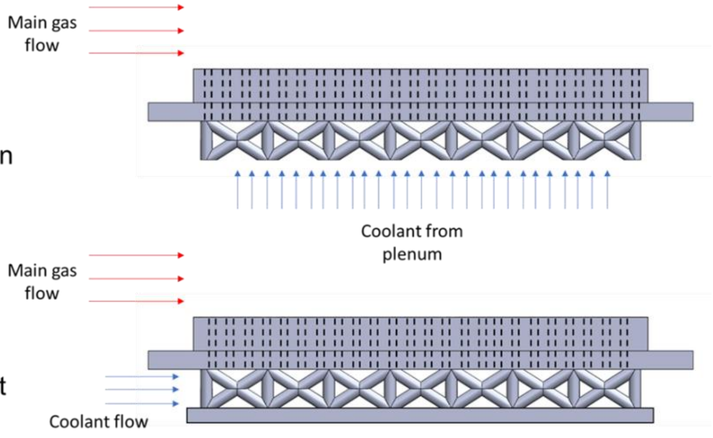
- Transpiration for outer cooling, lattice for internal cooling
- Two possible coolant flow configuration

Plenum fed

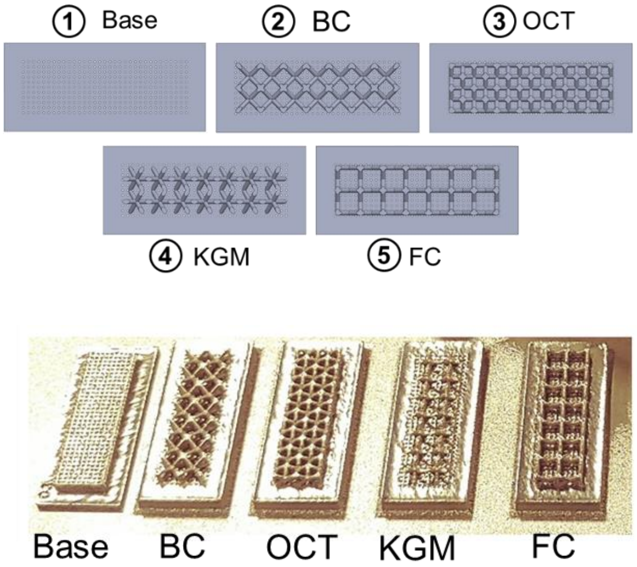
- Uniform pressure distribution

Crossflow fed

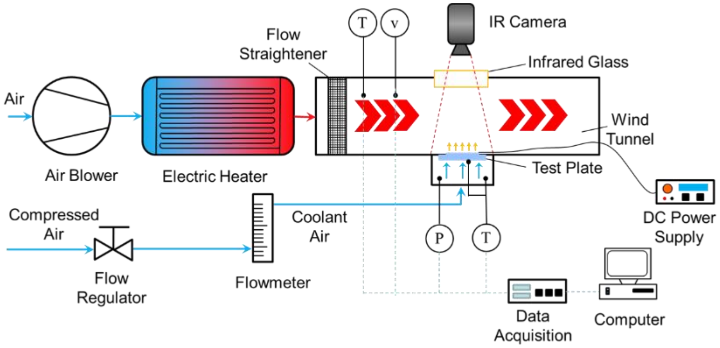
- Pressure drop along coolant direction



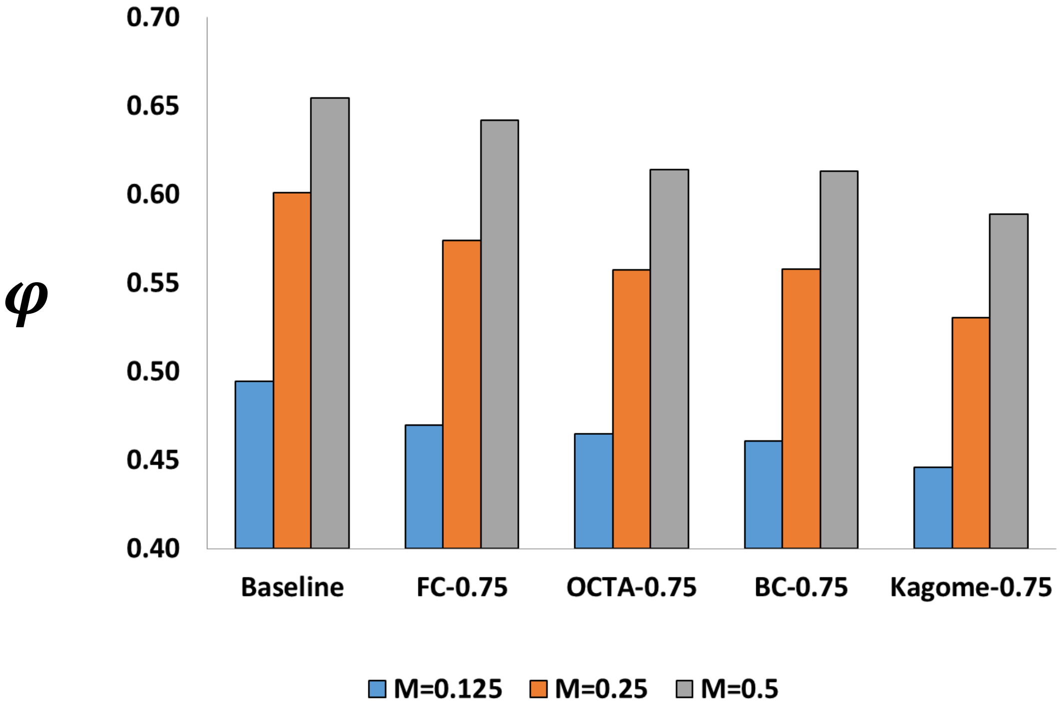
Plenum-Fed Test Coupons



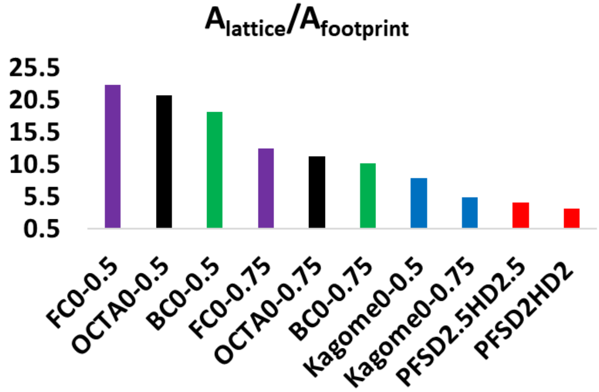
- Testing carried out in the already stablished wind tunnel



Overall Cooling Effectiveness



- Very small flow over backend lattices in plenum-fed configuration
- The φ value follows $A_{lattice}/A_{footprint}$ ratio



Summary

☐ Transpiration cooling

- The micro-lithography technique was employed to fabricate the surface heater on transpiration cooling target surface
- The adiabatic cooling effectiveness and HTC for the transpiration cooling structures were investigated for the first time
- Transpiration cooling with low blowing ratio (0.125) has higher adiabatic cooling effectiveness than multi-row film cooling and HTC ratio close to 1
- Although higher blowing ratio increases HTC significantly, the adiabatic cooling effectiveness of transpiration cooling is still higher than film cooling

☐ Lattice cooling

- Conjugate heat transfer study was performed for true-scale lattices, showing high heat transfer
- Both heat transfer and pressure drop depended on the ligament diameter, unit cell topology, as well as the lattice orientation

☐ Integrated cooling

- Two possible integrated design based on coolant flow direction being investigated
- The $A_{lattice}/A_{footprint}$ is an important parameter for overall cooling effectiveness in integrated designs, thus making lattices preferable to pin fins for backend cooling

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- ***ODS Powder Development***

Ni- and Fe-based ODS powder development for Additive Manufacturing of gas turbine component with better high temperature (up to 1200 °C) oxidation/corrosion resistance and material strength (i.e. creep and fatigue)

(i) (to produce) ODS powder for AM with lower manufacturing cost and better yttria dispersion using a combined Hosokawa mechano chemical bonding (MCB) and ball milling (BM) process:

(a) MCB + BM

(b) MCB only

(ii) AM processing optimization

(iii) (to achieve) a durable α-Al_2O_3 oxide layer with strong adhesion to substrate (via external oxidation) for better oxidation and corrosion resistance

Yttrium Agglomeration in Traditional Mechanical Alloying (MA) using Ball Milling

Yttrium agglomeration in ODS materials.

** ODS Ni-20Cr-5Al -xY_2O_3powder is prepared by Ball milling and then sintered in vacuum carbon tube furnace.

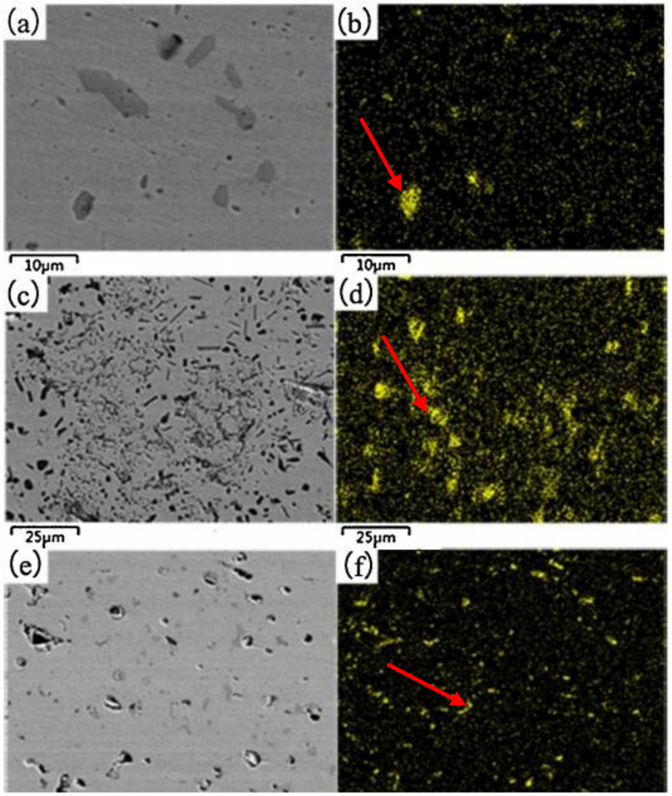
Agglomeration phenomenon:

When the percentage is over 0.6%, resulted in the decrease of mechanical properties.

The agglomeration of yttrium is owing to the limitation of ball milling.

Advantage of WVU-developed Mechano-Chemical –Bonding (MCB) method:

Breakdown and uniform distribution of nano-sized refractory elements, e.g. Y_2O_3 in ODS alloy



** (a), (b) Ni-20Cr-5Al-0.6%Y2O3; (c), (d) Ni-20Cr-5Al-0.8%Y2O3; (e), (f) Ni-20Cr-5Al-1.0%Y2O3.

** Sun, D.; Liang, C.; Shang, J.; Yin, J.; Song, Y.; Li, W.; Liang, T.; Zhang, X. Effect of Y2o3 Contents on Oxidation Resistance at 1150 ° c and Mechanical Properties at Room Temperature of Ods Ni-20cr-5al Alloy. *Applied Surface Science* **2016**, *385*, 587–596 DOI: 10.1016/j.apsusc.2016.05.143.

WV Task Outline

I. **Advantage of MCB-processed powders for AM:**

uniform distribution of nano-sized yittrim oxide;
uniform particle composition; near spherical shape

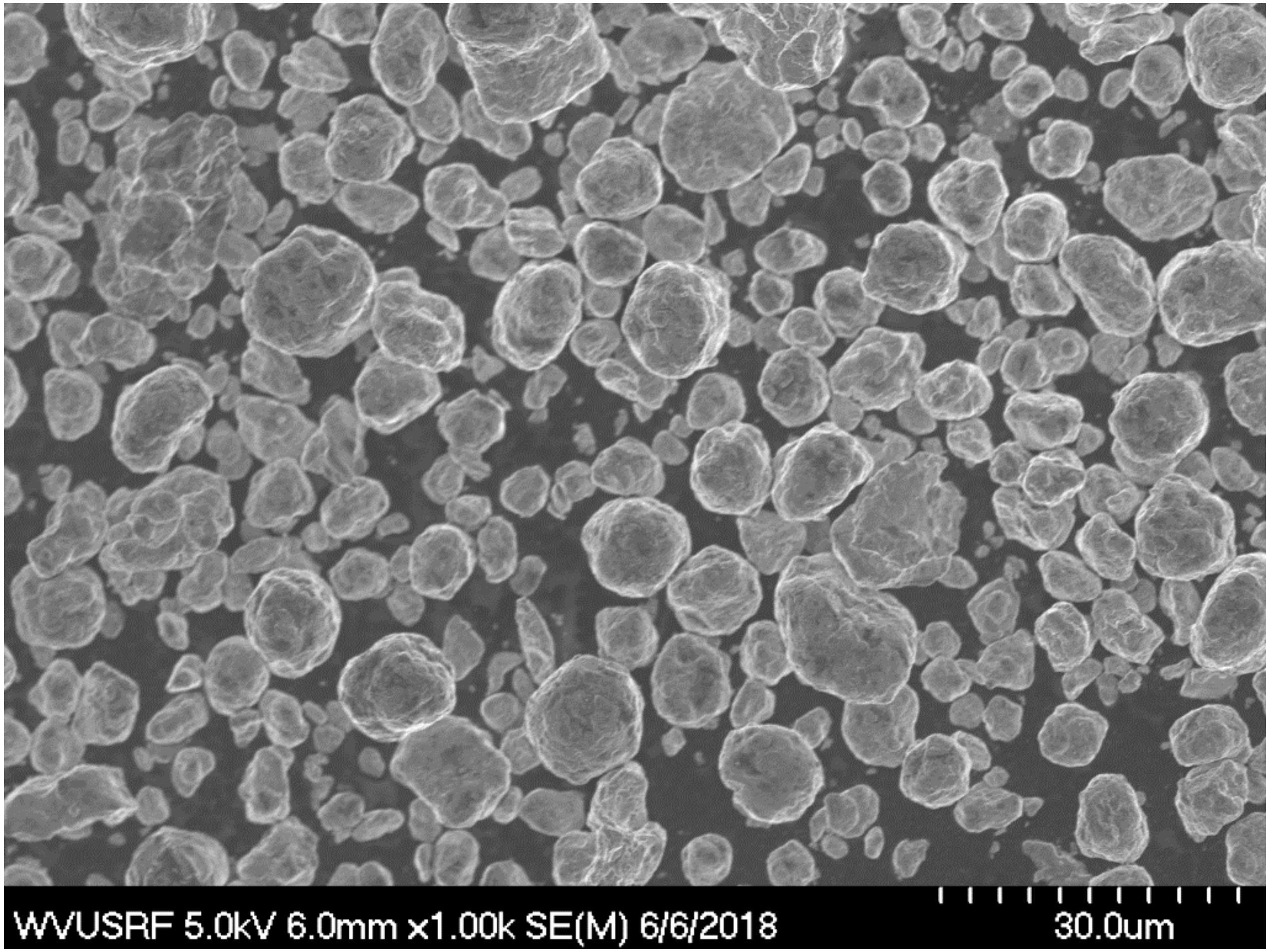
II. **Microstructure and oxidation resistance of ODS754**

III. **Surface oxide layer formation of ODS754**

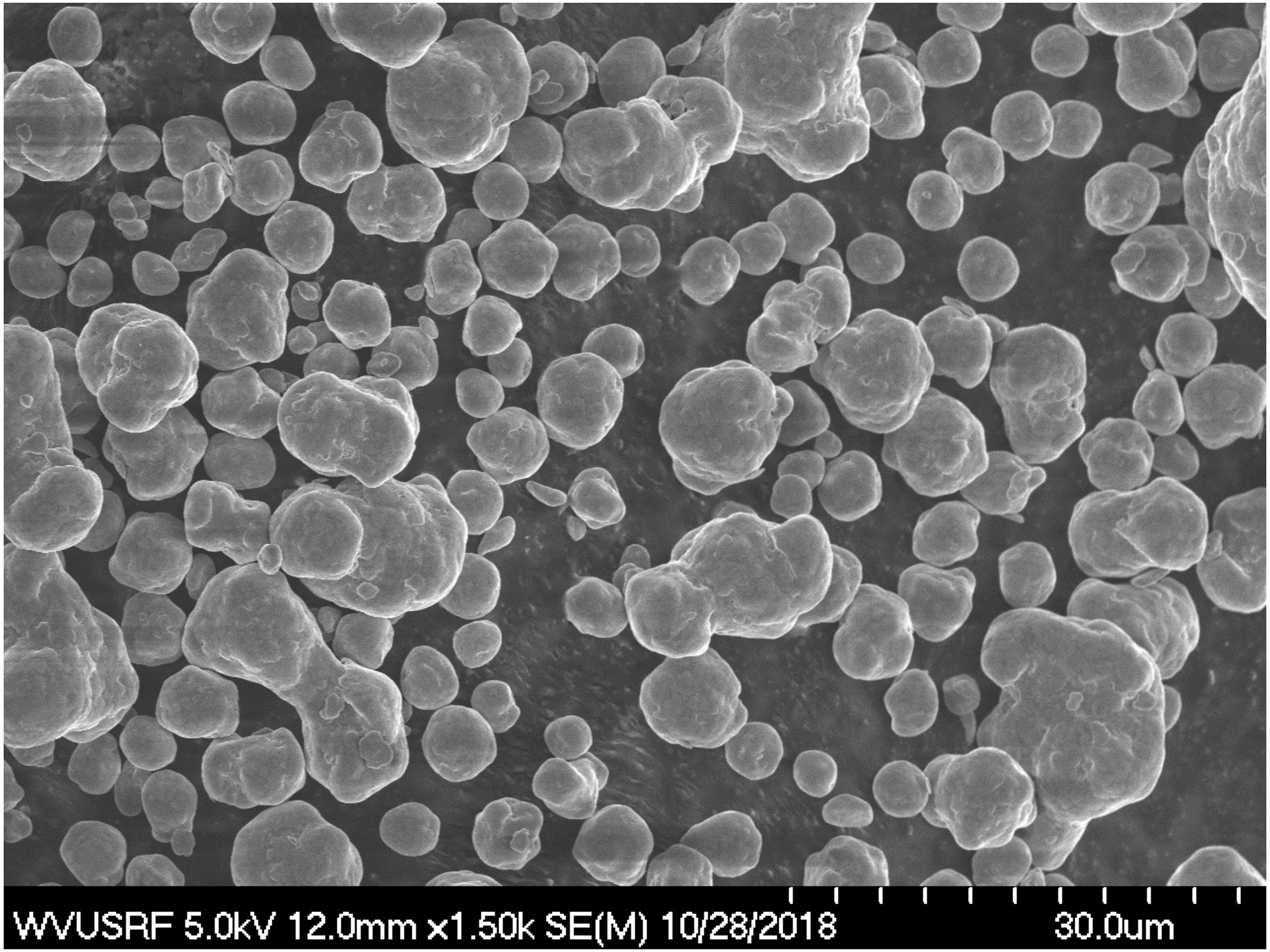
IV. **Summary**

ODS Powder processed by MCB+BM

Ni-20Cr-5Al-3W-1.5Y_2O_3 in Weight.%



MCB only



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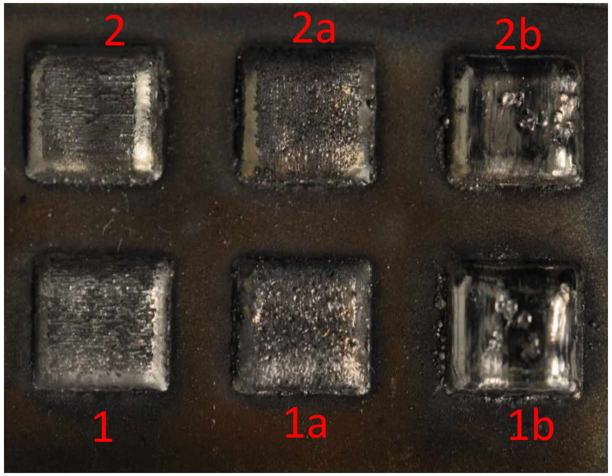
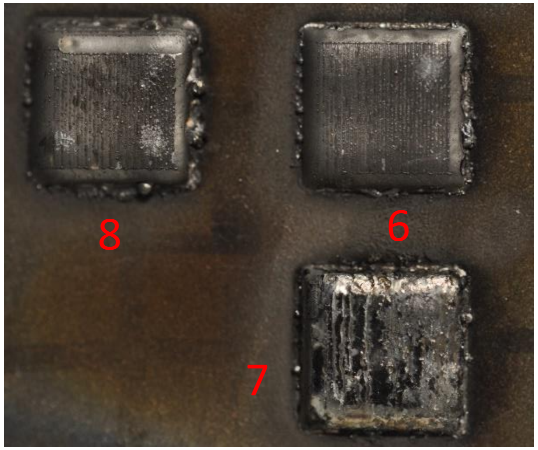
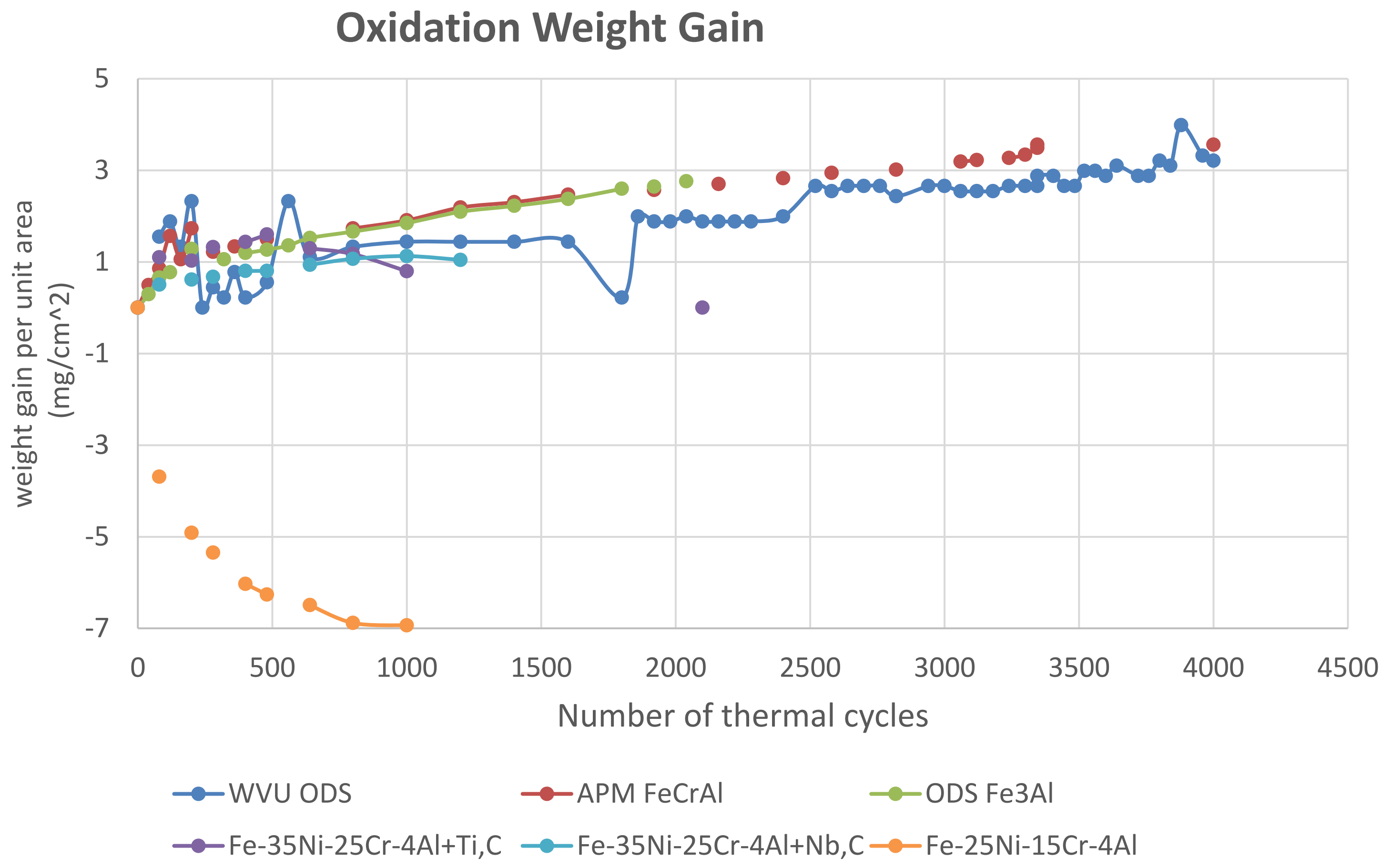
ODS Samples by LENS AM Machine





	Powder	Printing Parameters	Heat Treatment
2	MCB 4000rpm 1h, +BM 400rpm 40h	250W,10mm/s, 0°+90°	N/A
2a	MCB 4000rpm 1h, +BM 400rpm 40h	250W,10mm/s, 0°+90°	100W, 10mm/s
2b	MCB 4000rpm 1h, +BM 400rpm 40h	250W,10mm/s, 0°+90°	200W, 10mm/s
1	MCB 4000rpm 1h, +BM 400rpm 40h	250W,5mm/s, 0°+90°	N/A
1a	MCB 4000rpm 1h, +BM 400rpm 40h	250W,5mm/s, 0°+90°	100W, 10mm/s
1b	MCB 4000rpm 1h, +BM 400rpm 40h	250W,5mm/s, 0°+90°	200W, 10mm/s
8	MCB only, 5000rpm 11h, acid+sieved	250W,10mm/s, 0°+90°	N/A
6	MCB only, 5000rpm 11h, acid+sieved	250W,10mm/s, 0°+90°	100W, 10mm/s
7	MCB only, 5000rpm 11h, acid+sieved	250W,10mm/s, 0°+90°	200W, 10mm/s

Oxidation Weight Gain-ODS754



Comparison of weight gain percentage of WVU ODS sample with other AFA alloys [1,2]

[1] Wright, I.G., Pint, B.A. & Tortorelli, P.F. Oxidation of Metals (2001) 55: 333. https://doi.org/10.1023/A:1010316428752

[2] Brady, M.P., Muralidharan, G., Yamamoto, Y. et al. Oxid Met (2017) 87: 1. https://doi.org/10.1007/s11085-016-9667-3

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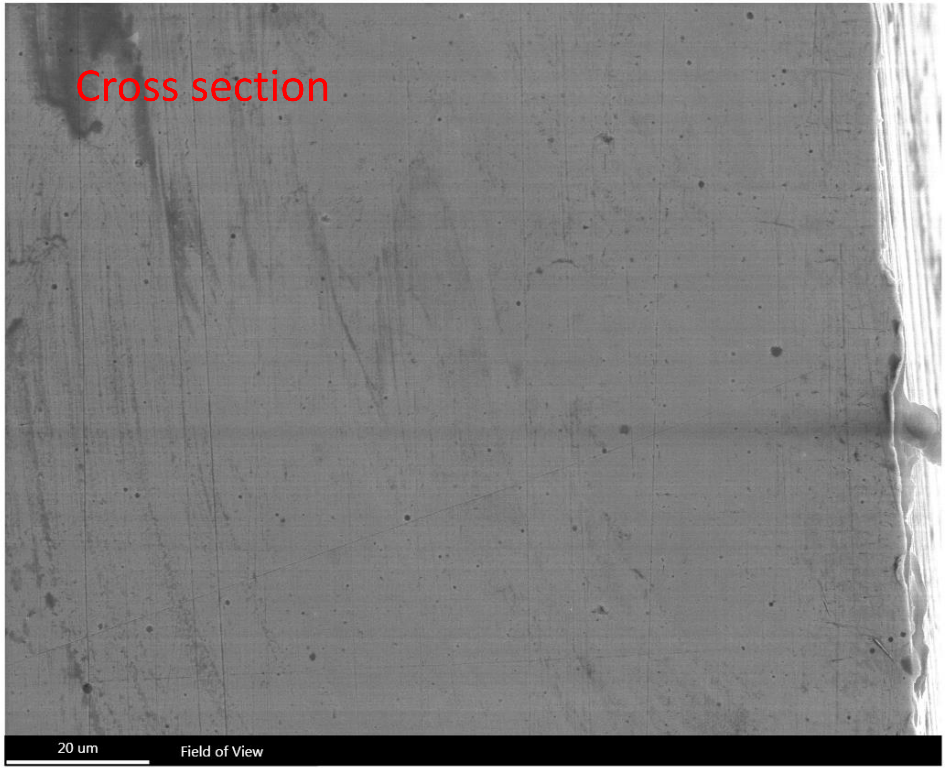
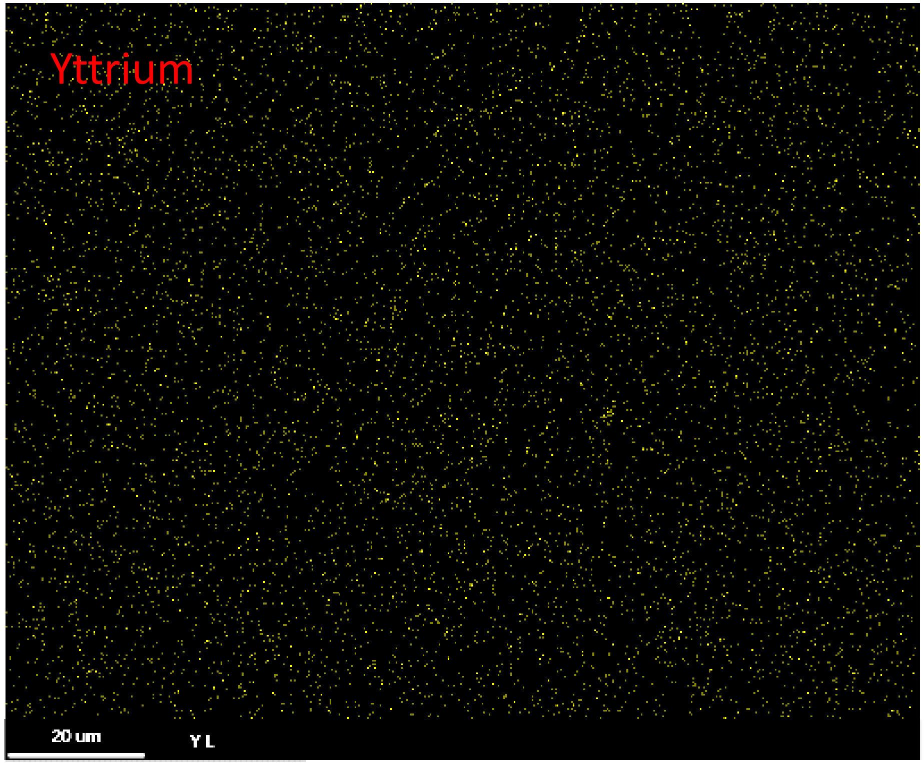
Yttrium Element Distribution under SEM

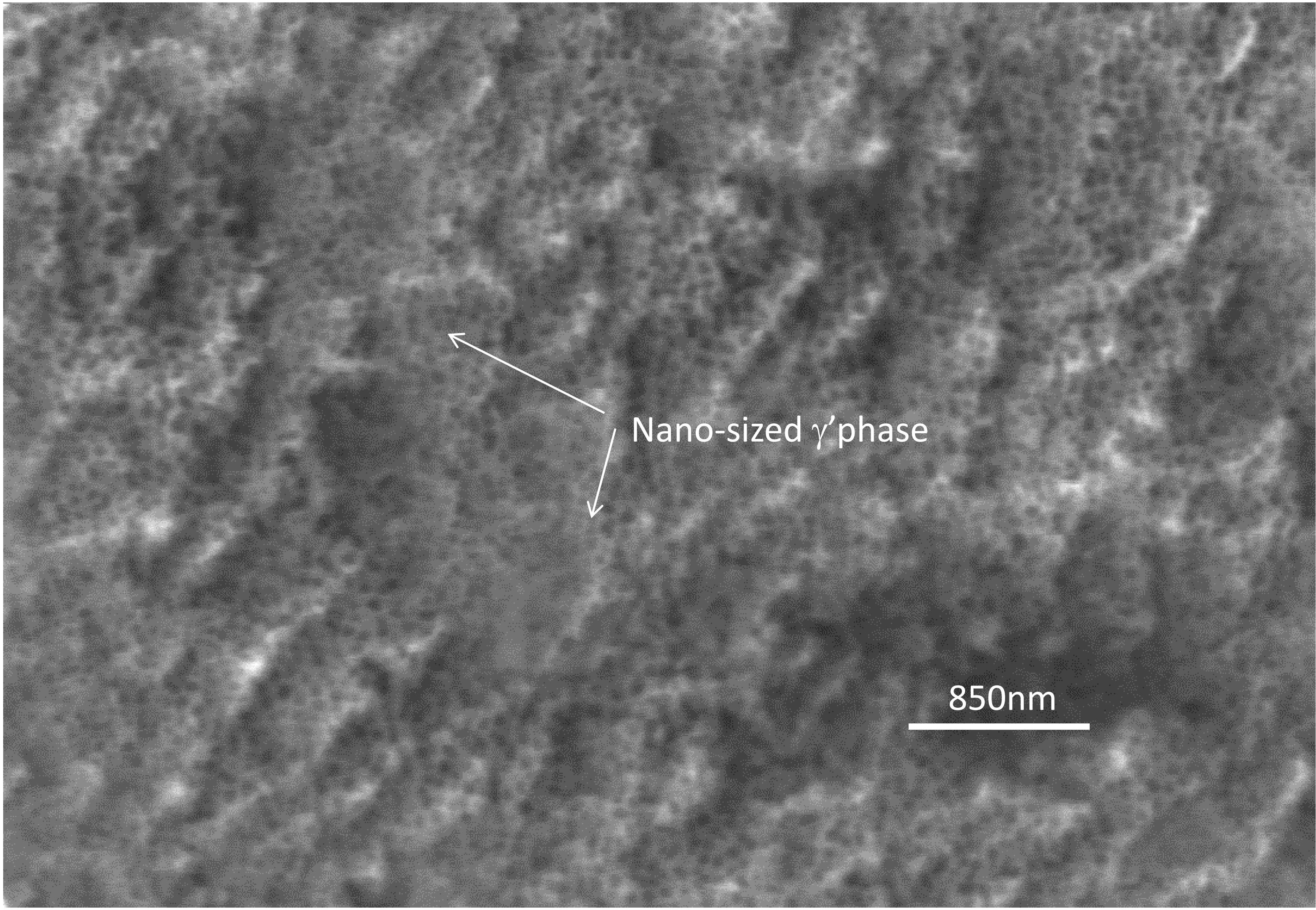
As-printed ODS754 (MCB+BM)

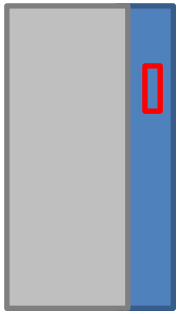




Excellent oxide layer stability due to the uniform distribution of yttrium by MCB.

200W ODS – AM Printed




ODS Coating

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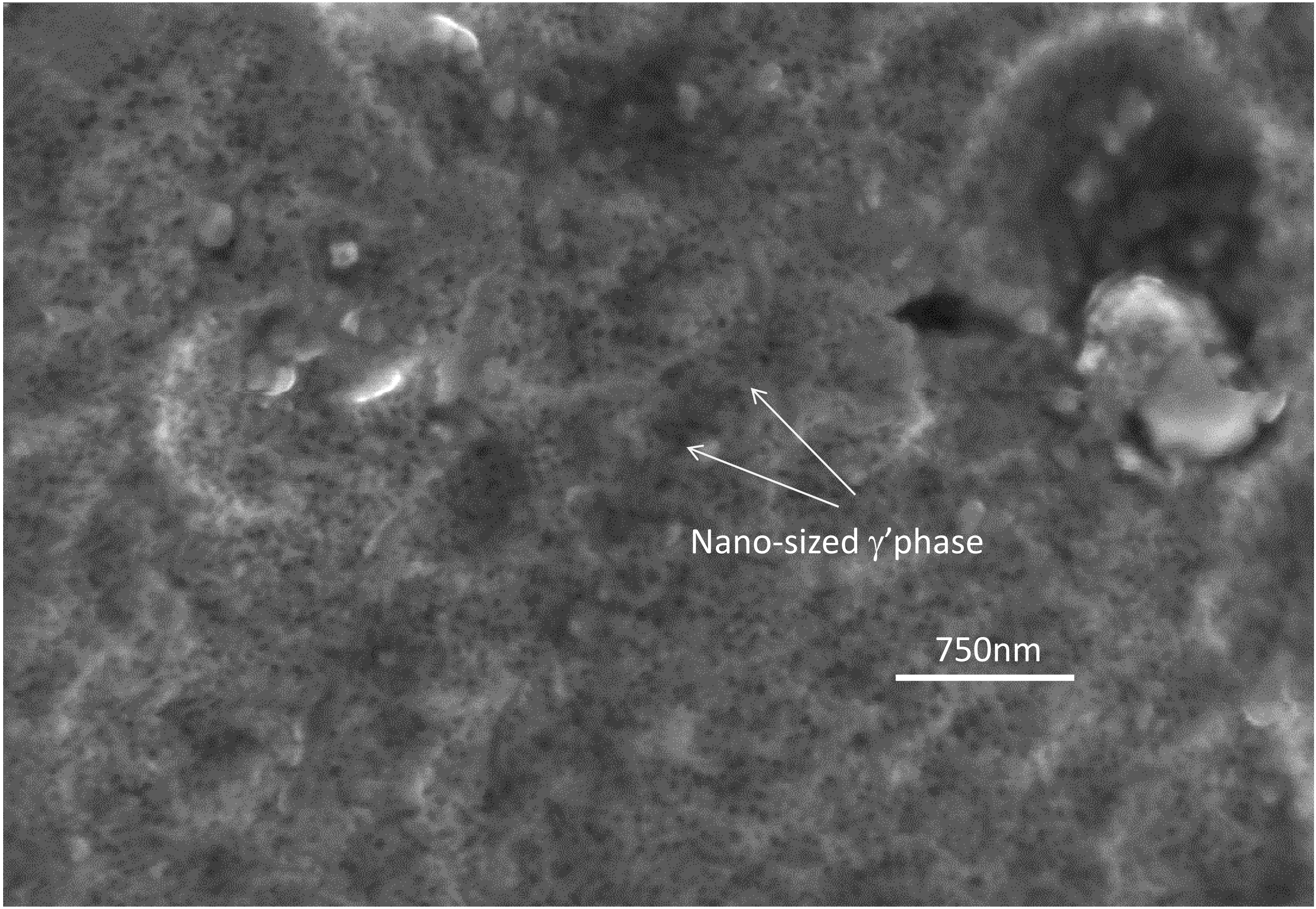
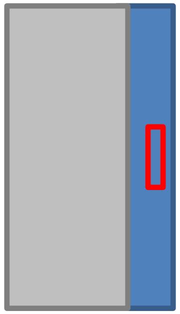
200W ODS – 2200cycles



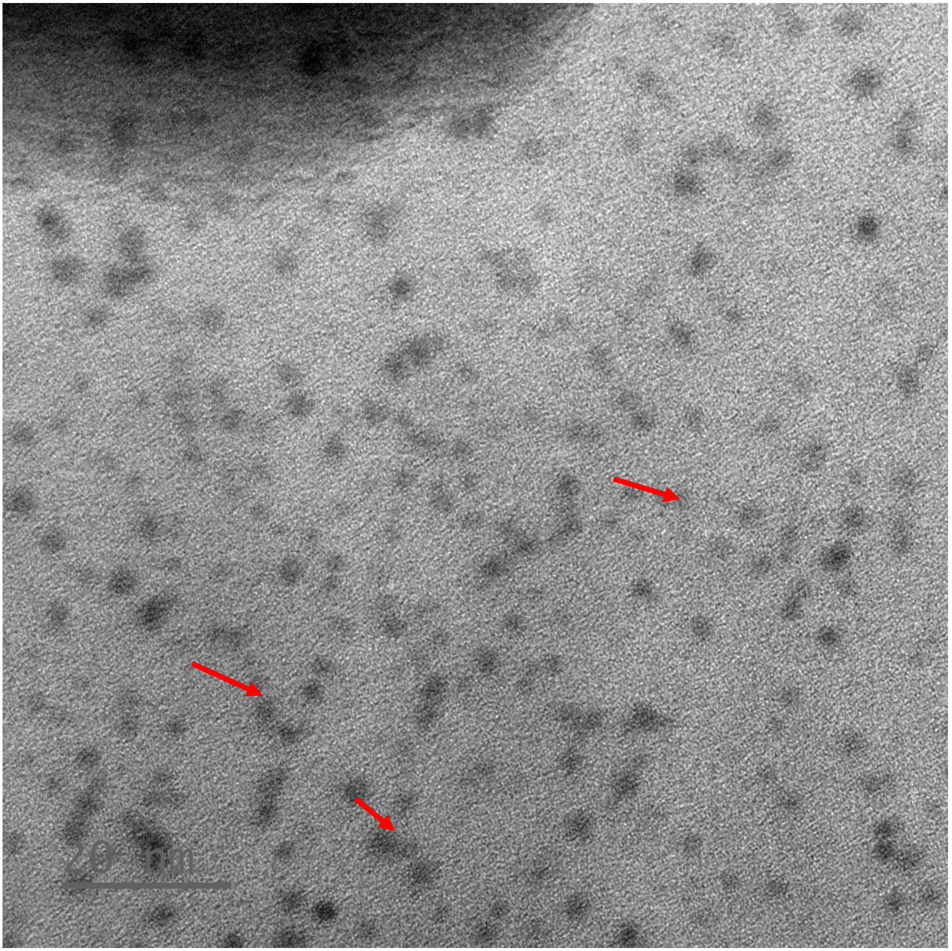


ODS Coating

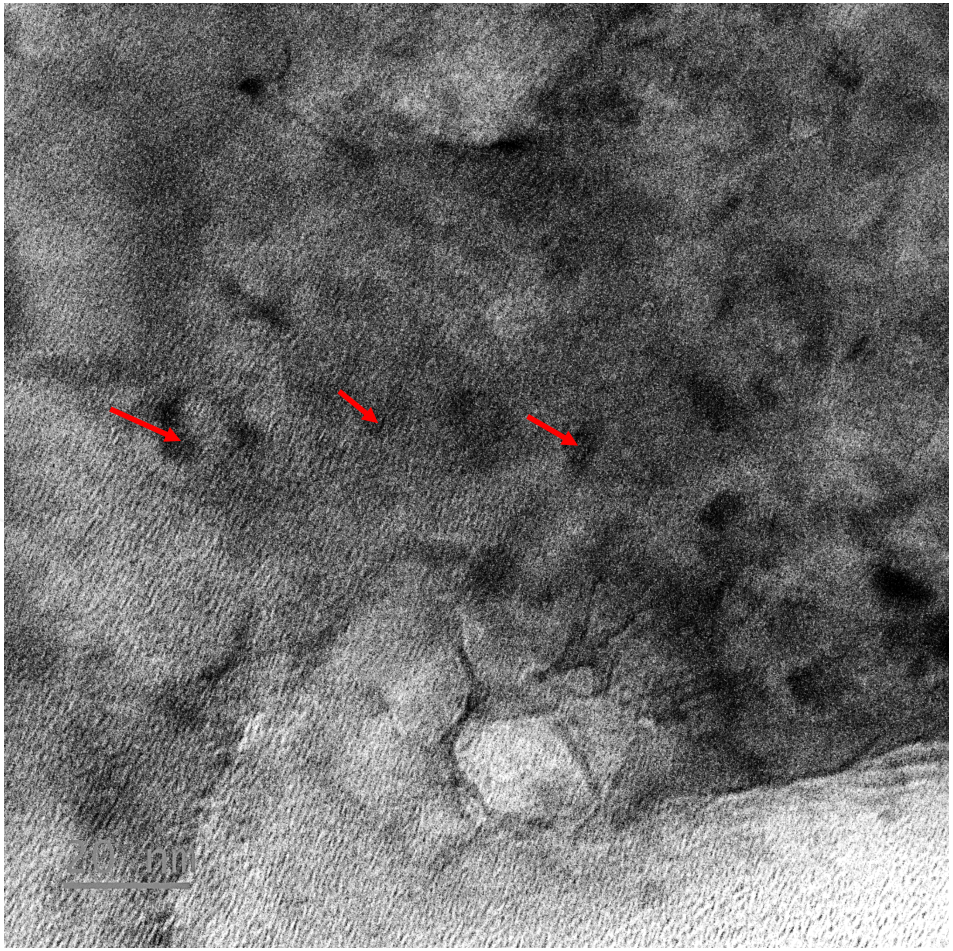
Nano-sized γ’phase preserved at ODS costing after 2200 cycles

ODS samples (MCB +BM)

Uniform distributed nanoparticles



Before thermal cyclic test

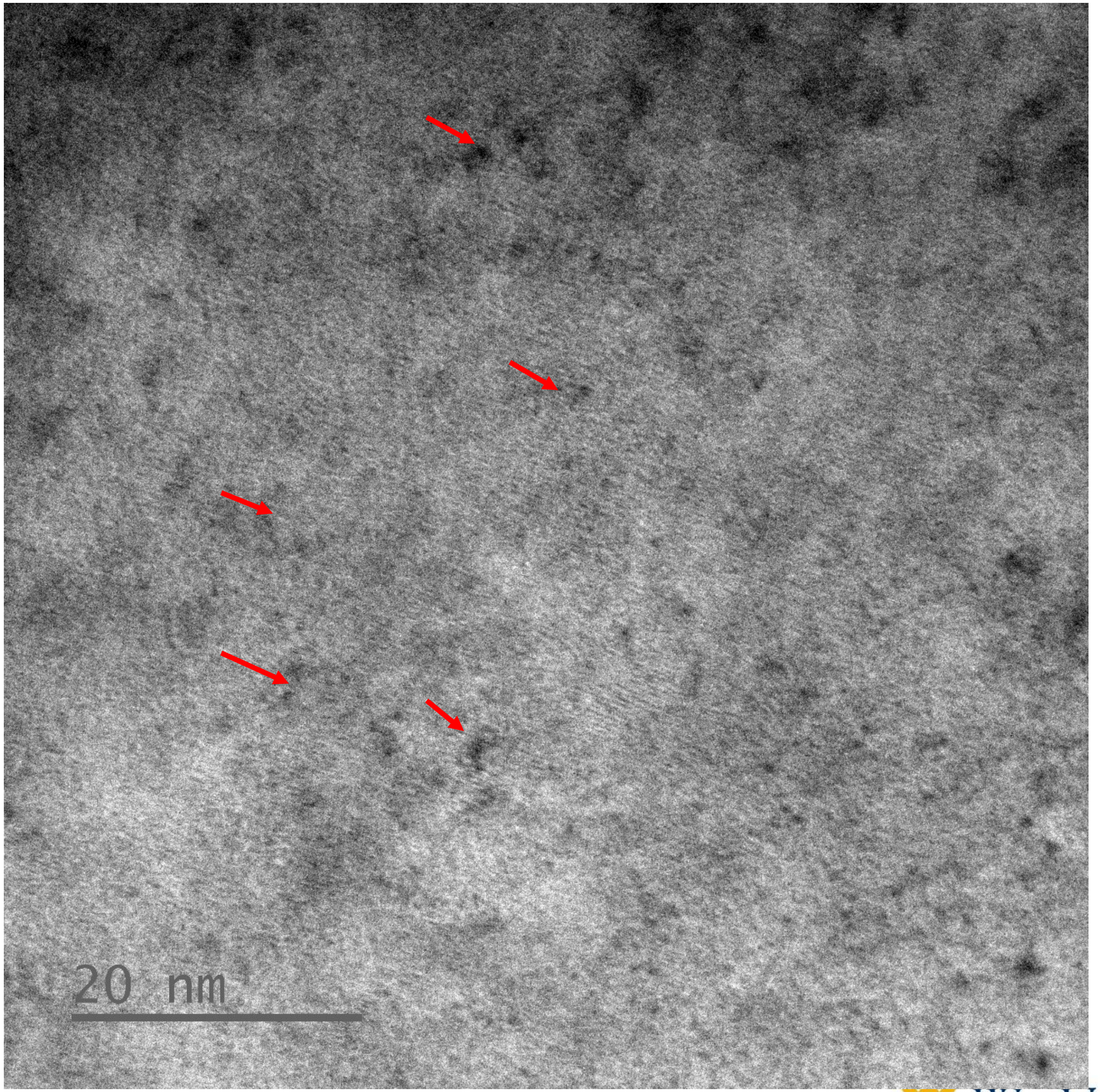


After 2000 cycles

***** Each cycle consists of moving test sample to the furnace within 15 minute and kept at 1100 ^{o}C for 45 minutes and moved out within 15 minutes, kept for 45 minute at room temperature.***

ODS samples (MCB only)

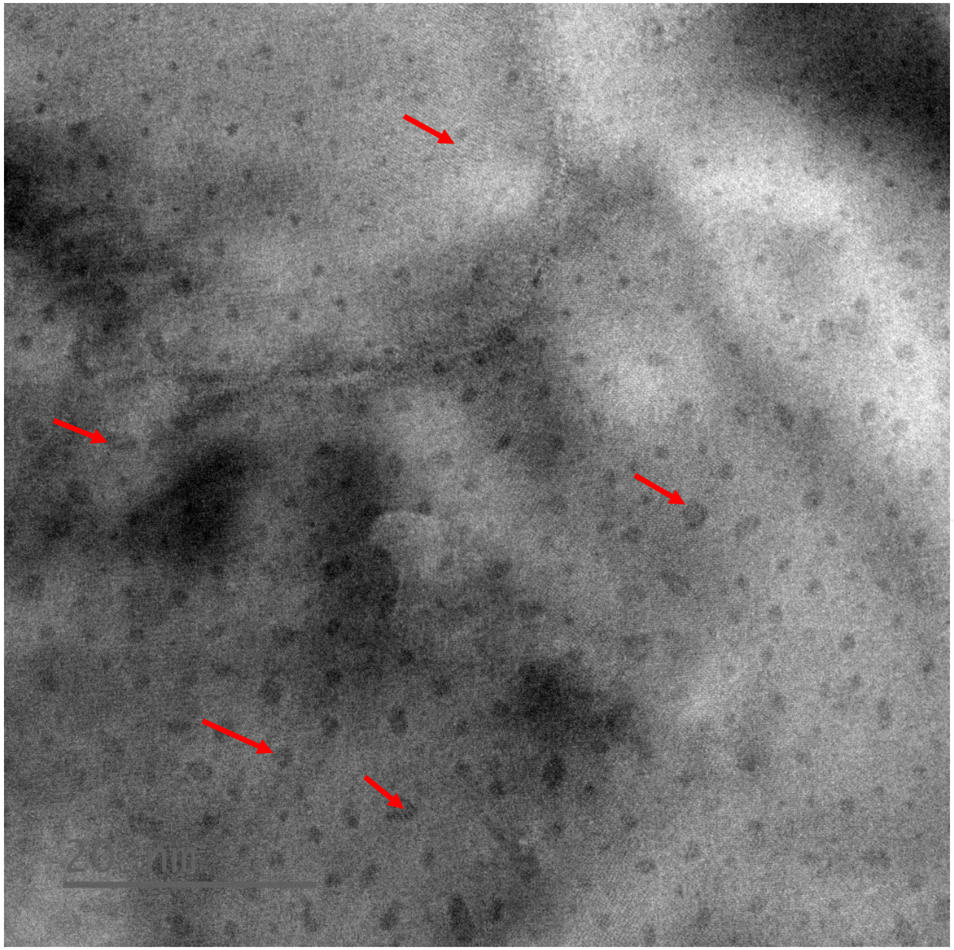
-As-printed



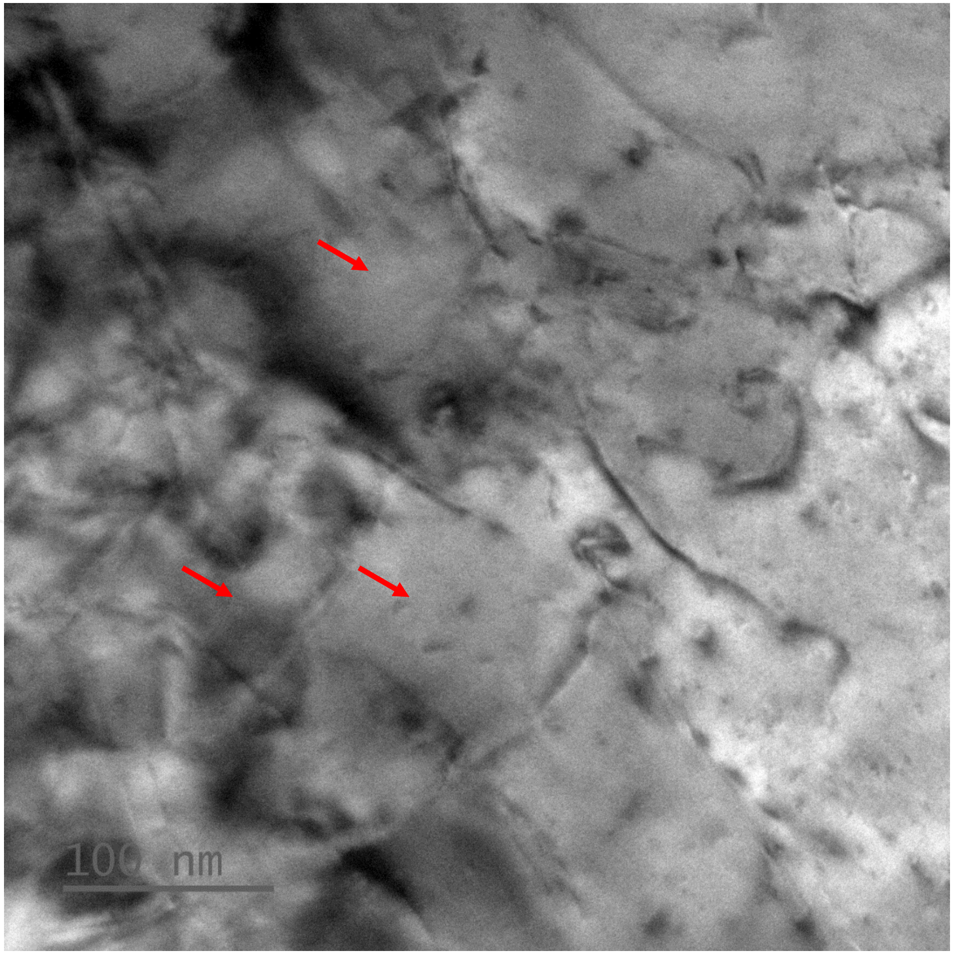
Uniform distributed nanoparticles

ODS samples (MCB only)

After 2000 thermal cycles



Uniform distribution of yttrium oxide nanoparticles



Fine grain structure

*** Each cycle consists of moving test sample to the furnace within 15 minute and kept at 1100 ^{o}C for 45 minutes and moved out within 15 minutes, kept for 45 minute at room temperature.*

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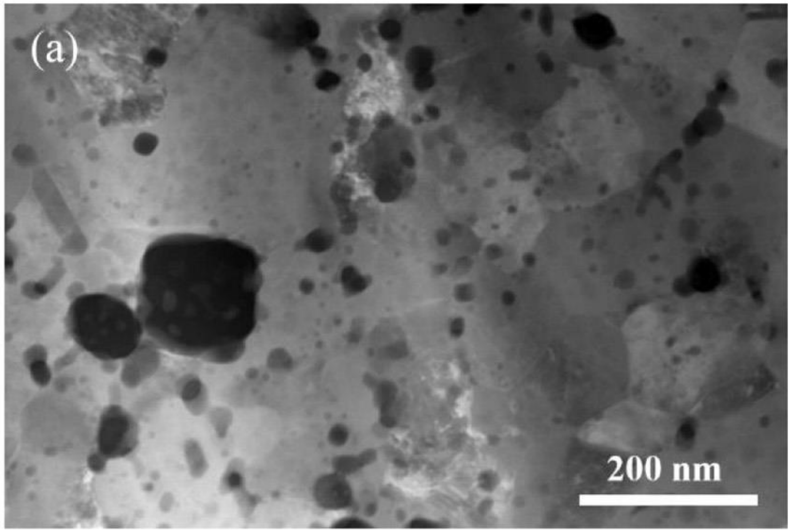
Comparison

- Material:
  Plasma rotating electrode process fabricated Rene 88DT powder with 5 vol.% Y_2O_3, ball milling for 48 hours
- Spark plasma sintering (SPS)
- Presence of coarse yttrium oxide particles



STEM bright field images of as-extruded R88-5Y alloy showing the dispersoids**

MCB-processed ODS alloys can achieve uniform dispersion of yttrium oxide nanoparticles.

** Xia, T.; Yang, C.; Zeng, W.; Xie, Y.; Zhang, Y.; Zhang, D.; Zhu, G.; Shu, D.; Lavernia, E. J. Dispersoids and γ′ Precipitates in an Ultrafine Grained René 88dt - 5vol.%y2o3 Alloy with Outstanding Thermal Stability. *Materials Characterization* **2018**, *141*, 139–147 DOI: 10.1016/j.matchar.2018.04.027

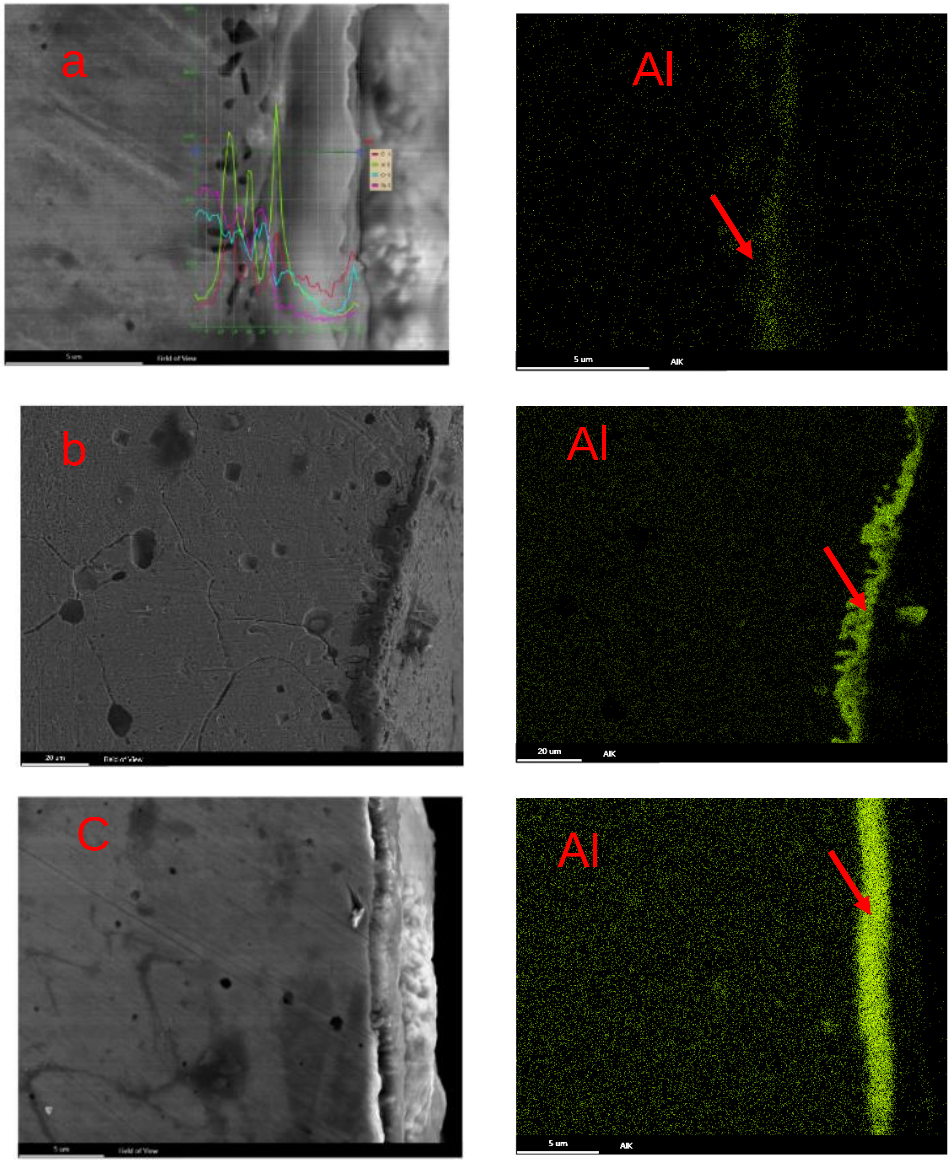
Long term oxidation

250W (MCB only(6)) ODS, 1280 cycles-Weight gain Scanning Speed 0.5 in/s,100 W In-situ Laser Heat Treatment

(a) 80 cycles

(b) 400 cycles

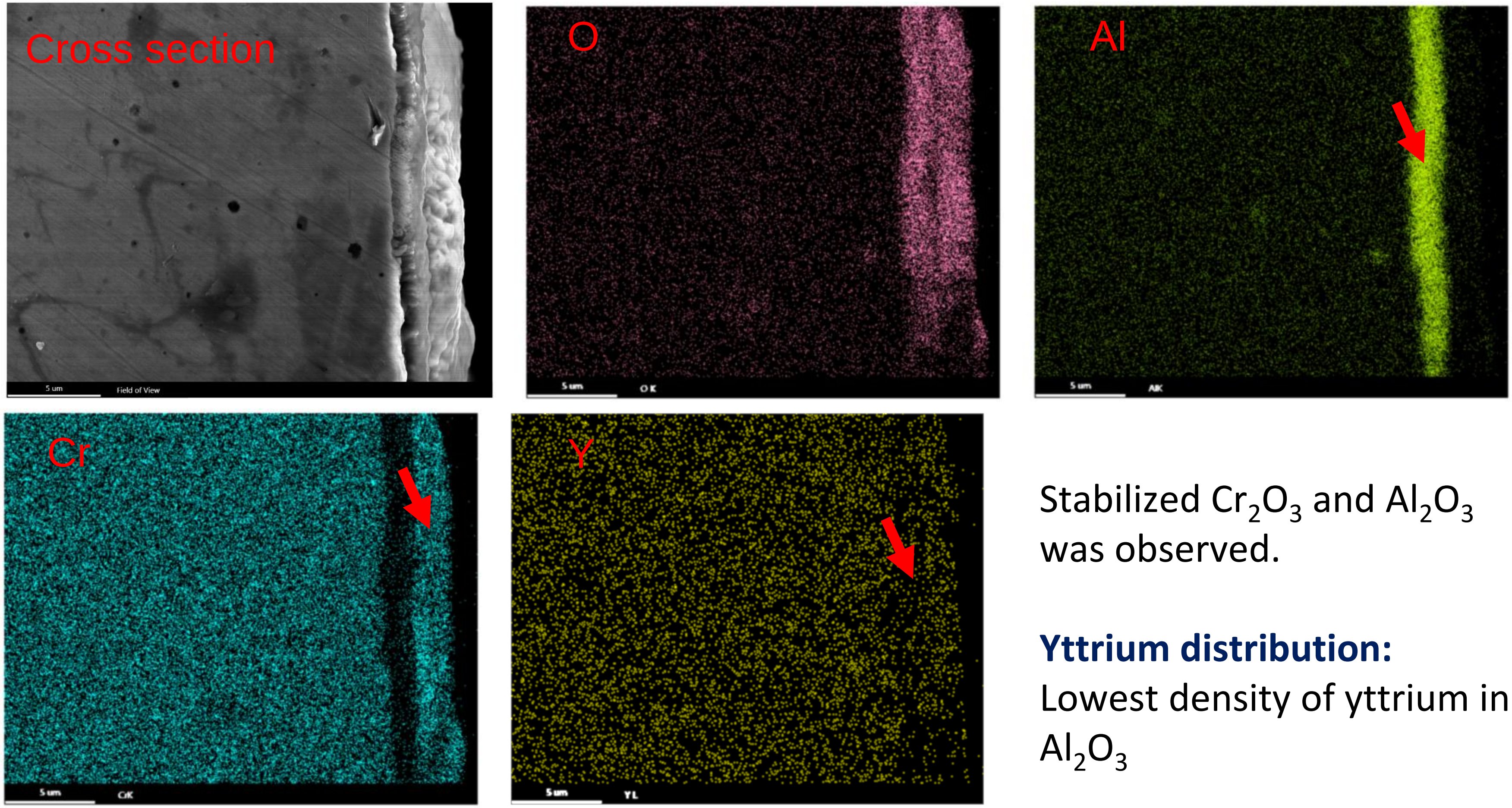
(c) 1280 cycles



External and continuous Al_2O_3 was detected at 80th cycles, 400th , and 1280th cycles.

Cross section of oxide layer

Long term oxidation (MCB only)
1280 cycles



Stabilized Cr_2O_3 and Al_2O_3 was observed.

Yttrium distribution:
Lowest density of yttrium in Al_2O_3

Oxide layer and corresponding elemental distribution

*** Each cycle consists of moving test sample to the furnace within 15 minute and kept at 1100 ^{o}C for 45 minutes and moved out within 15 minutes, kept for 45 minute at room temperature.*

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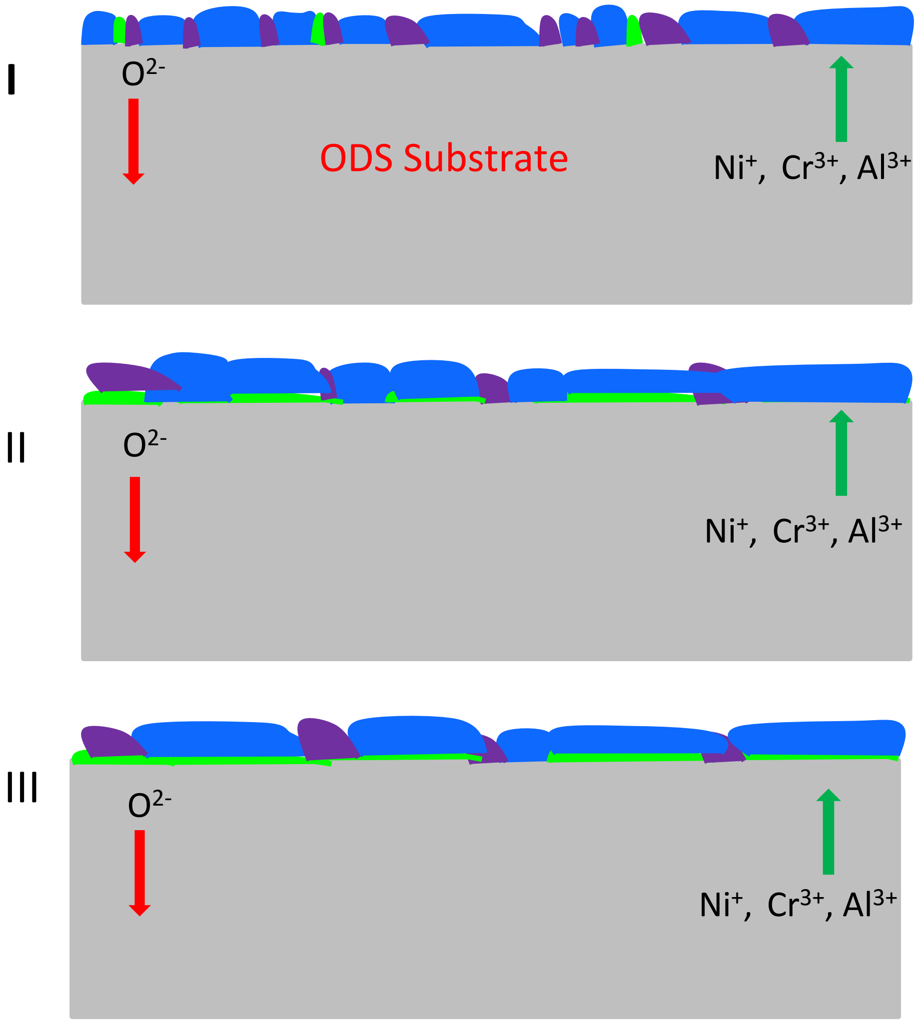
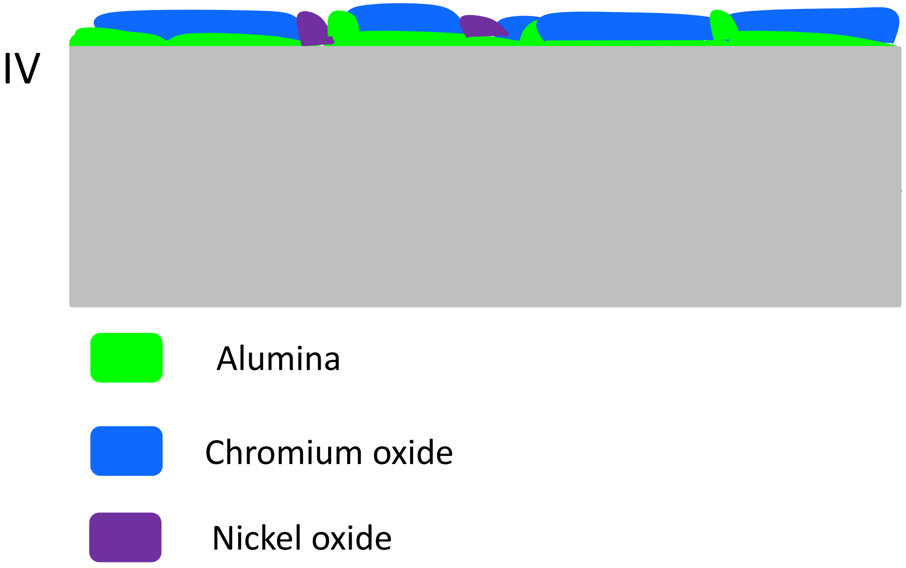
Schematic Diagram of Oxidation Process of AM-printed ODS alloys using MCB-processed ODS power



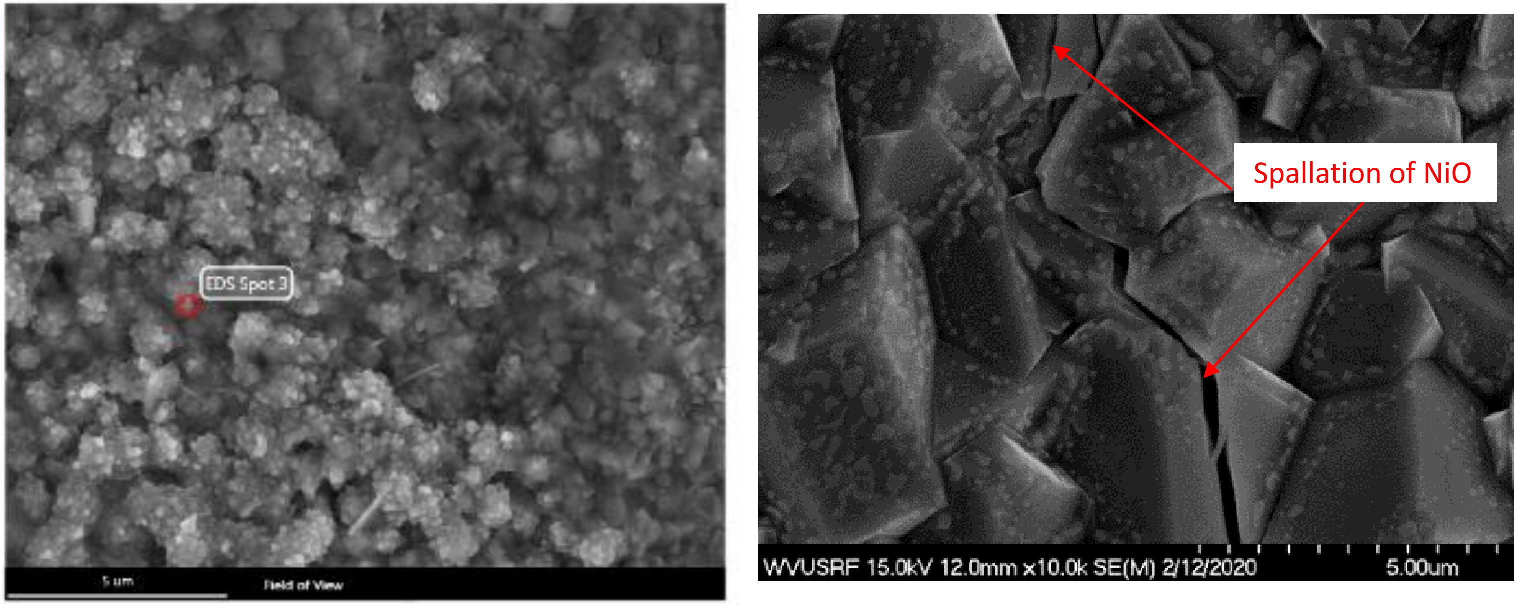


Oxidation mechanism：

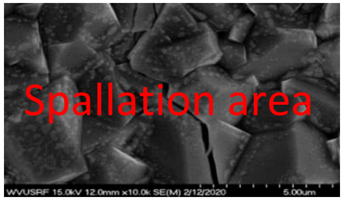
Chromium and nickel oxide formed first, then alumina exists underneath the chromium oxide layer. No internal oxidation (underneath surface oxide layer) was observed.

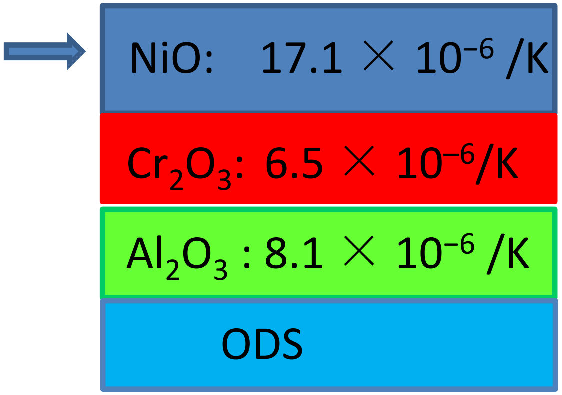
NiO Spallation analysis

Nio spallation along grain boundary and inside the grains



Oxide morphology at the (a) 1st cycles, and (b) 1280th cycle





NiO: 17.1×10^{-6} /K

Cr_2O_3: 6.5×10^{-6}/K

Al_2O_3 : 8.1×10^{-6} /K

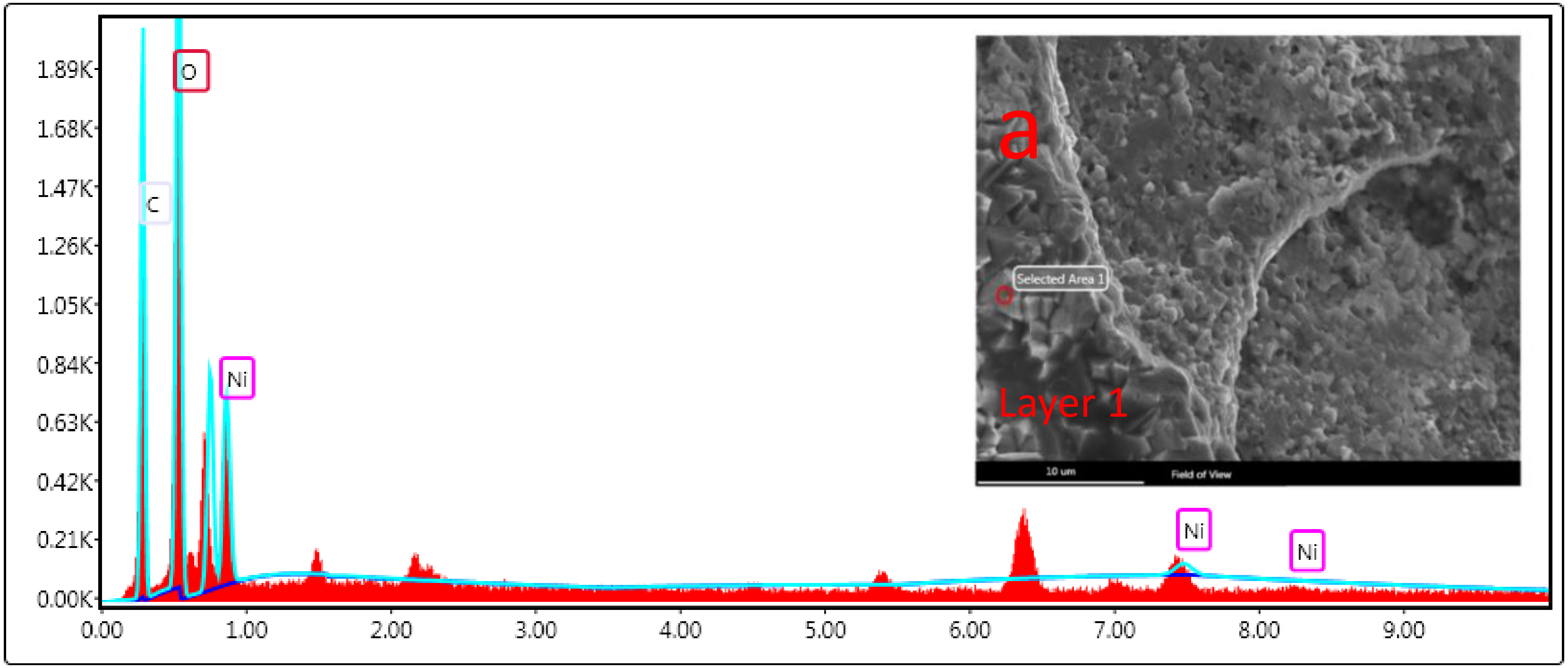
ODS

NiO spallation caused by thermal expansion mismatch.

How about the Cr_2O_3 and Al_2O_3 ?

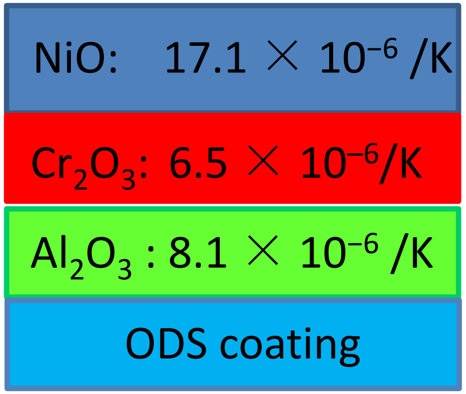
Schematic of oxide layer and corresponding thermal expansion coefficient of oxides

Spallation Analysis of Surface Oxide Layers



Status: Idle CPS: 720 DT: 2.6 Lsec: 108.4 926 Cnts 0.290 keV Det: Octane Pro

EDS analysis of surface oxides at the 1280th cycle



NiO: 17.1 × 10^{-6} /K

Cr_2O_3: 6.5 × 10^{-6}/K

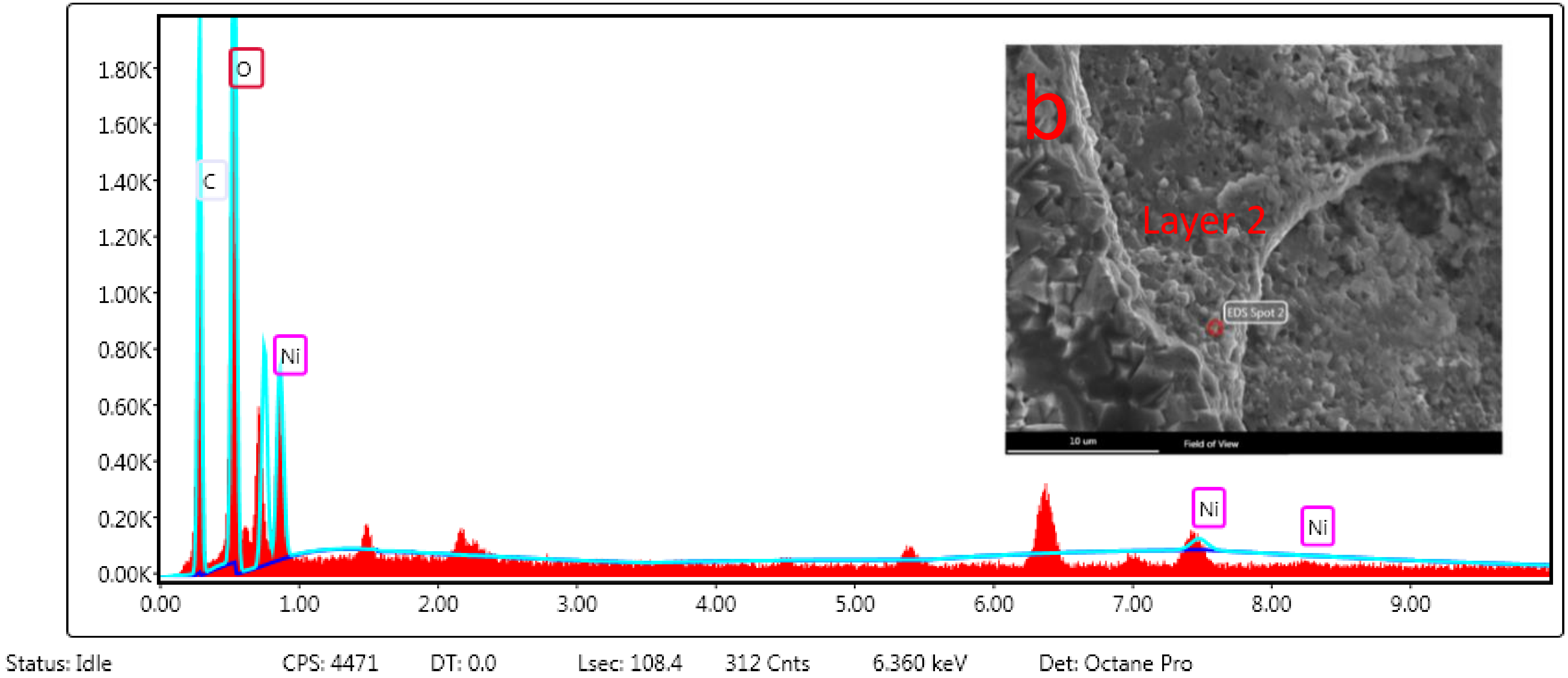
Al_2O_3 : 8.1 × 10^{-6} /K

ODS coating

Layer 1: Pure NiO

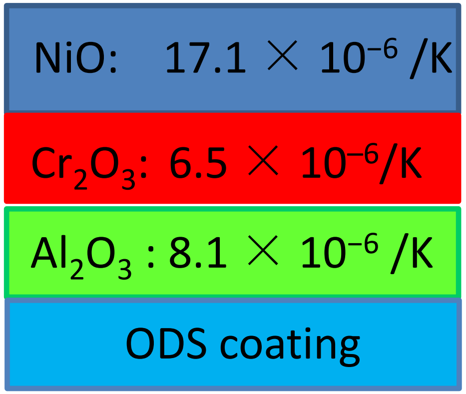
Schematic of oxide layer and corresponding thermal expansion coefficient of oxides

Spallation Analysis of Surface Oxide Layers



Status: Idle CPS: 4471 DT: 0.0 Lsec: 108.4 312 Cnts 6.360 keV Det: Octane Pro

EDS analysis of surface oxides at the 1280th cycle



NiO: 17.1 × 10^{-6} /K

Cr_2O_3: 6.5 × 10^{-6}/K

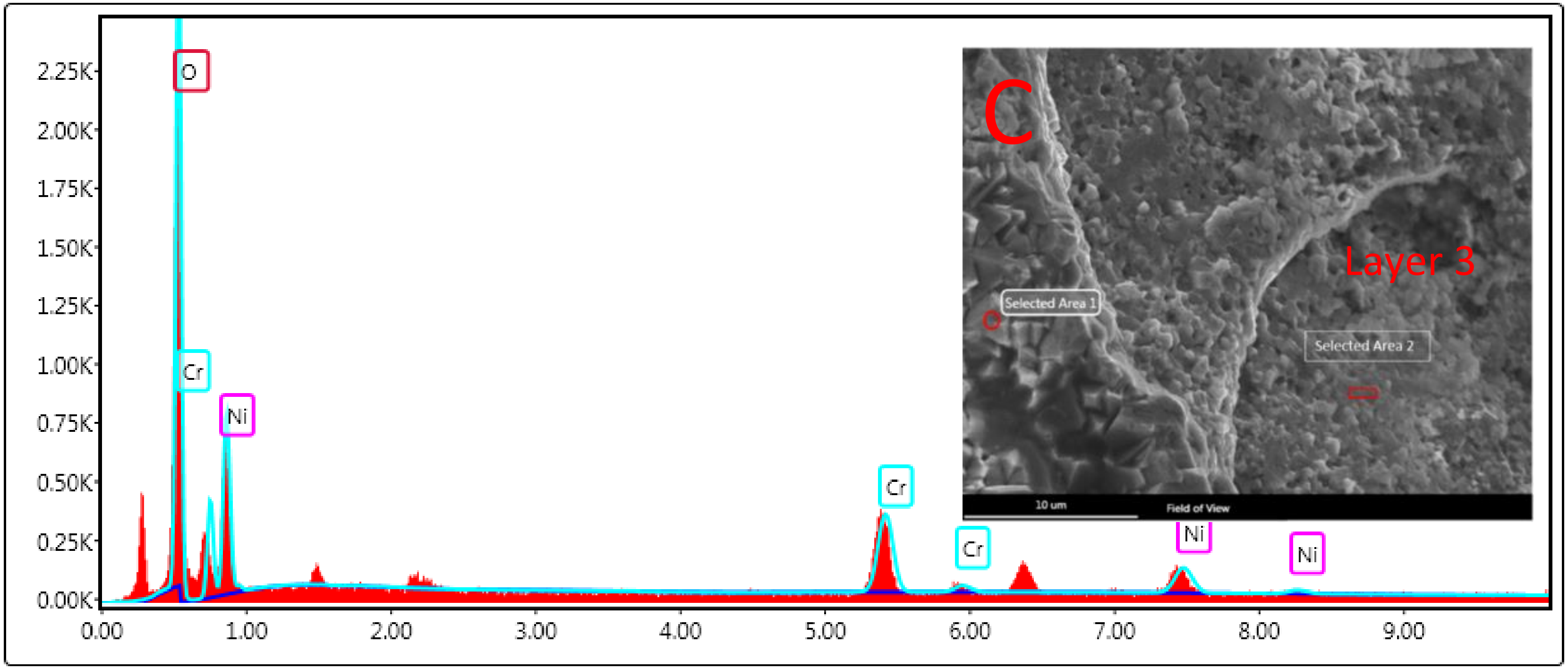
Al_2O_3 : 8.1 × 10^{-6} /K

ODS coating

Layer 2 :Pure NiO

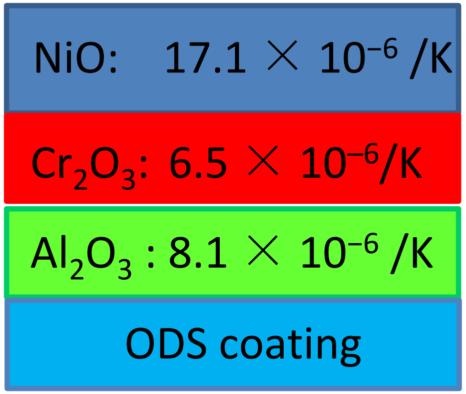
Schematic of oxide layer and corresponding thermal expansion coefficient of oxides

Spallation Analysis of Surface Oxide Layers



Status: Idle CPS: 677 DT: 1.9 Lsec: 90.6 116 Cnts 6.420 keV Det: Octane Pro

EDS analysis of surface oxides at the 1280th cycle



NiO: 17.1 × 10^{-6} /K

Cr_2O_3: 6.5 × 10^{-6}/K

Al_2O_3 : 8.1 × 10^{-6} /K

ODS coating

Layer 3: NiO and Cr_2O_3 mixture

Schematic of oxide layer and corresponding thermal expansion coefficient of oxides

Summary

- Developed MCB + BM and MCB only methods for production of ODS powders suitable for AM applications.

- AM-printed ODS 754 samples showed uniform distribution of strengthening nano-sized yttrium oxide particles in alloy matrix, with excellent thermal stability after over 1000 thermal cycles.

- Excellent oxidation resistance of AM-printed ODS 754 alloy owing to the presence of a continuous protective and durable alumina layer