NATIONAL ENERGY TECHNOLOGY LABORATORY

# 2015 University Turbine Systems Research Workshop

## Design, Fabrication and Performance Characterization of Near-Surface Embedded Cooling Channels with an Oxide Dispersion Strengthened (ODS) Coating Layer

Award Number: DE-FE0025793
Period of Performance :10/1/15 to 9/30/18

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***University of Pittsburgh***

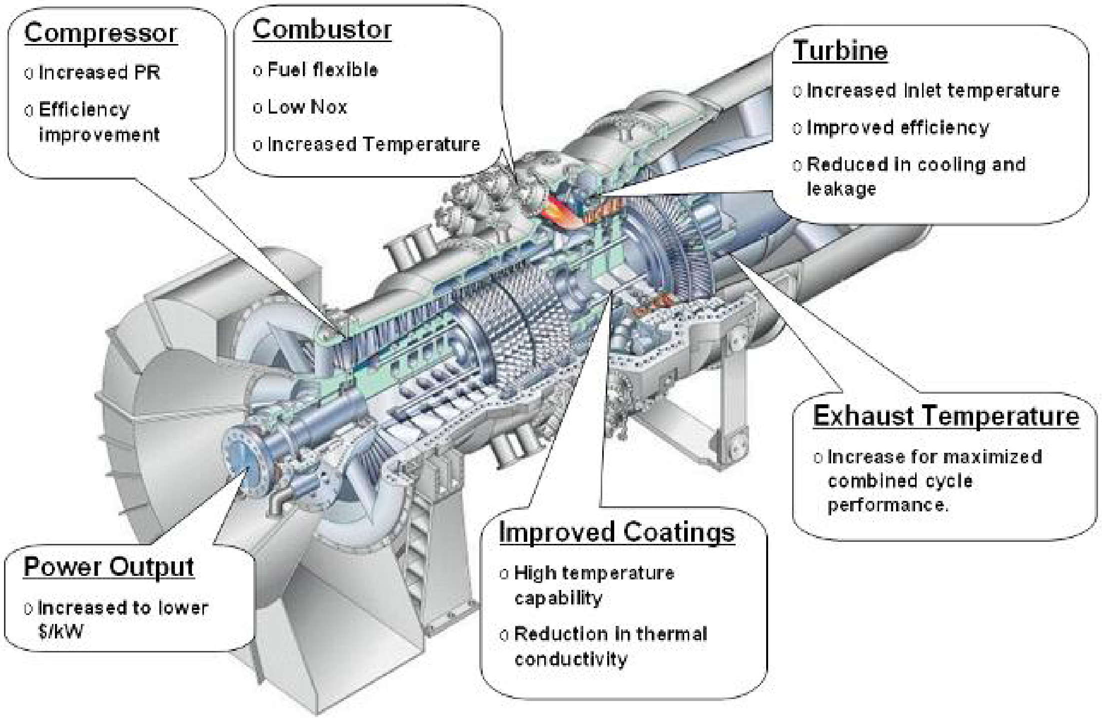
***Bruce S. Kang***
***West Virginia University***

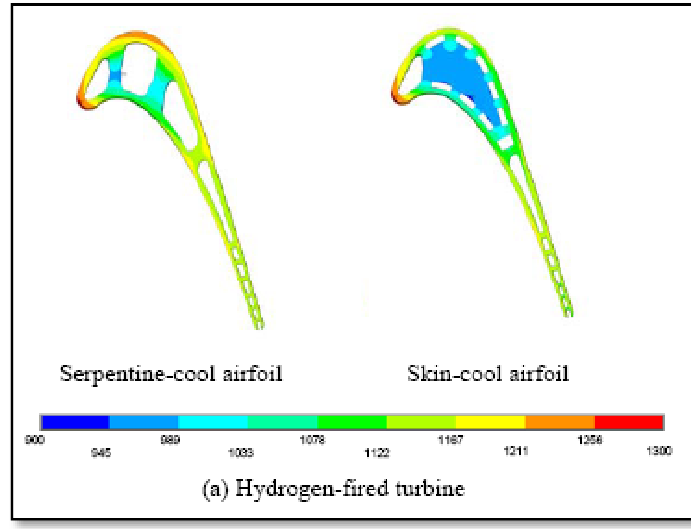
# Outlines

- ***Introduction and Background***
- *Challenges, Objectives, Benefits of Technology, Research Task Plan*
- *Tasks*
  1. *Advanced Impingement*
  2. *ODS Powders Fabrication and Characterization*
  3. *ODS Coating (AM Assisted) (Preliminary results)*
  4. *Microstructural and Mechanical Properties Characterization*
  5. *Detailed Experimental Measurement and Validation*

# Technical Background/Approach

## Targeted Areas of R&D



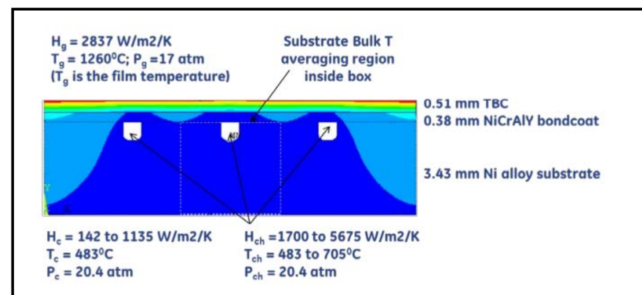




*Siw, S.C., Chyu, M.K., Karaivanov, V.G., Slaughter, W.S., and Alvin, M.A., 2009, "Influence of Internal Coolinjg Configuration on Metal Temperature Distributions of Future Coal-Fuel Based Turbine Airfoils," ASME Turbo Expo 2009, Paper No. GT2009-59829.*

***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℃, compared to conventional serpentine cooling designs***




*Skin Cooled Bulk Substrate Metal Temperature as a Function of Channel Heat transfer Coefficient and Coolant Temperature*

*Bunker, R.S., 2013, "Gas Turbine Cooling: Moving from Macro to Micro Cooling," ASME Turbo Expo 2013, Paper No. GT2013-94277*

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# Near Surface Embedded Channel Cooling

***Technical Challenges***

- *Design optimal aerothermal configuration*
- *ODS powder fabrication, ODS layer deposition processing*
- *Scale-up and commercial manufacturing of test articles*

## Project Objectives

- **To design highly-heat-transfer augmented and manufacturable internal cooling channels for the development of NSECC. The two heat transfer augmentation techniques to be explored first are:**
  **(a) advanced impingement cooling**
  **(b) zig-zag channel configurations**

- **To produce ODS particles within 45-105 microns which will be used in an additive manufacturing (AM) process based on laser deposition to build NSECC test modules**

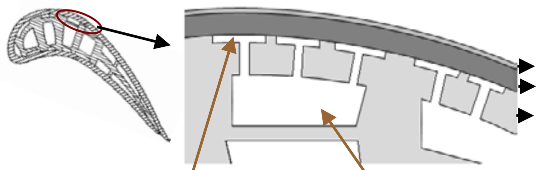
- **To develop fabrication process through additive manufacturing for coating either a densified ODS layer over a grooved single crystal superalloy substrate to form an enclosed NSECC, or an ODS layer with cooling channels embedded within the ODS layer atop a single crystal superalloy metal substrate**

- **To characterize the thermal-mechanical material properties and cooling performance of the AM produced ODS-NSECC protective module under high-temperature conditions. Comparison with the state-of-the-art cooling technology will be made and the performance improvements over the standards will be assessed**

# Project Work Breakdown Structure

## Enhanced Heat Removal Capability

*Current NSECC design leads to 50-70% over existing internal cooling technologies.*
*Additional improvement is projected.*



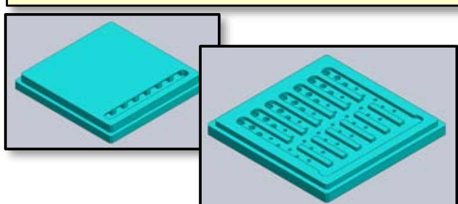

***Near Surface Embedded Cooling Channel (NSECC )***

## Novel Metallic ODS Surface Coating

- *Ultra-High Temperature (1200 °C) Strength*
- *Oxidation Resistance*
- *Significant challenges in traditional manufacturing*

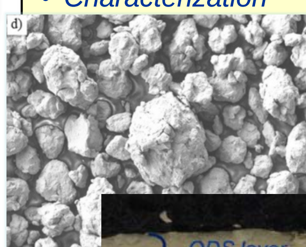
### Task 1 – Advanced Impingement

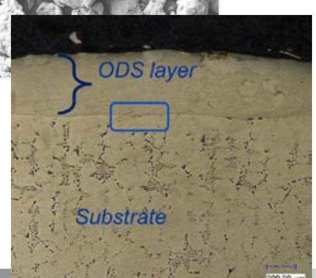
- *Design, CFD modeling & scaled testing*
- *Advanced impingement*



### Task 2 – ODS Powder Fabrication and Characterization

- *ODS powders fabrication*
- *Characterization*




### Task 3 – ODS Coating (AM Assisted)

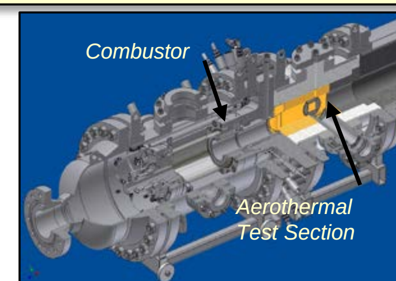
- *Process development and optimization*

### Task 4 – Microstructural and Mechanical Properties Evaluation

- *Thermal Cyclic Tests, Micro-Indentation Tests*
- *OM, EDX, SEM, XRD,TEM*

### Task 5 – Design Integration & Testing

- *High Temperature, Pressurized Testing (NETL)*
- *High Temperature Testing Facilities (Solar Turbines, Inc.)*



# Research Task Plan

Solar Turbines
A Caterpillar Company

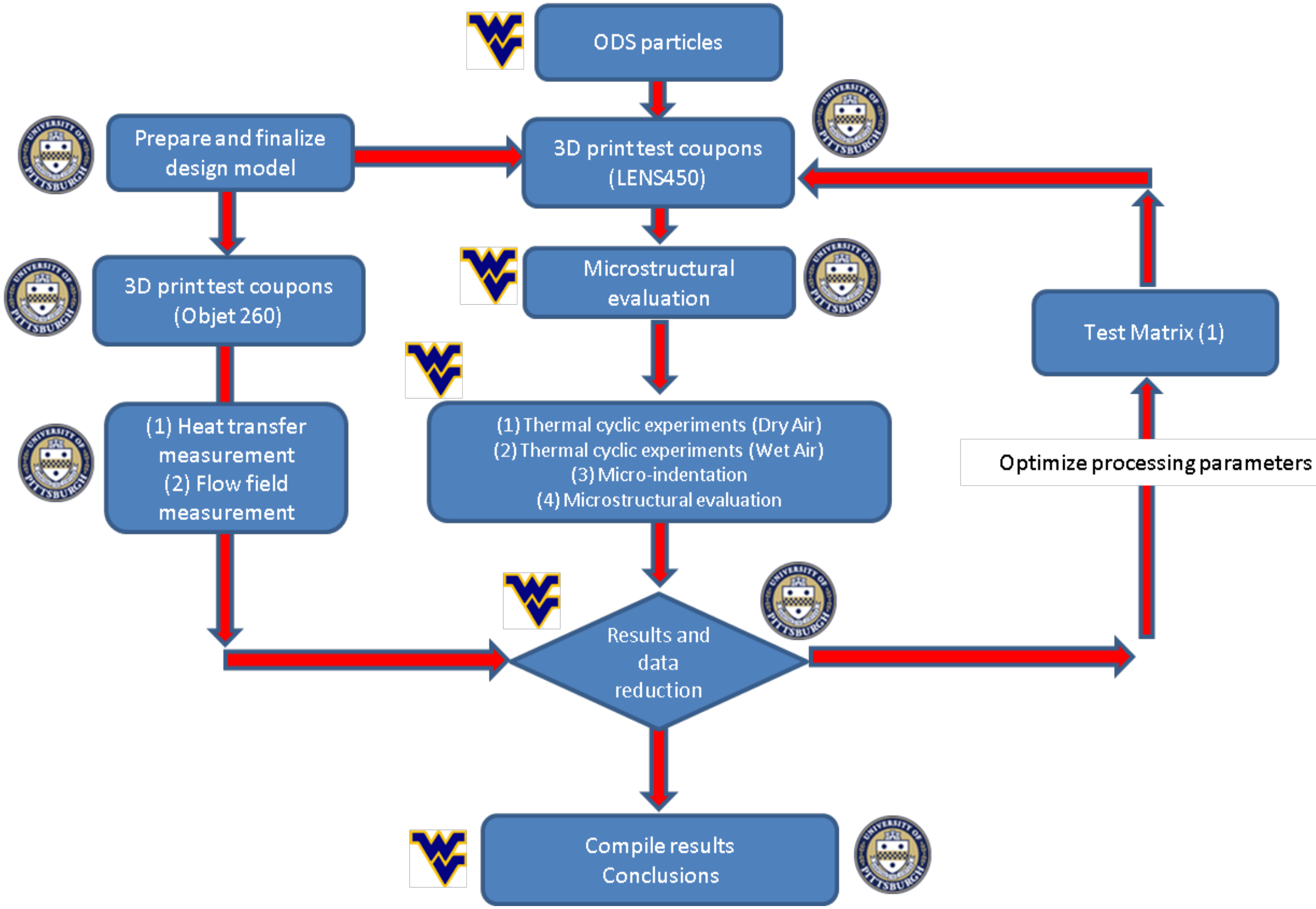
HOSOKAWA MICRON

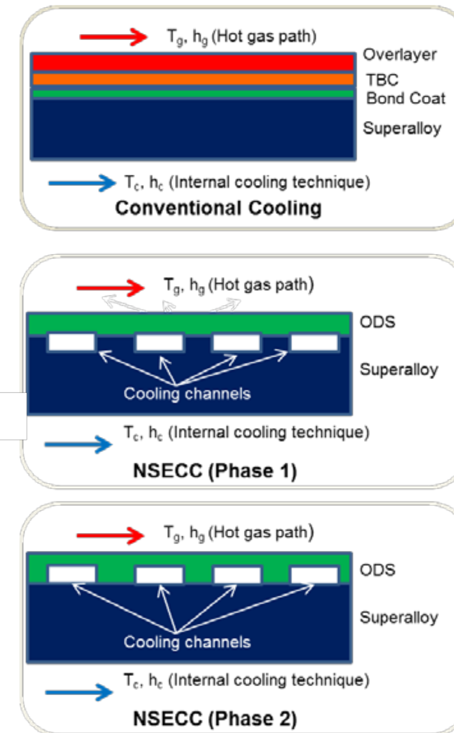
**Design, Fabrication and Performance Characterization of Near-Surface Embedded Cooling Channels with an Oxide Dispersion Strengthened (ODS) Coating Layer**

University of Pittsburgh

West Virginia University

**Research Task Plan (3 years)**



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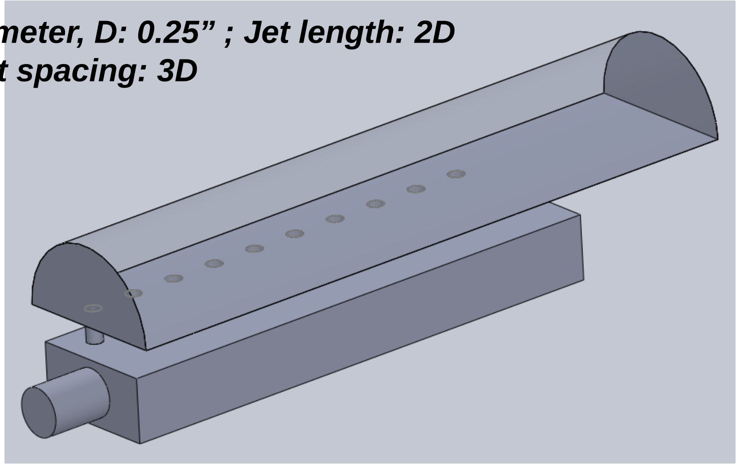
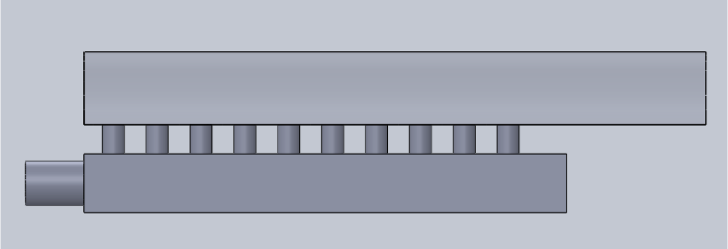
# Task 1 Advanced Impingement

## *Test #1 Inline $90^o$ jets*

***Jet diameter, D: 0.25" ; Jet length: 2D***
***Inter-jet spacing: 3D***



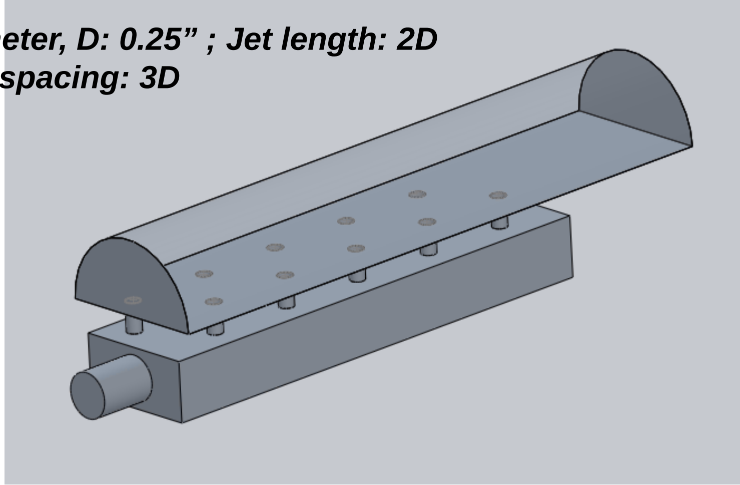
## *Test #3 Inline $90^o$ jets*



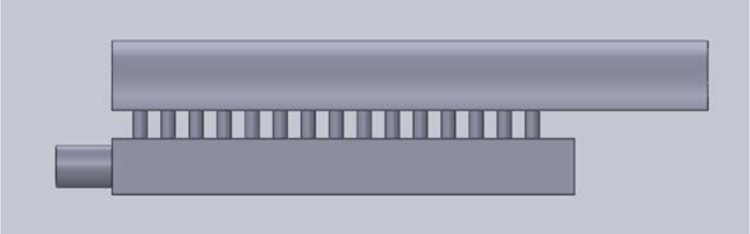
***Jet diameter, D: 0.375" ; Jet length: 1.33D***
***Inter-jet spacing: 2D***

## *Test #2 Staggered $90^o$ jets*

***Jet diameter, D: 0.25" ; Jet length: 2D***
***Inter-jet spacing: 3D***



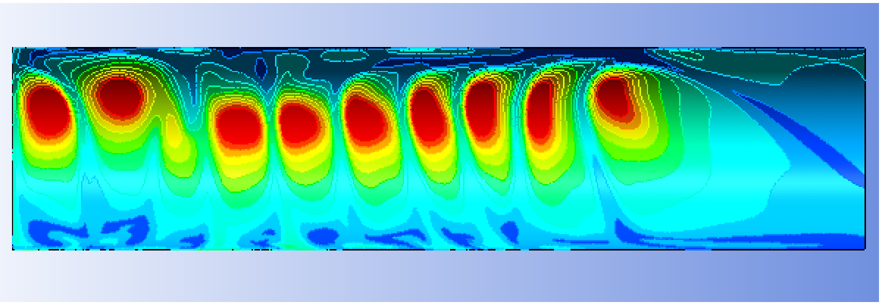
## *Test #4 Inline $90^o$ jets*



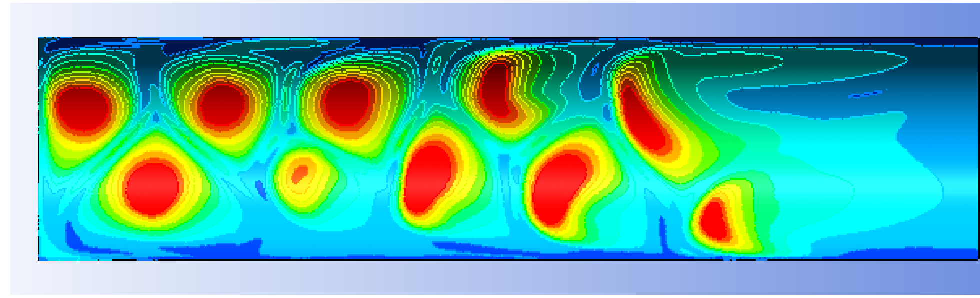
***Jet diameter, D: 0.25" ; Jet length: 2D***
***Inter-jet spacing: 2D***

# Task 1 Advanced Impingement

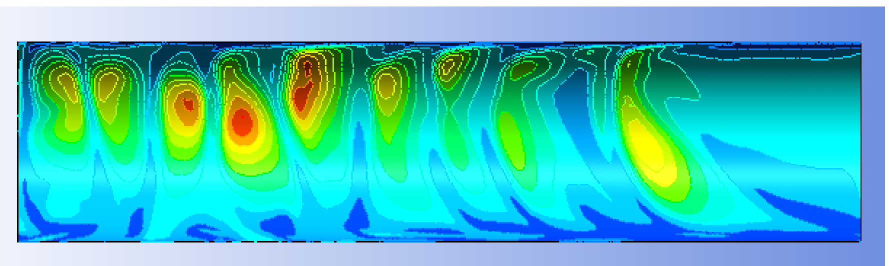
*Test #1*



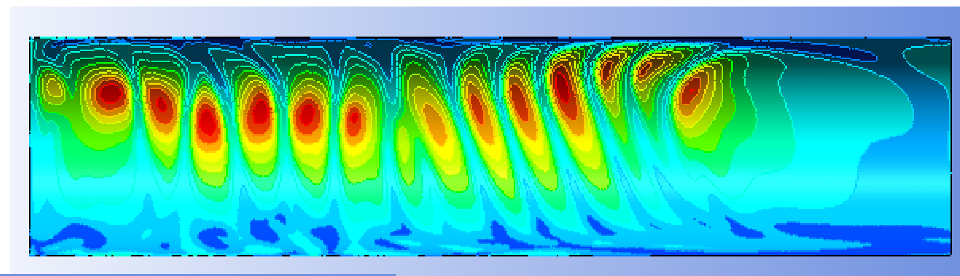
*Test #2*

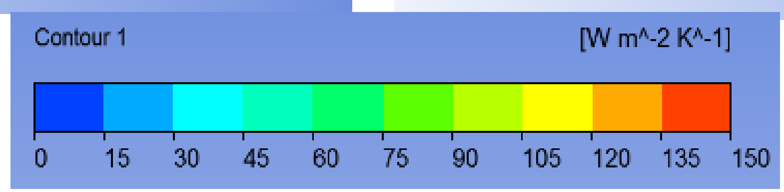


*Test #3*



*Test #4*






- *Test#1 and #2 – Significant impingement from the jets*
- *Test#3 - larger jet, reduce the bulk velocity, very minimal impingement effects, most uniform heat transfer distribution among all tested cases*
- *Test#4 - total number of jets is 50% more than other test cases - impingement effects are preserved*

- ***Design and fabricate scaled-up test section for detailed experimental study, for validation with CFD results***

# Task 1 Advanced Impingement

**Objective: Develop internal air foil cooling technologies capable of yielding an elevated heat transfer enhancement with reasonable manufacturability**

- Leading Edge Cooling - screw (helical) cooling

**Challenge:** Some of the promising intricate vortex generating geometries which were studied in the up-scaled research models cannot always be reproduced in actual size blade castings or are very sensitive to the manufacturing tolerances, particularly when small internal holes and sharp edge features are required

**Why? Advantages?**

- Swirling flow structure, move radially, generating 3D screw-shaped flow
- Optimized screw cooling configurations resulted in more uniform cooling ~ more efficient than direct impingement
- Less sensitive to fabrication tolerances than highly effective internal cooling technique,
  ~ more attractive for industrial applications

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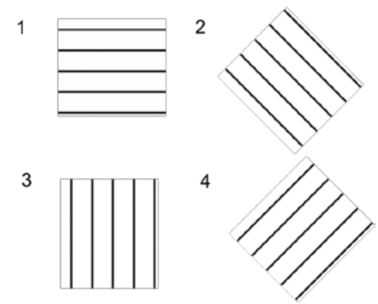
# Task 3: ODS Coating (AM Assisted)

**Objective: Develop and optimize processing parameters for fabricating an ODS layer atop of substrate**

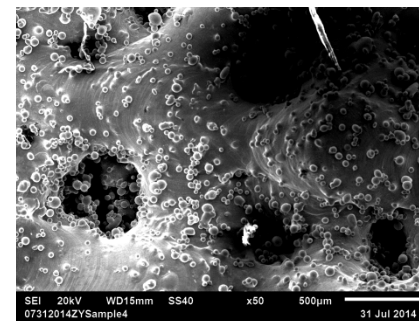
### *Approach*

- *Produce a series of test coupon with densified ODS layer atop of single crystal nickel based superalloy substrate using varying major parameters.*
  *- Laser power, powder feeding rate, deposition speed, hatch spacing, hatch pattern*

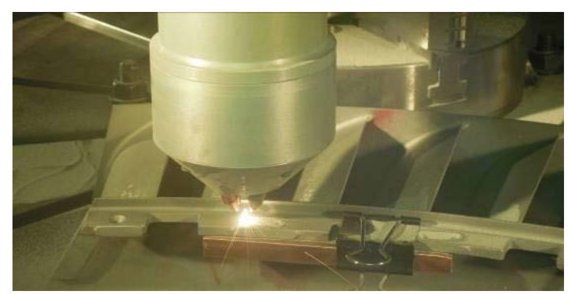

LENS450 (Direct Laser Deposition Process)




Hatch pattern

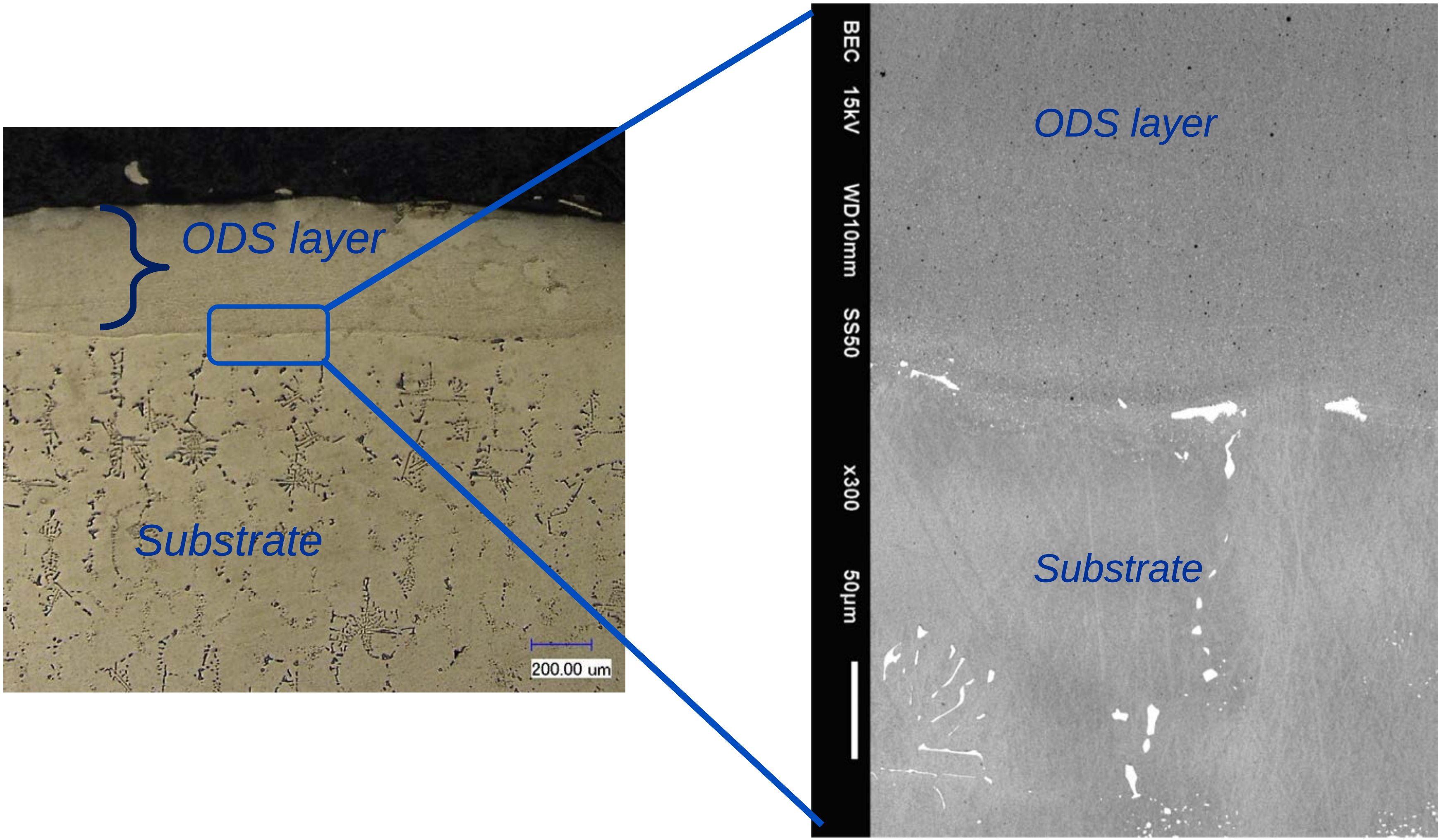



Hatch spacing



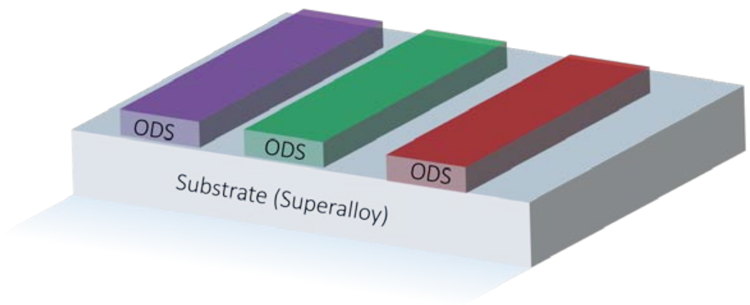
Laser power

# Task 3: ODS Coating (AM Assisted)

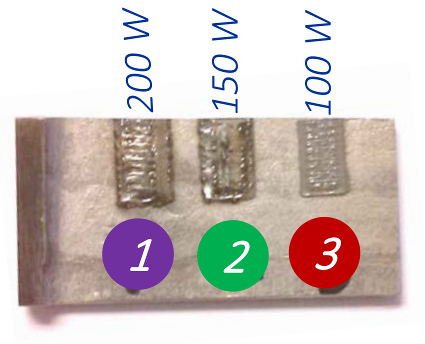



- Preliminary result
  Test coupon – single crystal nickel based superalloy with densified ODS layer

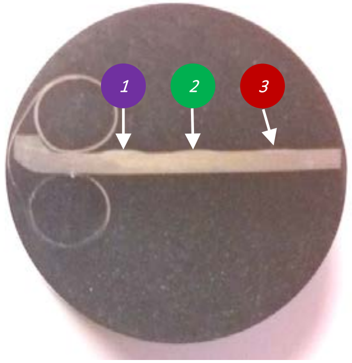
# Task 3: ODS Coating (AM Assisted)



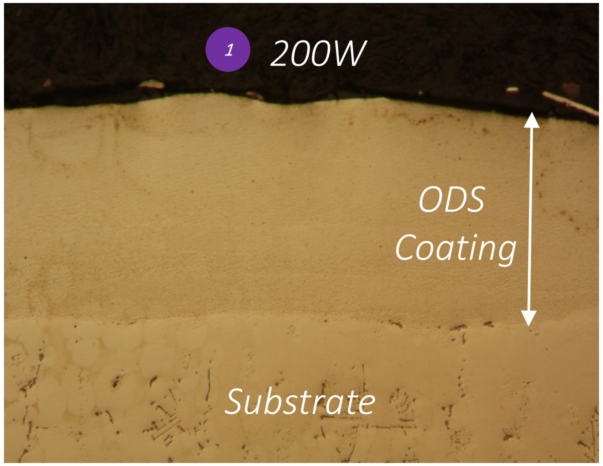

SCHEME

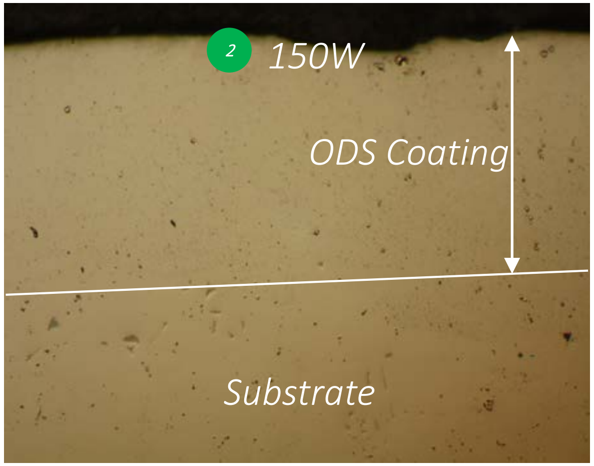


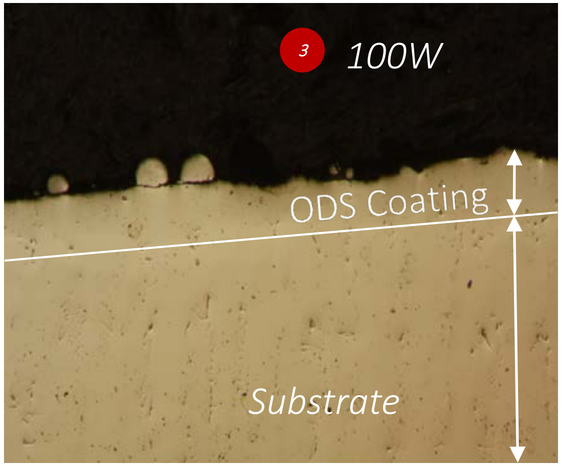

TOP VIEW




SIDE VIEW



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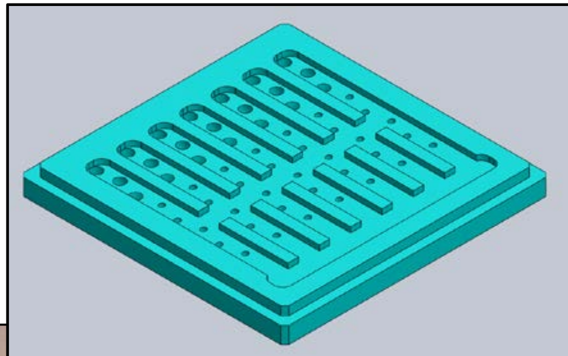
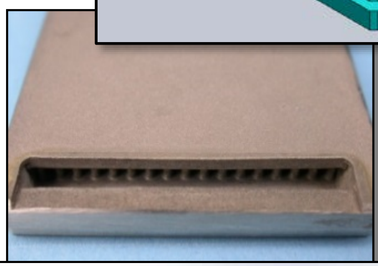
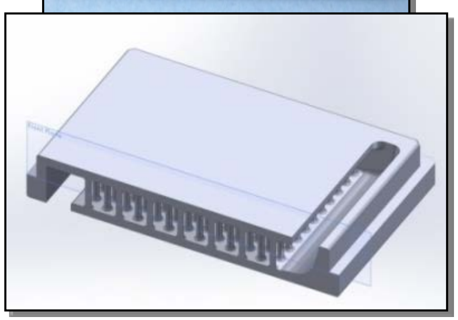
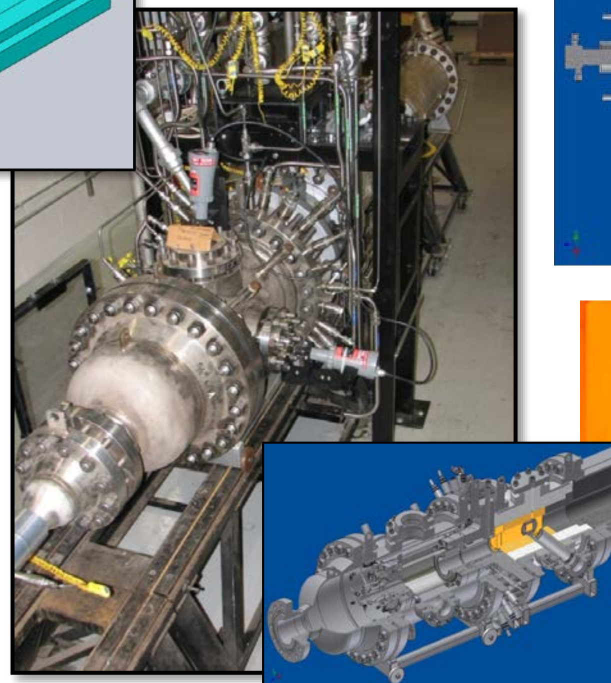
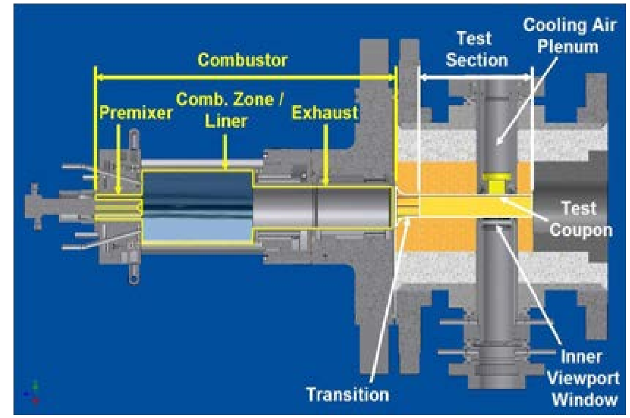
# *Task 5: Detailed Experimental Measurements and Validation*

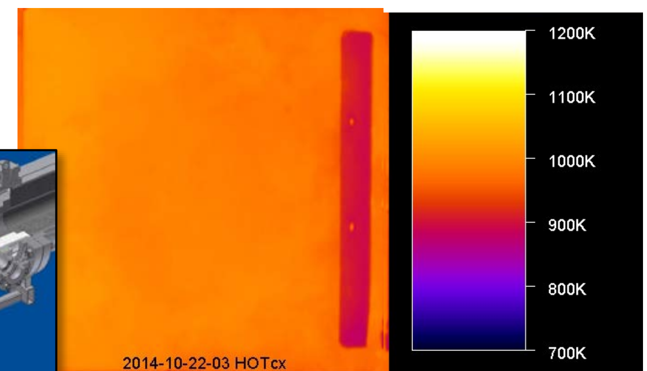











- Conduct HT/P testing at 1100°C demonstrating ~50-70% enhancement of NSECC over smooth channel and pin-fin arrays
- Further optimization of the NSECC configuration for enhanced cooling performance
- Address additive manufacturing capabilities for production of parts

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# Project Timeline

| Task Description | 1st Year | | | | 2nd Year | | | | 3rd Year | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1st quarter | 2nd quarter | 3rd quarter | 4th quarter | 1st quarter | 2nd quarter | 3rd quarter | 4th quarter | 1st quarter | 2nd quarter | 3rd quarter | 4th quarter |
| Bi-weekly, quarterly and final reports | | | | | | | | | | | | |
| **Dr. M.K. Chyu, Dr. S.C. Siw - University of Pittsburgh (Pitt)** | | | | | | | | | | | | |
| Project management and planning | | | | | | | | | | | | |
| Task 1.1 Heat Transfer Characterization of Optically Clear Scaled Test Section and Test Coupon | | | | | | | | | | | | |
| Task 1.2 Development and Process Optimization to Coat an ODS Layer on Single Crystal Superalloy Substrate | | | | | | | | | | | | |
| Task 1.3. Heat Transfer Characterization of ODS/NSECC Protected Single Crystal Superalloy Coupon under High Temperature Environment | | | | | | | | | | | | |
| **Dr. Bruce S. Kang - West Virginia University (WVU)** | | | | | | | | | | | | |
| Task 2.1 Fabrication approach to produce ODS powder | | | | | | | | | | | | |
| Task 2.2. Thermal cyclic experiment on ODS Alloy Specimens | | | | | | | | | | | | |

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# Task 2: ODS Powders Fabrication and Characterization

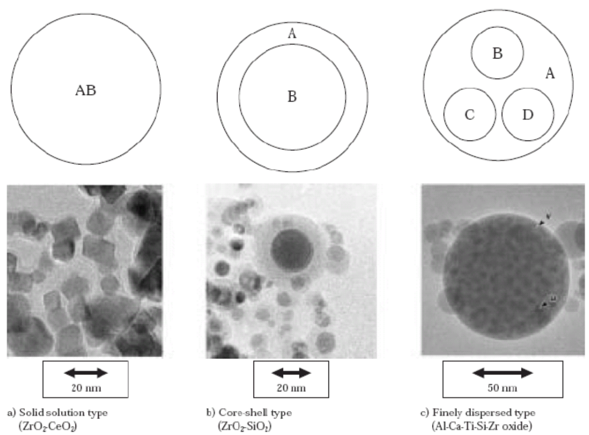
**Objective: Develop and optimize ODS fabrication process for additive manufacturing**

### *Approach*

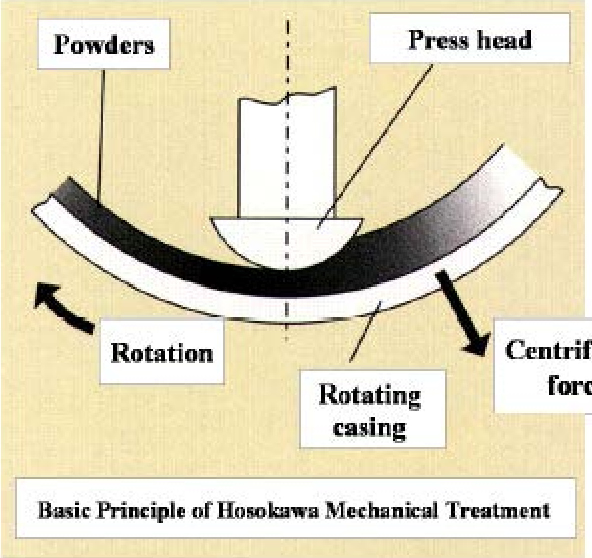
➢ Powder mechanical alloying using Hosokawa Mechano-Chemical Bonding (MCB) followed by Ball Milling (BM)

**Why MCB + BM?**

- For MCB, powders are subjected to substantial compression, shear, mechanical forces under high rotating condition (~4000 rpm), through a gap between chamber and press head
- Enable smaller particles to be dispersed uniformly and bonded onto base(host) particles without using binders.
- Improved particle sphericity, ideal for precision mixing of nano and submicron powders.
- Grain boundaries of host particles are pinned by nano-oxide particles,
- minimized grain growth during sintering.



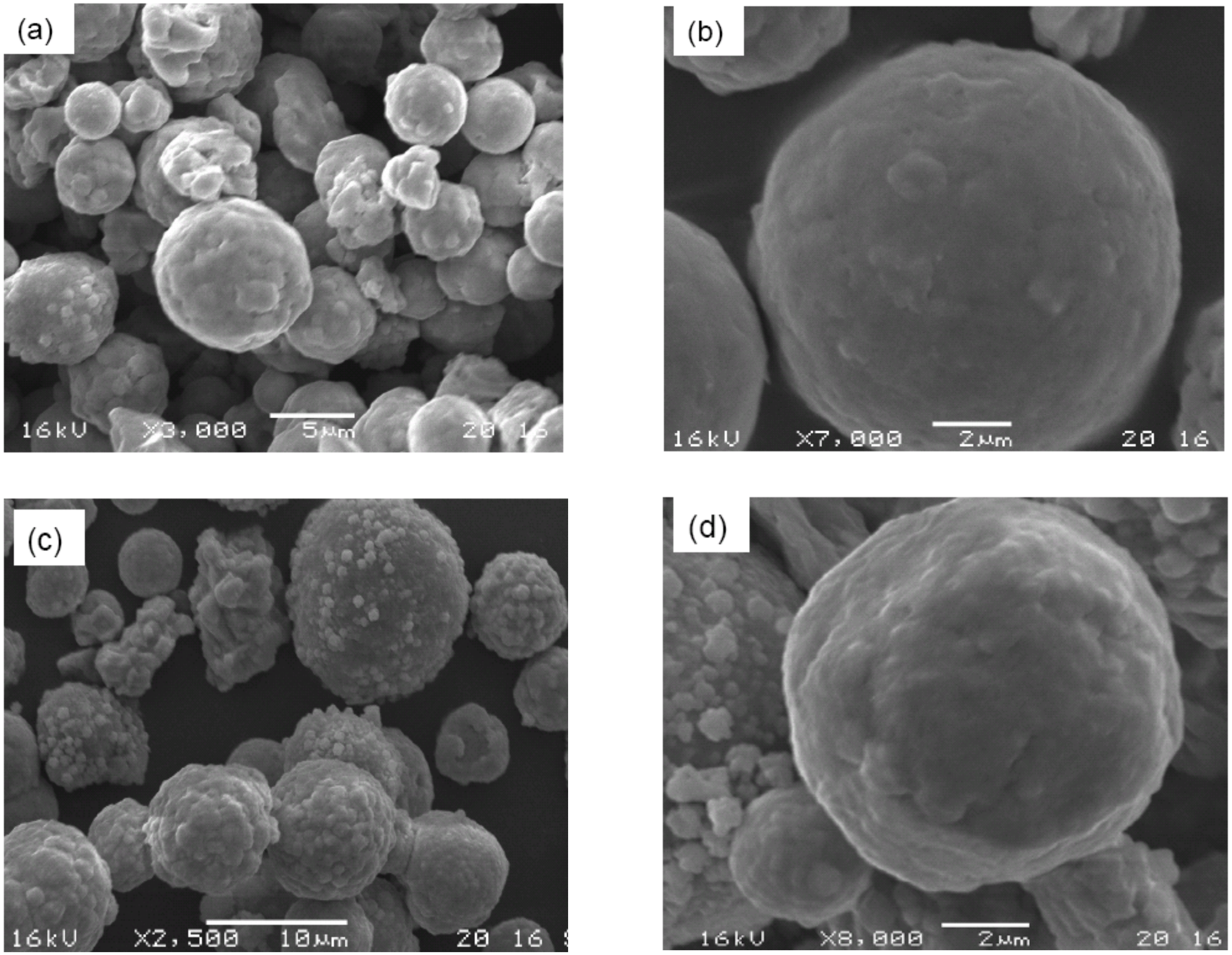

**Structural patterns of nanocomposite particles**
[ T. Yokoyama and C. C. Huang, KONA No.23 (2005) ]




*Kang, B.S., Chyu, M.K., Alvin, M.A., and Gleeson, B.M, "Method of Producing an Oxide Dispersion Strengthened Coating and Micro-Channels," US Patent 8609187 B1, 17, 2013*

# ODS Powder Compositions (in weight %)

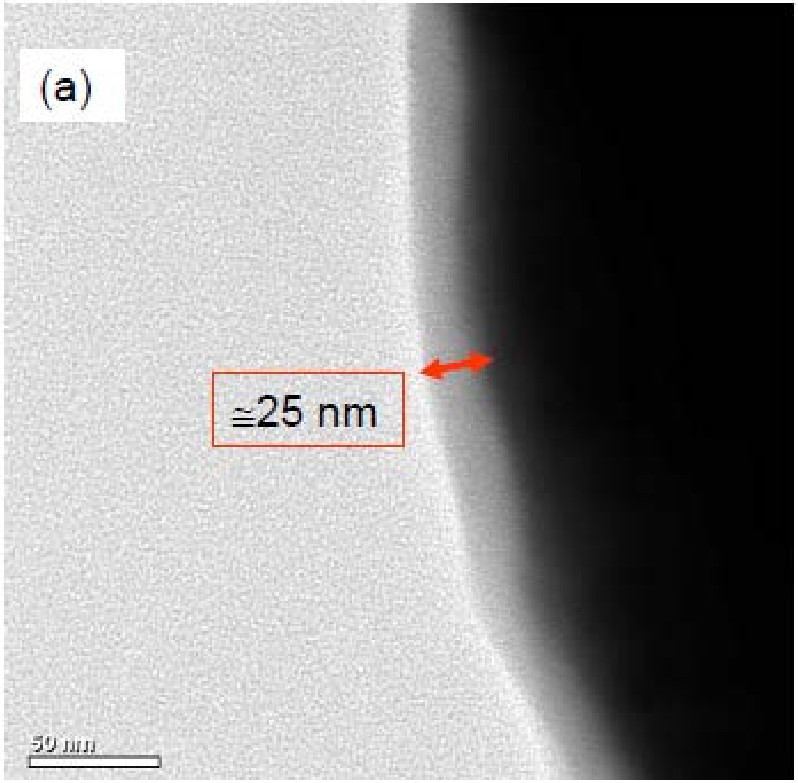
| | Cr (7.5~10 μm) | Al (4.5 ~ 7 μm) | $Y_2O_3$ < 50nm | W (~1 μm) | Ni (4 ~ 8 μm) |
|---|---|---|---|---|---|
| **A1** | **20** | **5** | **1.5** | **0** | **73.5** |
| **A2** | **20** | **5** | **1.5** | **3** | **70.5** |

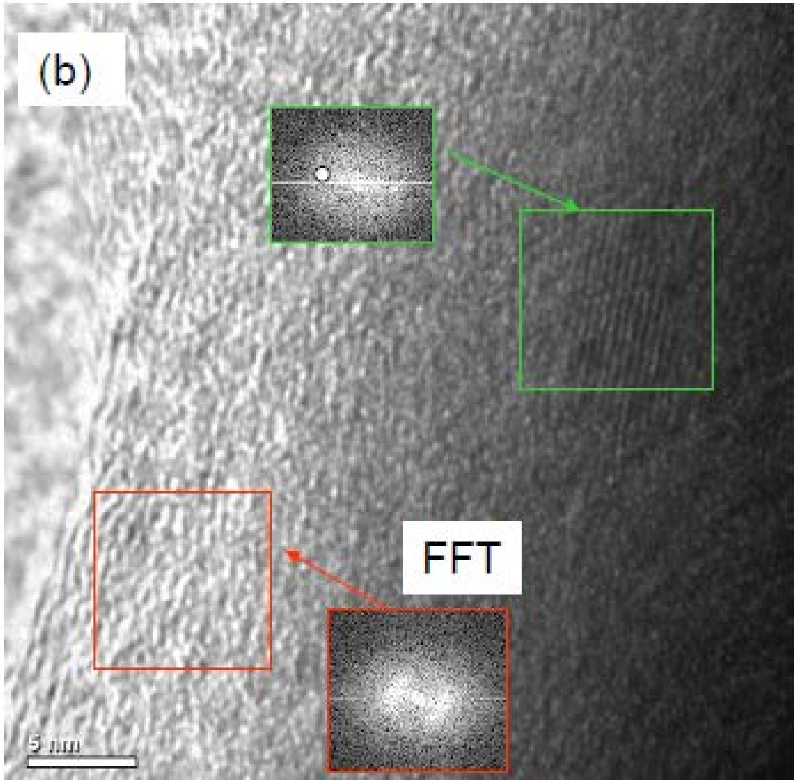



**SEM micrographs of MCB processed powder sample A1 and A2**
(a). Sample A1; (b) close view of (a); (c) sample A2; (d): close view of (c)

# ODS Powder Characterization

TEM BF and HREM imaging – A1 Sample

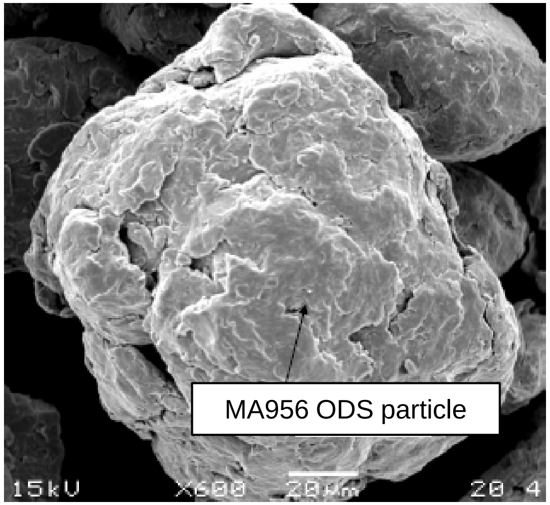


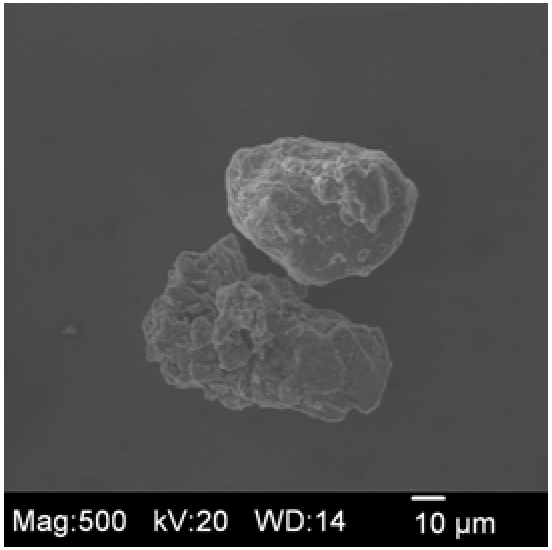



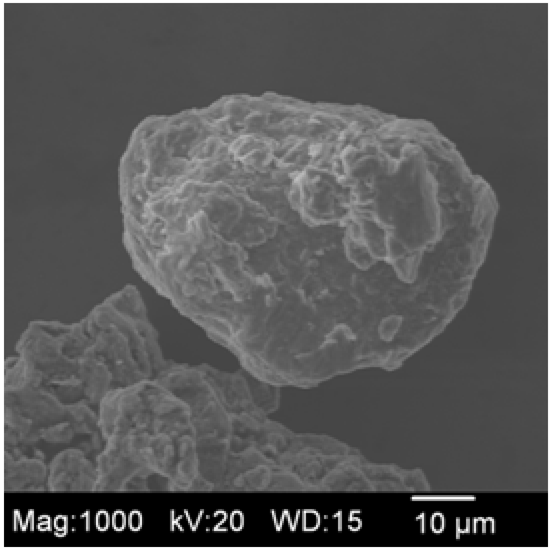

- TEM BF image (a) shows a layer of $Y_2O_3$ thin film with thickness about 25nm around the edge of particle. The film thickness is relatively homogeneous.
- HREM image (b) shows the fine structure of the thin film. Most area of the film is amorphous and the corresponding FFT (Fast Fourier Transform) image show the diffusive feature.
- There is crystal structure within film as FFT indicated. The embedded FFT shows the spots and image shows the orientation fringe. The growth of film may involve crystallization of $Y_2O_3$.

# ODS Powder Fabrication

***MA 956 ODS sample*** ***(Special Metals Inc.)***

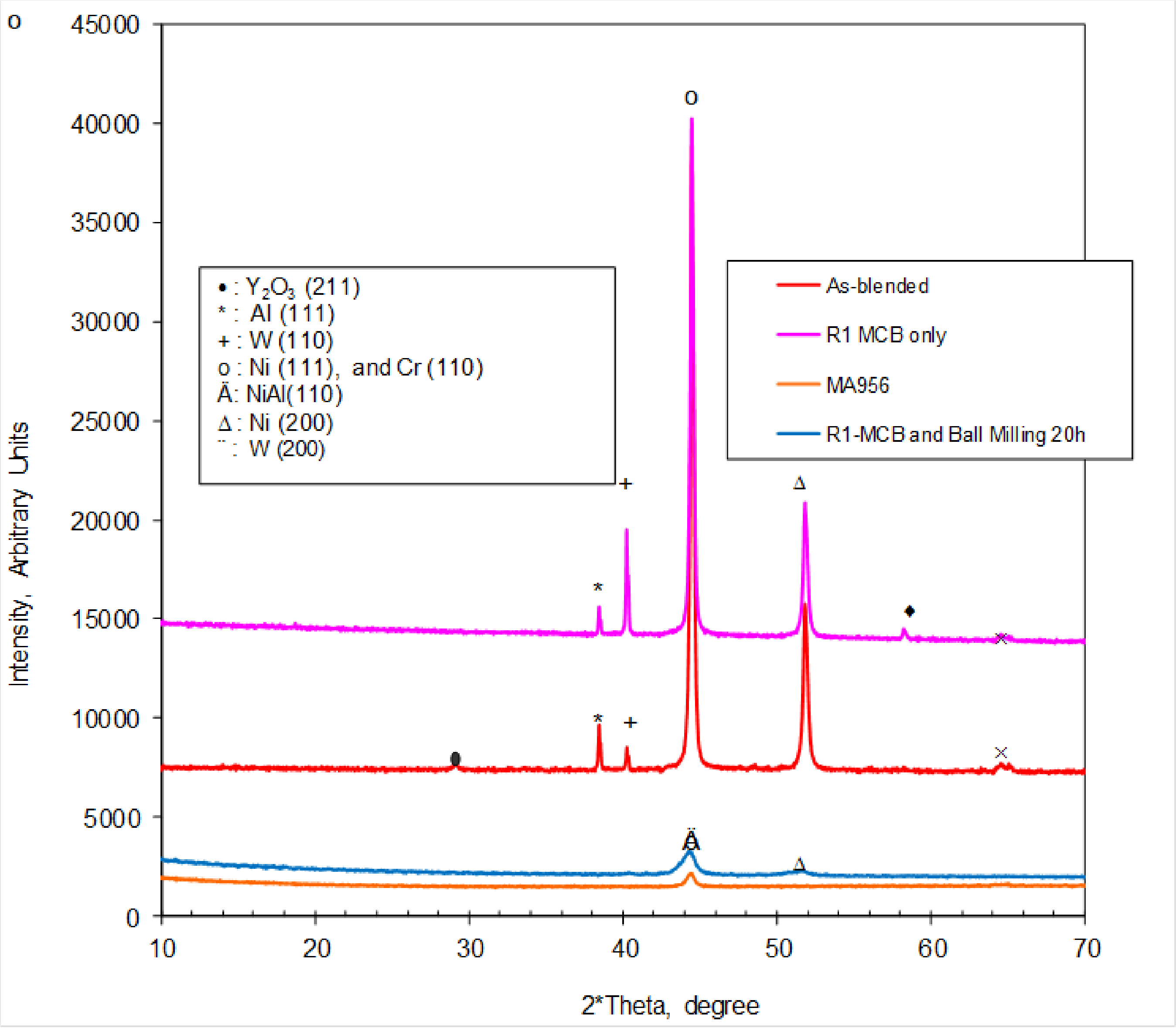



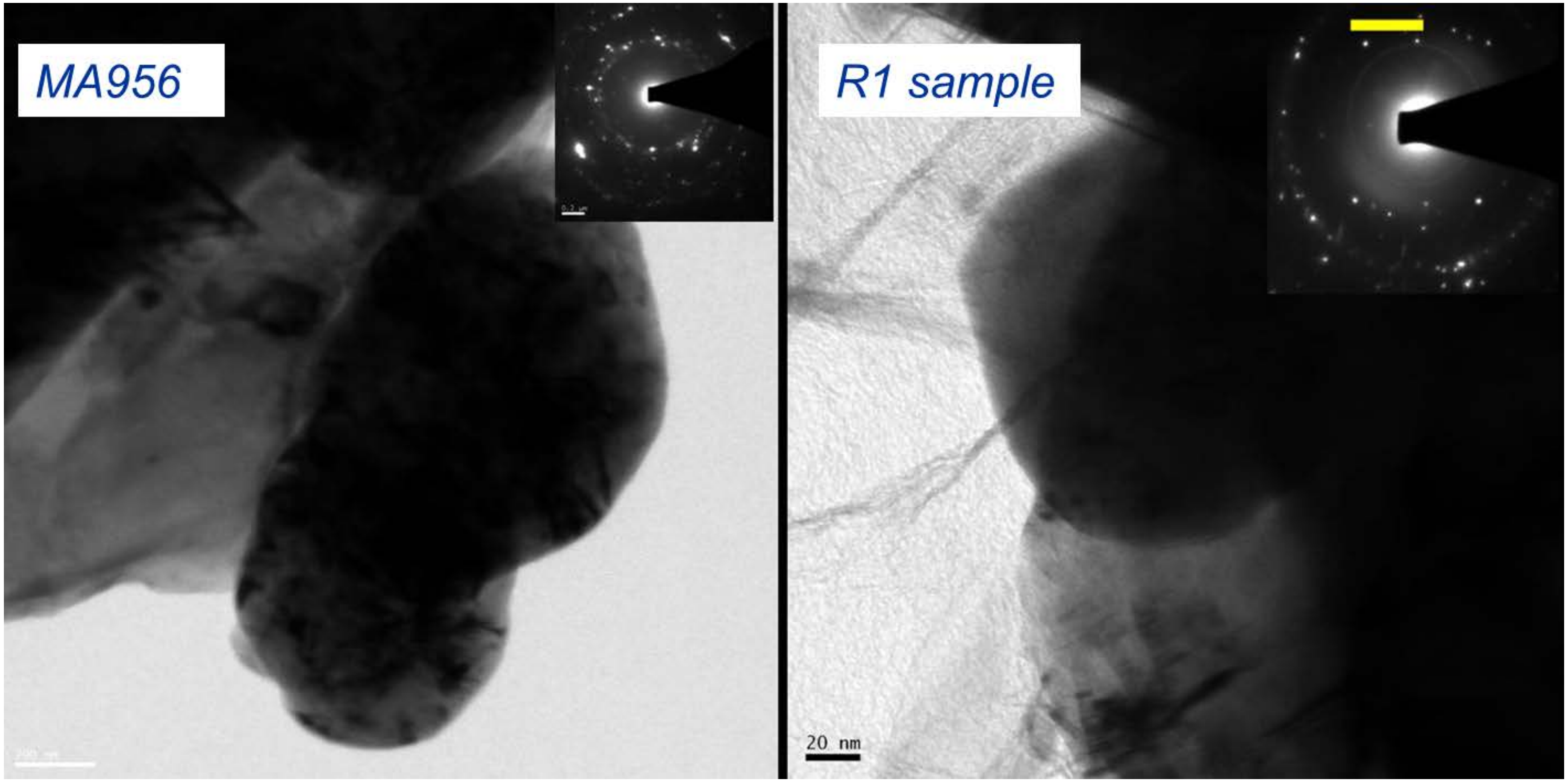




***R1 sample with 15 hrs ball milling***

# ODS Powder Characterization



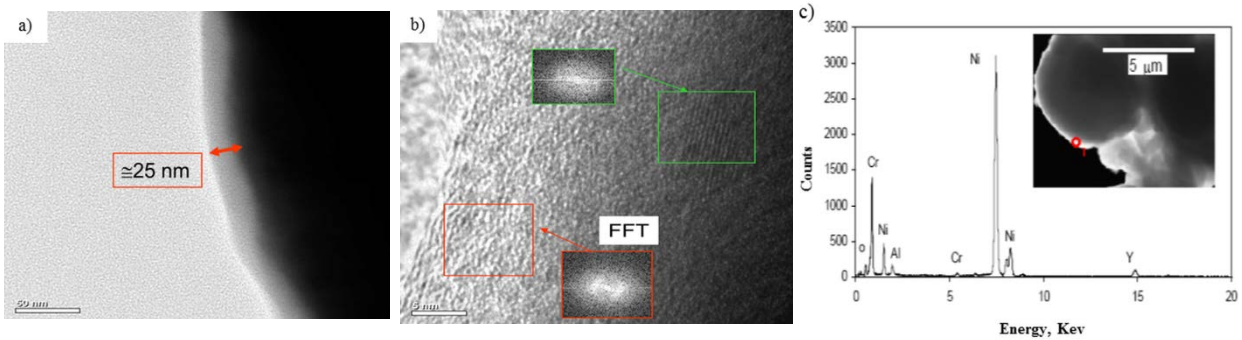
# ODS Powder Characterization



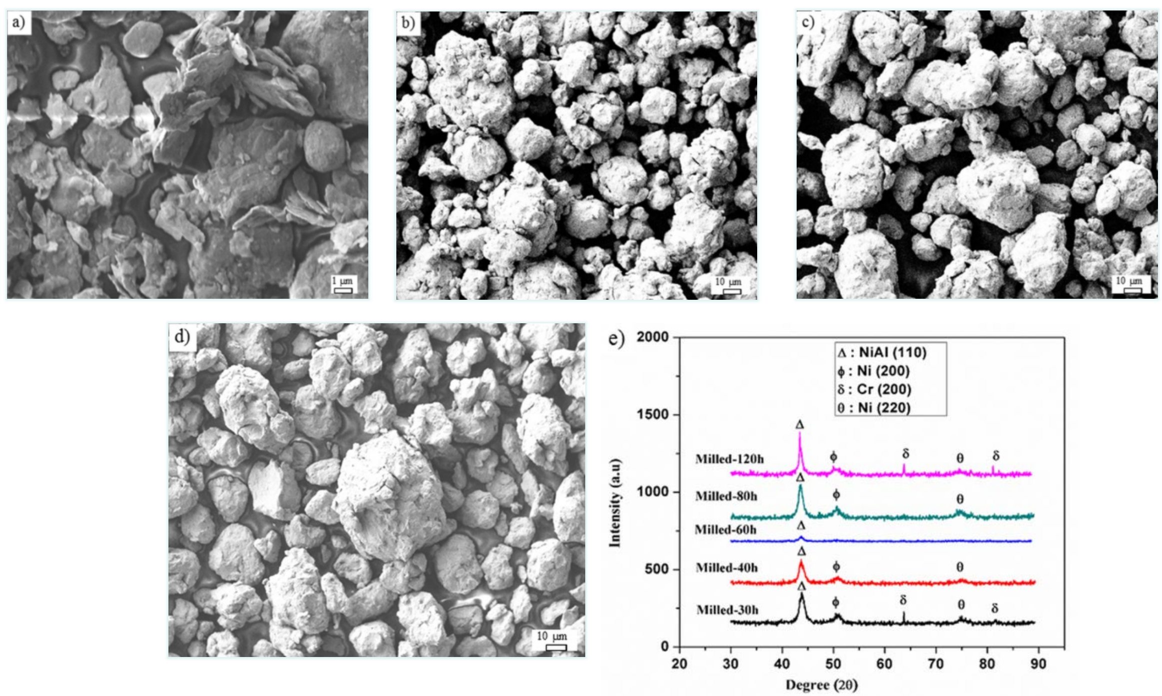

**TEM results**

- For MA 956, many dislocations were found inside particles, indicating heavy deformation during ball milling as well as many tiny particles were embedded into particles. SAD shows particle is polycrystalline.
- For R1 sample, TEM image and SAD show the similar structure to MA 956, indicating heavy deformation, well mixed and polycrystalline structure.

# Summary - ODS Powder Characterization




MCB processed ODS powders images, (a) TEM BF, (b) HR TEM, (c) STEM EDX




SEM micrographs of milled ODS powders for (a) 5 hrs, (b) 40 hrs, (c) 60 hrs, (d) 120 hrs, and (e) XRD spectrum.

# *Outlines*

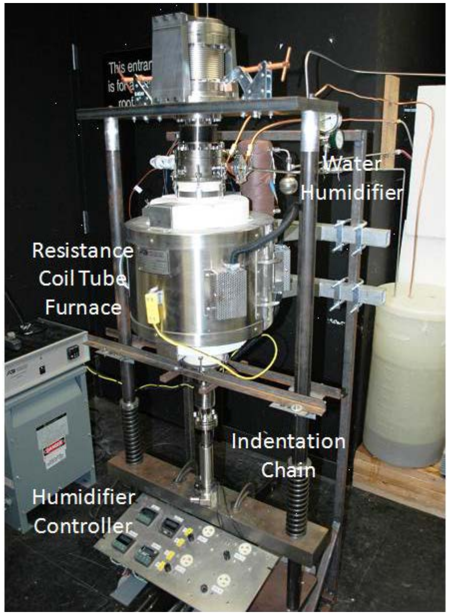
- *Introduction and Background*
- ***Challenges, Objectives, Benefits of Technology, Research Task Plan***
- *Tasks*
  1. *Advanced Impingement*
  2. *ODS Powders Fabrication and Characterization*
  3. *ODS Coating (AM Assisted) (Preliminary results)*
  4. ***Microstructural and Mechanical Properties Characterization***
  5. *Detailed Experimental Measurement and Validation*

# Task 4: Microstructural and Mechanical Properties Evaluations

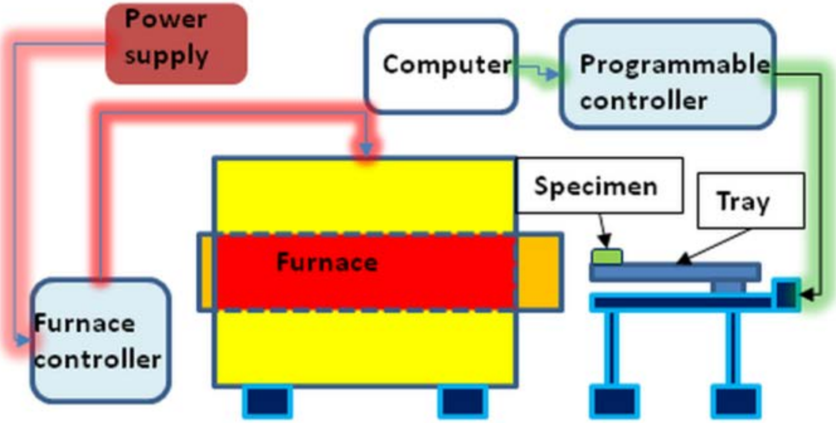
**Objective: Characterize the microstructural and mechanical properties of ODS coating under (1) dry air, and (2) highly moisture content**

## *Approach*

- Advanced microstructural characterization
  - OM, EDX, XRD, SEM, TEM
- *Micro-indentation using in-house test rig*
- *Thermal cyclic tests*



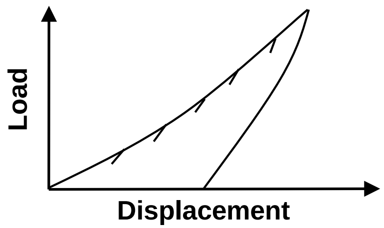

Controlled environment high temperature micro-indentation system

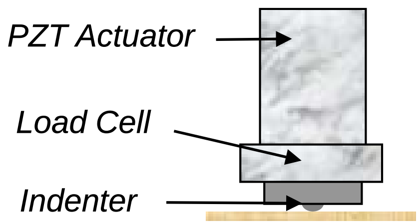



Schematic of the cyclic thermal exposure apparatus setup

# Durability/Damage Assessment of Advanced Turbine Components

## Multiple Loading/Partial Unloading Micro-Indentation




**Applications:**

- **Elastic Modulus**
- **Stress-Strain Curve**
- **Indentation Creep**
- **High Temperature Characterization**

**Potential:**

- **In Situ Material Characterization**
- **Portability**
- **Variable Influence Zone**

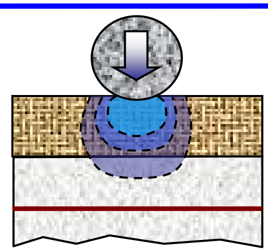
### Load Based vs. Contact Area Based

$$P=\frac{4}{3}\cdot\frac{\sqrt{R}}{k_0}h^{3/2}$$ *Hertzian Spherical Contact Mechanics*

*where,* $k_0=\frac{1}{E_r}=\frac{1-\upsilon^2}{E_d}+\frac{1-\upsilon^2}{E_i}$

$$\frac{dP}{dh}=\frac{2}{\sqrt{\pi}}E_r\sqrt{A}$$ ***Area Based***

$$\frac{dP}{dh}=(6RE_r^{\ 2})^{1/3}\cdot P^{1/3}$$ ***Load Based (WVU)***



$$\frac{dP}{dh}=(6RE_r^{\ 2})^{1/3}\cdot P^{1/3}$$

$$\frac{dh}{dP}=C\times\frac{1}{P^{1/3}}$$

$$\left.\frac{dh}{dP}\right|_1-\left.\frac{dh}{dP}\right|_2=C\times\left(\frac{1}{p_1^{\ 1/3}}-\frac{1}{p_2^{\ 1/3}}\right)$$

$$\left(\frac{dh}{dP}\right)=C\times\left(\frac{1}{P^{1/3}}\right)+C_s$$

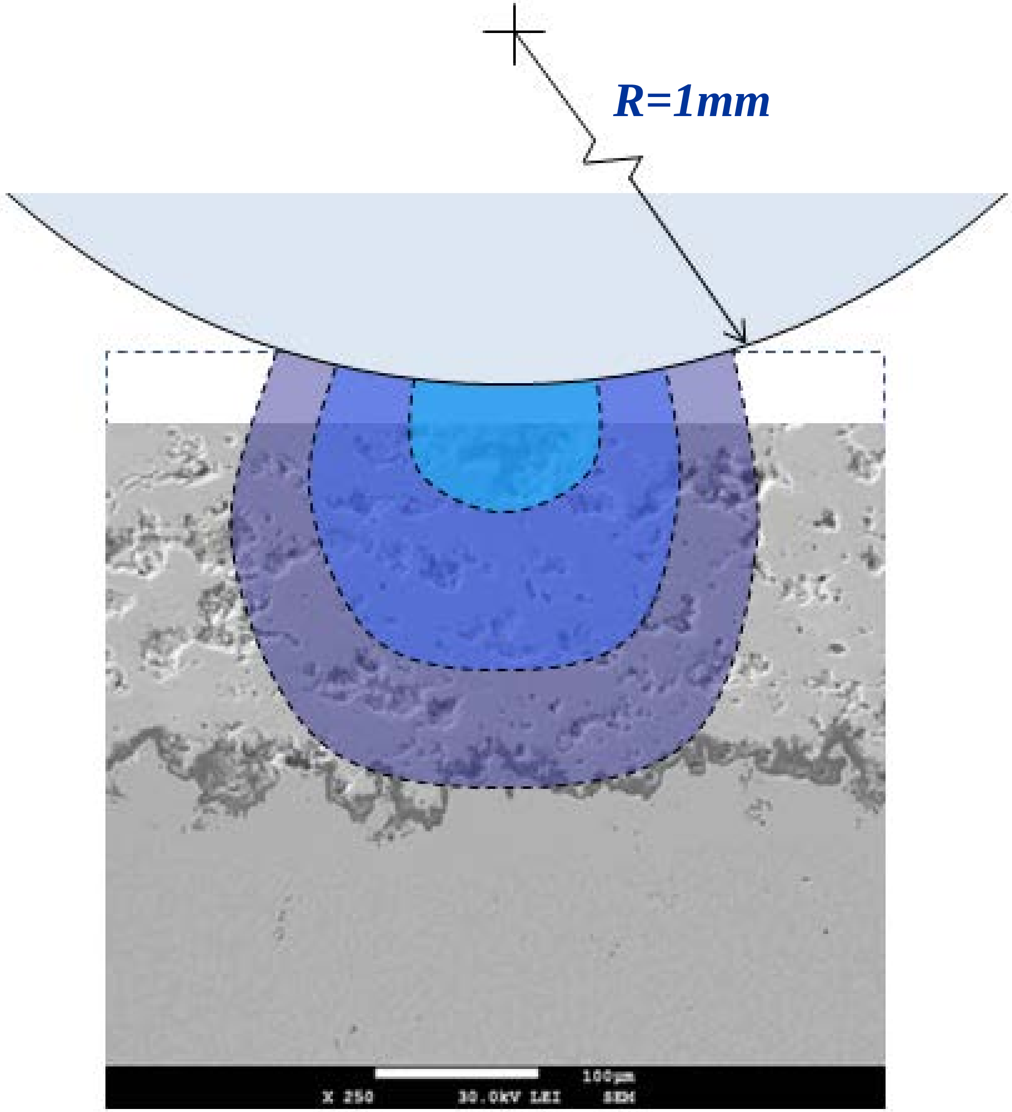
$$x=\frac{1}{p^{1/3}}\ ,\ y=\frac{dh}{dp}$$

$$y=mx+b$$ *where,* $a=\left(6RE_r^{\ 2}\right)^{-1/3},\ b=C_s$

### Technique Benefits

- **Designed for TBC/Bond and ODS Coating specimen**
  – Measurement of surface stiffness responses as a means to correlate the evolution of the microstructural changes of the coating layer/substrate subjected to high temperature thermal cycles.
  – Can also be correlated to the damping effect
  – No surface preparation needed
- **Controllable Influence Zone**
  – The overall response obtained is a contribution form different regions of the multilayered thermal barrier coating structure.
  – As load increases, the influence zone increase as well.

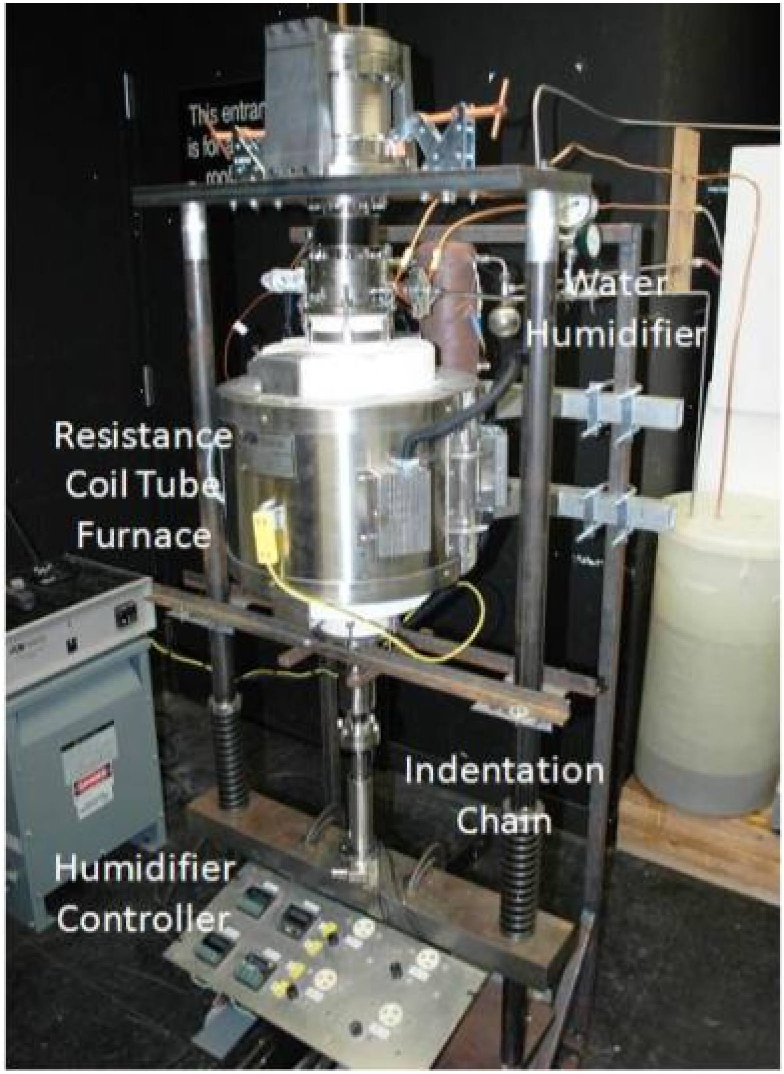
# Durability/Damage Assessment of Advanced Turbine Components



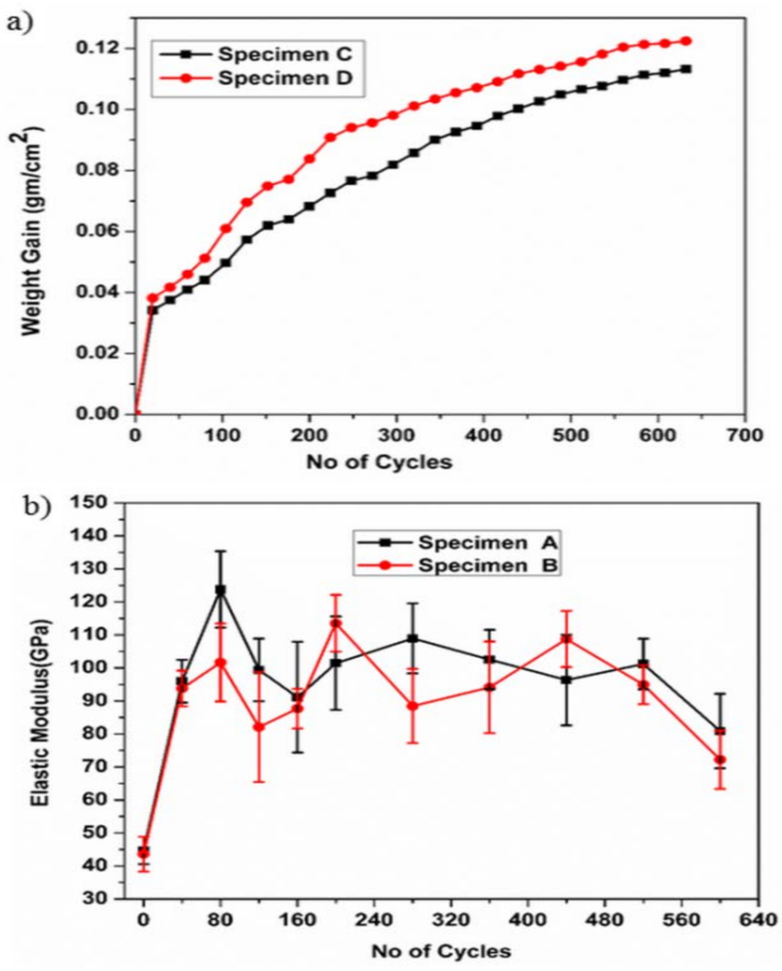
# Durability/Damage Assessment of Advanced Turbine Components

## Controlled Environment Indentation System

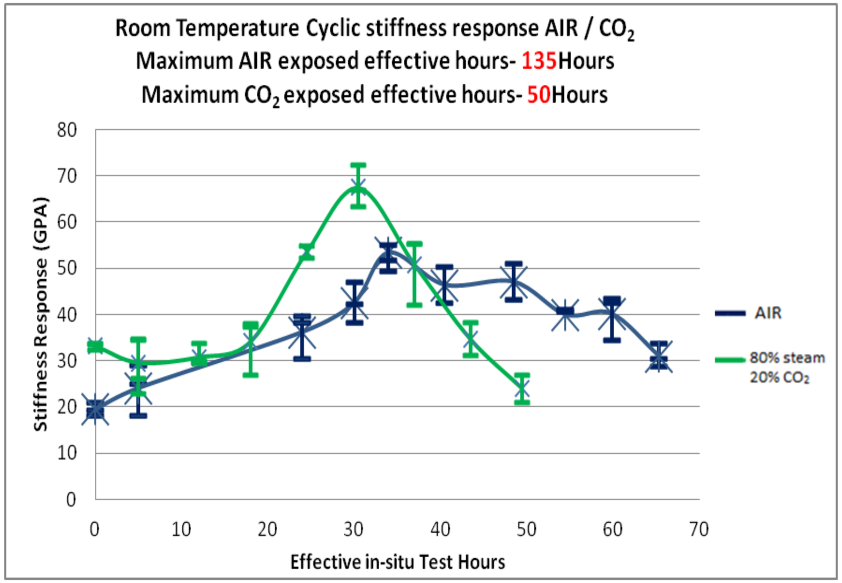
- Continuous Water or Air Cooling
- Applied Test Systems Resistance Coil Tube Furnace
- Humidifier Numerically Controlled by 3 Separate PIDs
- Copper Gaskets Insure Proper and Efficient Sealing at Elevated Temperatures
- Controllable Thermal Ramp Rate
- Potential to Vary Internal Pressure



# Mechanical Properties Evaluations

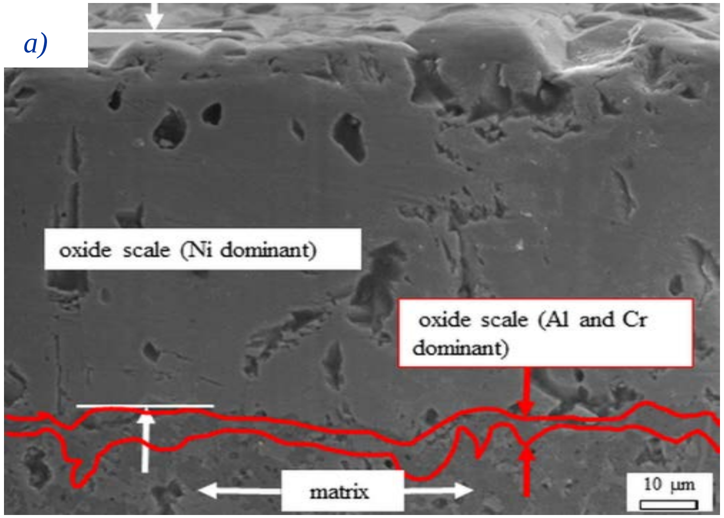



ODS specimen under cyclic oxidation room to 1100$^o$C in air:
(a) weight gain, (b) micro-indentation stiffness response

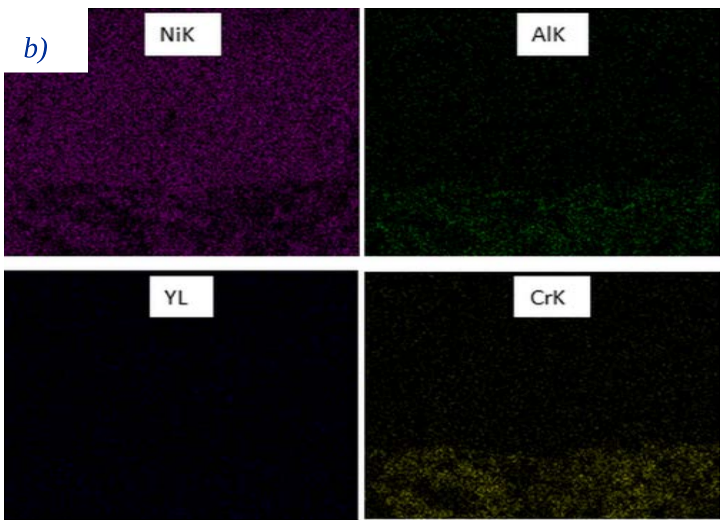



In-situ micro-indentation stiffness response of APS/MCrAlY/RenéN5 coupon under cyclic oxidation room to 1100$^o$C under air and wet (80% steam/20% $CO_2$) conditions

# Oxidation Kinetics – Stable NiO Formation

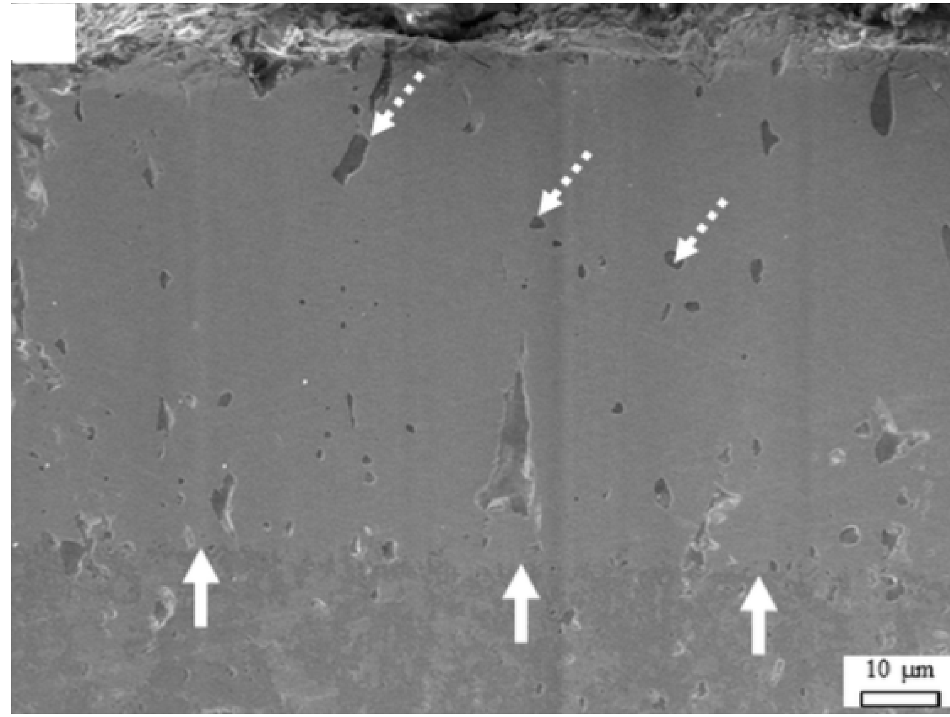



(a) oxide scale at 120 cycles



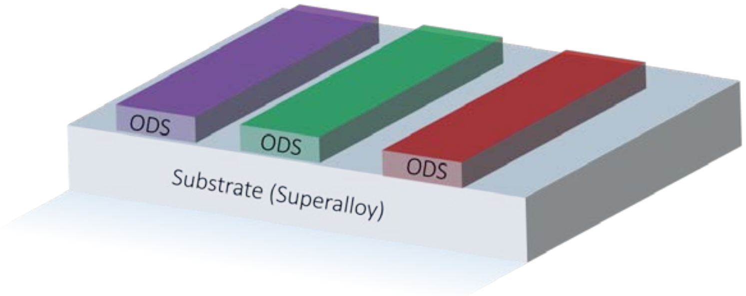

(b) EDX maps at 120 cycles

SEM metallographic cross-section micrographs

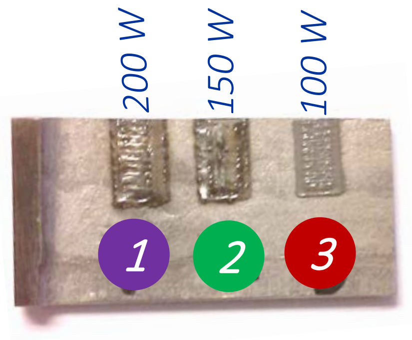



SEM metallographic cross-section micrographs of NiO oxide scale at 600 cycles

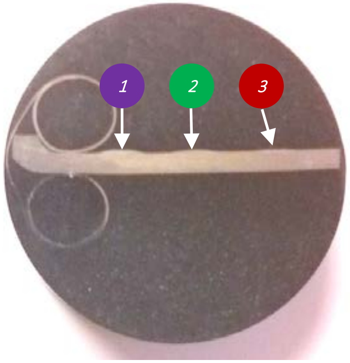
# Task 3: ODS Coating (AM Assisted)



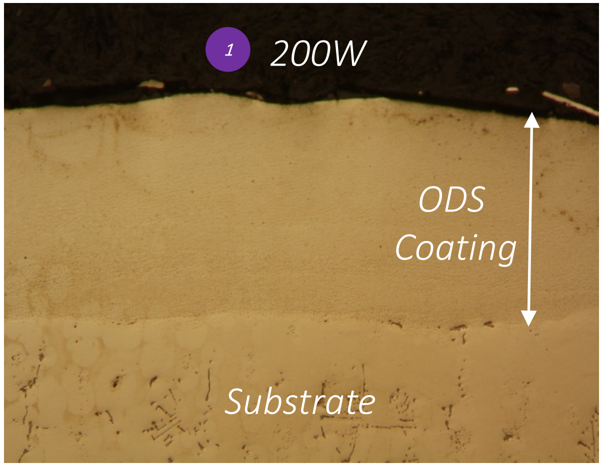

SCHEME

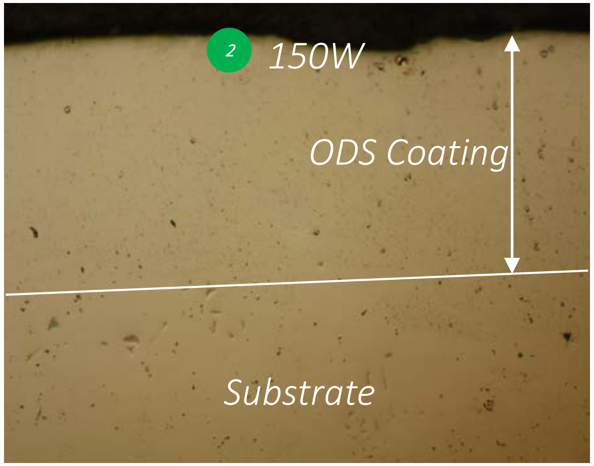


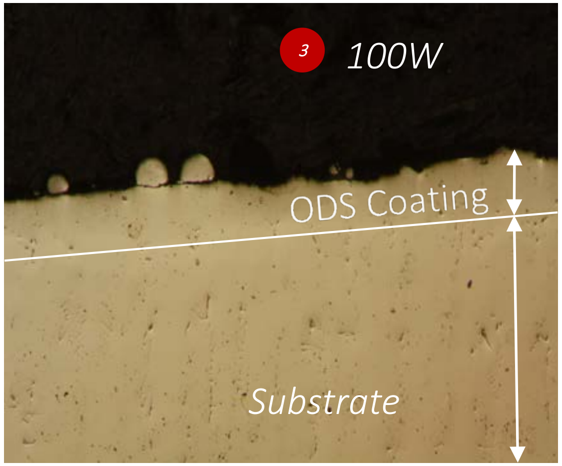

TOP VIEW




SIDE VIEW



*Thank You*