ODS coating development by cold spray method using powder mix prepared by combined MCB and ball milling processes
(ODS Coating Development)

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Chris Nung (MS Student) – ODS ball milling

Larry Graham (Manager, Advanced Technology, PCC Airfoils LLC) – advanced airfoil cooling manufacturability

Jack deBarbadillo – (Product & Process Development Manager, Special Metals Inc.) – ODS alloys technical feedback

Research Objective

This is an exploratory research with the aim to

(i) produce ODS powder mixture with reduced manufacturing cost and better yttria dispersion using a combined Hosokawa mechano chemical bonding (MCB) and ball milling process (FY09-10),

(ii) evaluate the applicability of ODS coating on superalloy substrate using Cold Spray method (FY09-10),

(iii) produce desirable ODS structural coating layer with proper surface heat treatment (FY10-11),

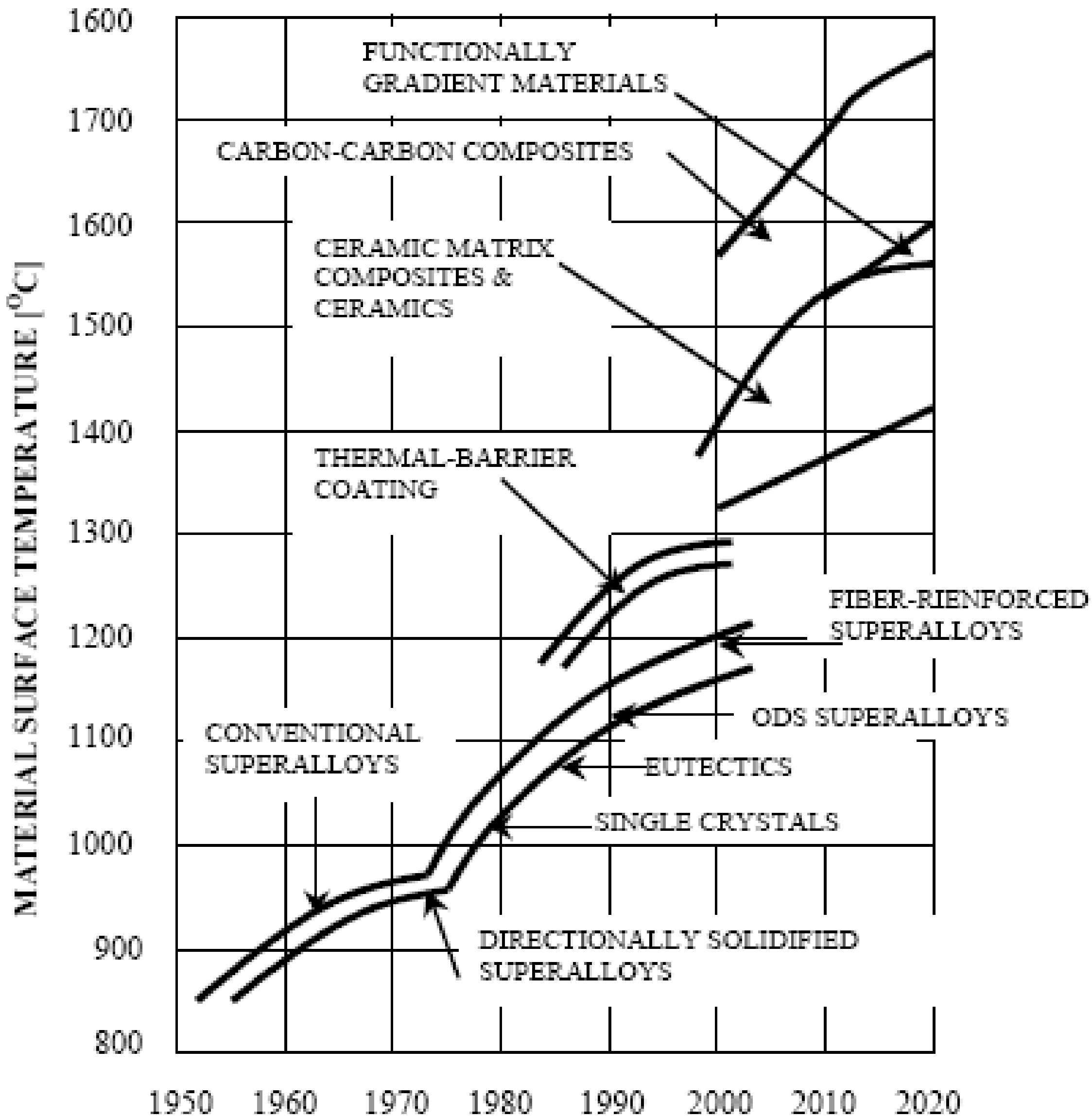
(iv) Mechanical property evaluation of ODS coating layer at elevated temperature (to 1250 ºC) using micro-indentation method (FY10-11).

DOE FE Coal Program
– Goals –

Advanced Power Systems
By 2010 Develop Advanced Coal-Based Power Systems Capable Of 45-50% Efficiency at <$1000/kw
Near Zero Emissions Energy From Coal
By 2015 Demonstrate Future Coal-Based Energy Plants That Offer Zero Emissions (Including CO_2 with Multi-Product Production (Electricity & H_2)

	Syngas Turbine 2010	Hydrogen Turbine 2015	Oxy-Fuel Turbine 2010	Oxy-Fuel Turbine 2015
Combustor Exhaust Temp, °C (°F)	~1480+ (~2700+)	~1480+ (~2700+)		
Turbine Inlet Temp, °C (°F)	~1370 (~2500)	~1425 (~2600)	~620 (~1150)	~760 (~1400) (HP) ~1760 (~3200) (IP)
Turbine Exhaust Temp, °C (°F)	~595 (~1100)	~595 (~1100)		
Turbine Inlet Pressure, psig	~265	~300	~450	~1500 (HP) ~625 (IP)
Combustor Exhaust Composition, %	CO_2 (9.27) H_2O (8.5) N_2 (72.8) Ar (0.8) O_2 (8.6)	CO_2 (1.4) H_2O (17.3) N_2 (72.2) Ar (0.9) O_2 (8.2)	H_2O (82) CO_2 (17) O_2 (0.1) N_2 (1.1) Ar (1)	H_2O (75-90) CO_2 (25-10) O_2, N_2, Ar (1.7)

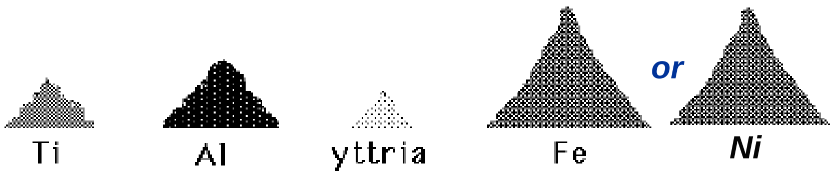
R.A.Dennis, "FE Research Direction – Thermal Barrier Coatings and Health Monitoring Techniques," Workshop on Advanced Coating Materials and Technology for Extreme Environments, Pennsylvania State University, State College, PA, September 12 - 13, 2006

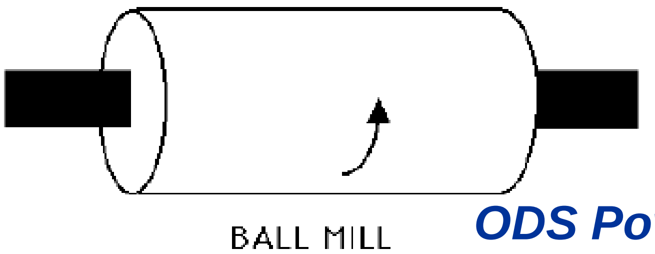


High-Temperature Materials for Turbine Blades

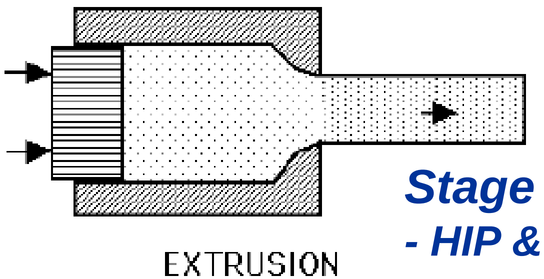
Ohnabe H., Wasaki S. and Imamura R., *Proceedings of the Third International Symposium on Ultra-high Temperature Materials*, Tajimi, 1993.

Typical Conventional ODS Alloy Production

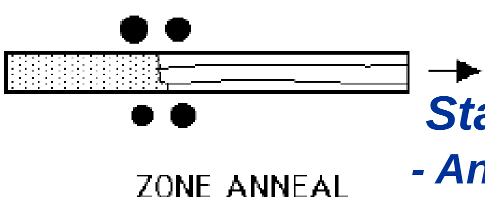




ODS Powder Production



Stage (1)
- HIP & Extrusion



Stage (2)
- Annealing/ Recrystallization

Stage (1) - consolidation of ODS powders thru combinations of hot isostatic pressing (HIP) and extrusion.

Stage (2) - heat treatment (either under isothermal or temperature gradient (i.e. zone annealing) to induce recrystallization.

The alloy produced at Stage (1) has very fine microstructures (usually in submicron size) with equi-axed grains that are characterized by high level of stored energy and very high hardness.

In Stage (2), the very fine grain structure is recrystallized to have coarsened and columnar microstructure, which is desirable in high temperature applications with enhanced creep strength.

(***Bhadeshia, 2000,*** *Proceedings of the 21st Riso International Symposium on Materials Science*)

The major processing parameters are:

Stage (1) - *Deformation* (i.e. kinetic strength or stored energy) prior to recrystallization (the higher the kinetic strength, the higher the Grain Aspect Ratio (GAR) or higher anisotropic columnar grains after recrsystallizaton).

Stage (2) - *Annealing temperature* (usually approaching 0.9 T_M , where T_M is the melting temperature, as compared to the typical recrystallization temperature of 0.6 T_M in similar casted metallic alloys).

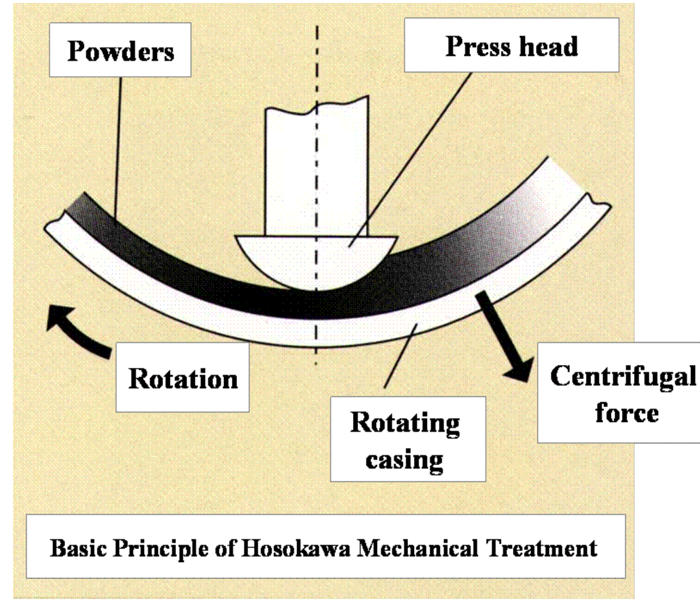
Research Outline

Part I: ODS Powder mixing using combined Hosokawa MCB and Ball Milling (FY09-10)

Part II: ODS Coating by Cold Spray with the follow-up Zone Annealing (FY10-11)

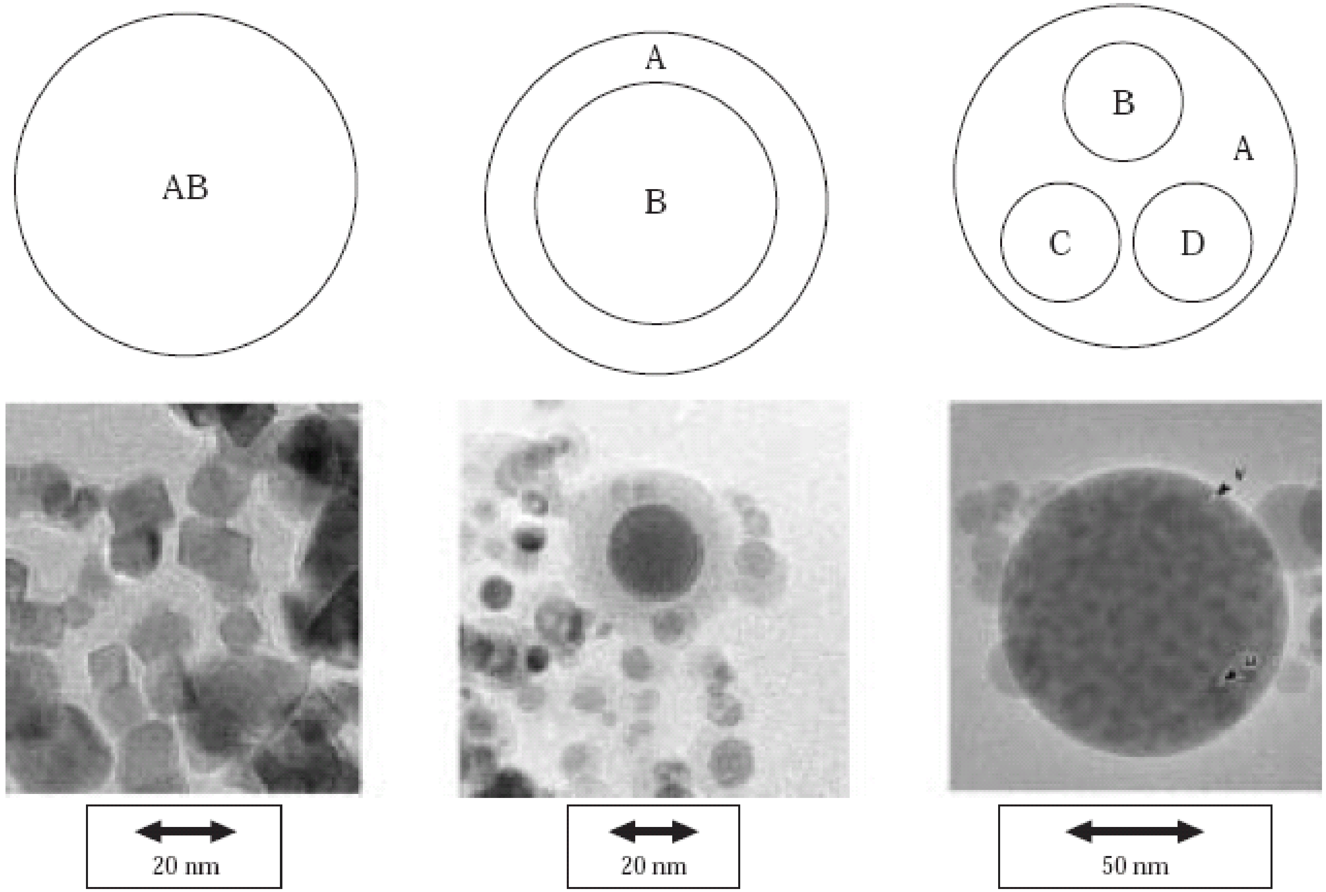
Powder Mixing Using Mechano-Chemical Bonding Technique

The powder mixture introduced into the internal cavity of the equipment is subjected to a centrifugal force which transports them to the inner wall of the rotating chamber. The powder mixture is then subjected to additional compression and shear mechanical forces as they rotate and pass through a gap between the chamber wall and a press head. This results in the smaller particles being dispersed and bonded onto the surfaces of larger base particles without using any binders. This technique can also be applied to improve particle sphericity and for precision mixing of nano and submicron powders.



Basic Principle of Hosokawa Mechanical Treatment

The MCB technique can disperse submicron and nano-size particles and bonds them onto the surfaces of larger host particles, which may result in oxide dispersion effects, i.e. this technique is applicable to make alloy powders suitable for ODS alloys at much lower cost.   Also, since the grain boundaries are pinned by the nano-oxide particles, grain growth will not occur during sintering, and therefore sintering of green compacts can proceed to full density. Also, the MCB process is much easier to scale up than ball mills.



a) Solid solution type (ZrO_2-CeO_2)

b) Core-shell type (ZrO_2-SiO_2)

c) Finely dispersed type (Al-Ca-Ti-Si-Zr oxide)

Structural patterns of nanocomposite particles

[T. Yokoyama and C. C. Huang, KONA No.23 (2005)]

(FY08-09) 1st Round ODS Powder Mixes (in weight %)
(30 minutes MCB Processing)

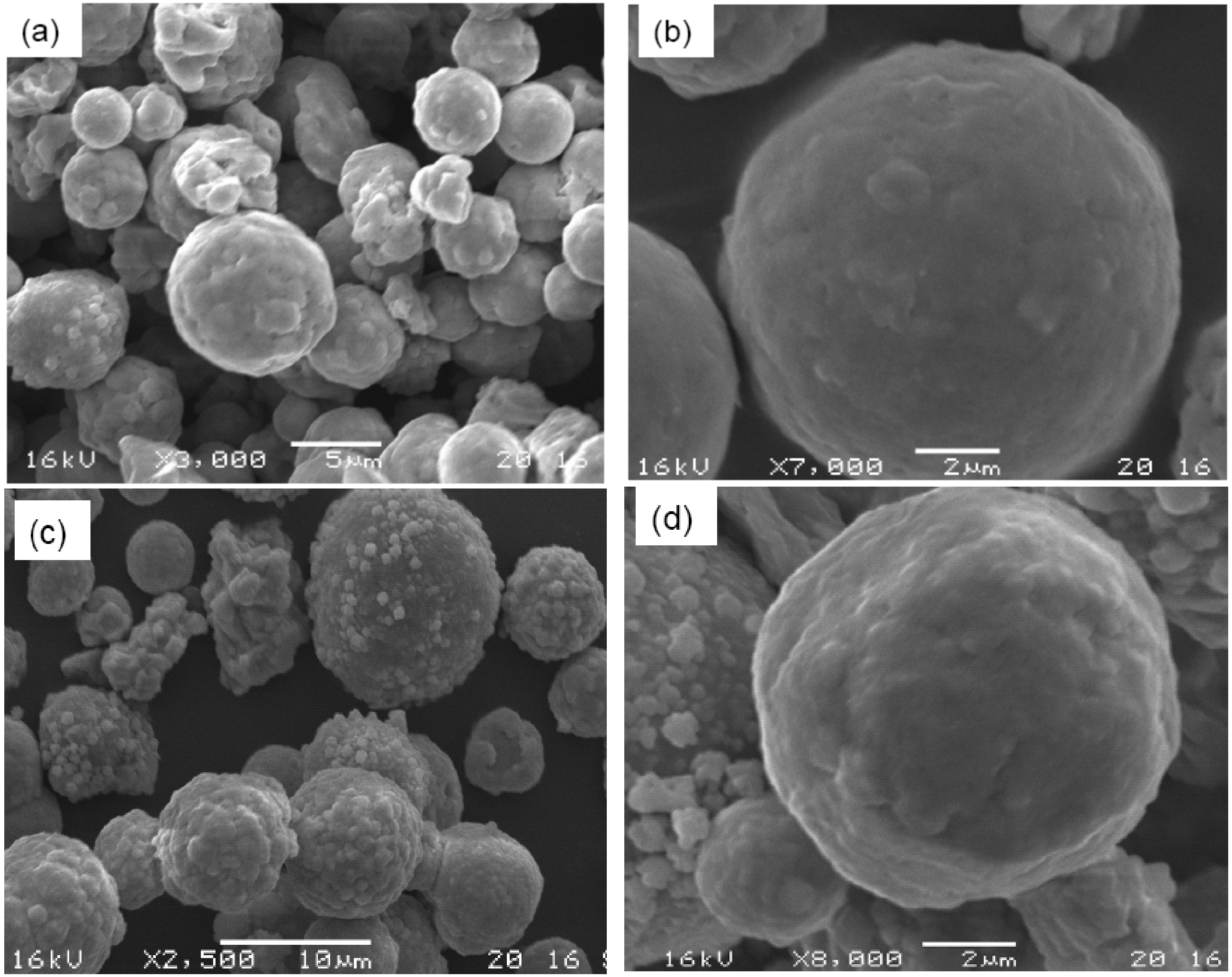
	Cr (7.5~10 μm)	Al (4.5 ~ 7 μm)	Y2O3 < 50nm	W (~1 μm)	Ni (4 ~ 8 μm)
A1	20	5	1.5	0	73.5
A2	20	5	1.5	3	70.5

	Cr (8~12 μm)	Al (4.5 ~ 7 μm)	Y2O3 < 50nm	W (2~4 μm)	Ni (8~15 μm)
B1	20	5	1.5	0	73.5
B2	20	5	1.5	3	70.5

(FY09-10) 2nd Round ODS Powder Mixes (in weight %)
(~ 1.5 hours high energy and ~ 30 minuutes lower energy MCB Processing)

	Cr (7.5~10 μm)	Al (4.5 ~ 7 μm)	Y2O3 < 50nm	W (~1 μm)	Ni (4 ~ 8 μm)
A1*	20	5	1.5	0	73.5
A2*	20	5	1.5	3	70.5

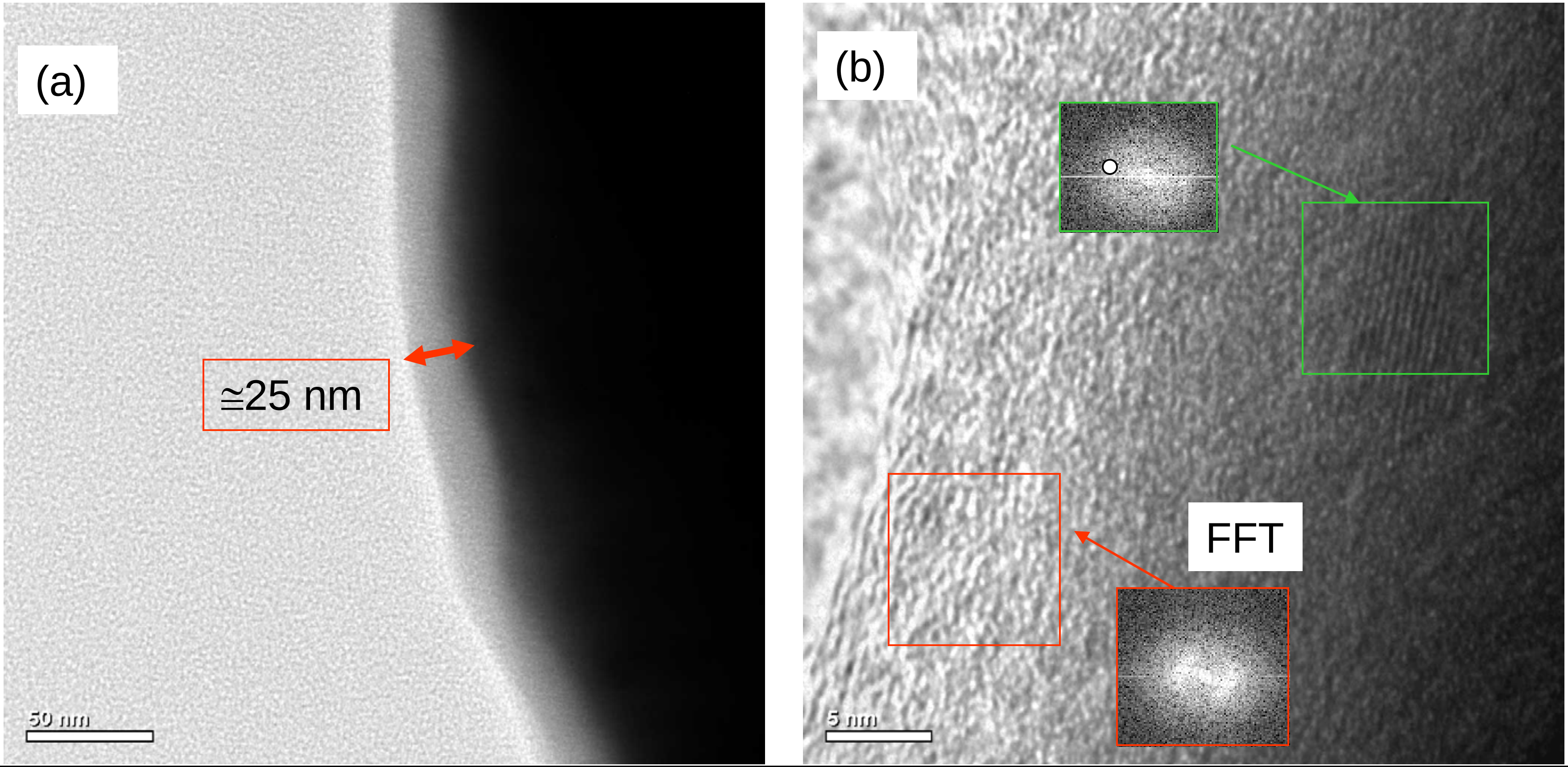
	Cr (1~5 μm)	Al (1 ~ 3 μm)	Y2O3 < 50nm	W (~1 μm)	Ni (1~5 μm)
C1	20	5	1.5	0	73.5
C2	20	5	1.5	3	70.5



SEM micrographs of MCB processed powder sample A1 and A2

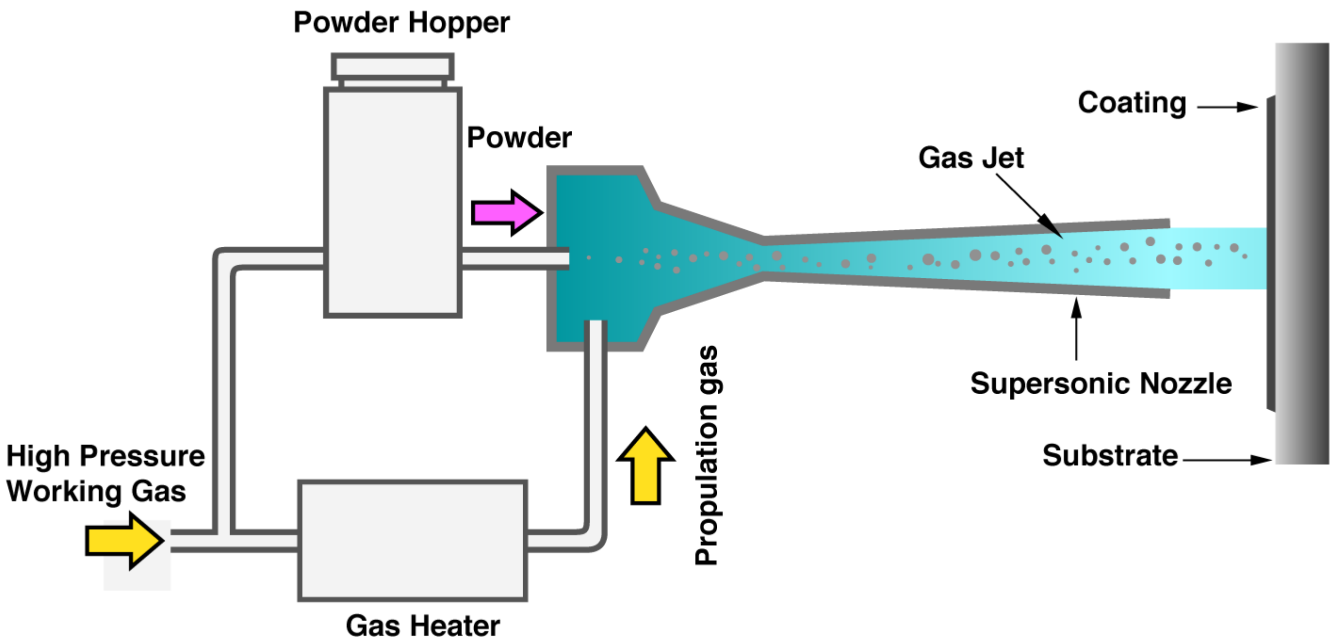
(a). Sample A1; (b) close view of (a); (c) sample A2; (d): close view of (c)

TEM BF and HREM imaging – A1 Sample



- TEM BF image (a) shows a layer of Y_2O_3 thin film with thickness about 25 nm 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 one orientation fringe. The growth of film may involve crystallization of Y_2O_3

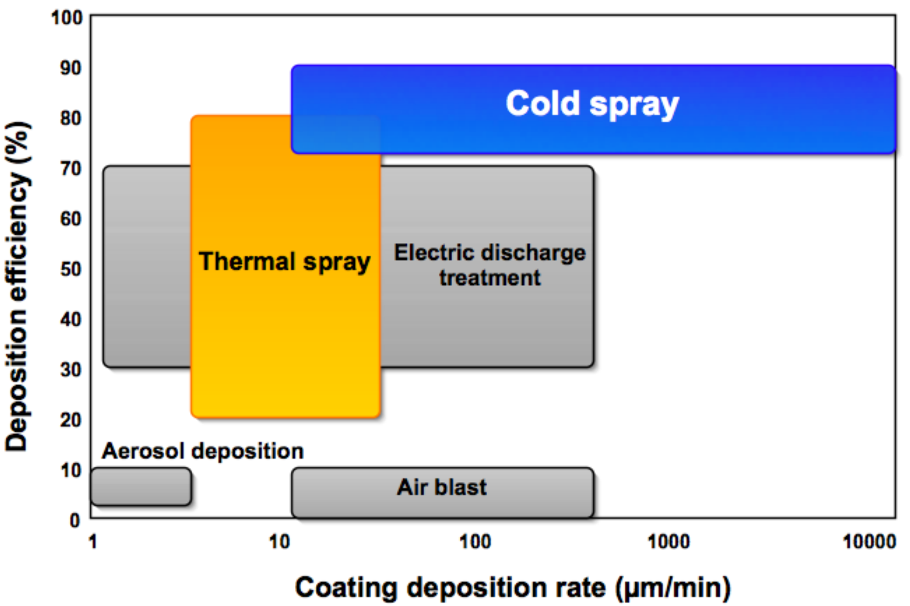
Cold-Gas Dynamic-Spray (Cold Spray)



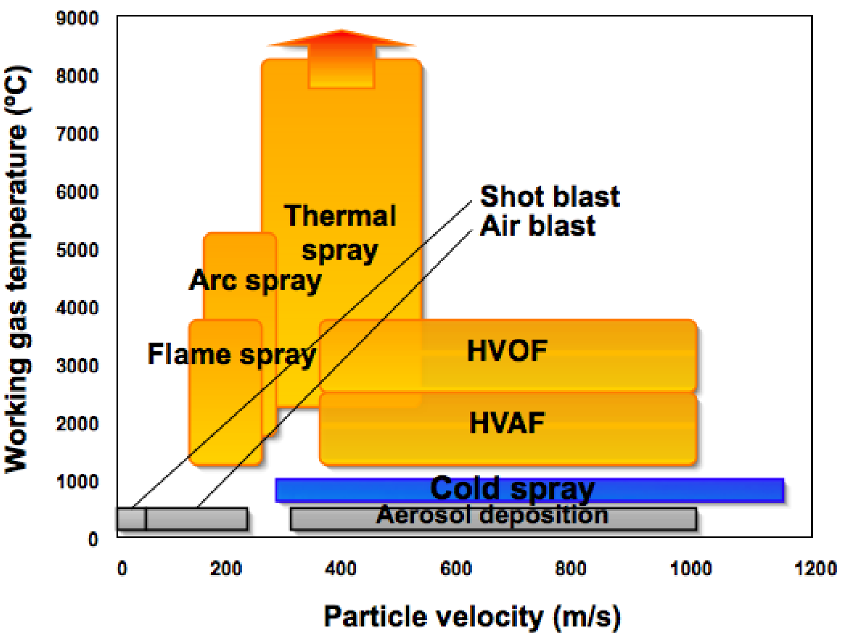
In conventional thermal spray technique, the coating material is heated to molten or semi-molten state. Therefore, thermal sprayed coatings have created some problems due to heating, i.e. high temperature oxidation and phase transformation. In the case of a cold spray technique, the particles are accelerated by the sonic/subsonic gas jet at the gas temperature, which is usually lower than melting temperature of powder materials.

Advantages of Cold Spray Technique

- **Low thermal effect (oxidation, phase transformation)**
  - **small microstructural changes on substrate surface**
  - **applicable for thermally and oxygen sensitive materials (e.g. Cr, Al, Cu, Ti)**
  - **Nanophase, intermetallic and amorphous materials can be cold sprayed**
  - **formation of embrittled phases, macro- and micro-segregation of the alloying elements (during solidification) can be avoided**
- **Peening effect - beneficial compressive residual stresses**
- Thick deposition
- Higher deposition efficiency and lower cost compared with thermal spraying



Deposition efficiency and rate



Working temp. and particle velocity

Cold Spray

High Pressure Type

High particle velocity (500-1500m/s)
30-40 bar (430 – 580 psi)
Possible hard material, such as WC, Ni alloy etc.
Expensive

Low pressure type

Low particle velocity
5-6 bar
Portable
Possible soft materials only, such as Al, Cu etc.
Low price and low maintenance cost

HP Cold Spray

Gas temperature increases.

Nitrogen 800°C, 40bar
Helium 600°C, 40bar

Heater

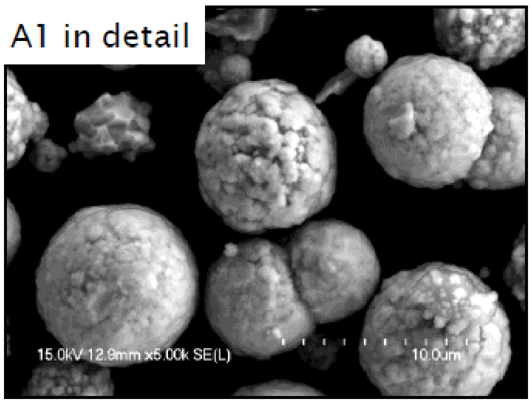
Gun

Reference: Web site of CGT corp.

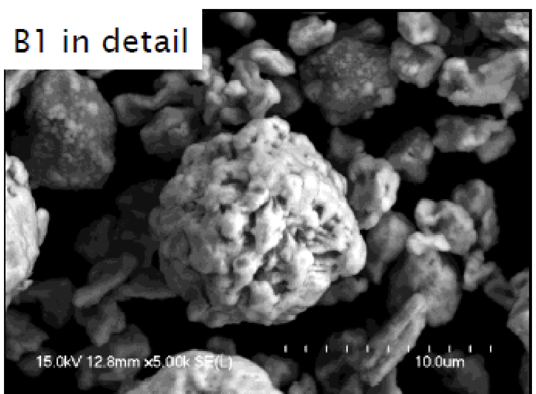
1st Round MCB-processed ODS Powders (FY 08-09)

ODS in stock
4-kinds (A1, A2, B1, B2), 400g per each powder pack.
Milled by Hosokawa MCB process.
73.5Ni-20Cr-5Al-1.5Y_2O_3 (sample A1) + W (only sample A2 & B2).

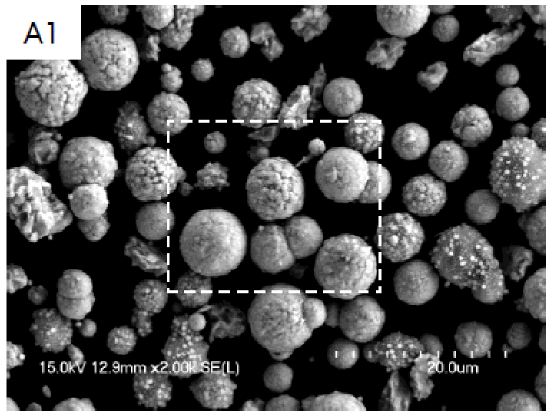
A1 in detail



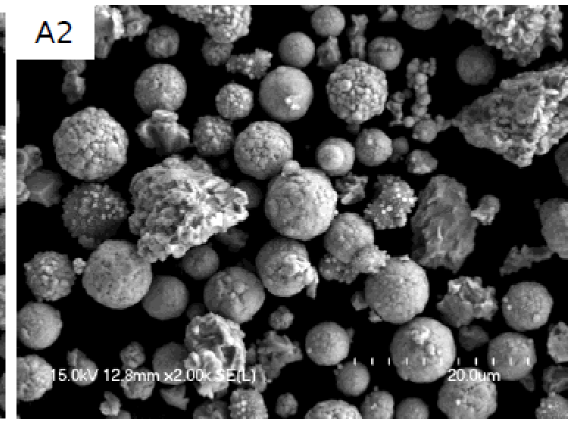
B1 in detail



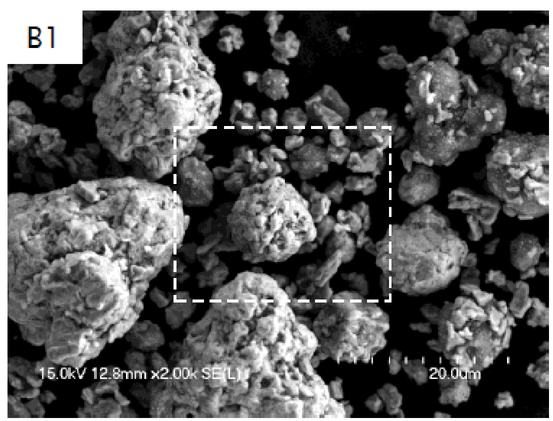
A1



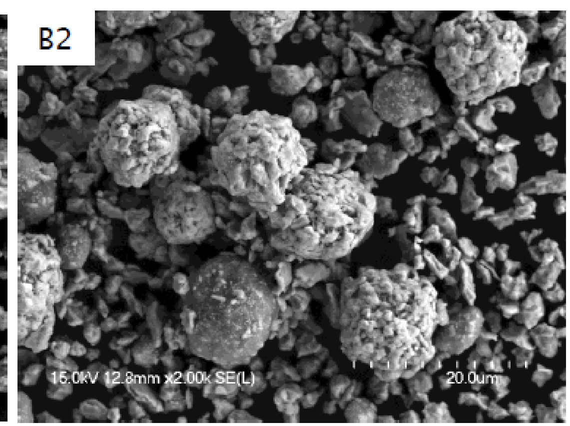
A2



B1

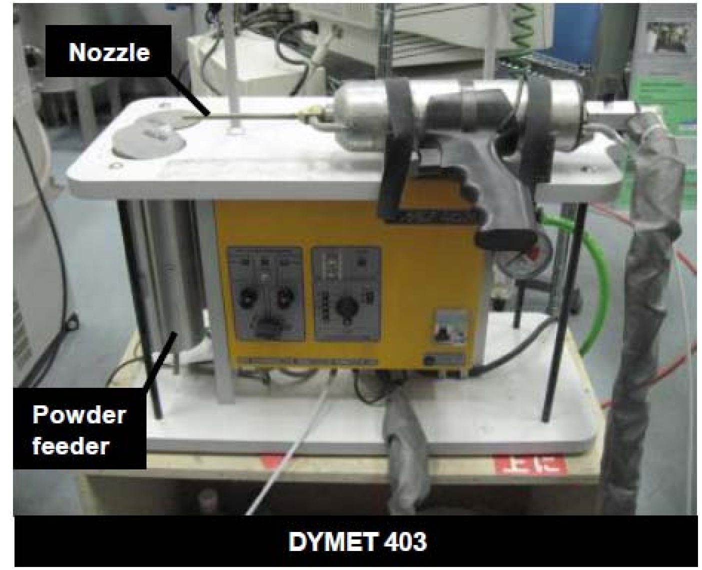


B2

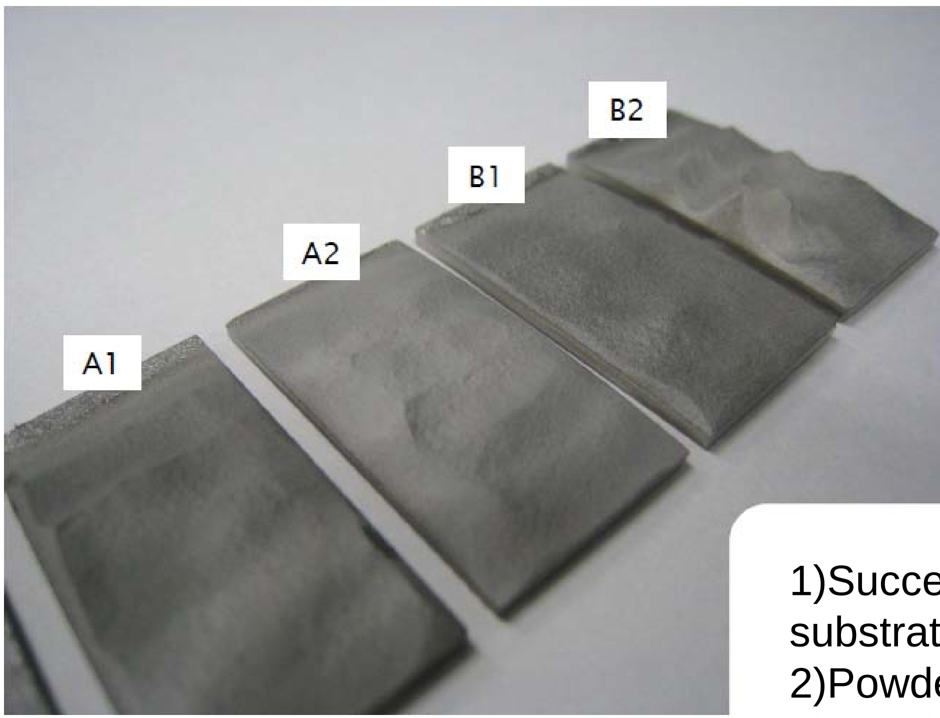


1st Trial Run of ODS Coatings on Inconel 625

Coating Conditions
A1, A2, B1, B2 coatings on sandblasted Inconel 625 by Dymet. Heating about 400deg.C; Powder Supply No. 8; 0.8MPa Air; 4 Pass; 10mm-off by Hands.



LP cold spray system used in this work

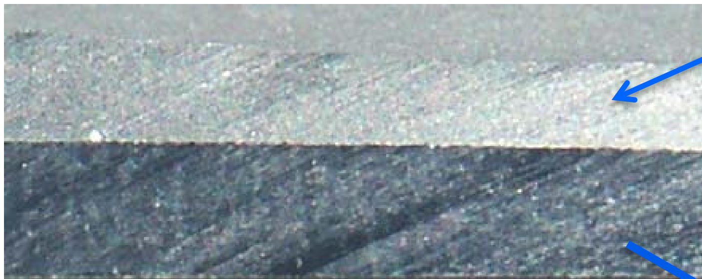


As-sprayed ODS coatings

1)Successful in making a thick coating on hard substrate.
2)Powder feeding is not smooth, it shows a discontinuous flow. Powder dry process is desired additionally.

ODS Coating

1.85 mm



A1

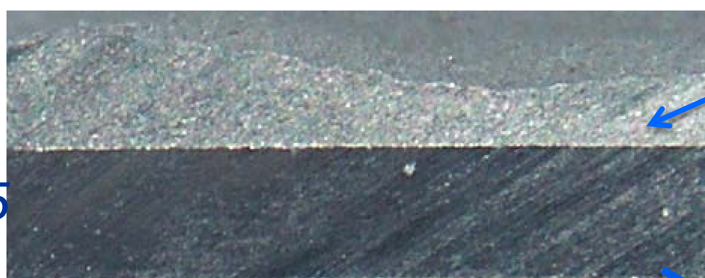
1.30 mm

A2

Inconel 625 substrate

ODS Coating

1.75 mm

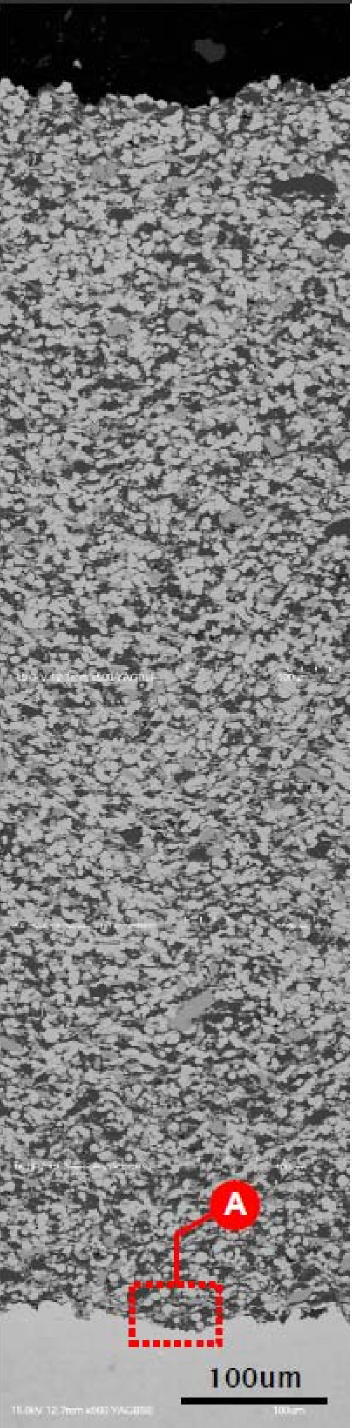


B1

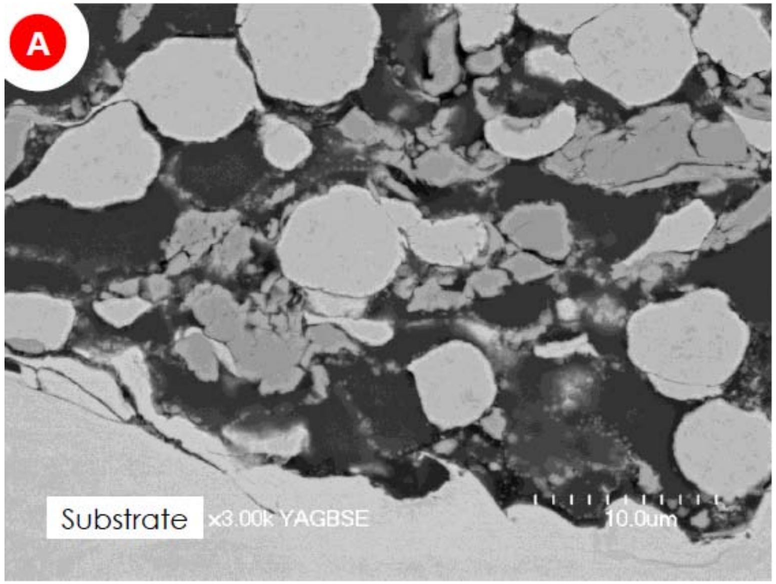
1.75 mm

B2

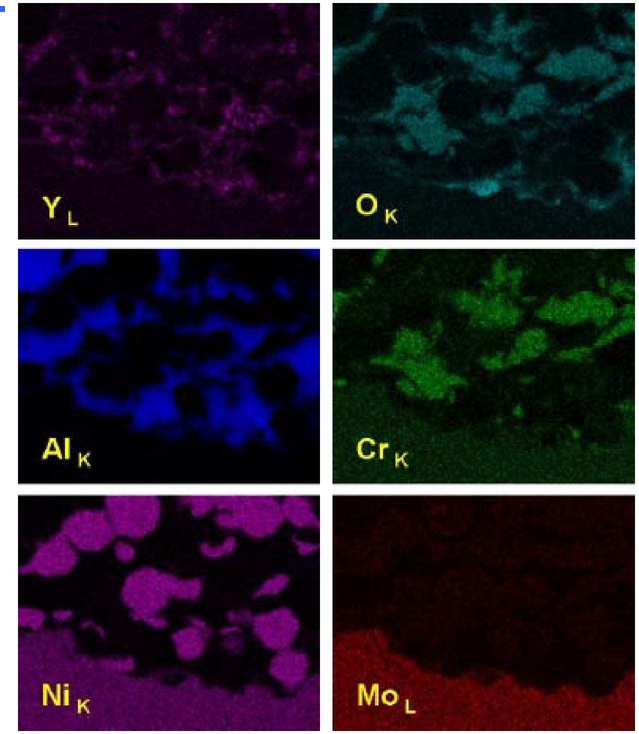
Inconel 625 substrate



A1 coating & its EDX analysis



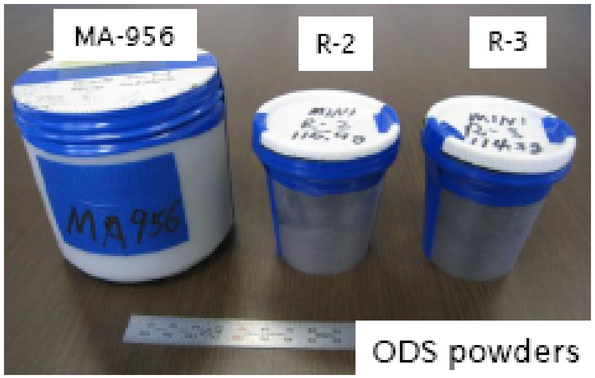
BSE image of region A, located at the coating/substrate interface



EDX element mapping of left region A

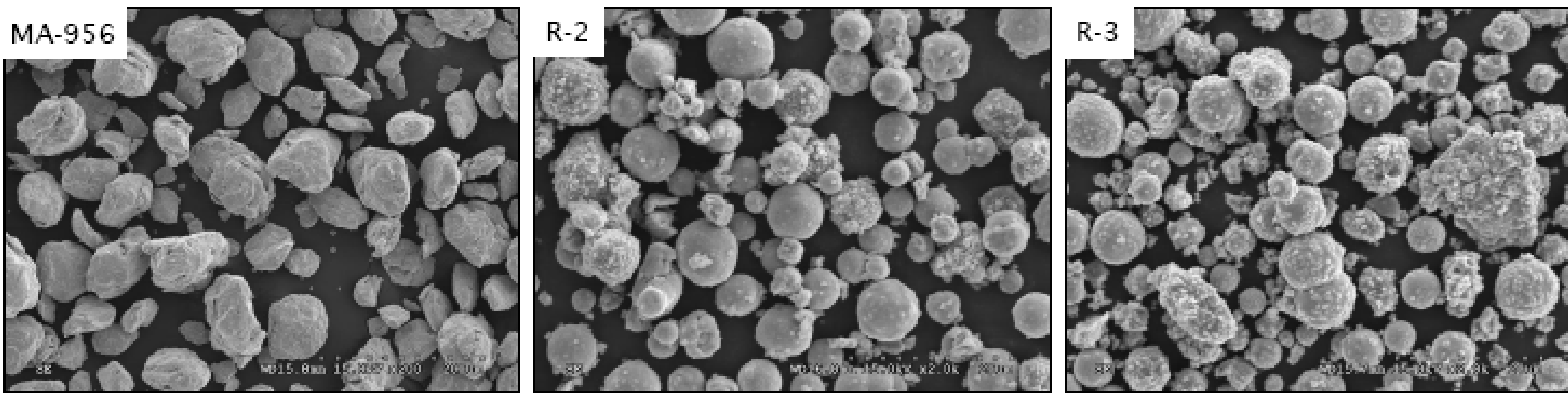
1)Coating is about 850um thick, dense, and well-adherent to each other particles. Most particles are well-distributed in whole coating layer, relatively uniform.
2)Most Al and Cr particles are full-deformed, especially the plastic deformation of Al particles may play an important role in the deposition mechanism.
3)Yttrium oxides are distributed uniformly at the interfaces among Al, Cr, Ni splats.
4)Most nickel particles keep their spherical shape with partial deformation only.
5)Chrome oxide showing in EDX maps results from the pre-holding compositions, may not from oxidizing in cold-spray gas stream.

2nd Round MCB-processed ODS Powders (FY09-10)



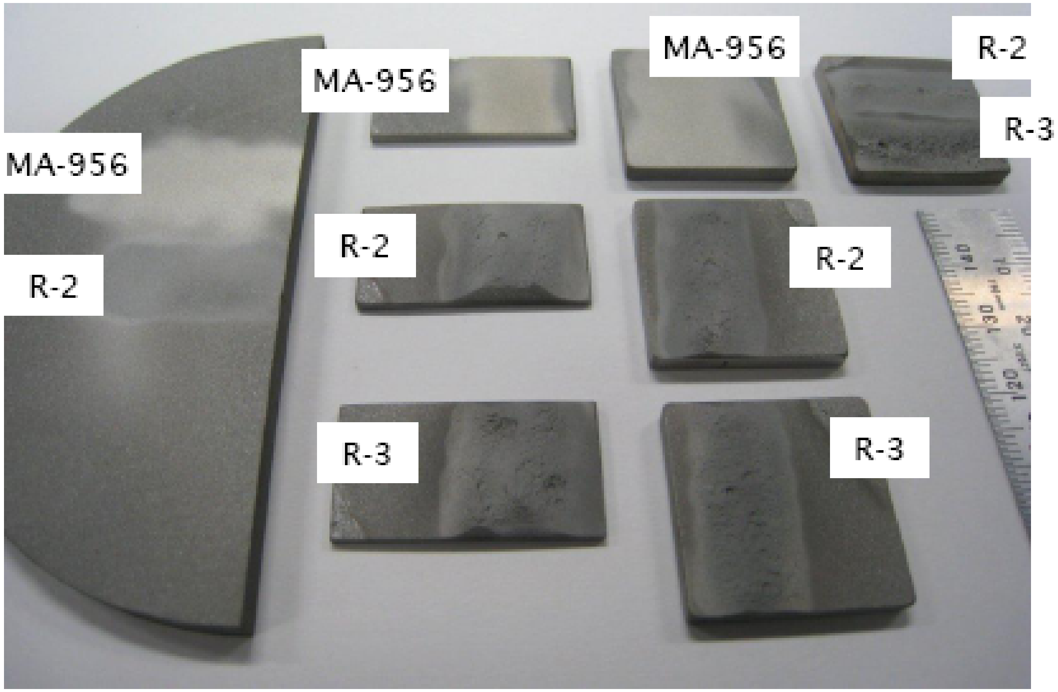
Nominal composition (wt%)	
MA-956	Fe-20Cr-4.5Al-0.5Ti-0.5Y_2O_3-0.02C

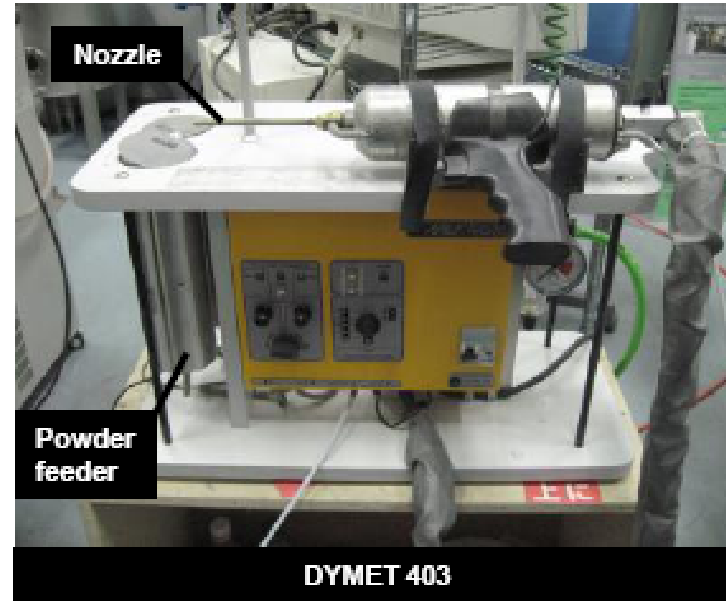
Particle size distribution (um)					
	20Cr	5Al	1.5Y_2O_3	3W	70.5Ni
R-2	7.5-10	4.5-7	<50nm	0.5-1	4-8
R-3	1-5	1-3	<50nm	-1	1-5



2nd Round Cold Spray Coating

Substrate;
Inconel 738LC   Inconel 625   Haynes 230   ReNe N5





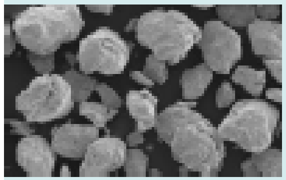
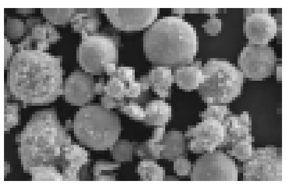
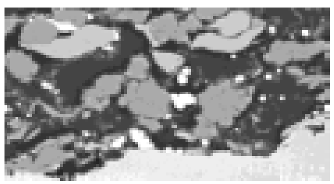
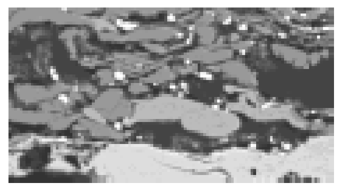
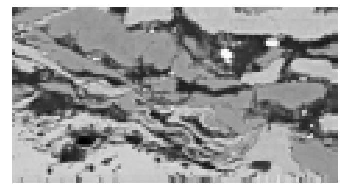
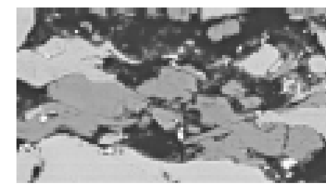
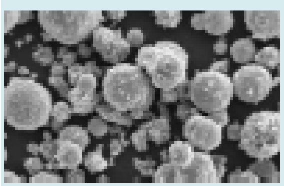
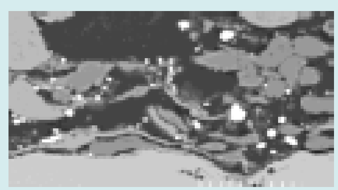
LP cold spray system used in this work.

Cold spray condition
Model: Dymet 403J, OCPS, Russia Supplying gas: Air / 0.8MPa Heating temperature: 400degC Stand-off distance from nozzle: 15mm Layer: approx. 2mm pitch, 4 pass

Pretreatment of substrate
Sandblasted by media size of 106-125um

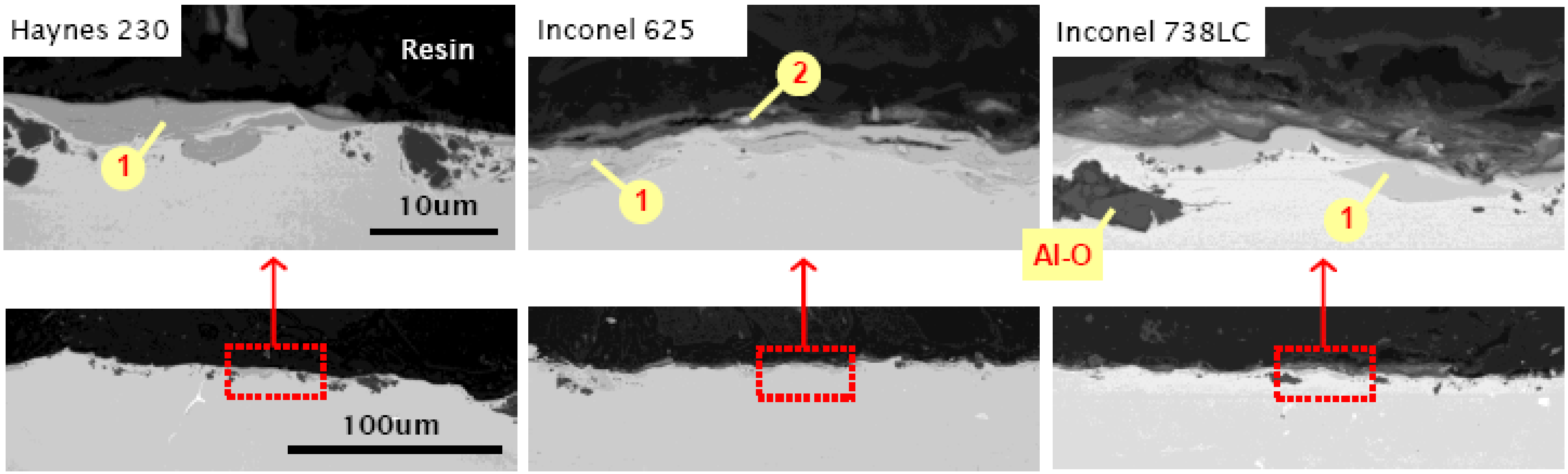
1) R-2 and R-3 were deposited well on the Haynes 230, ReNe'N5 and Inconel 625 substrate, with a good efficiency.
2) MA-956 powder was slightly deposited on all kinds of substrate, and the feeding trouble happened during spray.

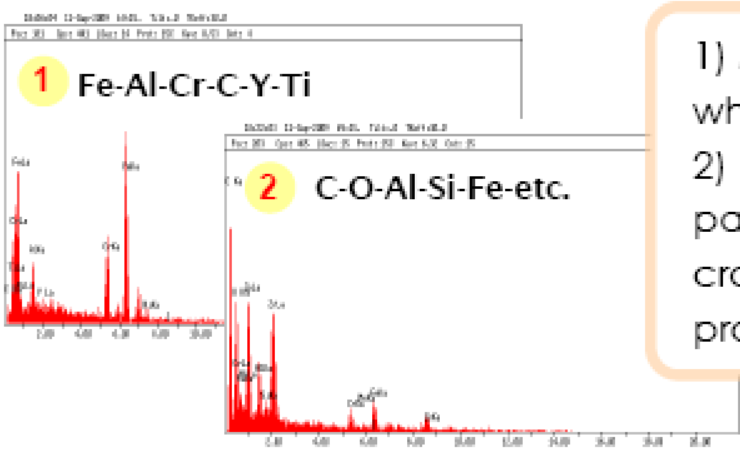
Overview of 2nd ODS coating by LP CS

Substrate / ODS Powder	Haynes 230	ReNe'N5	Inconel 625	Inconel 738LC
MA-956 	Failed [1] Limited in <4um-thick 	– [2]	Failed [1] Limited in <5um-thick 	Failed [1] Limited in <4um-thick 
R-2 	Successful Ave 870um-thick 	Successful Ave 700um-thick 	Successful Ave 1200um-thick 	Half-successful [3] Ave 30um-thick 
R-3 	Successful Ave 570um-thick 	Successful Ave 800um-thick 	Successful Ave 780um-thick 	– [4]

1) Discontinuous and limited in thickness of 4-5 micro.
2) Not tried due to the insufficiency of ReNe'N5 substrate.
3) Has a low deposition efficiency.
4) Not tried due to the insufficiency of R-3 powder.

1) MA-956 coatings & EDX



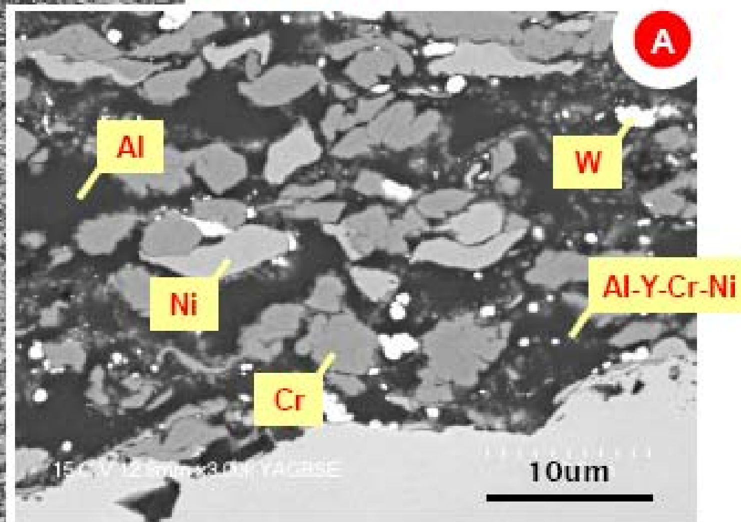


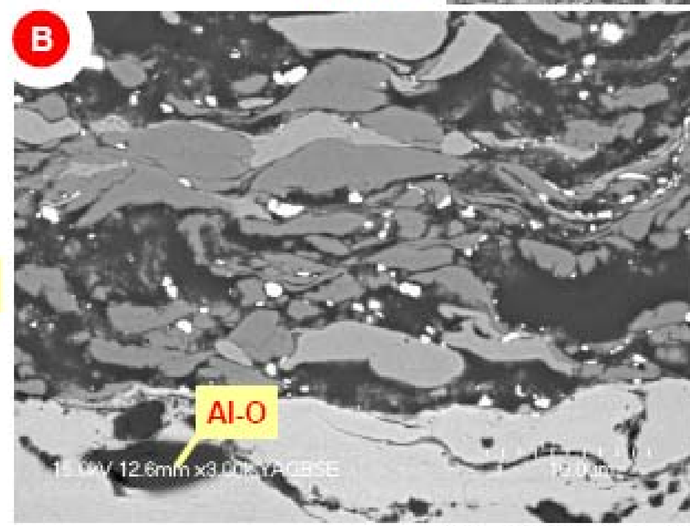
1) MA-956 coatings are not continuous to cover the whole surface of substrates, and too thin (<5um).
2) Al_2O_3 (suggested from Al-O peak; <10um) particles @ the substrate interface could be the crashed pieces of media used in the sand-blasting process.

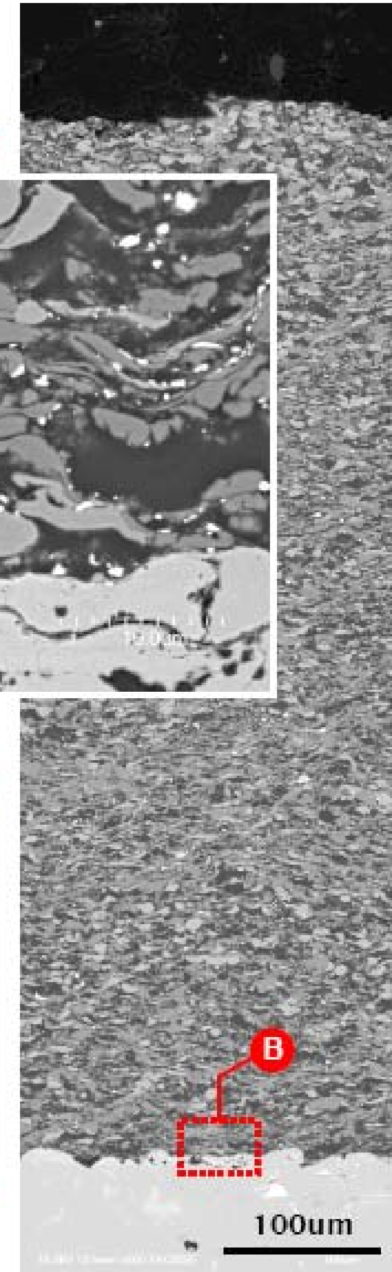
2) R-2 coatings *on Haynes & ReNe*

Haynes 230

ReNe'N5







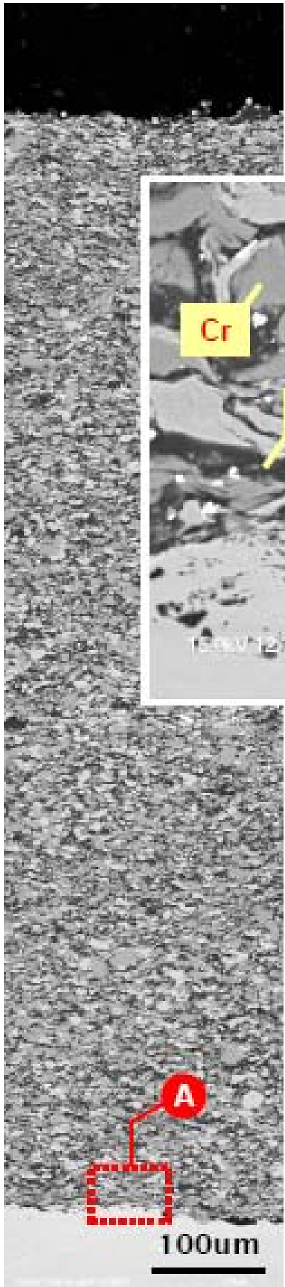
1) Coating is about 870 and 700um-thick, dense, and well-adherent to each other particles. Most particles are well-distributed in whole coating layer, relatively uniform.

2) Most Al particles are full-deformed, and their plastic deformation may play a important role in the deposition mechanism.

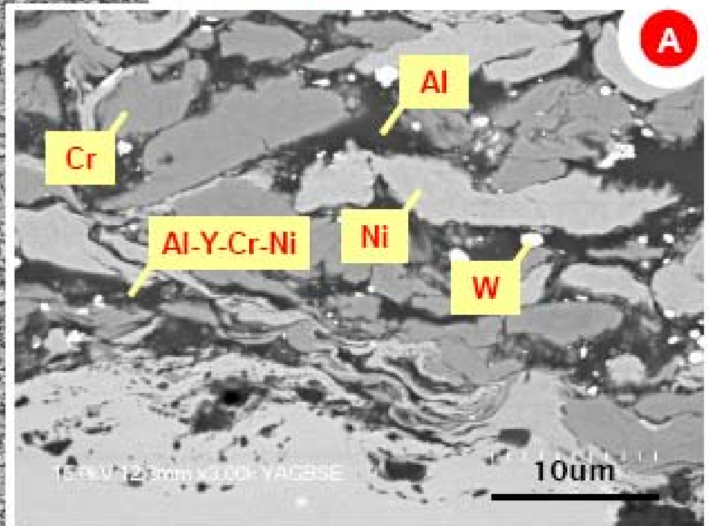
3) Yttrium oxides are distributed uniformly at the interfaces among Al, Cr, Ni splats.

4) Tiny tungsten particles are observed with half-deformed form.

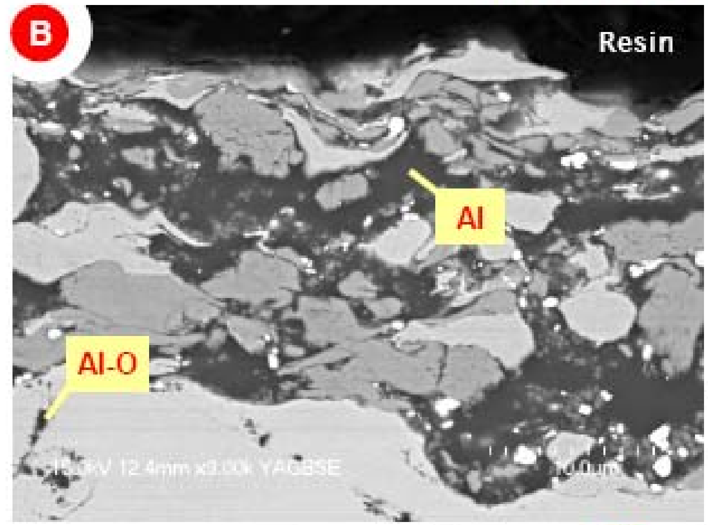
3) R-2 coatings *on Inconels*

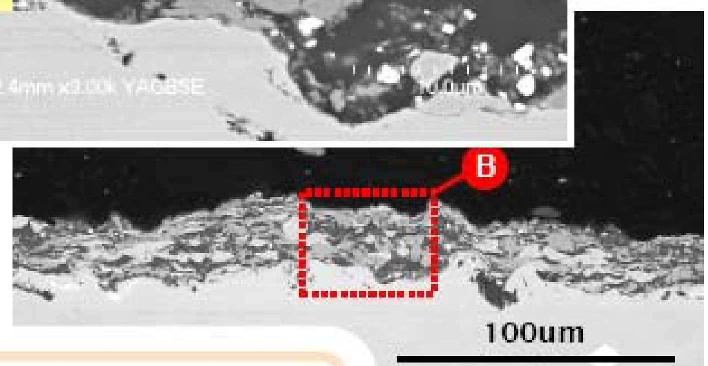


Inconel 625



Inconel 738LC





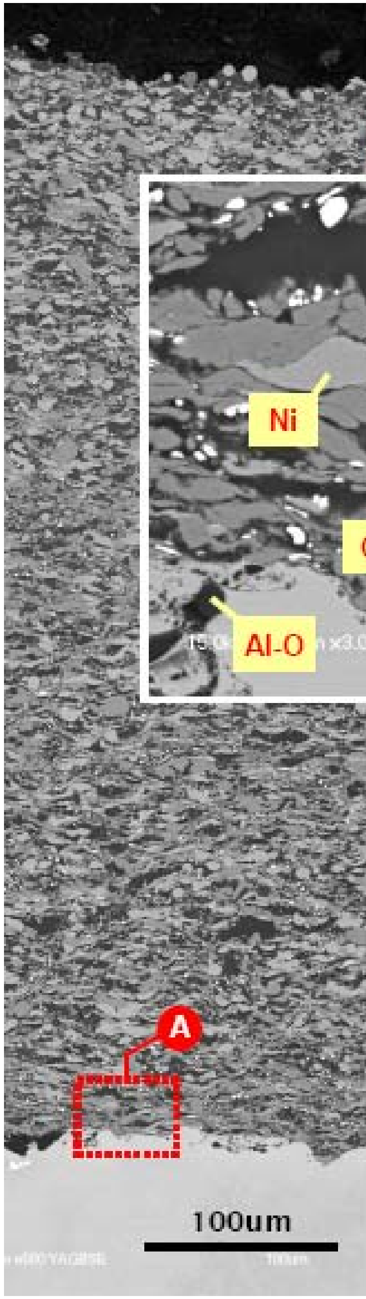
1) R-2 coating on Inconel 625 is about 1200um-thick, dense, and well-adherent to each other particles.
2) In case of Inconel 738LC is relatively thin (below 30um) and most of layers consist of the deformed Al particles. Deposition is possible, but it shows the low deposition efficiency.

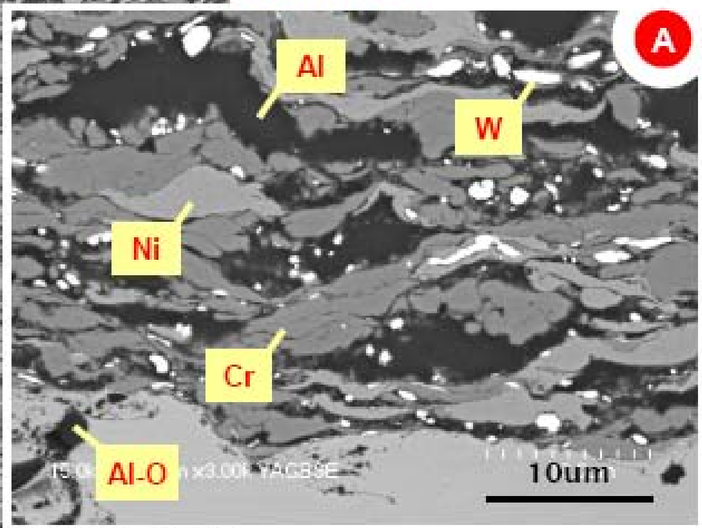
2009/09/17

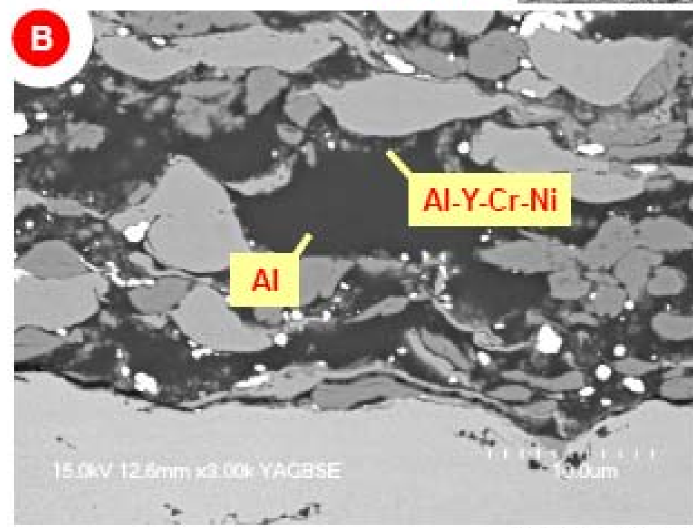
4) R-3 coatings *on Haynes & ReNe*

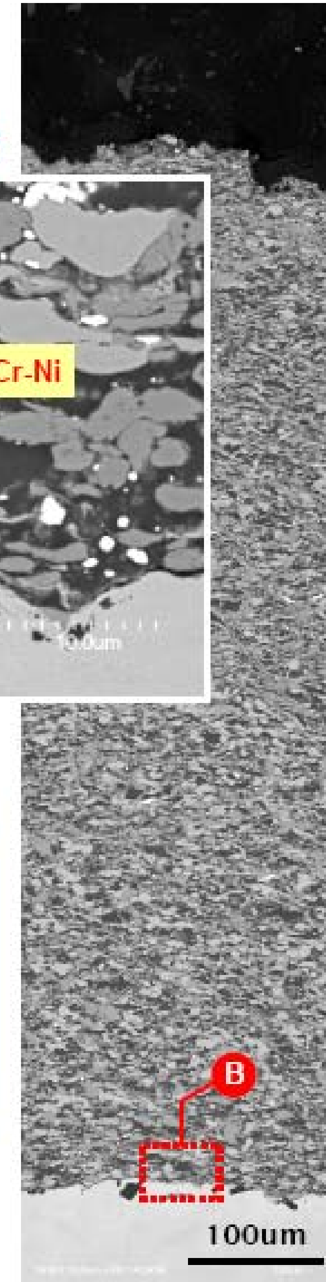
Haynes 230

ReNe'N5









1) Coating is about 570 and 800um-thick, dense, and well-adherent to each other particles.
2) In case of R-3 coating on Haynes, Most of metallic particles (Cr, Ni, Al) are full-deformed well comparing the ReNe coating. Tungsten particles in the Haynes coating are also distributed uniformly.

MCB plus Ball-Milling

ODS Powders Ball Milling Plan:

Samples: R1, R4, R5, R7 (2nd Round MCB powders).

Ball-to-powder ratio: 30:1, with 0.5 wt.% stearic acid as PCA.

Rotating speed: 300 rpm.

Duration: ***2 hrs, 6 hrs, 15 hrs, 30 hrs, 60 hrs, 84 hrs, 120 hrs***.

Ball Milling Procedure:

1): Sample preparation

a. pump and feed the glove box with argon for 2 hrs to remove air

b. weigh each sample under inert atmosphere with ball-to-powder ration of 30:1 and 0.5 wt.% stearic acid as PCA.

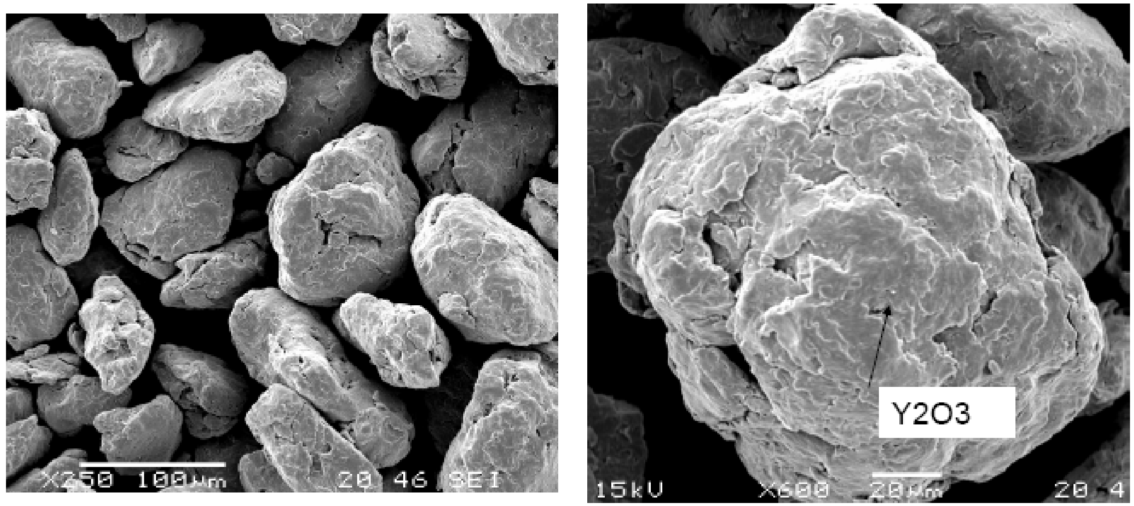
2): Ball milled samples (R1, R4, R5, R7) under ***argon*** *atmosphere;* ***at selected hours, extract a few powders from each sample under argon atmosphere which were analyzed using SEM and TEM.***

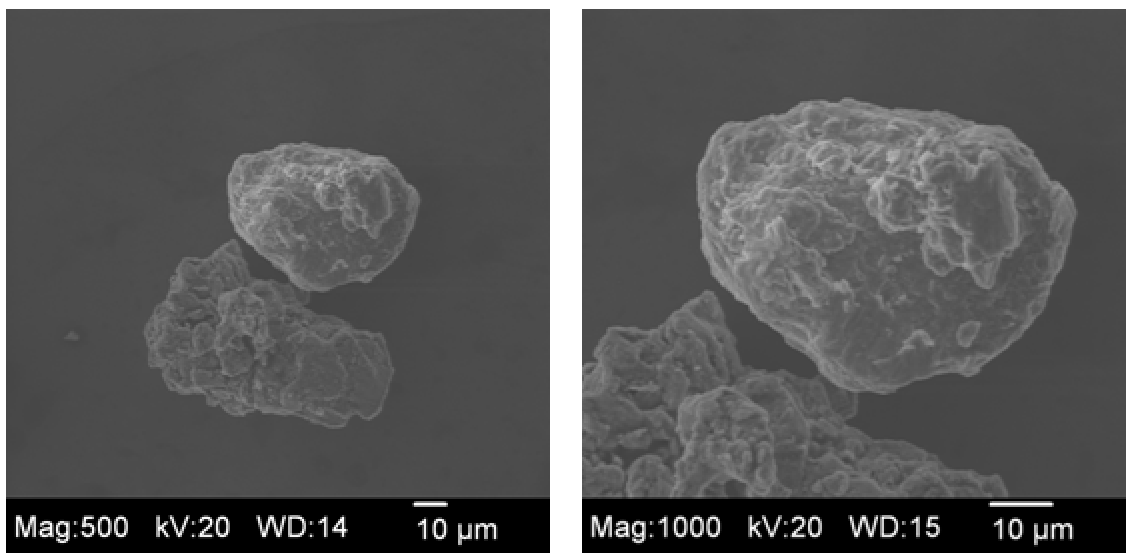
Sample	Total Ball Milling Time (hours)								
	0	2	4	6	15	30	60	84	120
*R1	√	√	√	√	√	√	√	√	
*R4	√	√	√	√	√	√	√	√	
**R5	√	√	√	√	√	√	√	√	
**R7	√	√	√	√	√	√	√	√	
A1 (without MCB)	√	√	√	√	√	√	√	√	√
A2 (without MCB)	√	√	√	√	√	√	√	√	√

*: R1, R2, R3, and R4 were processed using high energy MCB (~ 90 minutes)

**: R5, R6, R7, and R8 were processed using lower energy MCB (~ 30 minutes)

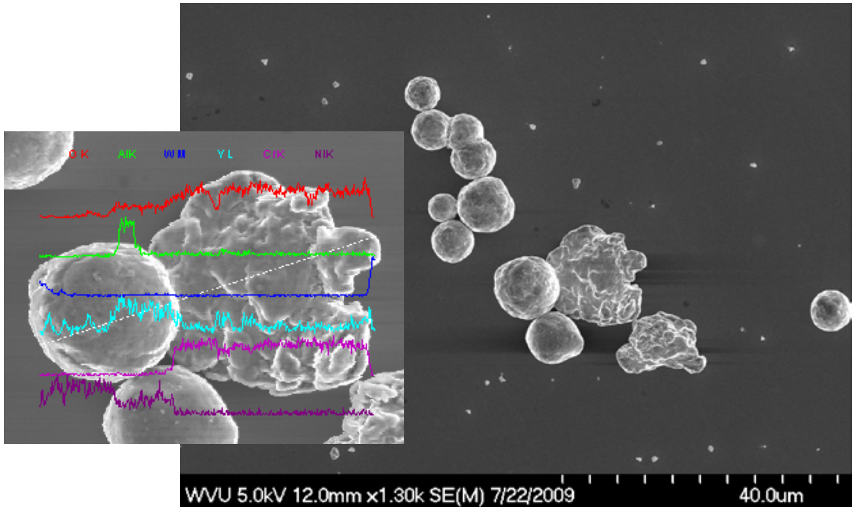
MA 956 ODS sample *(Special Metals Inc.)*



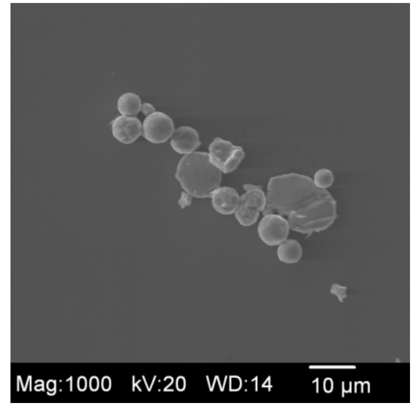


R1 sample with 15 hrs ball milling

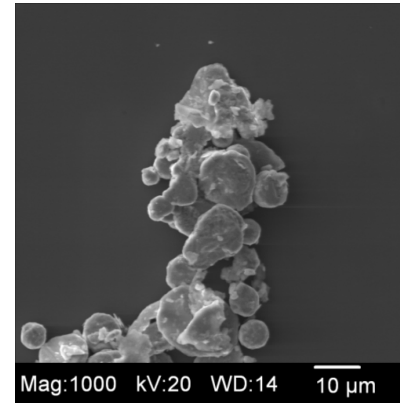
Sample R1 *(MCB + Ball Mill)*



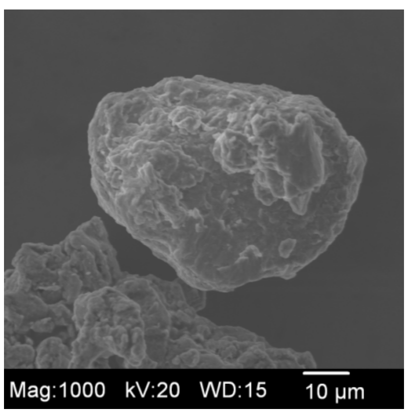
Before ball milling



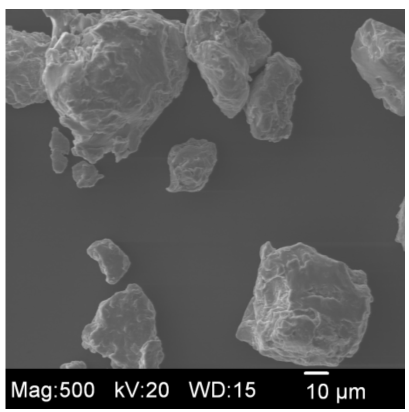
Ball milling for 2 hrs



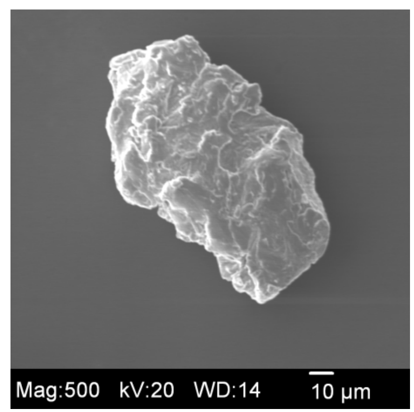
Ball milling for 6 hrs



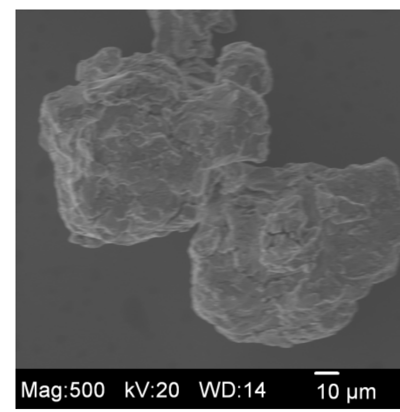
Ball milling for 15 hrs



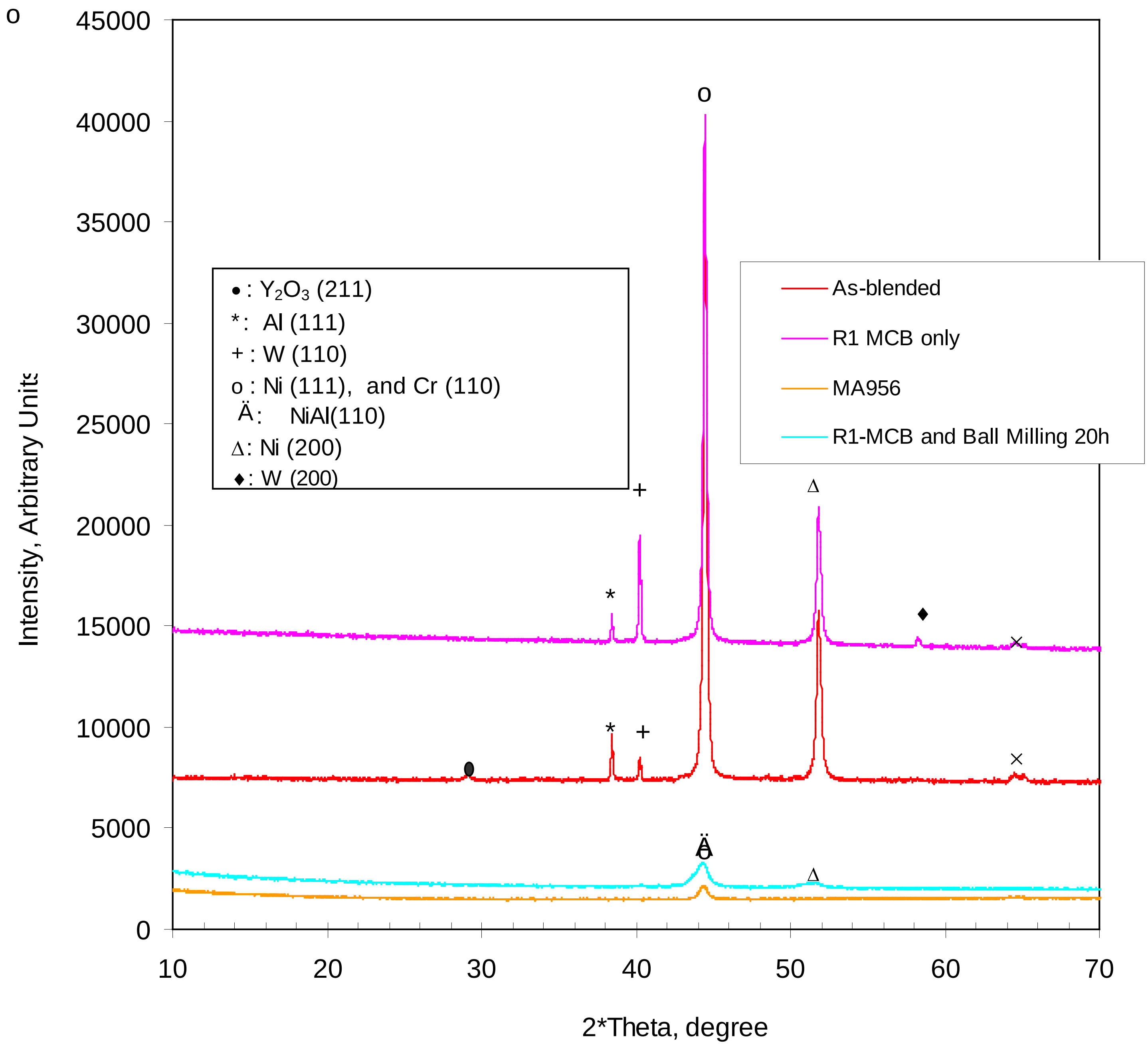
Ball milling for 30 hrs

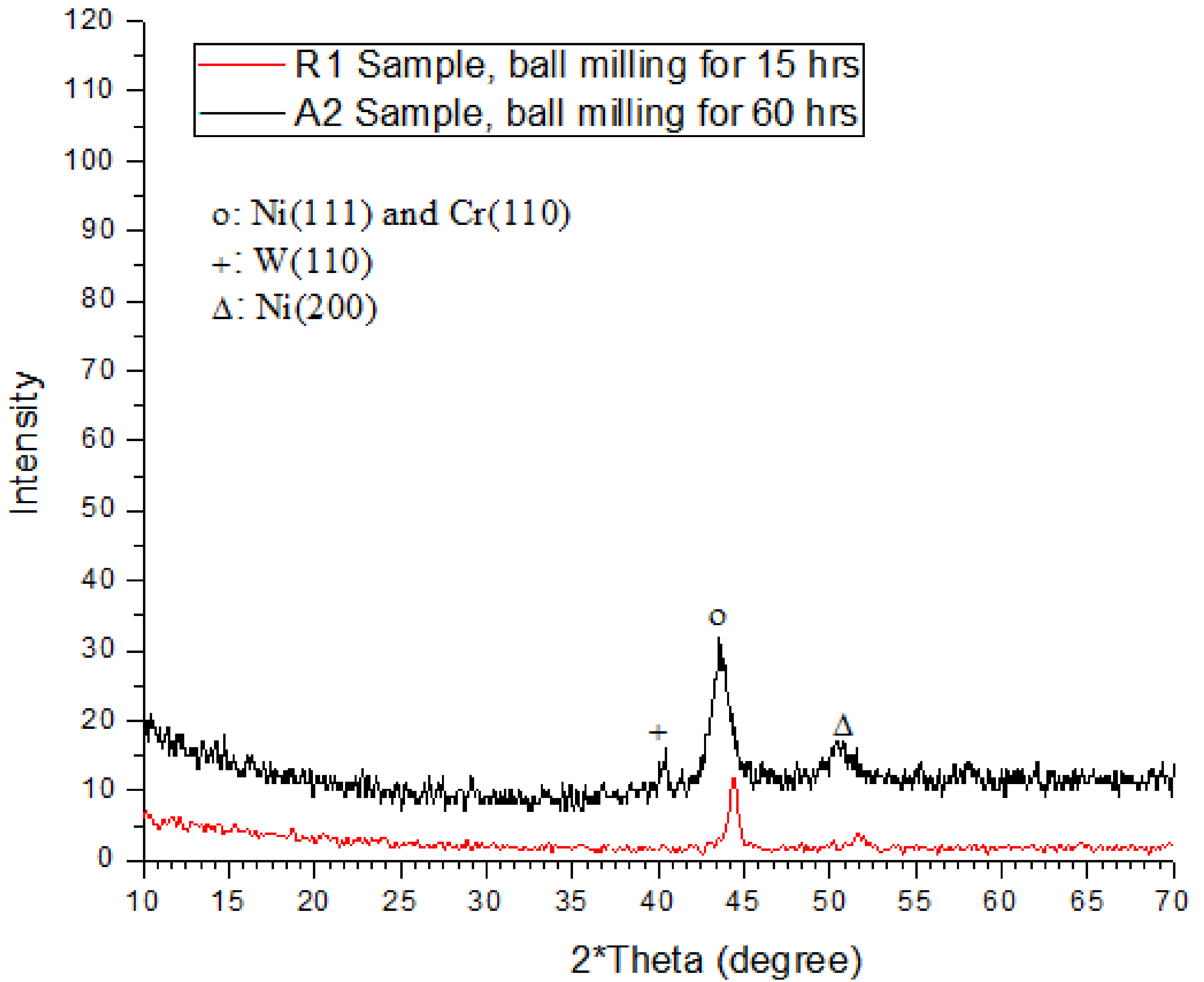


Ball milling for 60 hrs

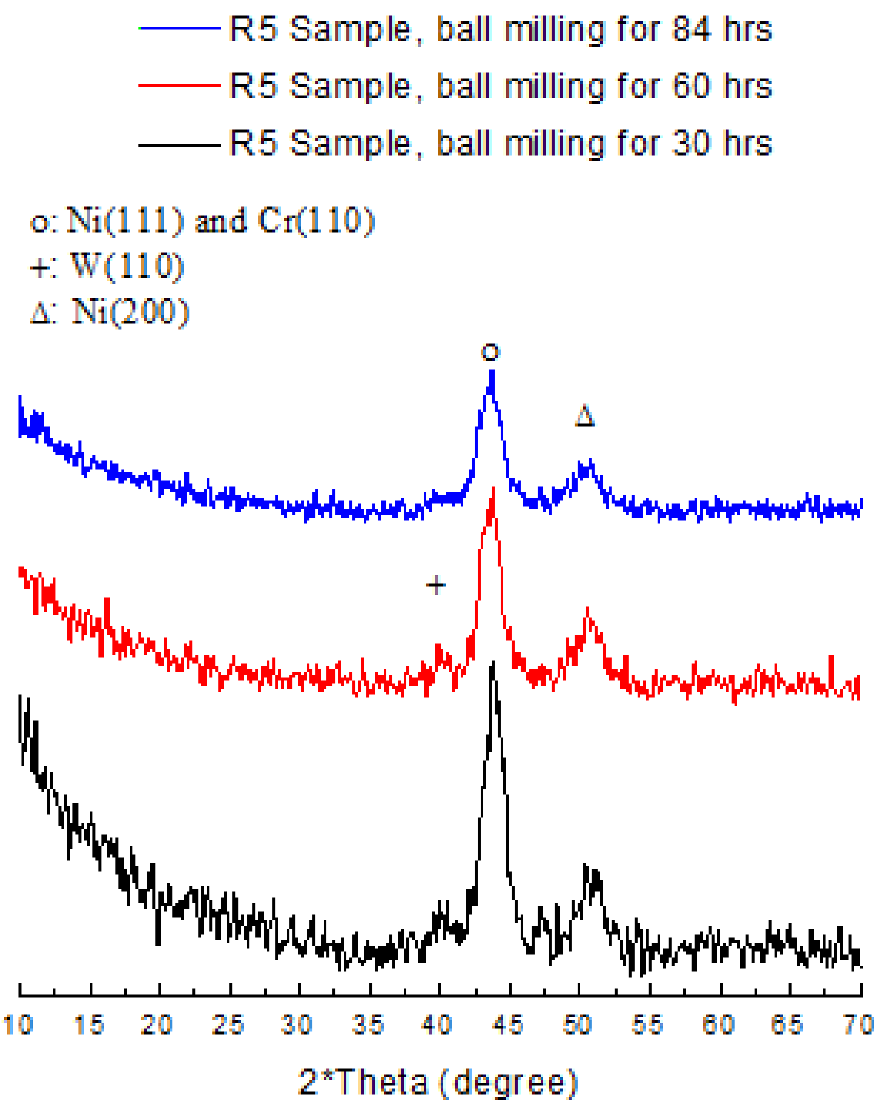


Ball milling for 84 hrs

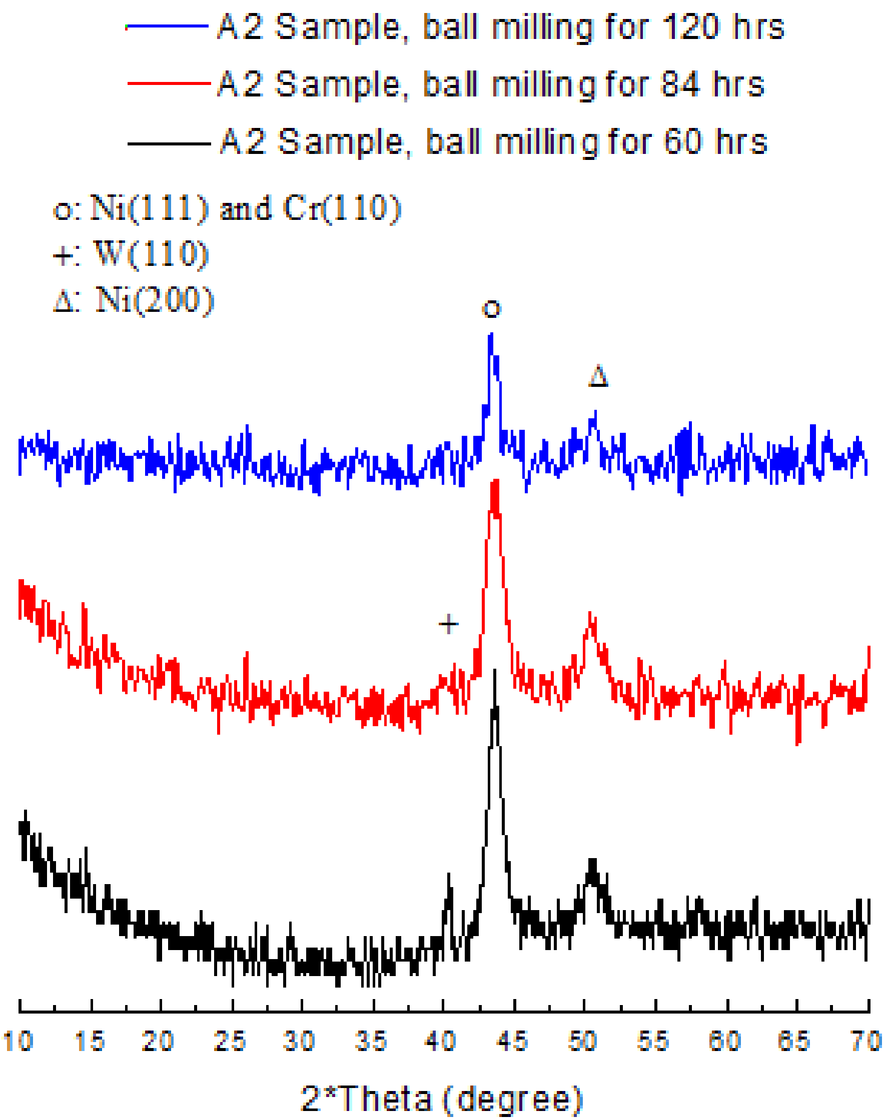

o
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
Intensity, Arbitrary Units
10
20
30
40
50
60
70
2*Theta, degree
●: Y_2O_3 (211)
*: Al (111)
+ : W (110)
o : Ni (111), and Cr (110)
Ä: NiAl(110)
Δ: Ni (200)
♦: W (200)
As-blended
R1 MCB only
MA956
R1-MCB and Ball Milling 20h

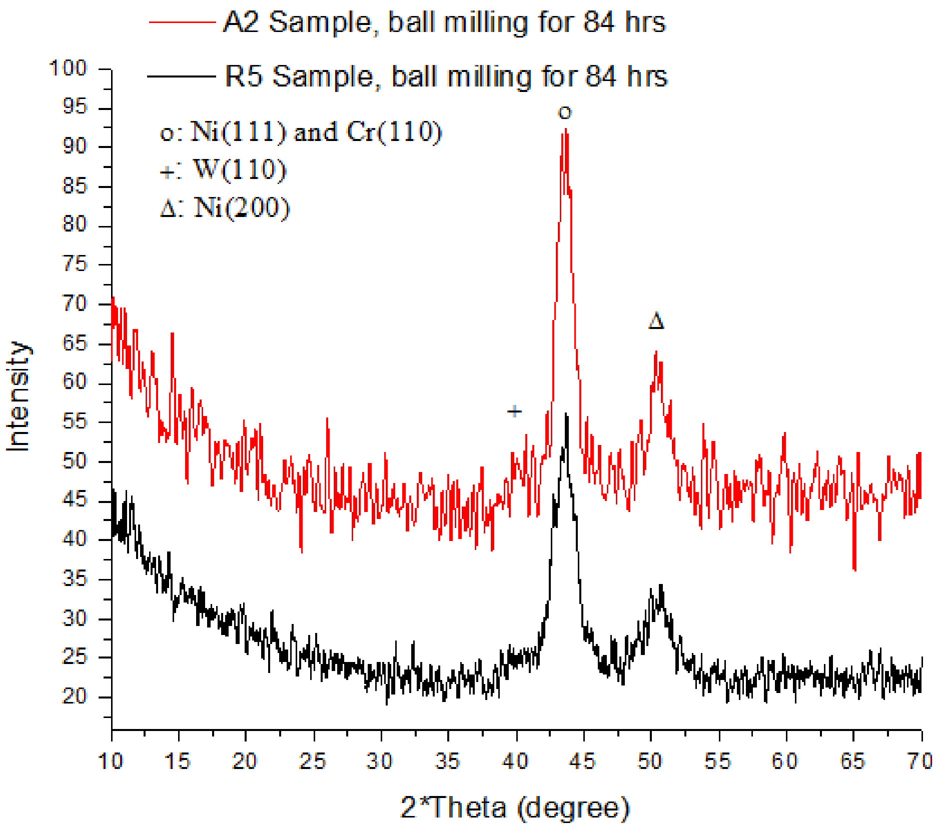

R1 Sample, ball milling for 15 hrs
A2 Sample, ball milling for 60 hrs
o: Ni(111) and Cr(110)
+: W(110)
Δ: Ni(200)
Intensity
2*Theta (degree)
120
110
100
90
80
70
60
50
40
30
20
10
0
10
15
20
25
30
35
40
45
50
55
60
65
70
o
+
Δ

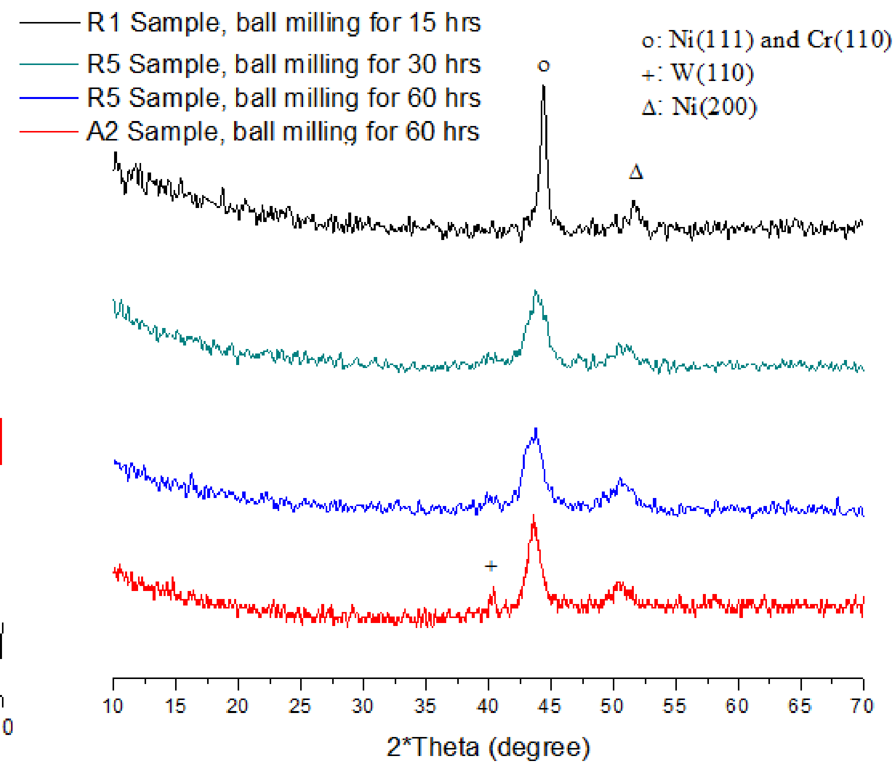
(MCB + Ball Mill)

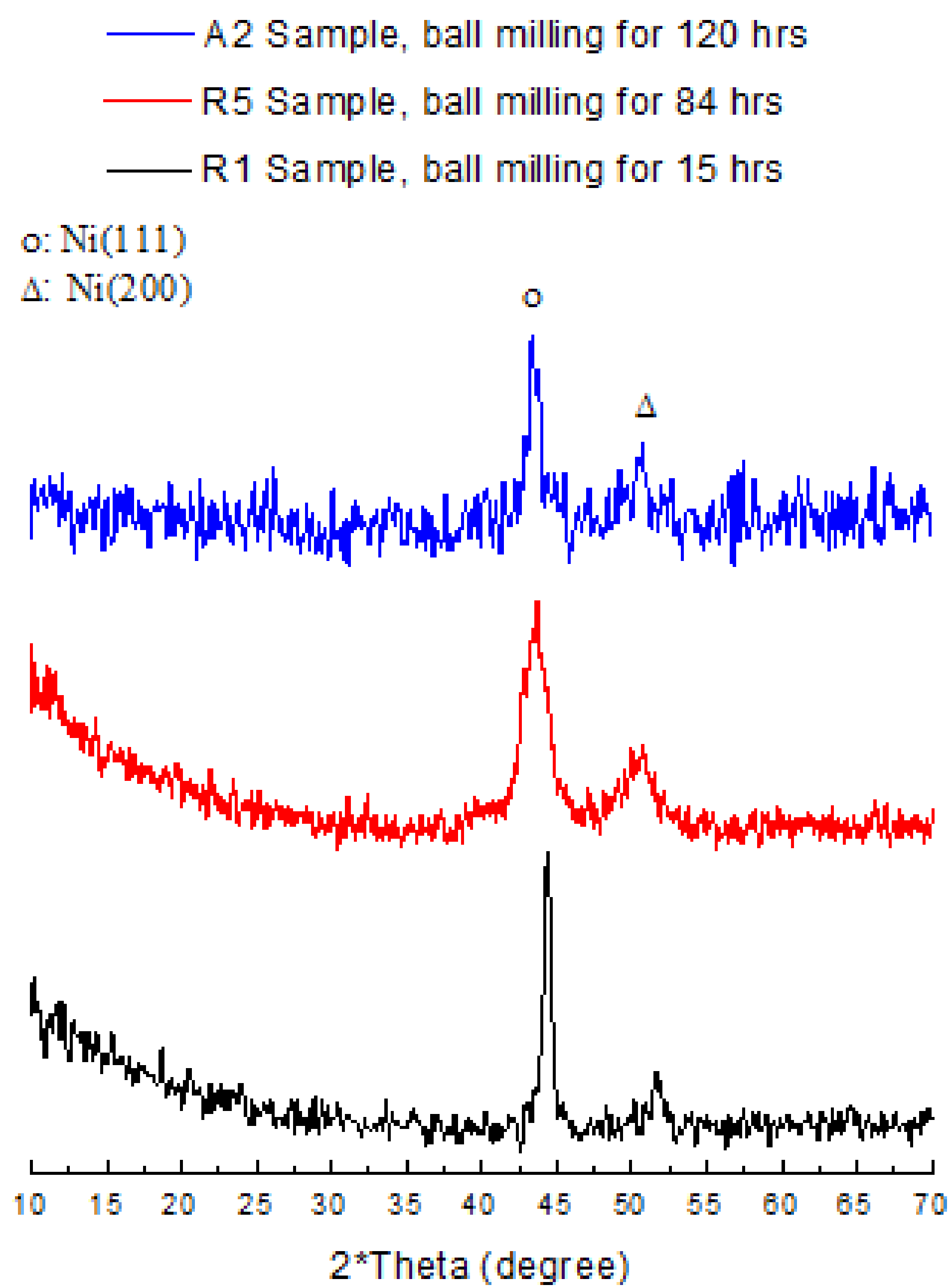


(Ball Mill only)

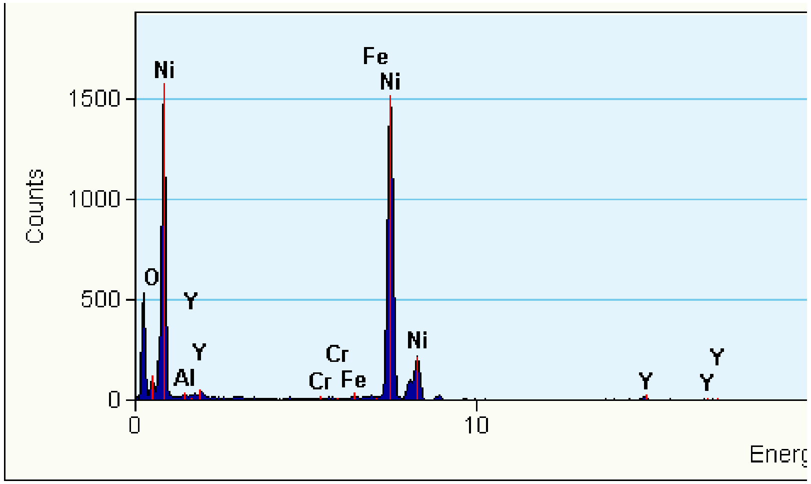


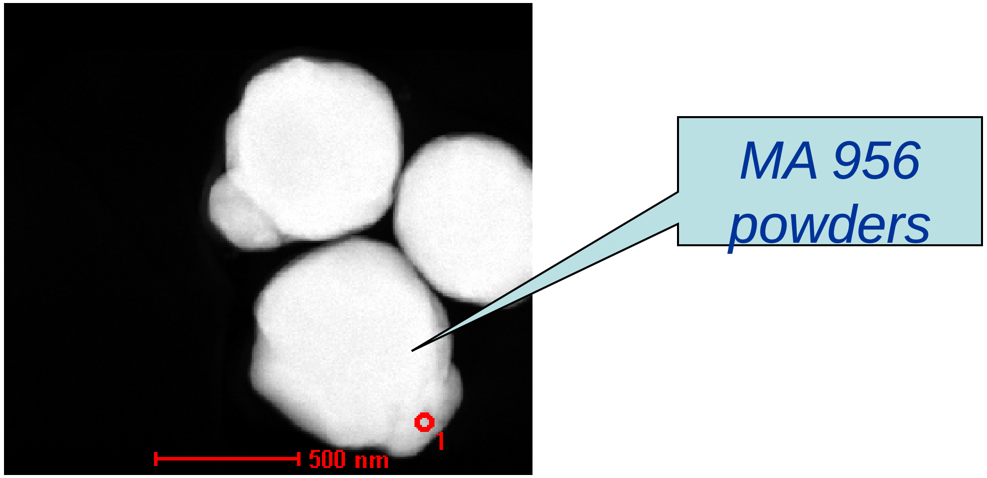

A2 Sample, ball milling for 84 hrs
R5 Sample, ball milling for 84 hrs
o: Ni(111) and Cr(110)
+: W(110)
Δ: Ni(200)
Intensity
2*Theta (degree)

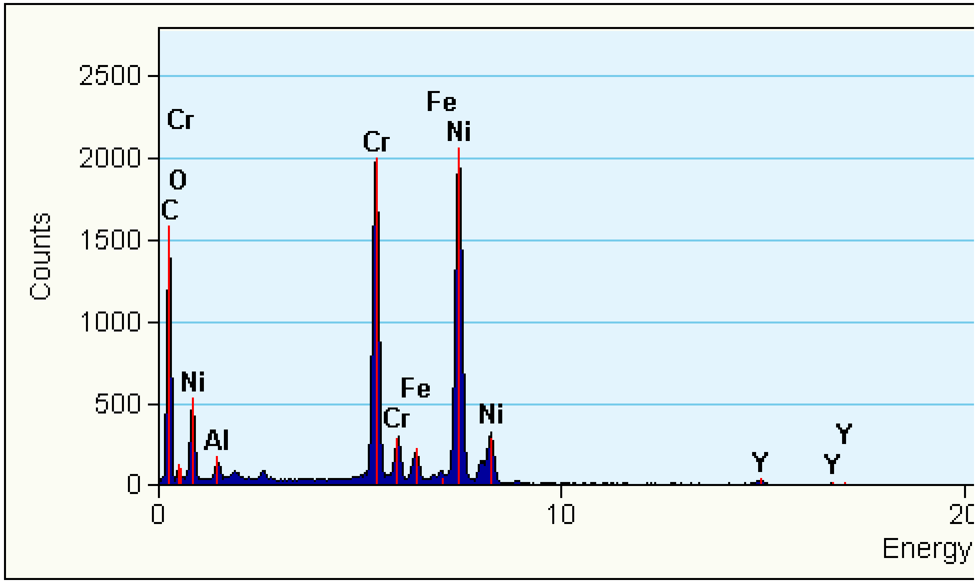

R1 Sample, ball milling for 15 hrs
R5 Sample, ball milling for 30 hrs
R5 Sample, ball milling for 60 hrs
A2 Sample, ball milling for 60 hrs
o: Ni(111) and Cr(110)
+: W(110)
Δ: Ni(200)
2*Theta (degree)

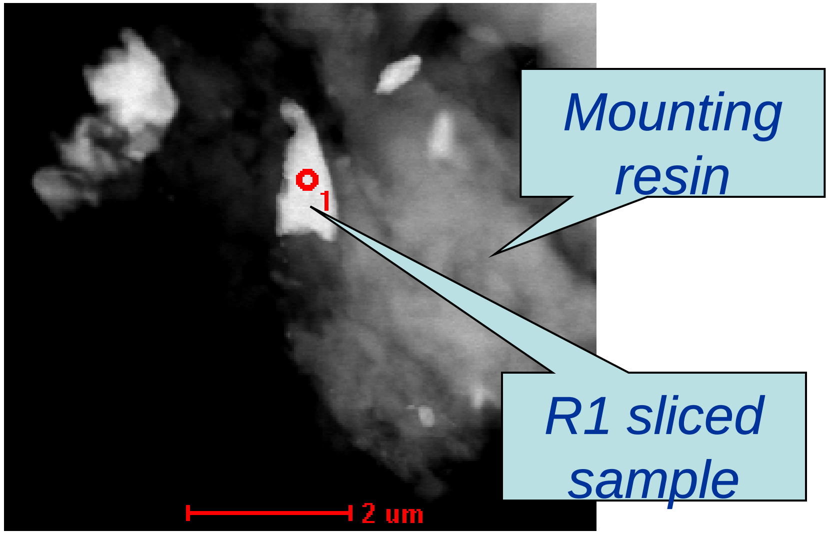


A2 Sample, ball milling for 120 hrs
R5 Sample, ball milling for 84 hrs
R1 Sample, ball milling for 15 hrs
o: Ni(111)
Δ: Ni(200)
o
Δ
10 15 20 25 30 35 40 45 50 55 60 65 70
2*Theta (degree)



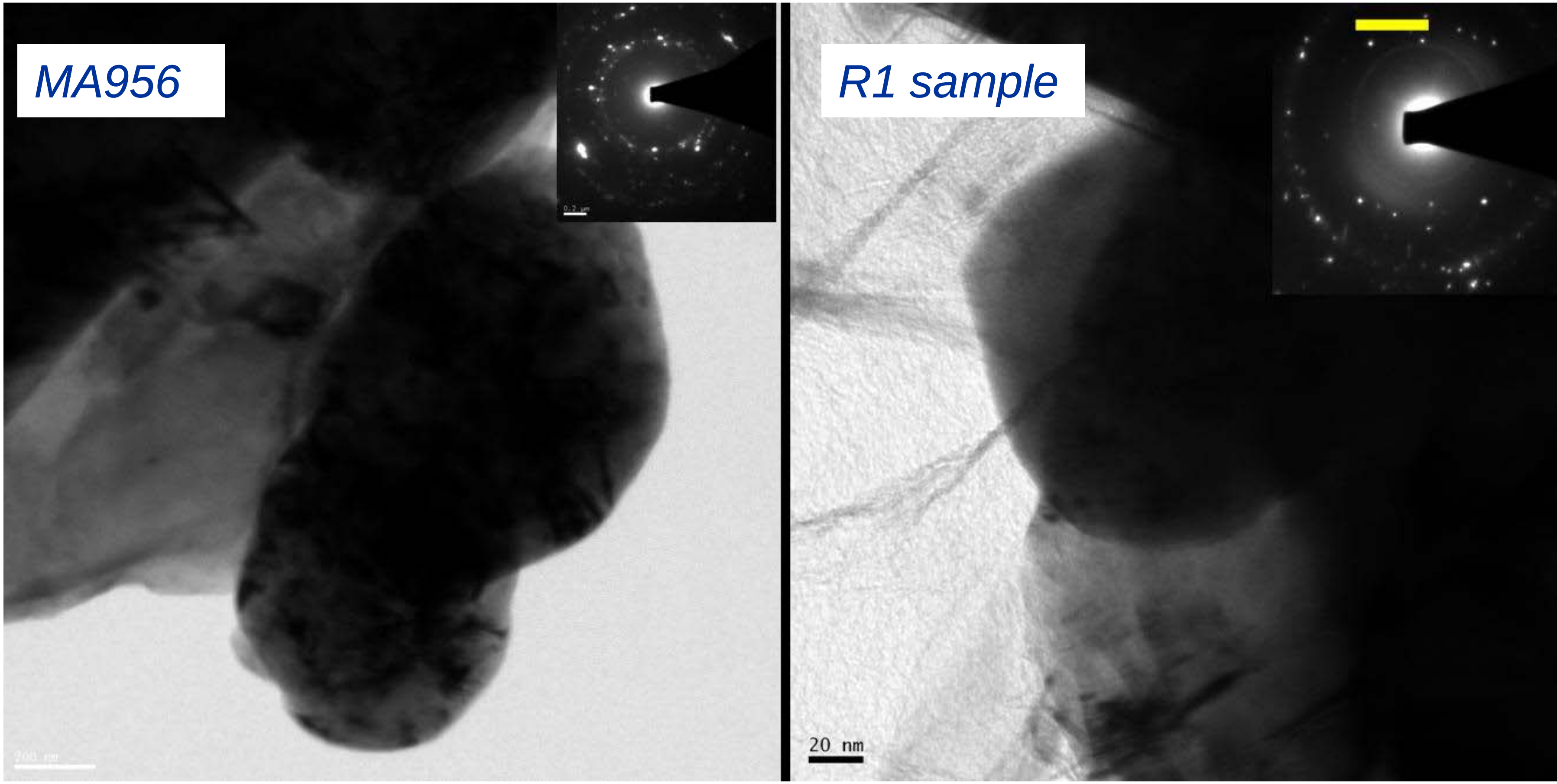






STEM and EDX

- *For MA 956, hosting particles consist of Ni and Fe, and a few of Cr, Al, indicating Fe and Ni were well mixed.*
- *For R1 sample, cross section of powder shows Fe, Cr, Al, Ni and Y were well mixed. There are relatively higher Al, Cr and Y counts in R1 sample then in MA 956*



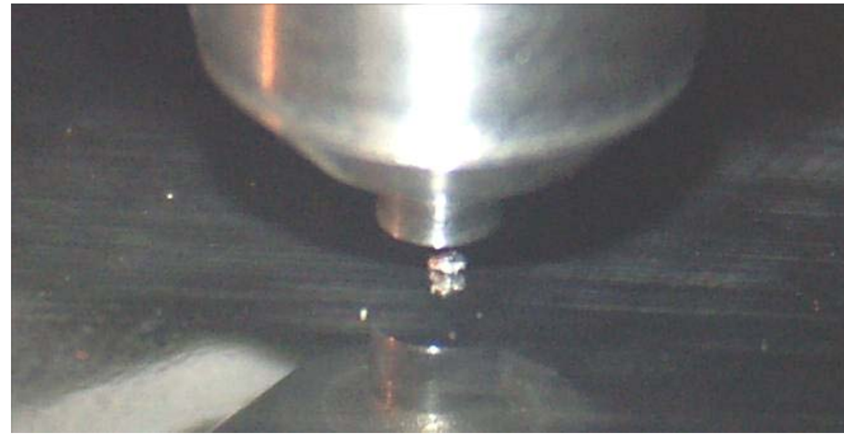
TEM results

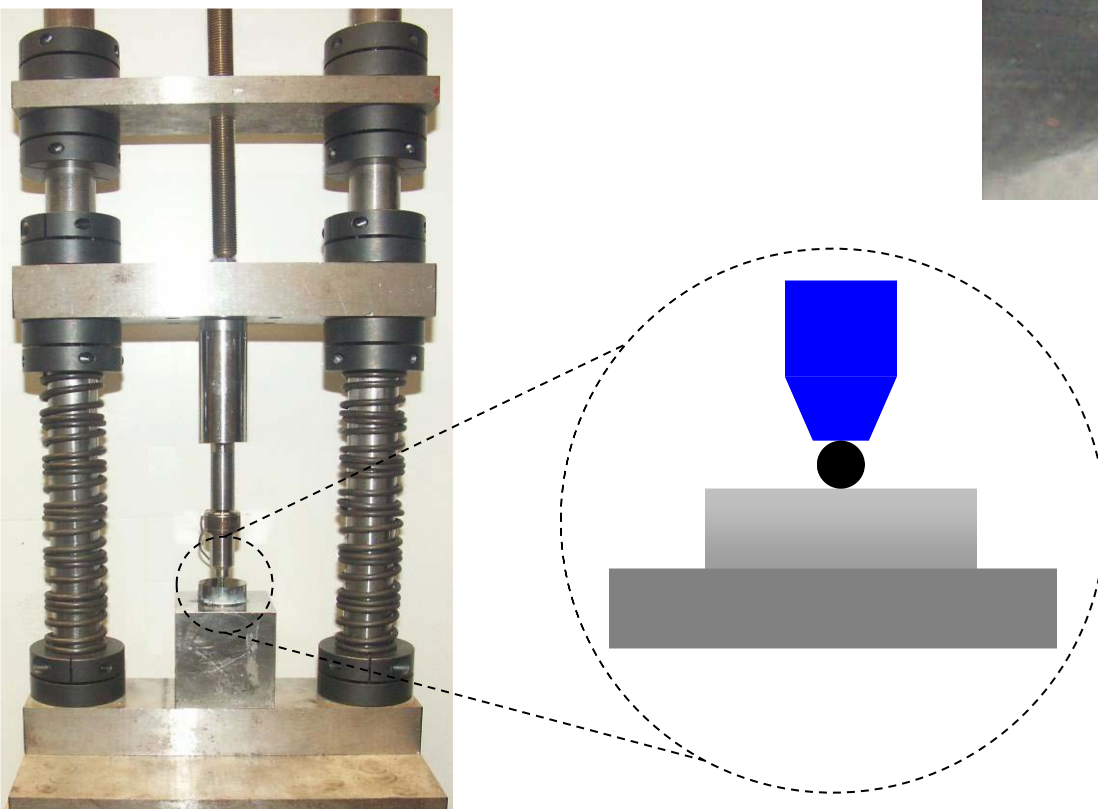
- For MA 956, many dislocations were fund inside particles, indicating heavy deformation during ball milling as well as many tiny particles were embedded into particles. SAD shows particle is polycrystalline.
- For R1 sample, TEM image and SAD show the similar structure to MA 956, indicating heavy deformation,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

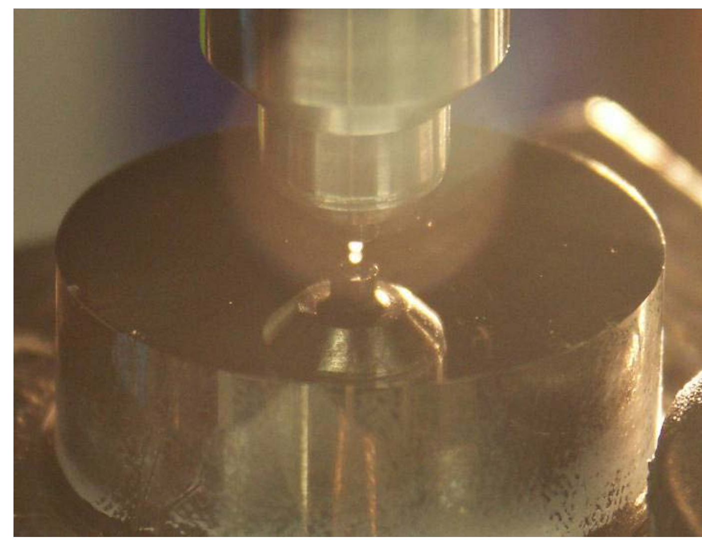
ODS Powder Instrumented Micro-Indentation

MA956 ODS Powder

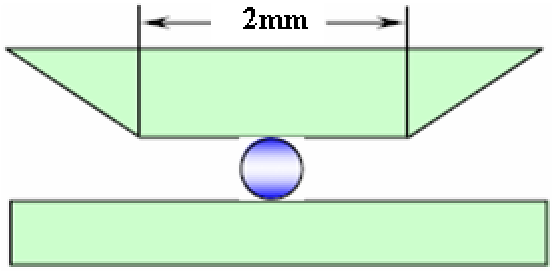
Flat punch tungsten carbide indenter and indentation surface are used.





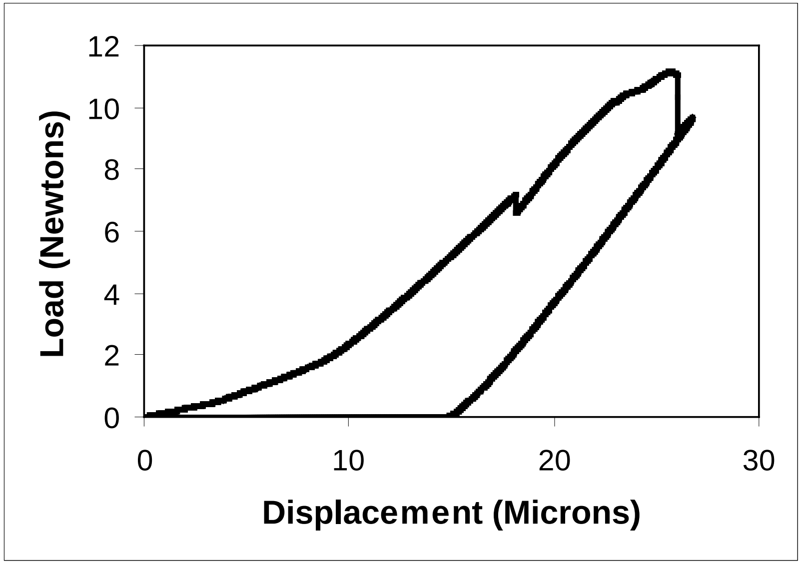


Micro-Indentation



Flat Punch Indenter

MA 956 ODS Sample Particle



R1 Sample Particle (MCB with 15 hrs Ball Milling

(Critical ODS processing parameters)

Stage (I) - Deformation (i.e. kinetic strength or stored energy) prior to recrystallization (the higher the kinetic strength, the higher the Grain Aspect Ratio (GAR) or higher anisotropic columnar grains after recrsystallizaton),

Stage (II) - *Annealing temperature* (usually approaching 0.9 T_M , where T_M is the melting temperature, as compared to the typical recrystallization temperature of 0.6 T_M in similar casted metallic alloys).

Q: *Similar to Stage (1),* ***can cold spray consolidate ODS powders with grains ?***

Example of HP-Cold Sprayed CoNiCrAlY bond coat

(Noted that nano-size grains are formed)

Experimental Example

Material preparation

Coating material: CoNiCrAlY
(SULZER Metco AMDRY9951)

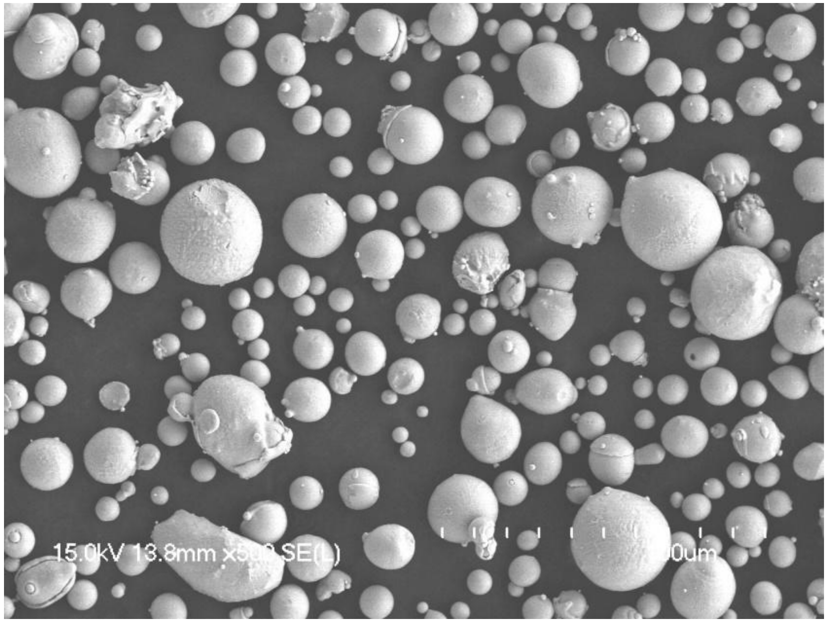
Co	Ni	Cr	Al	Y
Bal.	32	21	8	0.5

(wt.%)

Substrate: Inconel 625

Ni	Cr	Mo	Nb	Fe
Bal.	24	9	4	3.5

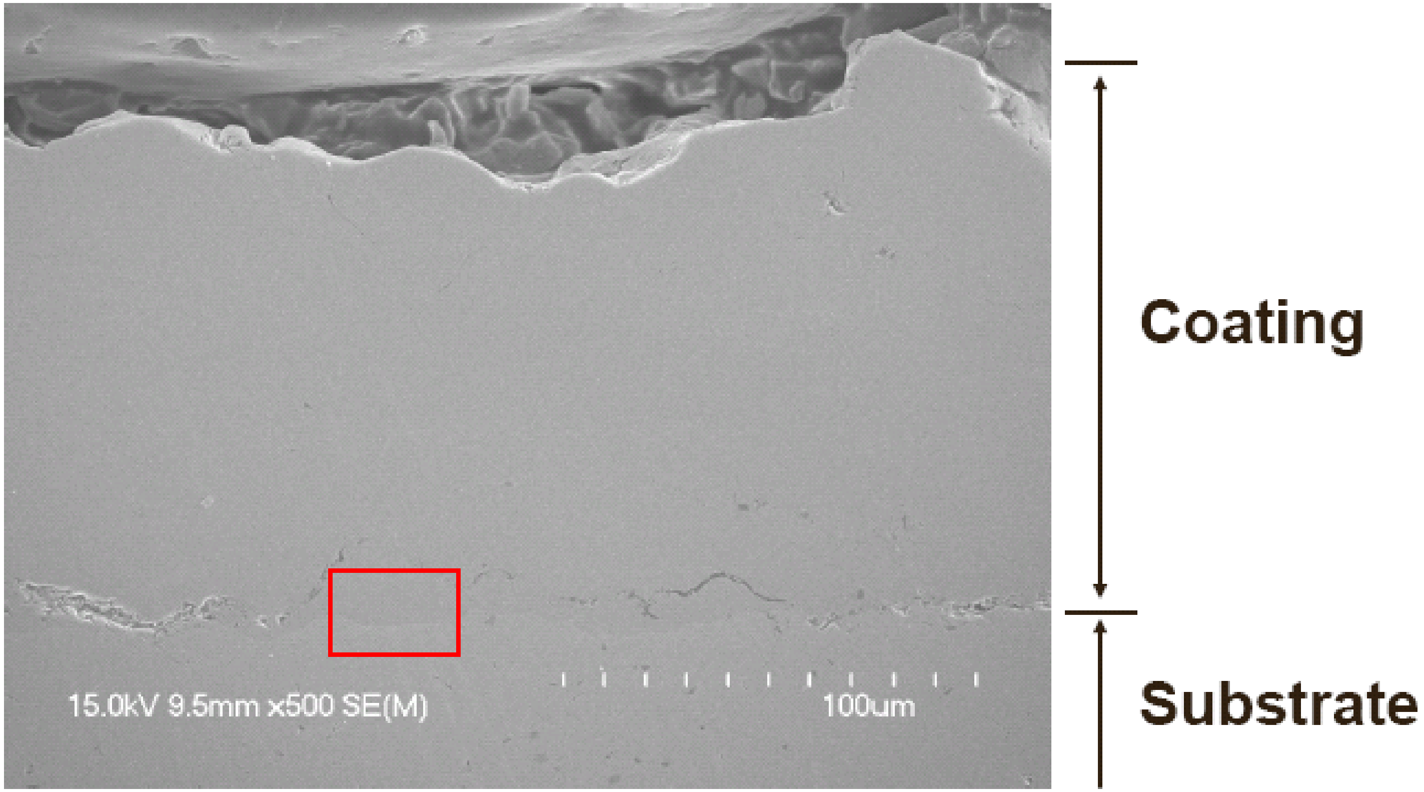
(wt.%)



SEM image of AMDRY9951 powder(-37+5.5μm)

Facility: KINETIKS 3000 (CGT-Cold Gas Technology GmbH)
Gas: Nitrogen (3 MPa)

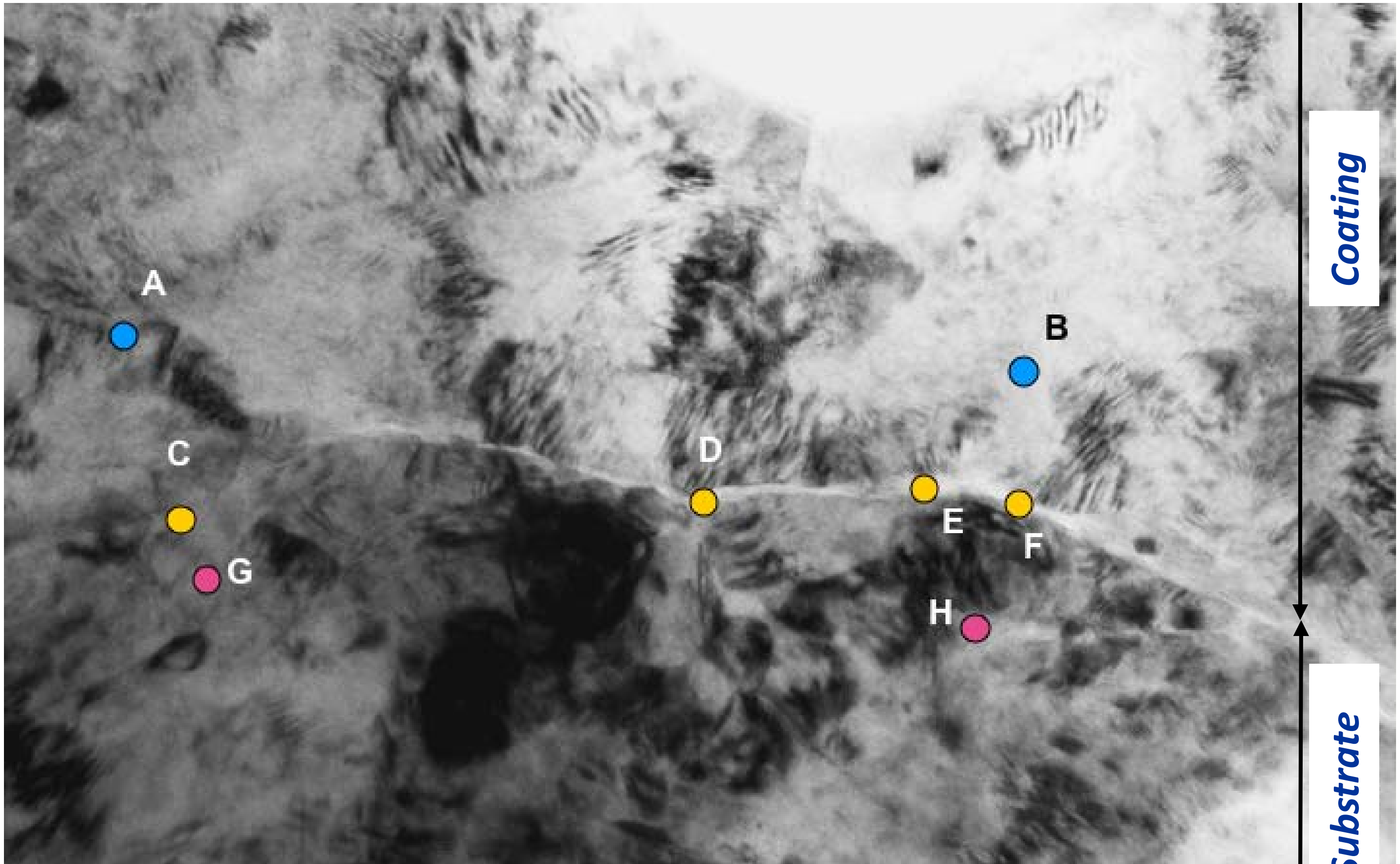
As-sprayed Coating



As-sprayed MCrAlY coating is high density due to high velocity impingements

=> Higher magnified observation of interface between coating and substrate is required.

TEM observation and EDX point-analysis results of the interface



Thin (0.5 to 3nm) oxide film exists at the substrate/coating interface.

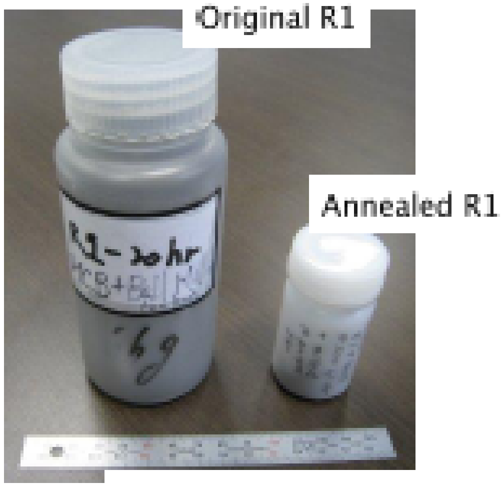
25 nm

Higher magnified TEM image of interface between cold sprayed coating and substrate

Analysis results

- ***CoNiCrAlY coatings***
- ***Oxide film***
- ***Inconel substrate***

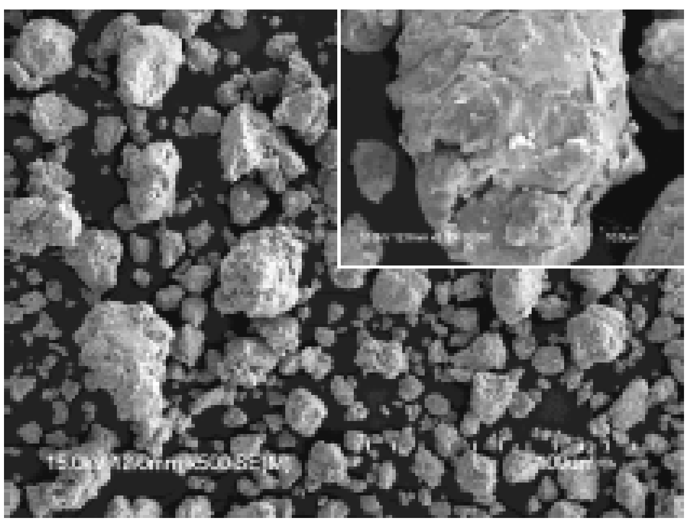
WVU ODS Powder (R1) – MCB and Ball Milling



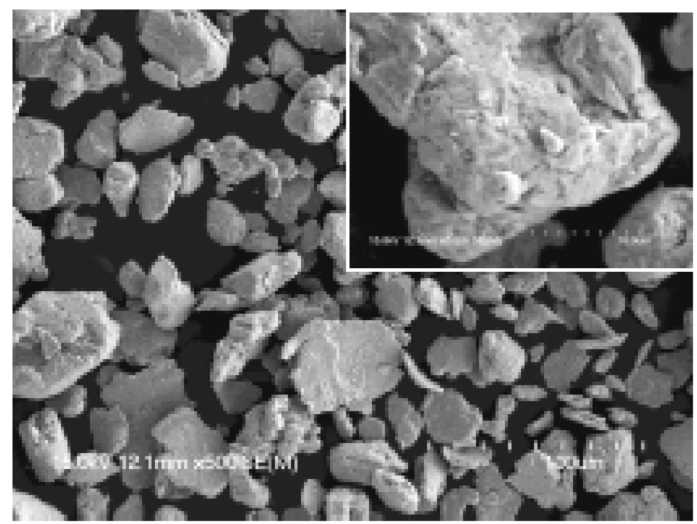
ODS R1 powders

Original R1 powder (by MCB+20hr ball-milled)					
	Cr	Al	Y_2O_3	W	Ni
Chemical wt.%	20	5	1.5	3	70.5
Particle size, um	7.5-10	4.5-7	<50nm	0.5-1	4-8

Post treatment of R1 powder	
1) Annealing	1hr @1200degC, in Ar gas (heating of 20degC/min; cooling of 3degC/min)
2) Milling	5hr @ 300rpm by planetary ball mill

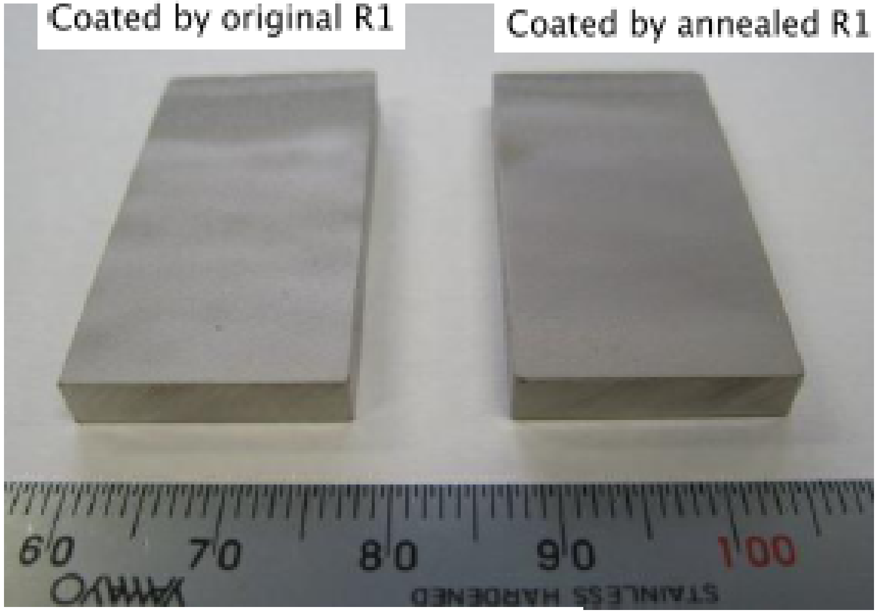


Original R1 powder (+0.5–35um)



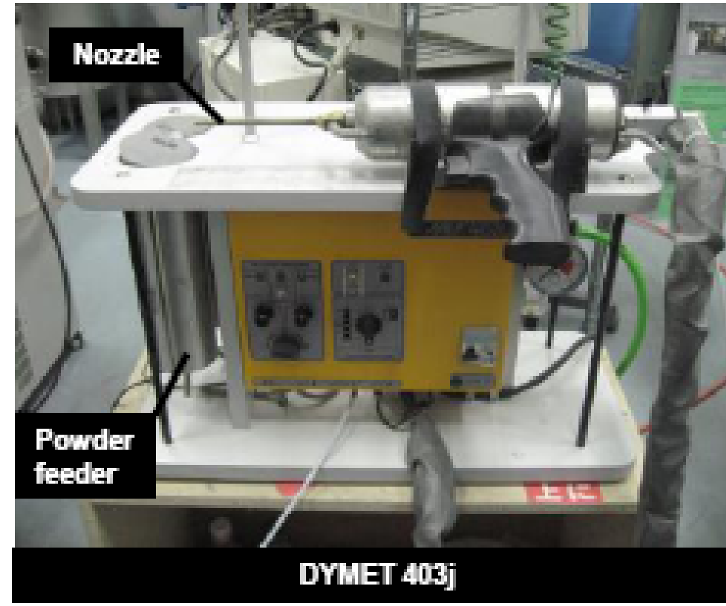
Annealed R1 powder (+3–40um)

R1 coatings by subsonic air



Substrates are Inconel 625 both.

1) Vey thin (<50um) coatings were obtained on both with low deposition efficiency.
2) There is no big difference between the original and annealed R1 coatings.

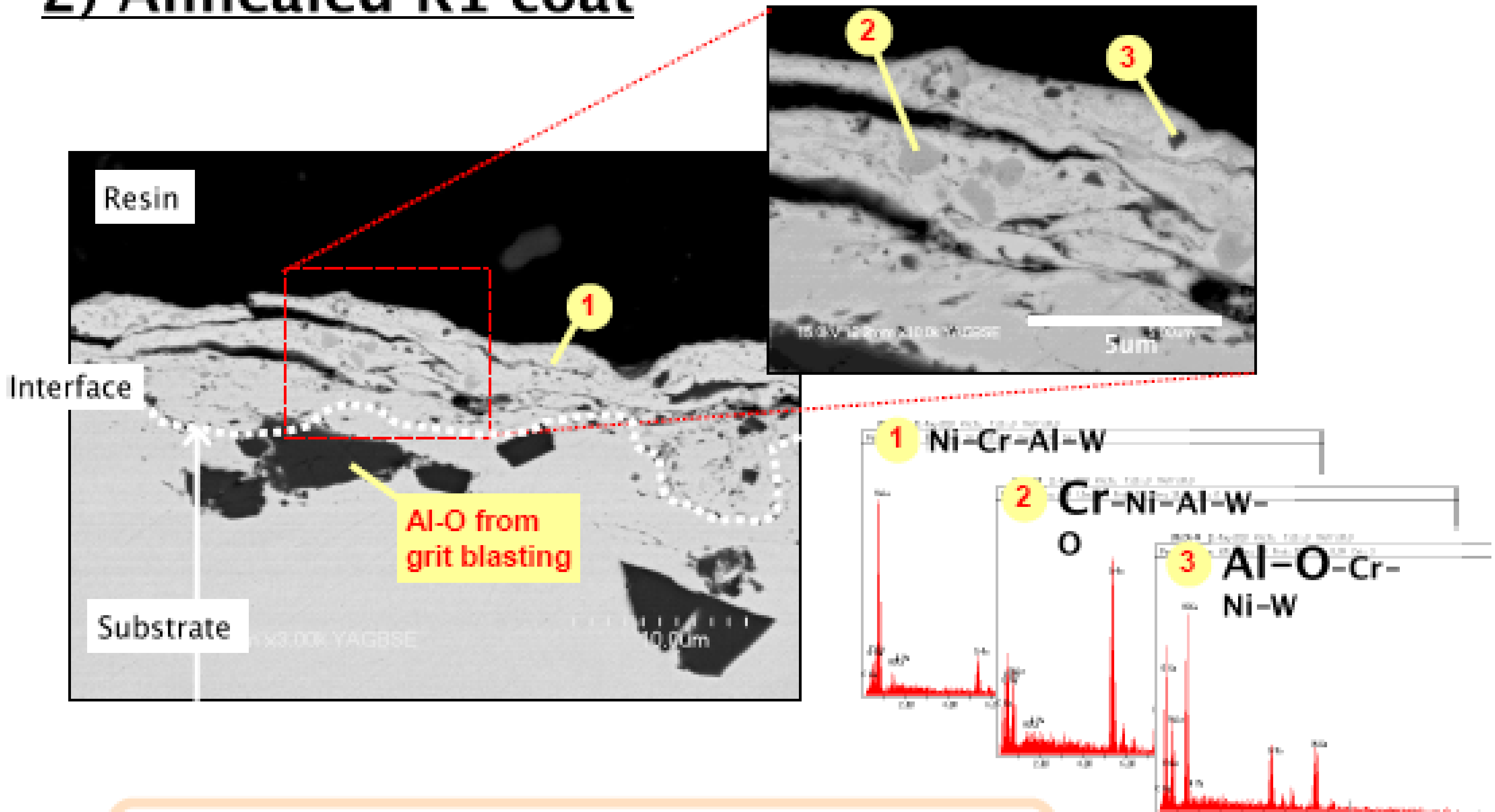


LP cold spray system used in this work.

Cold spray condition
Model: Dymet 403j, OCPS, Russia Supplying gas: Air / 0.8MPa Heating temperature: 400degC Stand-off distance from nozzle: 15mm Layer: approx. 2mm pitch, 3 pass

Pretreatment of substrate
3mm-thick Inconel 625 Grit-blasted by media size of 106-125um

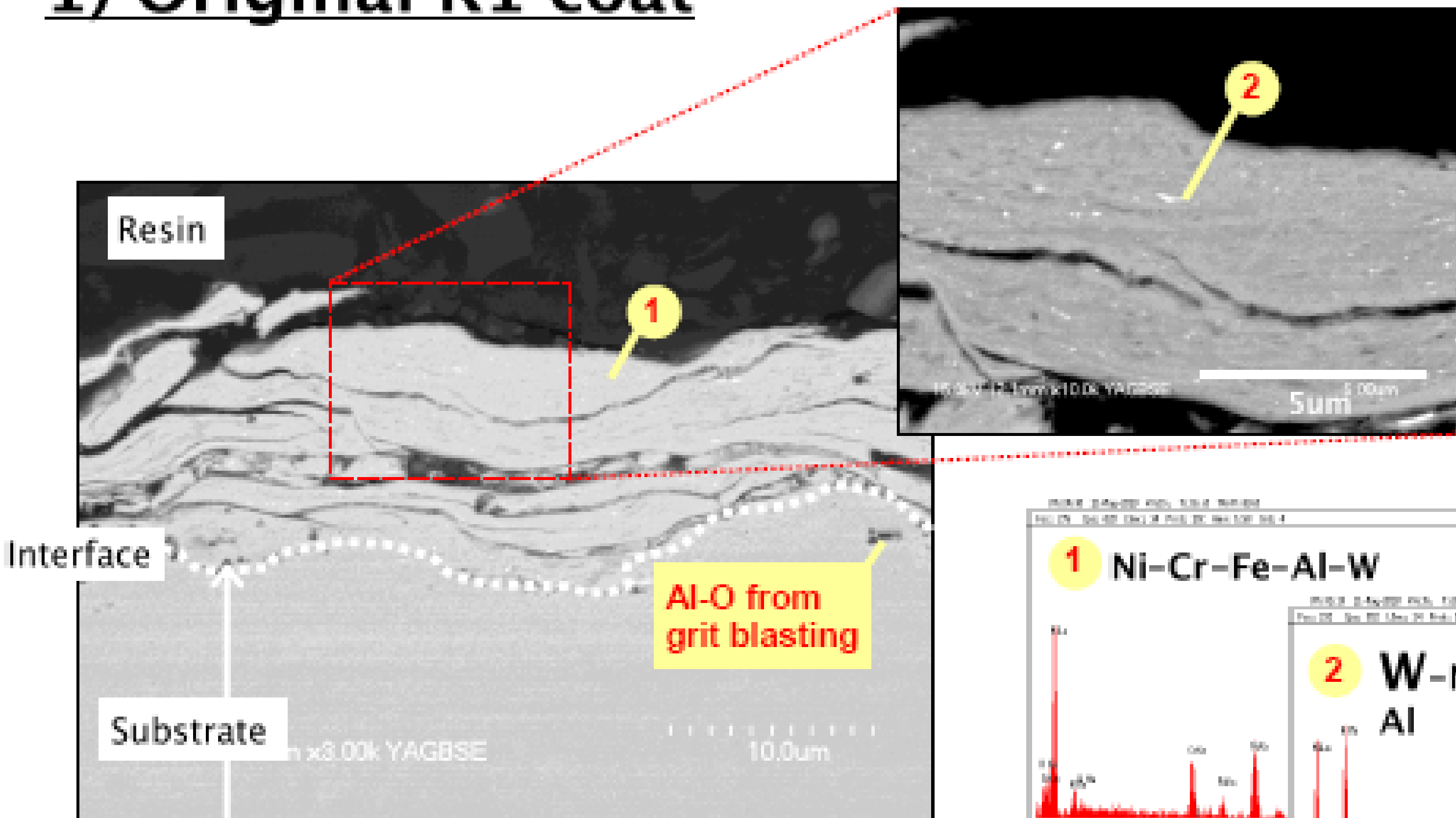
2) Annealed R1 coat

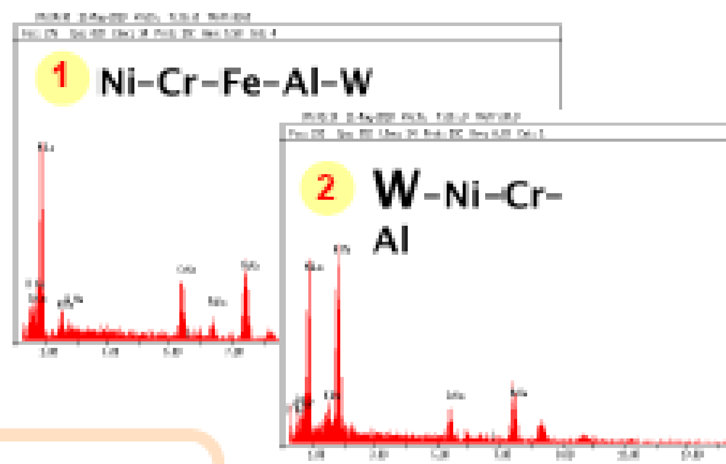


1) Coating by the annealed R1 powder is similar with one by the original R1; i.e., laminated and delaminated. Its thickness is <10um.

2) Cr-rich points are observed in the laminas, but W particles are not observed.

1) Original R1 coat





1) Coating by the original R1 powder shows a laminated structure with a lot of splat delamination, and it's too thin (<10um), not enough thickness.

2) Tiny W particles (suggested from W peak @ graph No.2) are well embedded into the laminated splats.

Summary of FY09-10 Accomplishment

(i) Produced ODS powder mixture with reduced manufacturing cost and better yttria dispersion using a combined Hosokawa mechano chemical bonding (MCB) and ball milling process,

(ii) Demonstrated the applicability of ODS coating on superalloy substrate using Cold Spray method. However, further processing improvement is needed to produce the desirable thicker ODS coating, e.g. high pressure Cold Spray under helium or nitrogen gas condition.

Planned Research for FY 10-11

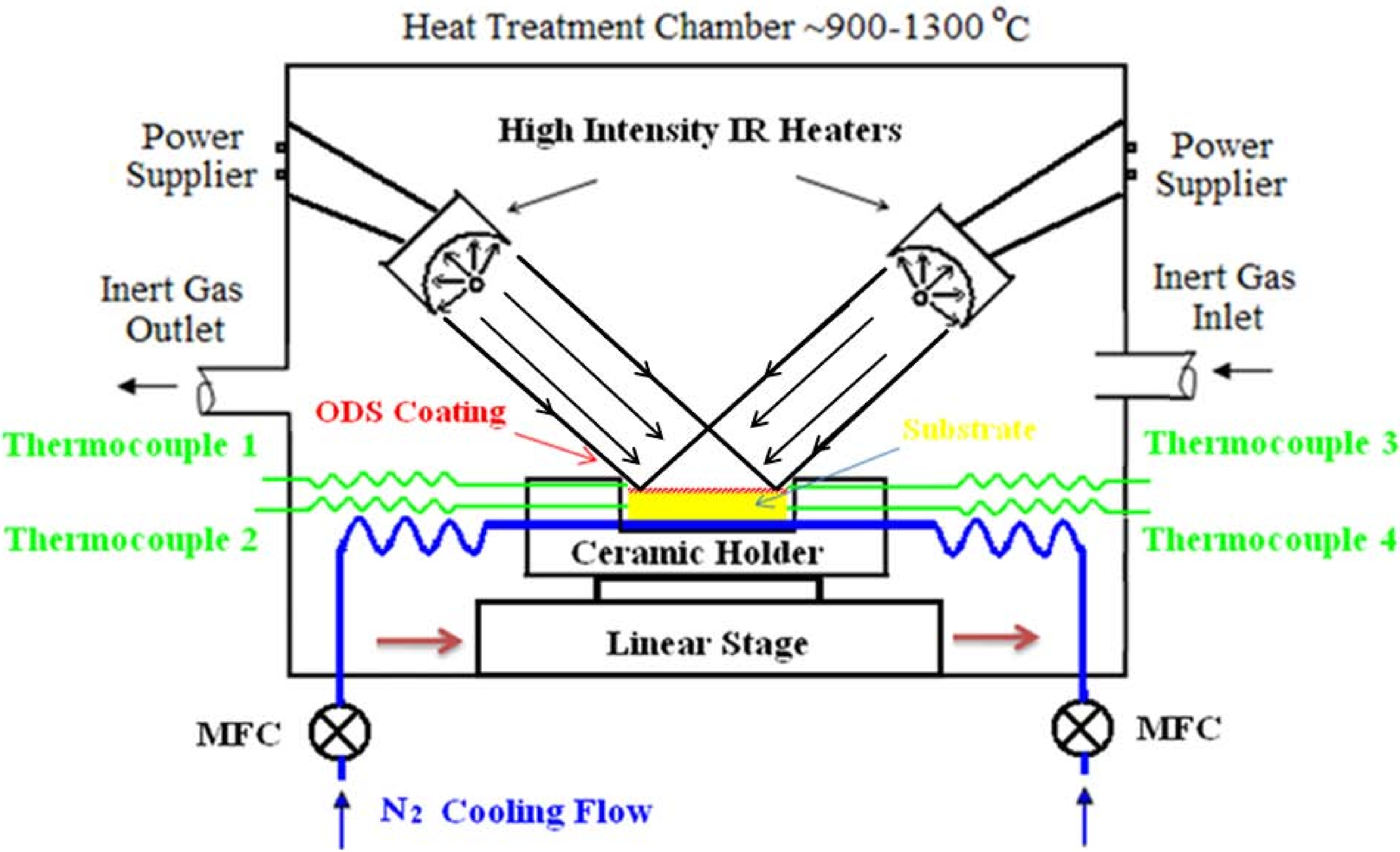
(i) Produce new batch of ODS powders using the optimized combination of MCB and ball milling process,

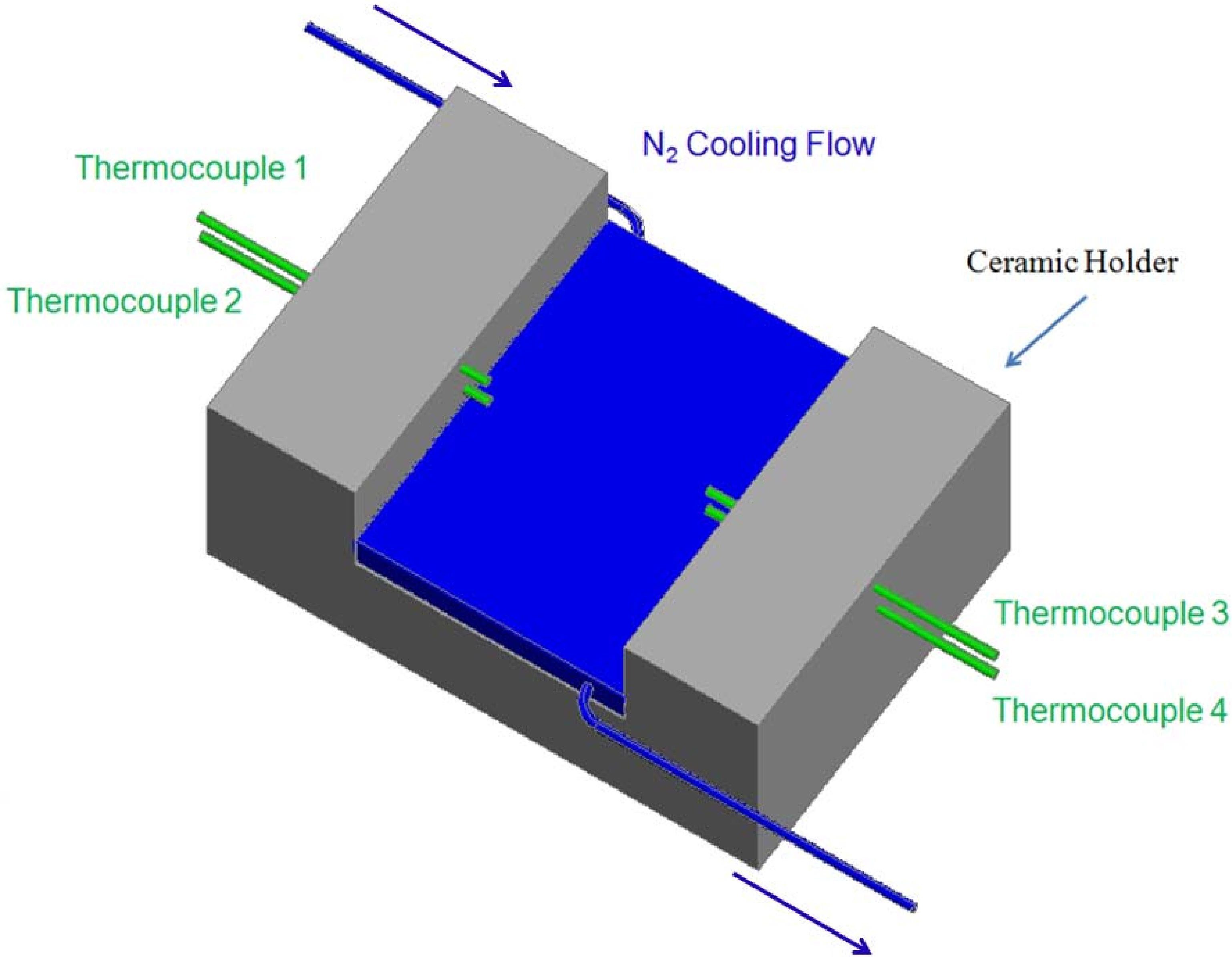
(ii) Comparative ODS coating evaluation (WVU ODS powders and MA 754) on superalloy substrate using HP Cold Spray,

(iii) Produce desirable ODS structural coating layer with proper surface heat treatment,

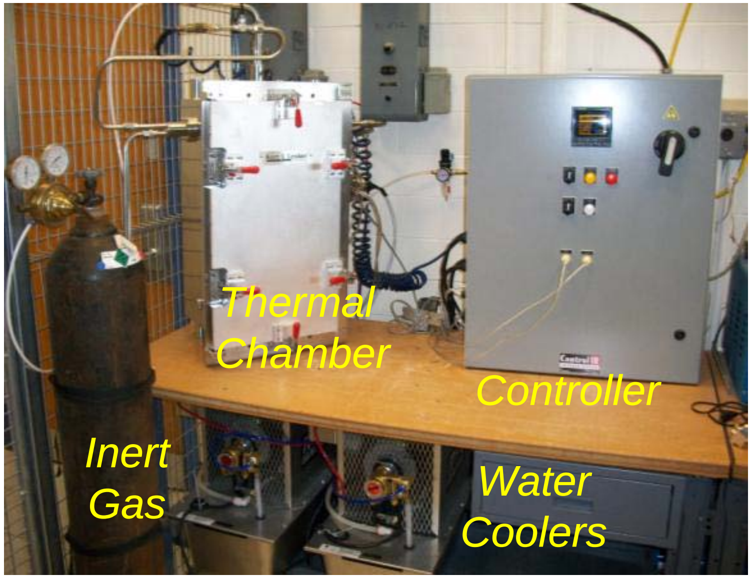
(iv) Mechanical property evaluation of ODS coating layer at elevated temperature (to 1250 ºC) using micro-indentation method.

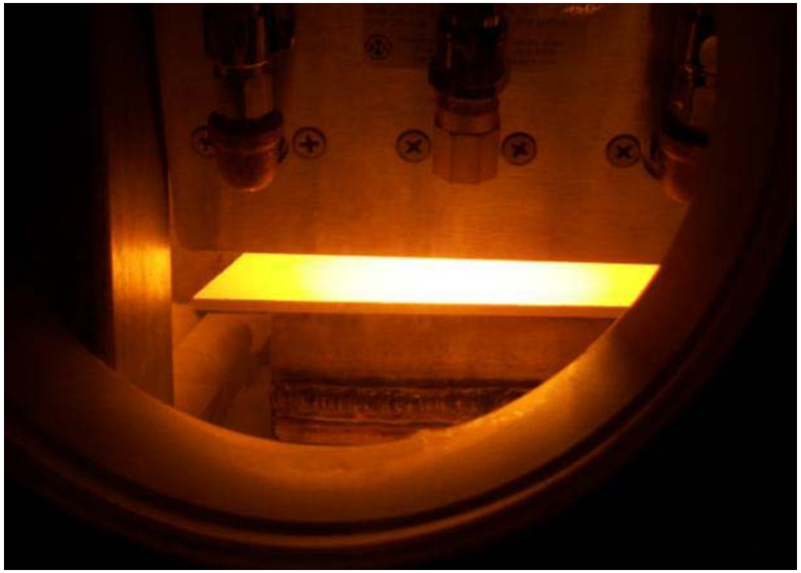
ODS Coating Surface Heat Treatment (Columanar grain growth stimulated by recrystallizing thru zone annealing)




N2 Cooling Flow
Thermocouple 1
Thermocouple 2
Ceramic Holder
Thermocouple 3
Thermocouple 4

High Intensity IR Surface Heat Treatment System

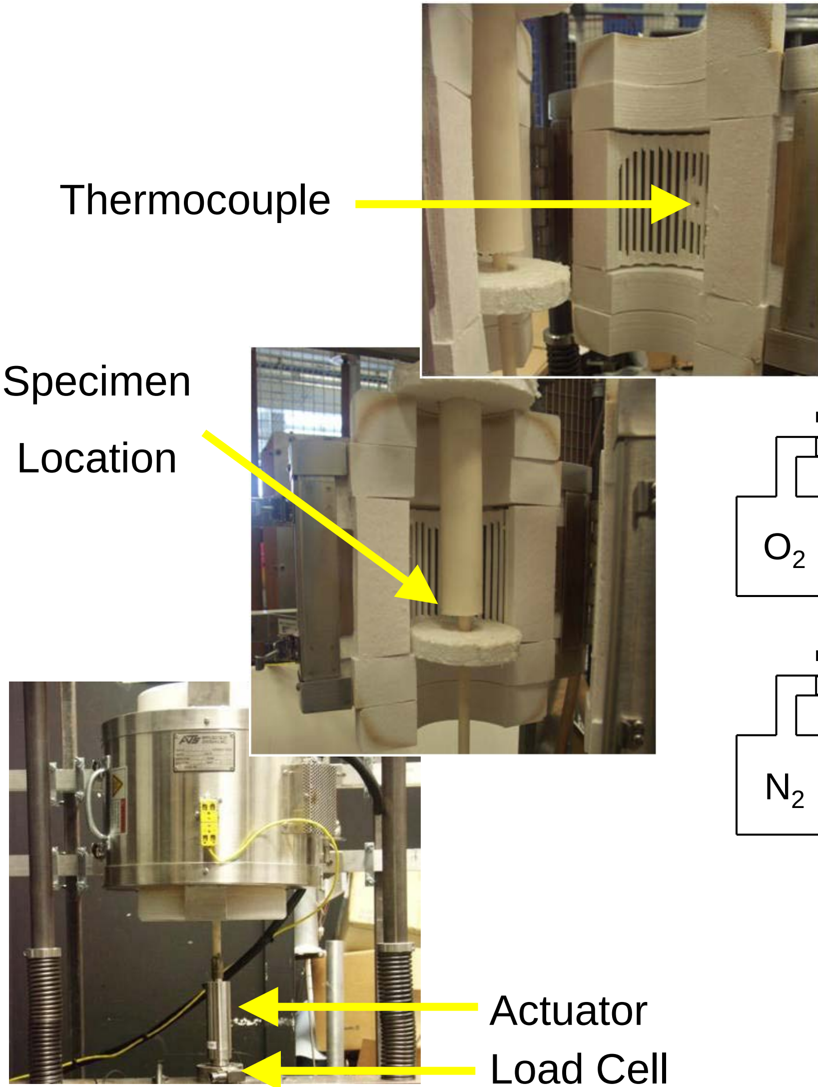


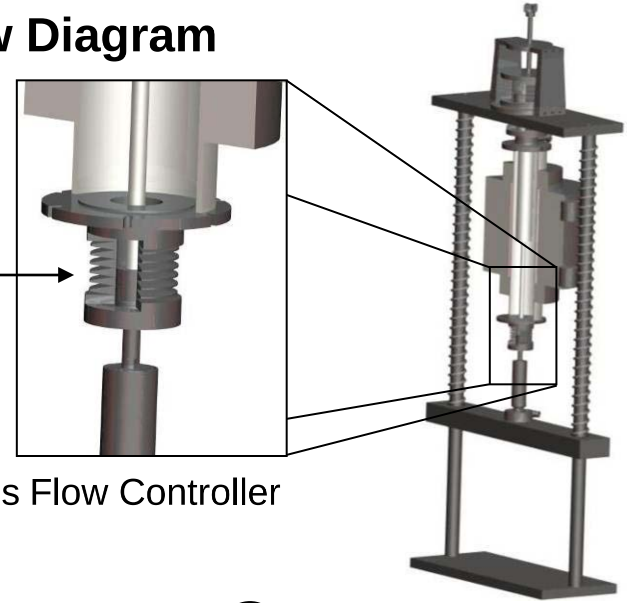


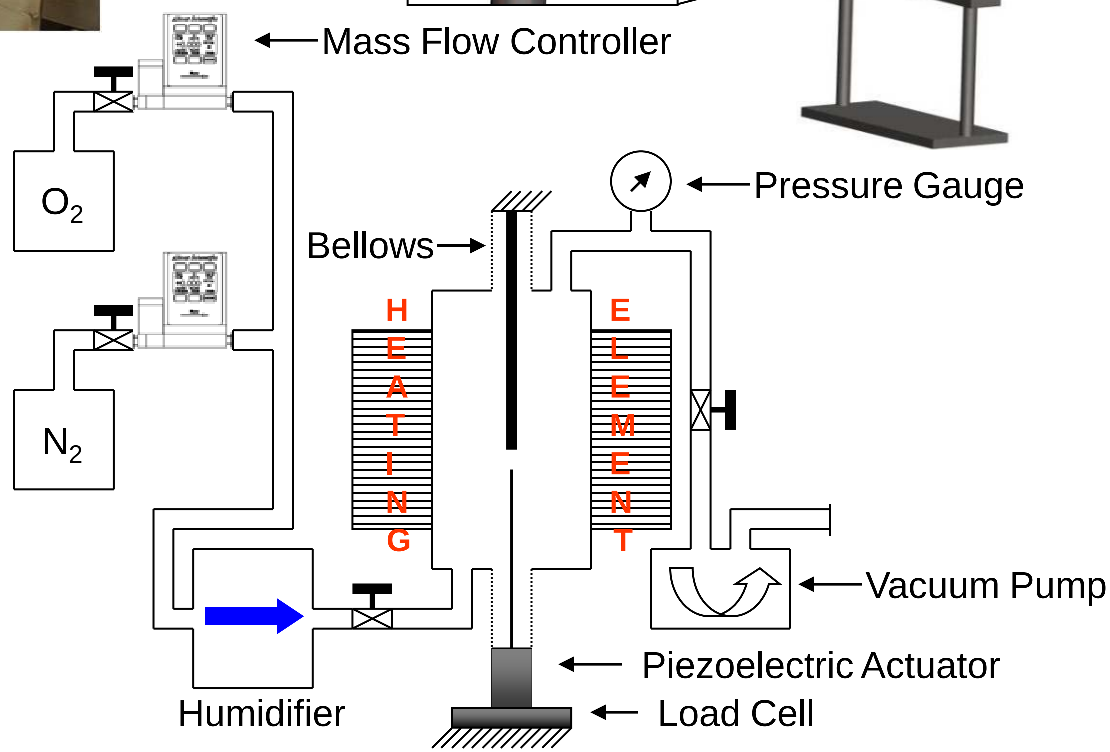
- *The system is capable of performing surface heat treatments in an inert gas environment (up to 1350 ^{o}C).*
- *The coupon* ***substrate can be cooled*** *with controlled flow rate for closed-loop temperature control.*
- *Through the implementation of infrared lamps, a compact and controlled* ***thermal loading area*** *(~ 4 in x 2 in) is produced and can be observed thru a* ***viewing port****.*
- *Logic control allows the user to define custom constant and/or cyclical heating operations.*

High Temperature Micro Indentation System

Furnace, Controller and Flow Diagram



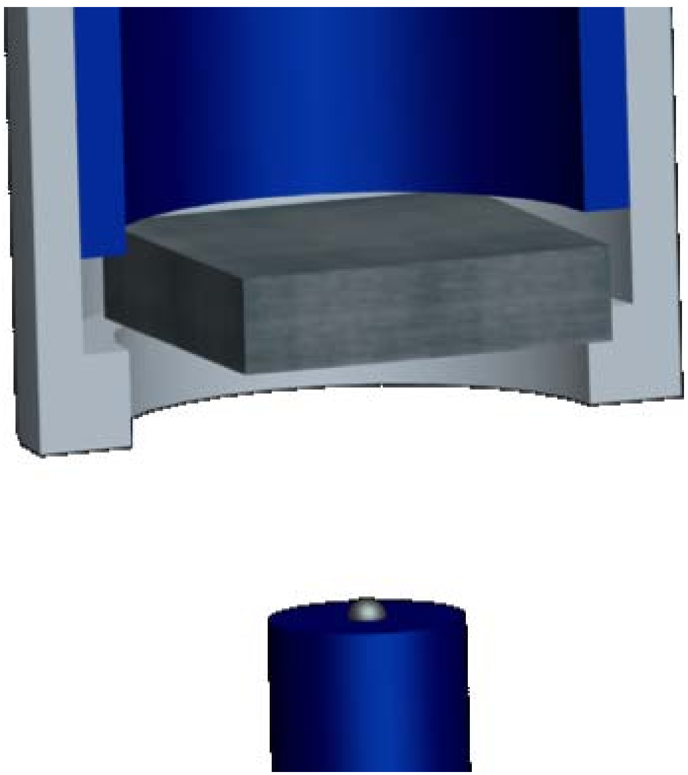




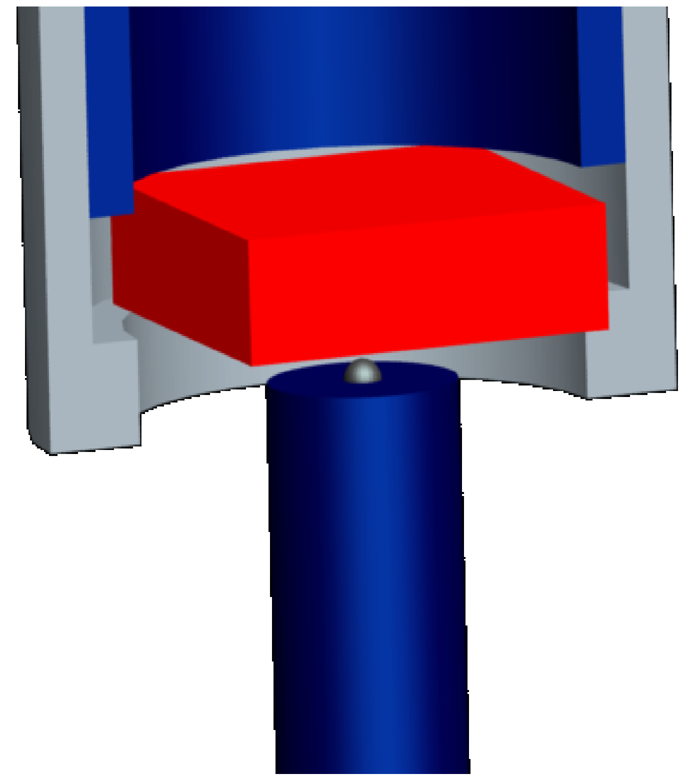
High Temperature Micro Indentation System

Specimen Mounting Procedure

Room Temperature



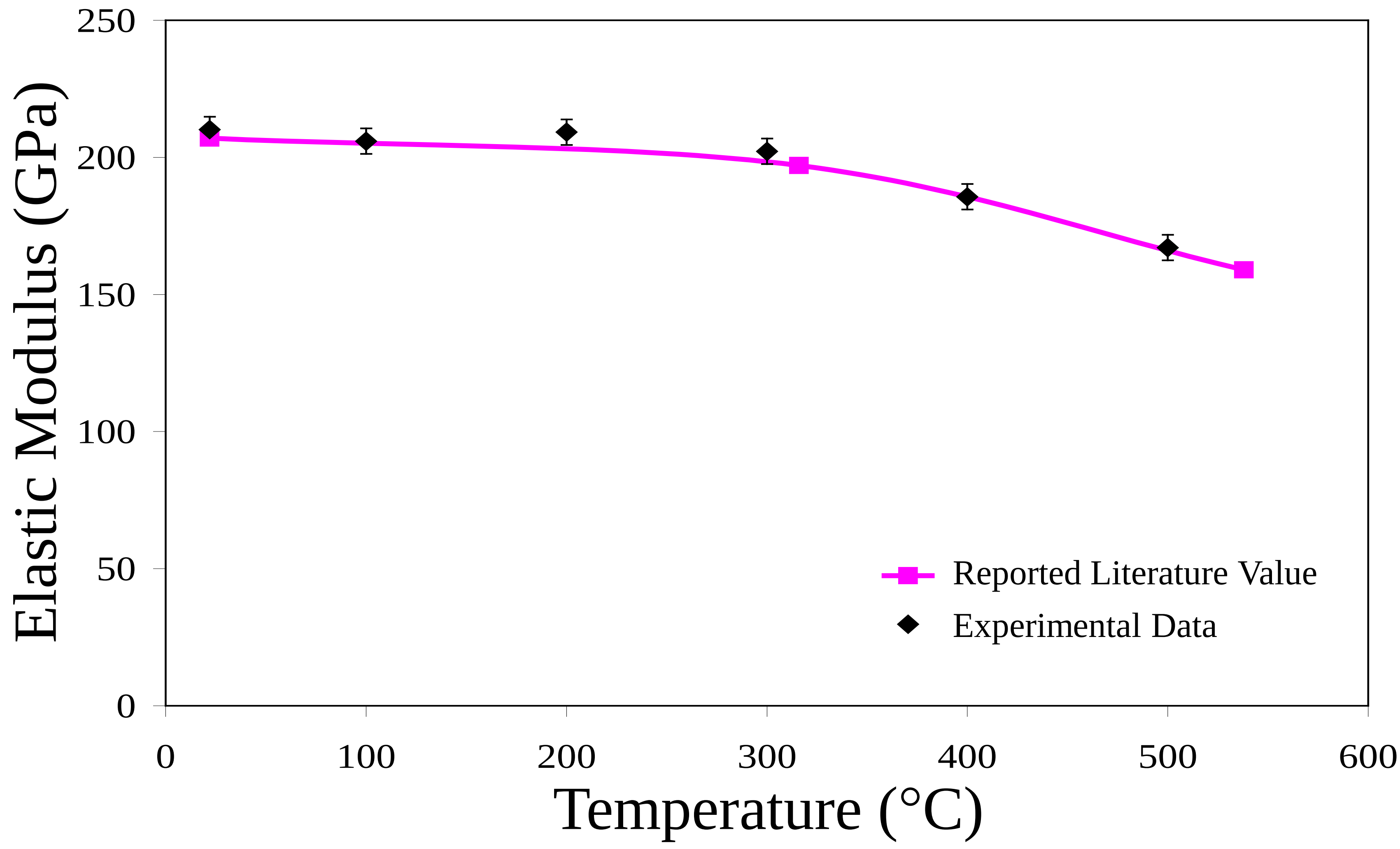
High Temperature



Note: Due to thermal expansion/drift, a system steady state condition must first be met before initial contact can be made. A load controlled multiple partial unloading/loading procedure further reduces the effects of thermal drift.

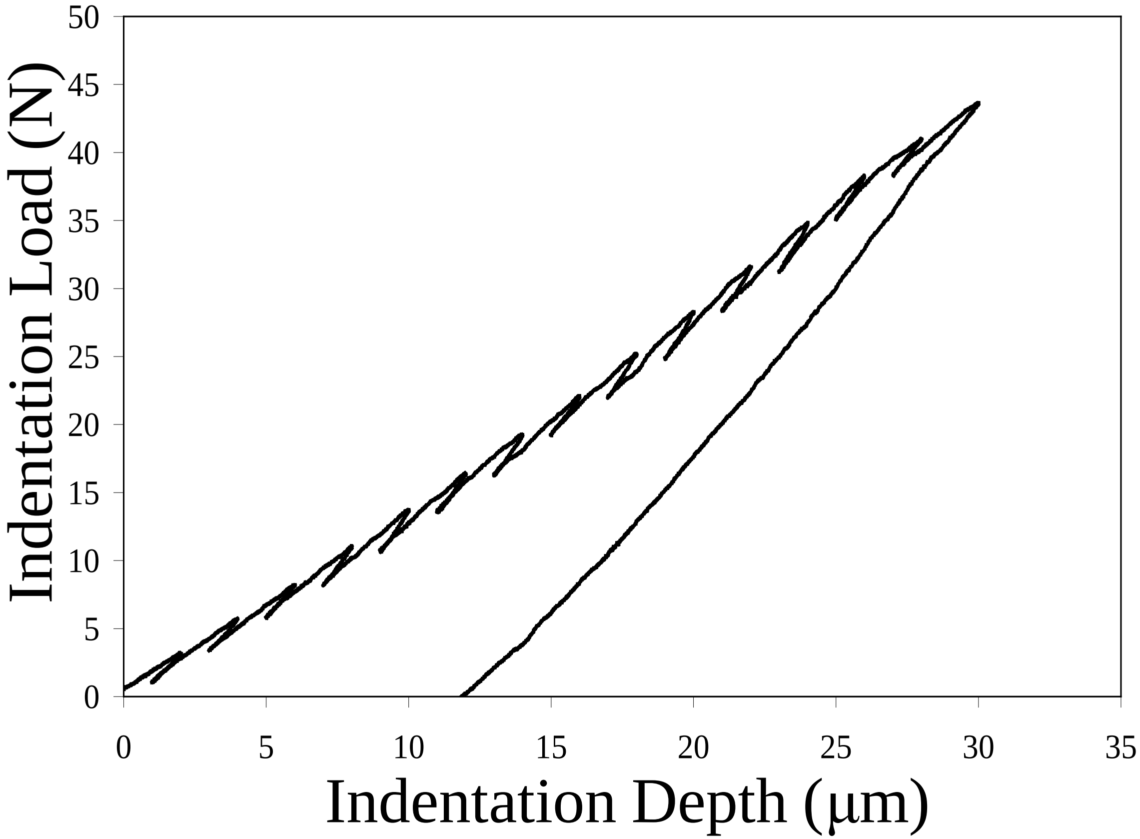
High Temperature Micro Indentation System

H13 Tool Steel Modulus vs. Temperature



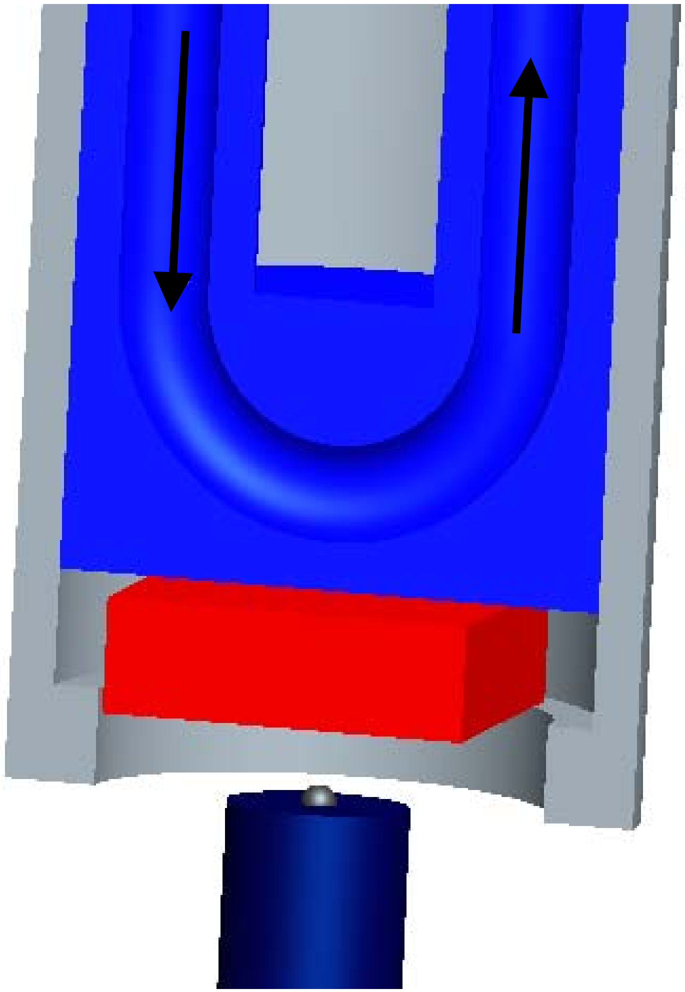
High Temperature Micro Indentation System

Haynes 230 at 1000°C



Test	*Haynes 230*
Number	*GPa*
Literature Value	150
1	150.1
2	147.3
3	145.5
4	147.4
5	153.1
Average	148.7
Standard Deviation	3.0

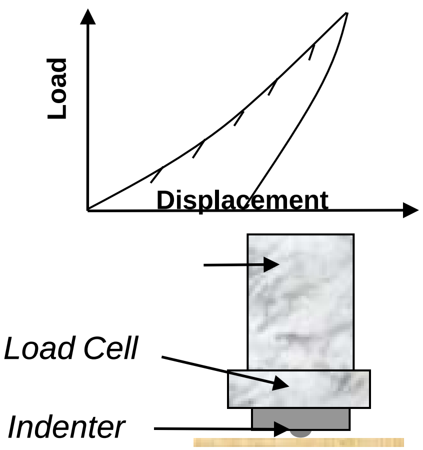
High Temperature Micro Indentation System



- Implementation of a simple yet effective coupon substrate water cooling systems allows for high temperature instrumented indentation to be conducted in harsh thermal environments exceeding 1250°C.
- Variable coolant flow rates allow the user to vary the temperature at which the substrate is kept.
- The thermal gradients across the coupon's cross-section induced by the cooling plate more accurately simulate those experienced by turbine compnents in working conditions.

Thank You !!!

WVU Micro-indentation Technology



Applications:

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

Potential:

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

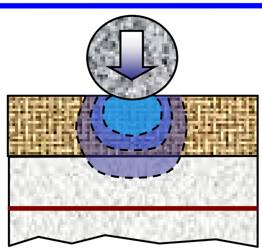
Load Based vs. Contact Area Based

$$P = \frac{4}{3} \cdot \frac{\sqrt{R}}{k_0} h^{3/2}$$ *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)***



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

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

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

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

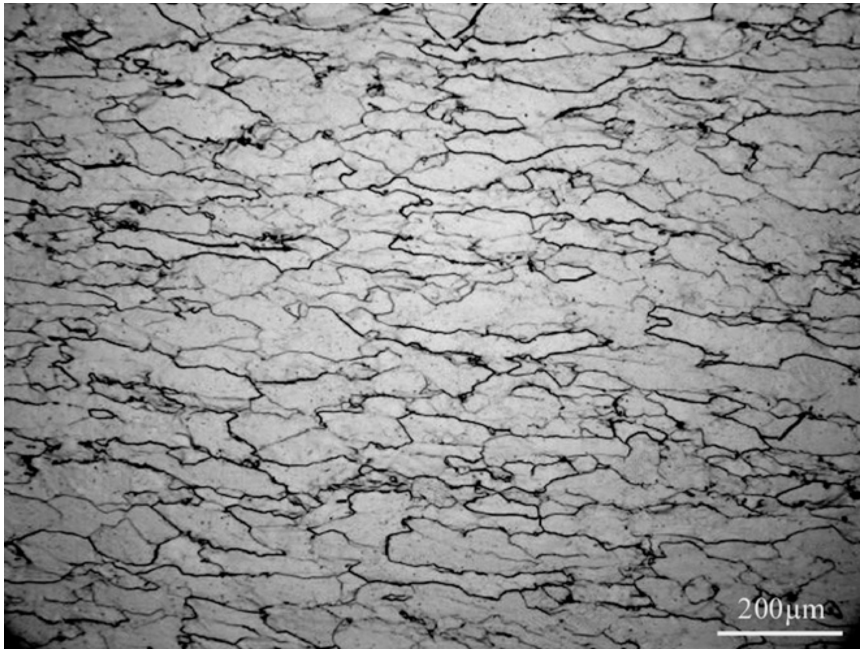
$$x = \frac{1}{p^{1/3}}, \quad y = \frac{dh}{dp}$$

$$y = mx + b$$

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

Capability

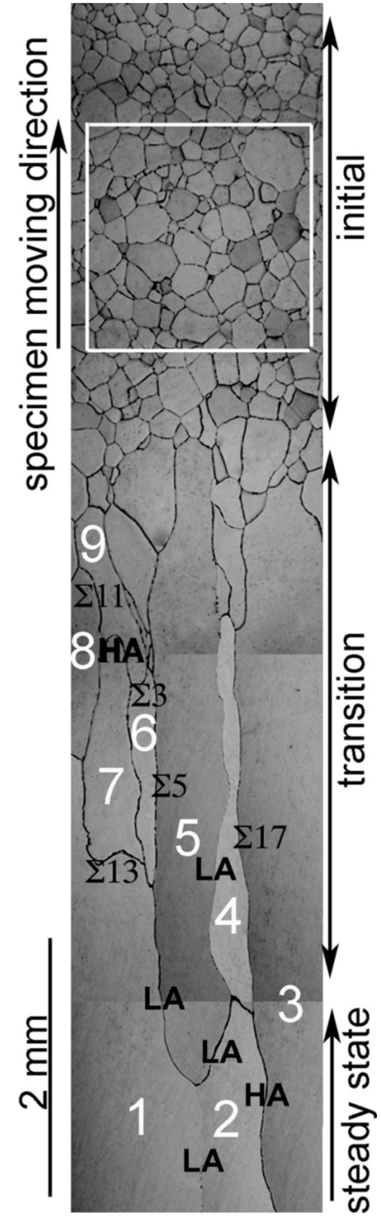
- **Designed for TBC/Bond Coat specimen**
  - Measurement of **surface stiffness responses** as a means to correlate the evolution of the microstructural changes of the TBC bond coat/substrate subjected to high temperature thermal cycles.
  - Can also be correlated to the damping effect
  - No surface preparation needed
- **High-temperature (to 1200 ºC) micro-indentation testing with test specimen under control gas/steam environments.**



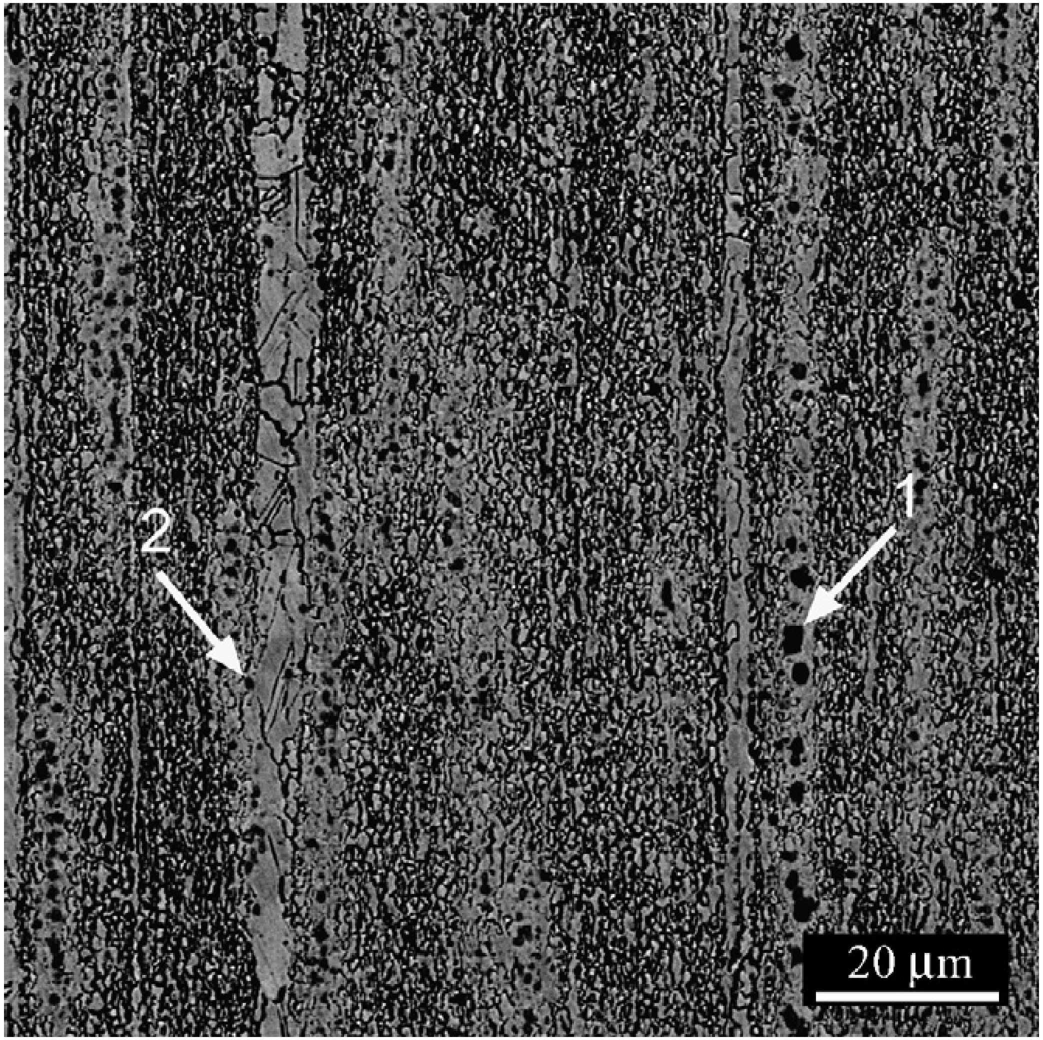
Starting Material

Fe–6.5wt%Si alloy with the composition (in wt%) of 0.05 C, 6.31 Si, 0.11 Mn, 0.008 P, 0.006 S, 0.005 Al, 0.043 B, and the balance Fe

From: Z. Zhang, G. Chen, H. Bei, F. Li, F. Ye, G. Chen, C-T Liu, "Directional recrystallization and microstructures of an Fe–6.5wt%Si alloy" , J. Mater. Res., Vol.24, No.8, pp. 2654-2660, Aug (2009).



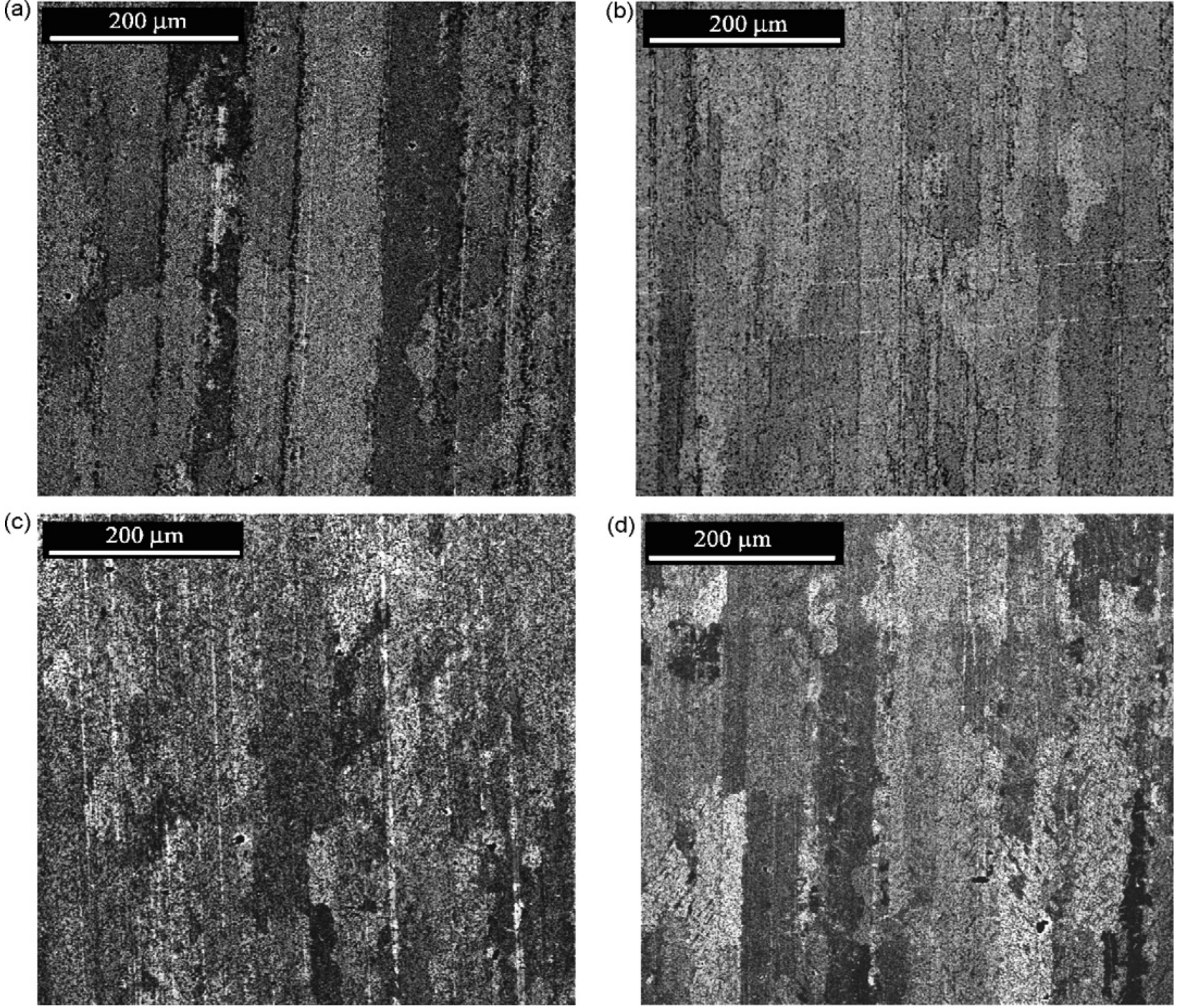
Recrystallized at 1200 ^{o}C and 3 μm/second



Starting Material , MA 754

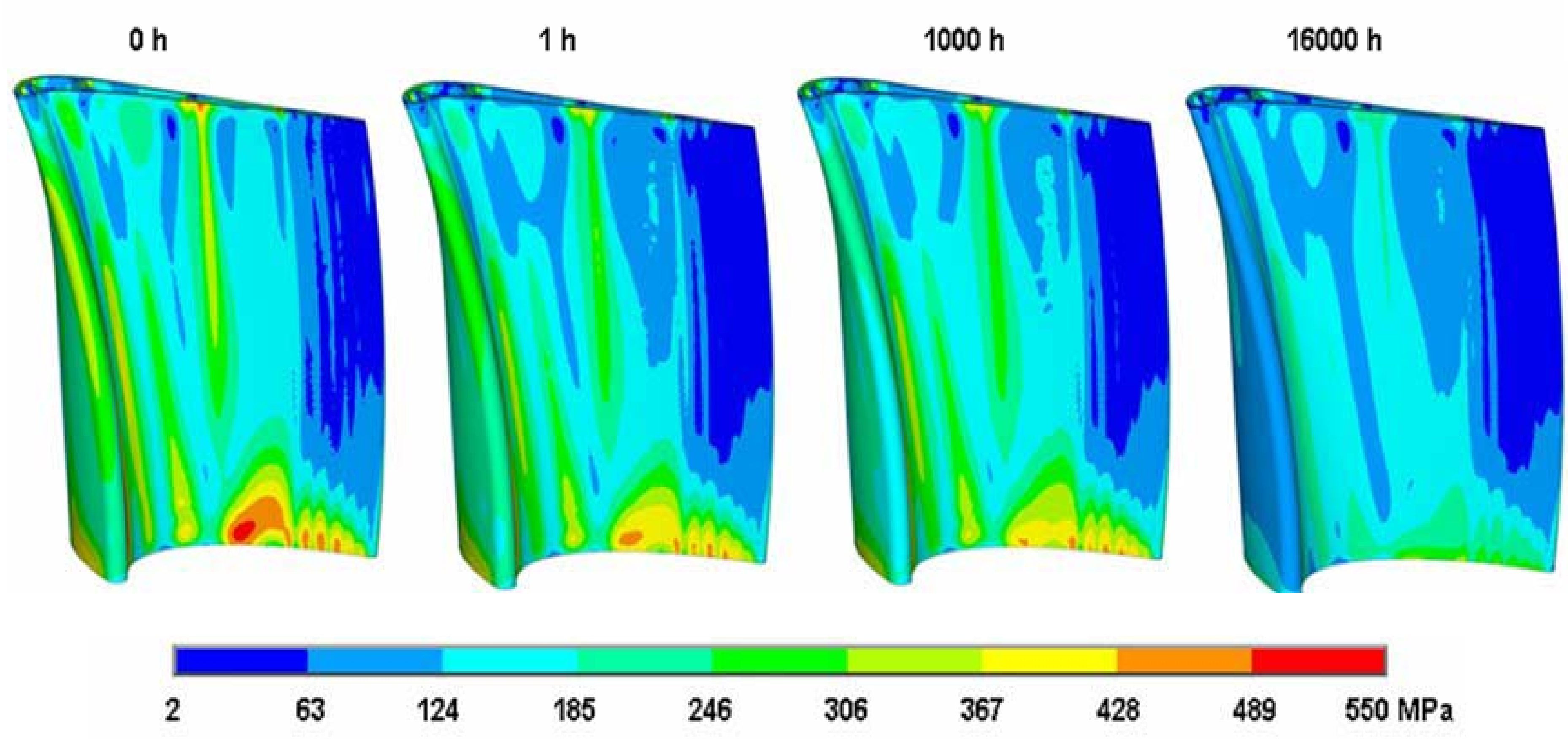
(in wt%, 20Cr, 0.3 Al, 0.5 Ti, 0.05 C, 1 Fe, 0.6 Y_2O_3, 77.55 Ni)
Arrow 1 points to yttrium oxide, Arrow 2 points to grains

From: I. Baker, B. Lliescu, J. Li, H.J. Frost, "Experiments and simulations of directionally annealed ODS MA 754", Materials Science and Engineering A, 492, pp. 353-363, (2008).



MA 754, directionally annealed at 1100 ^{o}C at various zone velocities (a) 2 mm/h, (b) 10 mm/h, © 50 mm/h and (d) 100 mm/h

From: I. Baker, B. Lliescu, J. Li, H.J. Frost, "Experiments and simulations of directionally annealed ODS MA 754", Materials Science and Engineering A, 492, pp. 353-363, (2008).



Von Mises Stress Profile with an angular velocity of 376.8 rad/s (3600 rpm), CMSX-4 substrate.

V. G. Karaivanov, Lifetime Prediction Modeling of Airfoils for Advanced Power Generation, Doctoral Thesis, University of Pittsburgh, Pittsburg, Pa (2009).