Testing and Qualification of CS Processes- A Focused Approach

CSAT 2015

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Sauer Engineering
IMR Test Labs

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Company Backgrounds

- Sauer Engineering
  - 25 yrs in TS
  - Work on TS Process Qualification
  - Stakeholder in HCAT Program
  - Work with plume sensors on process control
- IMR Test Labs
  - 25 yrs in materials testing and TS
  - Capabilities in all major test methods
  - All NADCAP and OEM approvals
  - Teach classes on TS Evaluation techniques

You Want Consistency……

- Want to thank everyone for setting up my talk
- Message is consistency-use the same procedure every time you test
- Having time constraints in the talks
- Any questions??

Design Strcutural Engineering

- Go talk to the DESIGN AND STRUCTURAL ENGINEERS
- And now the fun begins…..
  - Well we have never used coatings on that part of the engine, sub, aircraft before….
  - Will the coating have EXACTLY the same properties as the substrate
    - Coating must be AS GOOD OR BETTER
  - We will have to develop Mil Hbk 5 data for that
  - BOY ARE WE GOING TO DO SOME TESTING

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Coatings

- Really an afterthought in design at the beginning
- That wear coating does not really affect anything else in performance
- If it comes off, it is not prime reliant……*the engine or aircraft won't fail*
- *Those plasma coatings might affect the surface but since this is not a fatigue critical component we are not worried*

Coatings

- Coatings never carry any load……..????
- Never a structural repair….
- Is CS really a thermal spray coating process??
- *We want to charactrrize the process as "additive" manufacturing that caries load*

HVOF Coatings

- One area in coatings where some traction has been achieved with regard to using coatings in critical applications
- Lots of fatigue testing to look at debits
- Actually used on fatigue critical components
- Spray coatings in compression using Almen strips to monitor residual stress
- Strictly monitor process temperature during coating to minimize surface affects

HCAT Program

- Hard Chrome Alternative Team (HCAT)
- Given the task of replacing hard chrome plating with an alternative technology
- Many technologies reviewed and some are still under testing
- Turns out the main replacement has been the HVOF thermal spray coatings mentioned earlier
- It was a long journey that as I said above continues to this day on some applications

HCAT Program

- AS GOOD OR BETTER THAN CHROME
- And what is the modulus of elasticity for chrome plating……????
- Do you know HOW LONG we have been using hard chrome plating??
- You want to know what was considered when we were thinking about using chrome plating
- Yeah the design engineers from 1945 are still around and eager to talk with you….☺

HCAT Program

- DOD funded program
- Military and industrial stake holders
- Divided the applications into categories and developed Joint Test Protocols (JTP's) for each category
  - Landing Gear
  - Engine
  - Actuators
  - Propeller Chrome Replacement Project
- LESSONS LEARNED….

HCAT Program

- Normal coating testing
  - Metallography
  - Tensile or bond testing
  - Macrohardness testing
  - Microhardness testing
  - Bend testing
  - Erosion testing
  - Other
    - Thermal cycling
    - ????????

ANY Program

- Types of qualification testing
  - Fatigue
    - Axial
    - Bending
  - Corrosion
    - Salt spray
    - Galvanic
  - Wear
    - Sliding wear
  - Drop testing

- Types of qualification testing
  - Shear testing
  - Functional testing
  - Modulus testing
  - ????????

Testing Issues

- Metallography
  - Always a debate about whether we have the “true” structure of the coating
  - Cryogenic fracturing
  - FIB (Focused Ion Beam)
  - Etching
  - Process reliability
    - Establish a “baseline” early
    - Use of Metallographic standards

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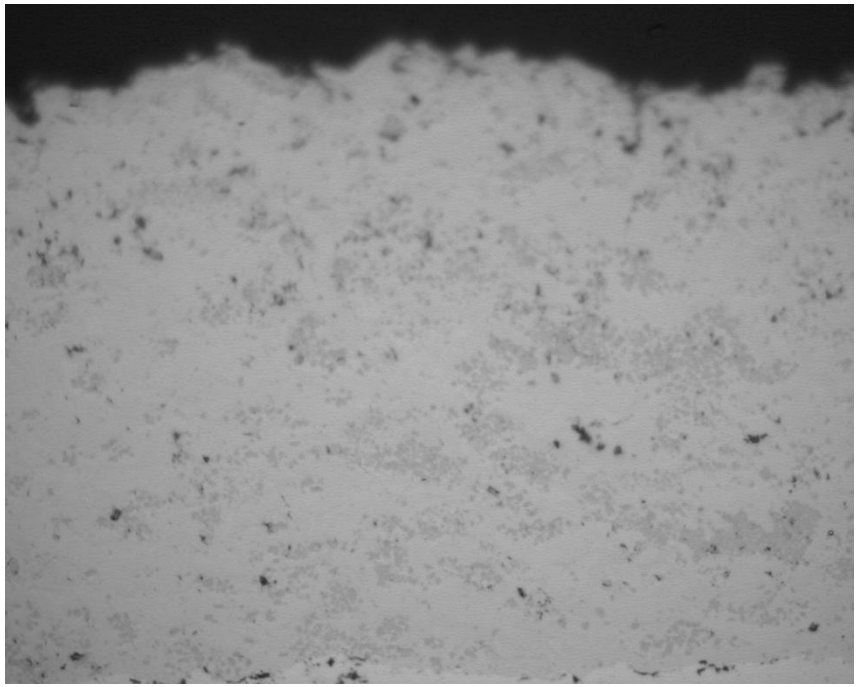
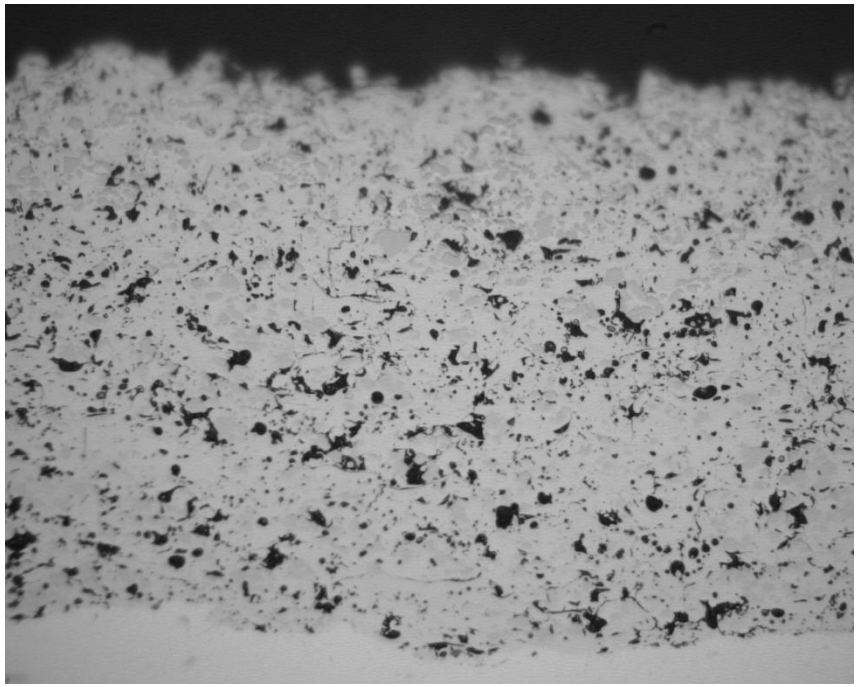
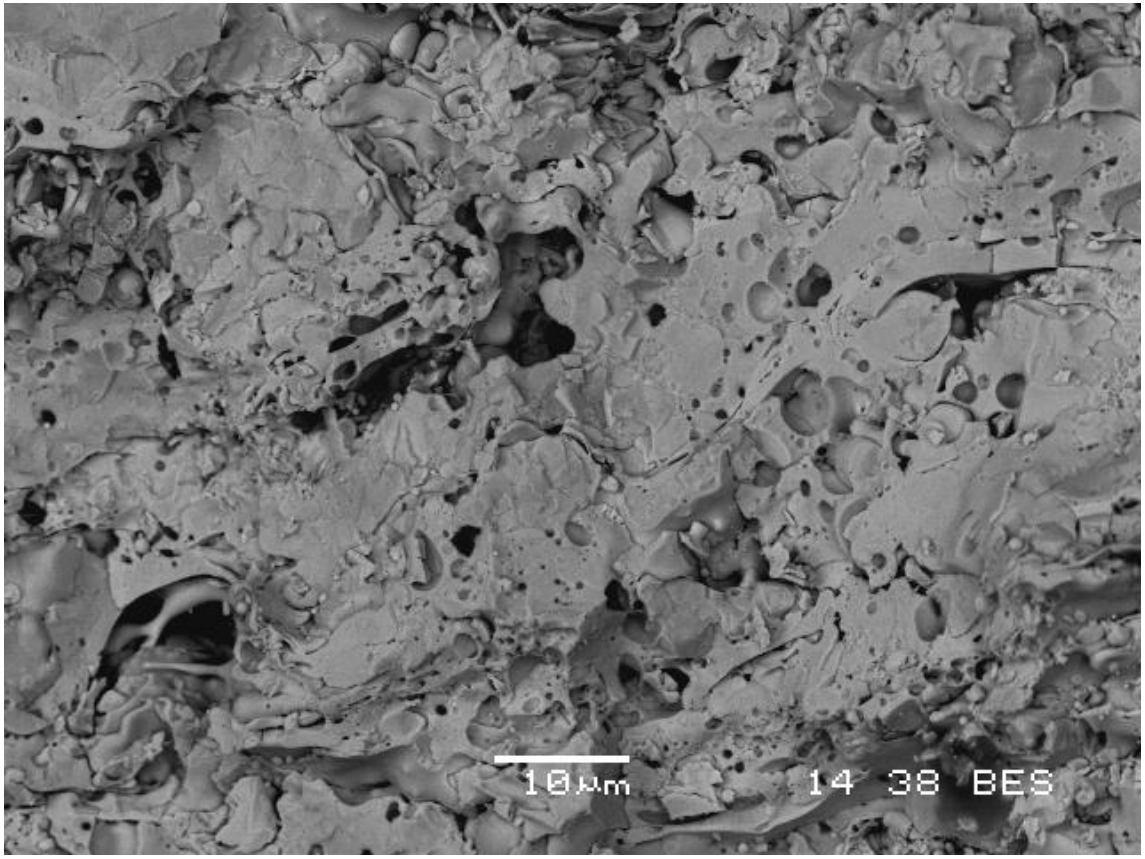
Tungsten Carbide (WCCo)





Identical plasma spray WCCo samples prepared by two different preparation methods. Which is the true structure?

WCCo – Fracture Analysis



Cryogenically produced fracture surface of a plasma sprayed WCCo coating. This method can be used to qualitatively assess porosity levels inherent to the coating.

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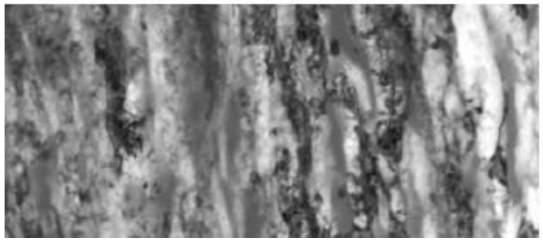
Focused Ion Beam

Fig. 3 Stages of the TEM Lamellae preparation using FIB in both milling and deposition mode and subsequent nano-manipulation of the prepared lamellae (x3500).



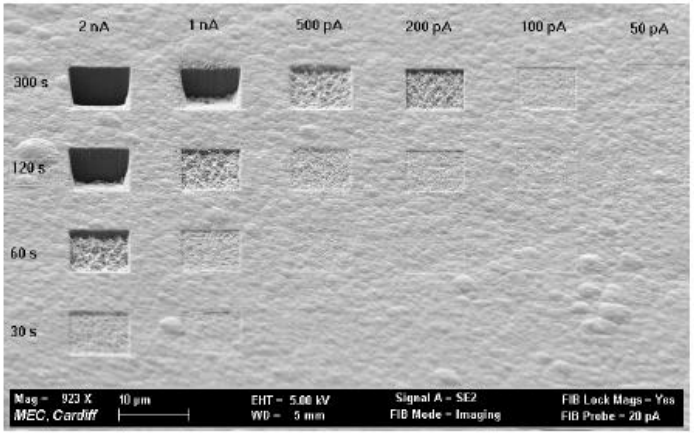


Fig.6 FIB milled patterns produced with different ion beam current (I_{ion}) and time (t_s). Number of layers NL=1

(a) (b)

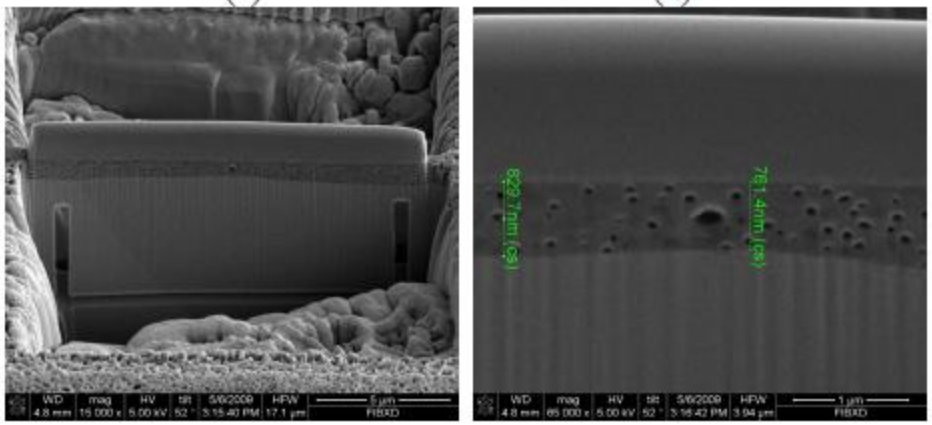


Fig. 2. SEM morphology of cross-sectional coating A. (a) 15,000X; (b) 65,000X.

Focused Ion Beam

Analysis of Microstructure for Insulating Coating on Non-Oriented Electrical Steel

CHING-KUO KUO AND JINN-CHERN WU

New Materials Research & Development Department
China Steel Corporation

The microstructure of the two insulating coatings on non-oriented electrical steel is investigated. The experimental methods included preparation of a cross-section of the coated specimens utilizing a focus ion beam, observation of cross-sectional morphologies by transmission electron microscopy, and the study of the relationship between interfacial reactivity and coating properties by X-ray photoelectron spectroscopy and glow discharge spectrometer. The results can be summarized as follows: (1) the existence of holes in the cross-sectional morphologies of coating A is ascribed to unexhausted air in the film; coating B is uniformly dispersed with deformed organic component; (2) depth profile analysis shows that coating A contains trivalent chromium, whereas coating B is chromium free. As compared to the coating A with weaker reactivity, the adhesion of coating B is better due to formation of P-O-Fe bonding in the interface; (3) depth profile analysis by glow discharge spectrometer shows that the quantity of iron salt in the coating and the reactivity between the paint and the steel increased upon lowering the pH value of the paint. The above results exhibit that the adhesion of the coating is correlated with the reactivity of the paint; however, corrosion resistance may be deteriorated by an excessive acidity of the paint.

FIB Milling and Characterization of CrC Coatings on Tool Steel Substrate

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Abstract

In micro tools, the base of a die should be ductile and the surface layer that will undergo processing should have a good machining response to various tool making processes. At the same time the resulting working surfaces of the tooling cavities should be hard, having low roughness, low wettability and high erosion resistance. To achieve such diverse properties, nano-crystalline CrC coatings deposited onto 12% Cr tool steel were investigated in this research. To verify the properties of this coating various metallographic techniques were applied. In particular, the corrosion resistance was studied by means of potentiodynamic anodic polarization. A STEM analysis of the structure was performed on samples prepared with Focused Ion Beam (FIB). The mechanical properties and grain size distribution were determined and statistically analysed. In addition, X-ray diffraction and scanning electron microscopy were used in studying the surface properties of this coating.

To investigate the response of the CrC coatings to FIB milling a series of rectangular trenches were produced using FIB/SEM cross-beam system. Especially, the effects of the ion beam current, exposure time and ion fluence on the sputtering yield and roughness of the produced micro structures were studied. Some essential parameter' windows for performing FIB milling with relatively high sputtering rates, higher than 1 μm/min, and at the same time achieving the best possible surface integrity were determined during the experiments.

Keywords: CrC, PVD, FIB, micro-tooling, STEM analyses

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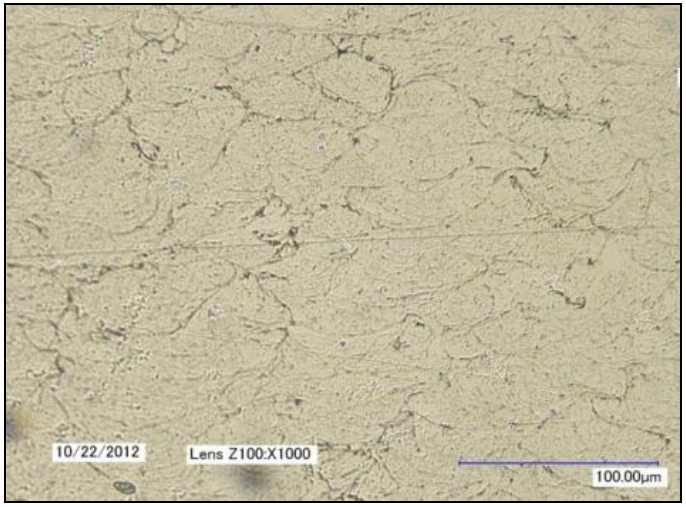
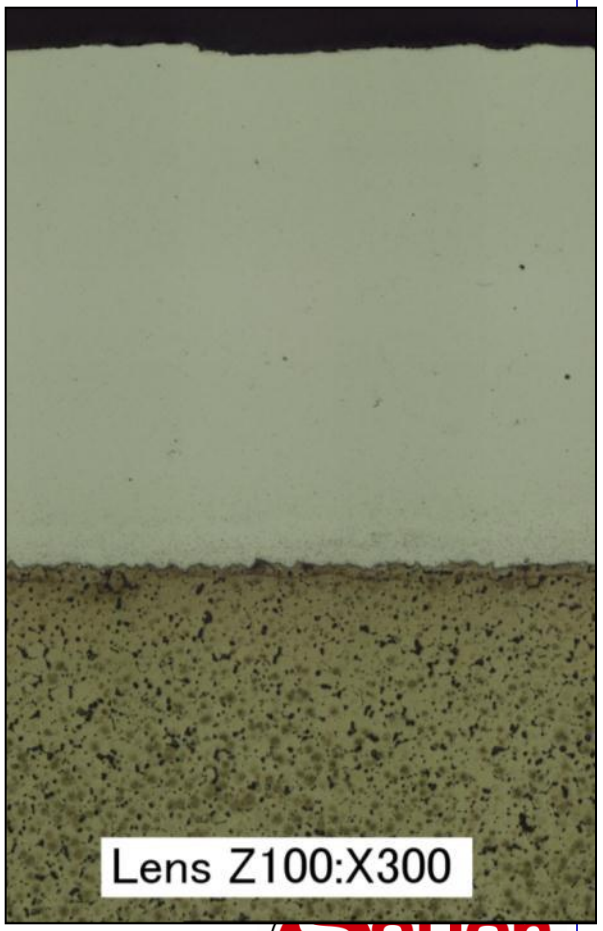
Magnesium Repair with 6061 Al Cold Spray

- 6061 aluminum deposited on ZE41 magnesium
- Porosity: **<1%**
- Adhesion Strength (ASTM C633-01)
  - **>11,000 psi** (limited due to glue)
- Hardness: **≈100 HV**



6061 coating etched to reveal splat deformation (1000X)



6061 cold spray coating (300X)

Testing Issues

- Tensile
  - Machine a “real “ tensile bar from a CS deposit
  - Thickness limits
  - Structural repair or a coating??
- Bond
  - “True” strength of the coating
  - Thickness limits
  - Flatness and alignment
  - Glue penetration-porosity??
  - Other methods besides ASTM C-633??

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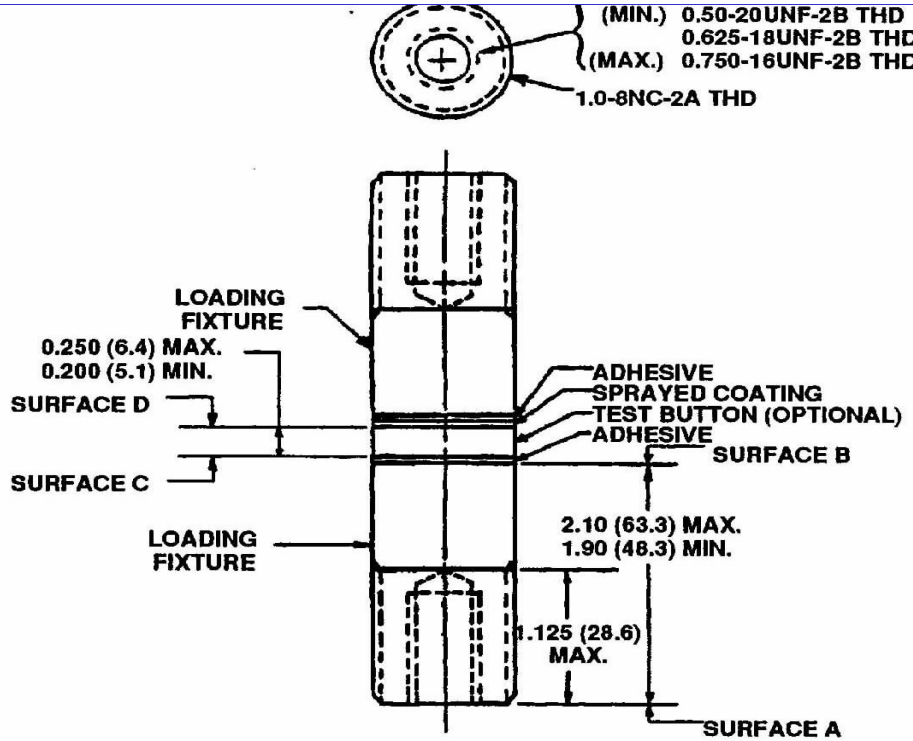


(A) ALL DIMENSIONS ARE IN INCHES (mm)

(B) ALL MACHINED SURFACES SHALL HAVE A SURFACE FINISH OF 125 MICROINCHES (3.2 MICROMETERS) OR SMOOTHER

(C) THE DIAMETERS OF TEST BUTTONS AND LOADING FIXTURES SHALL BE 0.995 ± 0.005 (25.273 + 0.127) AND SHALL BE CONCENTRIC WITH THEIR RESPECTIVE CENTERLINES WITHIN 0.003 INCH (0.076 mm) TIR

(D) LOADING FIXTURES SHALL BE THREADED ON THE OUTER DIAMETER OR DRILLED AND TAPPED

(E) LOADING FIXTURE THREADS SHALL BE CONCENTRIC WITH THE LOADING FIXTURE CENTERLINE WITHIN 0.003 INCH (0.076 mm) TIR

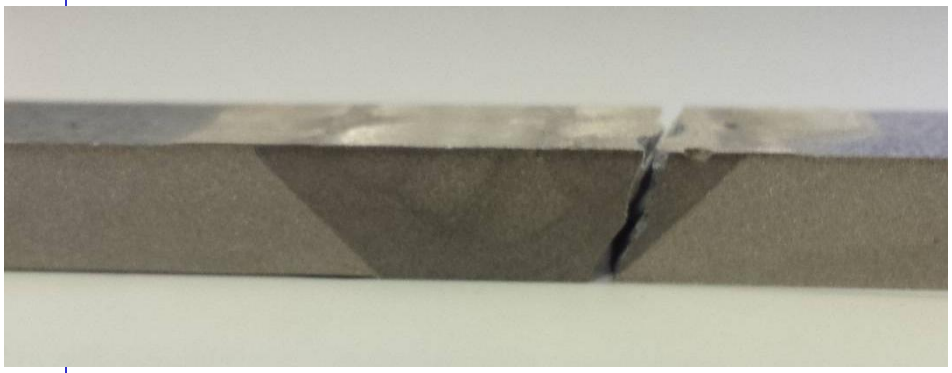
(F) SURFACES A AND B SHALL BE PERPENDICULAR TO THE LOADING FIXTURE CENTERLINE WITHIN 0.003 INCH (0.076 mm) TIR

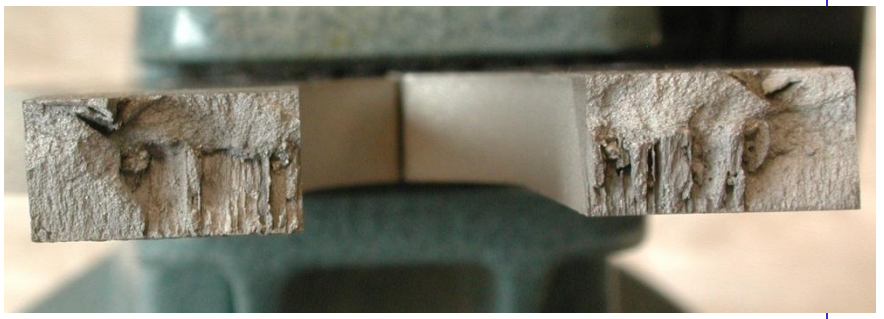
(G) SURFACES C AND D SHALL BE PERPENDICULAR TO THE TEST BUTTON CENTERLINE WITHIN 0.003 INCH (0.076 mm) TIR

(H) THE ASSEMBLED SPECIMEN DIAMETER SHALL BE CONCENTRIC WITH THE CENTERLINE WITHIN 0.015 INCH (0.381 mm) TIR

Tensile Tests– Cold Spray Repair

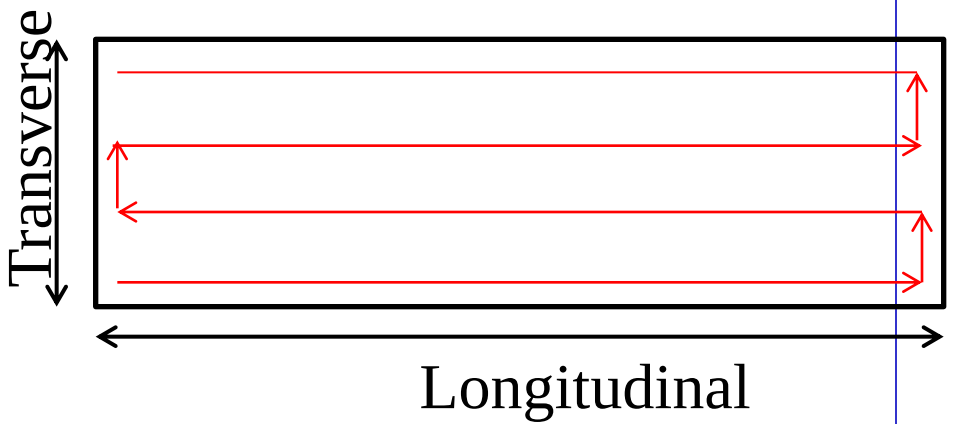
- Fracture consists of both transgranular and intergranular cracking
- No preferential fracture at root or AA5456-Al_2O_3 coating interface
- Fracture cohesive through cold spray repair

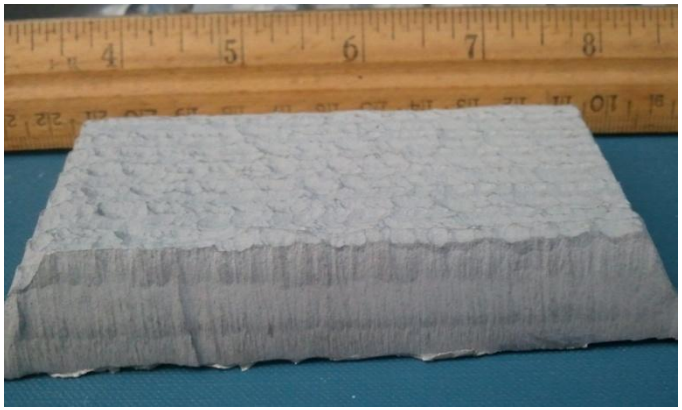




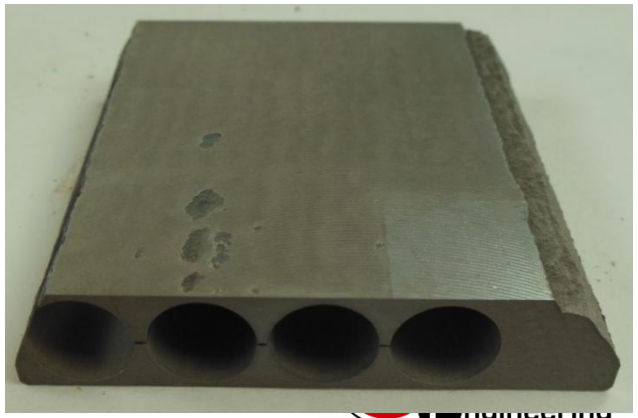
Tensile Tests-All Cold Spray

- Tensile specimens machined out of bulk build
- Initial gauge diameters ~0.25 in
- Specimens tested in both transverse and longitudinal directions





Bouffard



Testing Issues

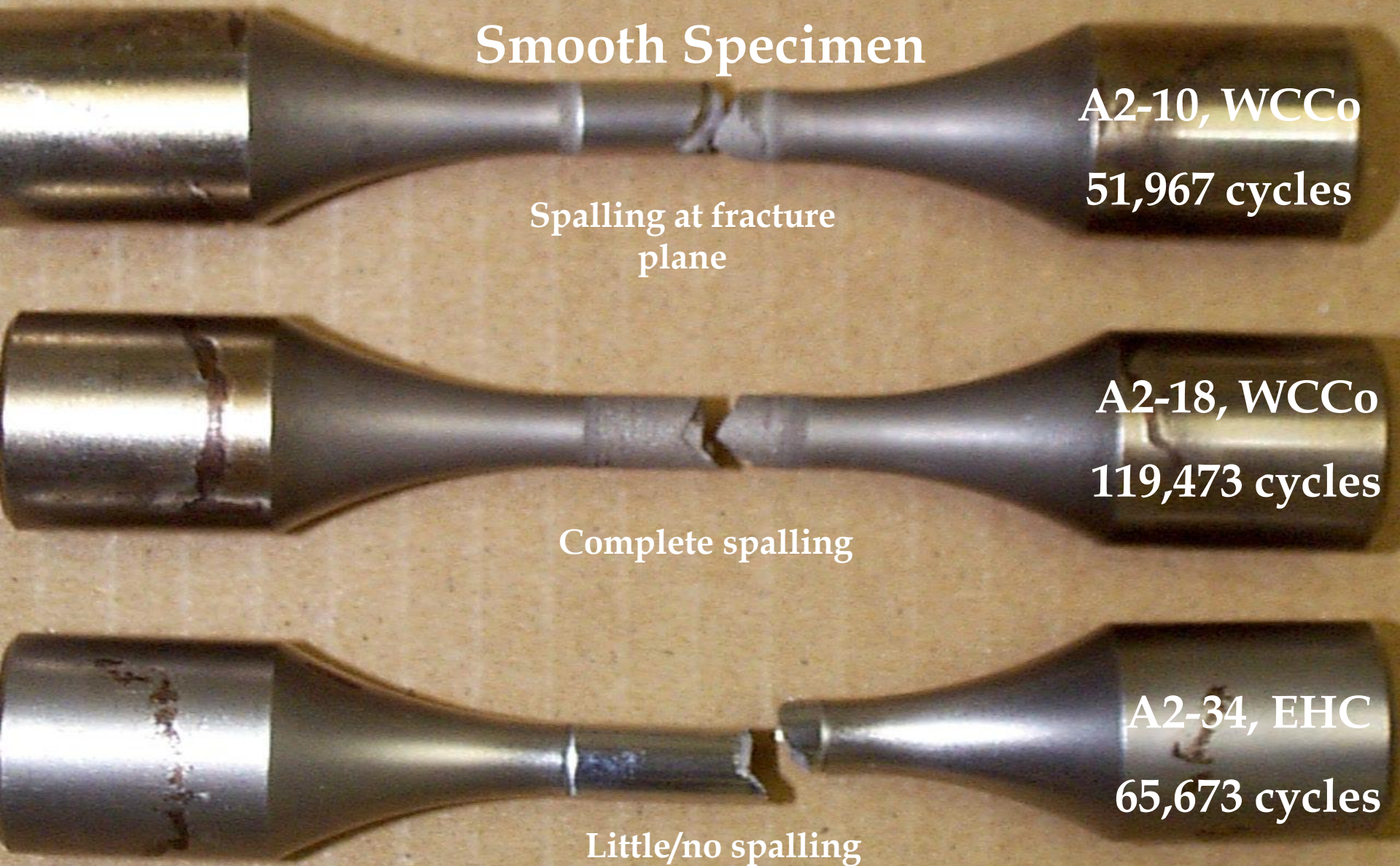
- Fatigue
  - Does a “baseline” exist??
  - High cycle or low cycle??
  - Axial
    - Most common baseline
    - R ratio…….1, -1 fully reversed??
    - Unfortunately, many parts in service don’t see axial loading

Testing Issues

- Fatigue
  - In the HCAT program especially with landing gear applications, the mode for fatigue was bending
  - In the axial fatigue testing, the HVOF coating kept "spalling" while the hard chrome never flaked off even at failure
  - Real issue…HVOF was going to fail….☹

NaCl, A100, 150 ksi, R = -1

Smooth Specimen



A2-10, WCCo

51,967 cycles

Spalling at fracture plane

A2-18, WCCo

119,473 cycles

Complete spalling

A2-34, EHC

65,673 cycles

Little/no spalling

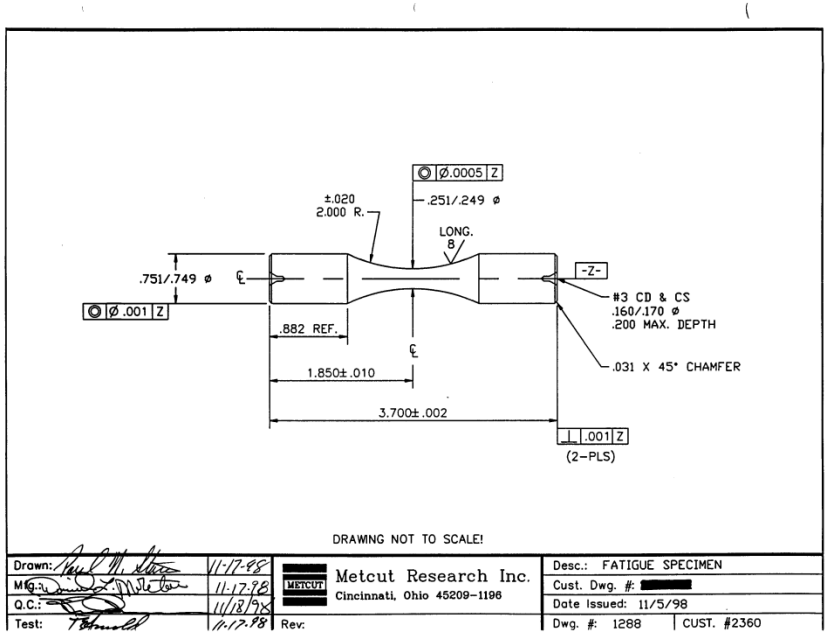
Fatigue frames

Testing Issues

- Bending
  - R ratio…….1, -1 fully reversed??
  - Unfortunately, many companies developed their "own" fatigue configuration for qualifying suppliers
  - CS community has to understand the needs for the applications and develop the best most standardized methods….

Fatigue Bars

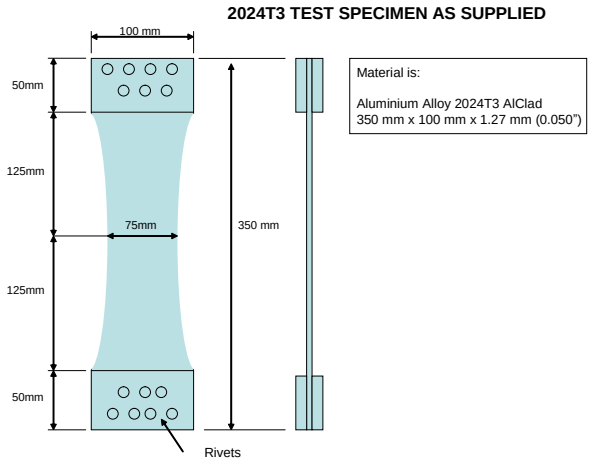
Axial Fatigue Bar

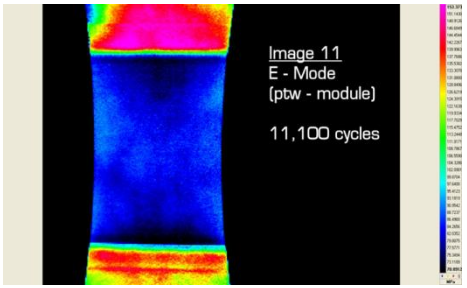


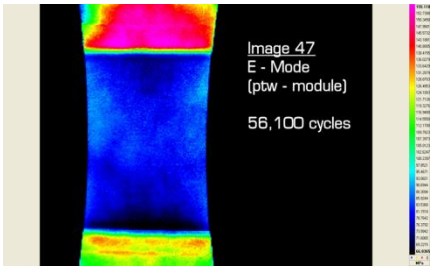
Bending Fatigue Bar

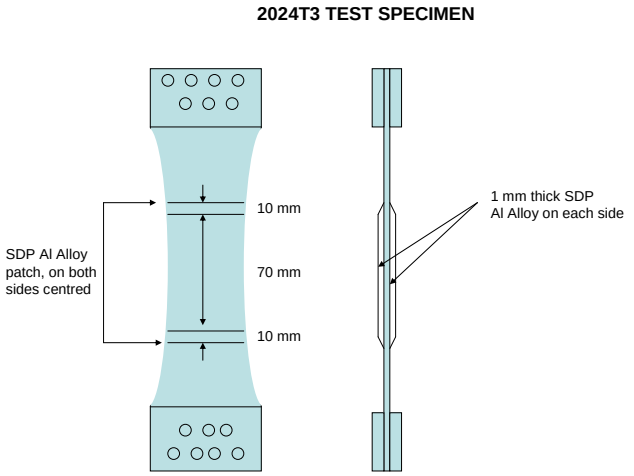
Fatigue Life Enhancement of Thin Skins with Pre-existing Defect

Experiment 1









Geometry of the Single edge notch tension (SENT) panel. An initial 2mm long edge crack

Baseline test specimen with notch and no CS Doubler

- Failure at 35,000 cycles

Geometry of the panel with an 7075 CS doubler

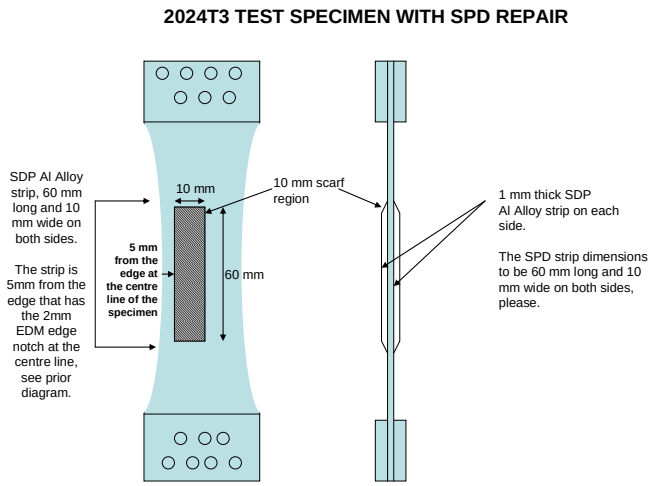
Applied peak stress was σ_{max} *= 180 MPa, R = 0.1 MPa*

For test specimen with notch and the CS Doubler

There was no growth, or damage, after 60,000 cycles. Test stopped

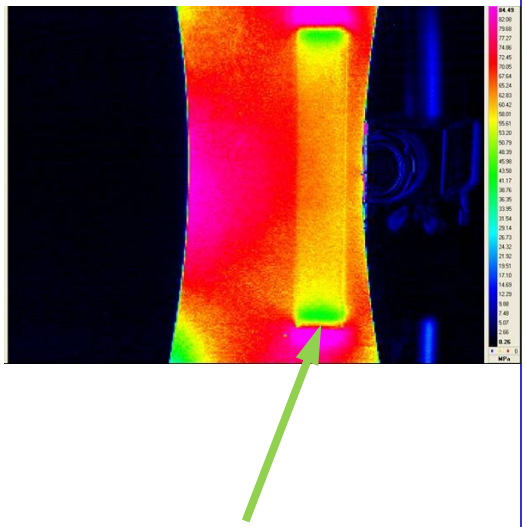
Fatigue Life Enhancement of Thin Skins with Pre-existing Defect

Experiment 2



Geometry of the panel with an 7075 CS doubler

Applied peak stress was σ_{max} *= 180 MPa, R = 0.1 MPa*



Baseline specimen (no CS) failed after ~ 40,000 cycles

This test was terminated after ~ 345,00 cycles *with no growth* from the edge crack *or damage in the CS*

Note: The CS is performing structurally and is pulling load from the skin.

Testing Issues

- Corrosion
  - Does a “baseline” exist??
  - Salt spray
    - Preparation of panels
      - HCAT lesson..panels bowed in spraying ..ground flat….less coating in center when ground…failed testing..☹((
    - Standard method
  - Galvanic corrosion
    - Exposed interface between coating and substrate

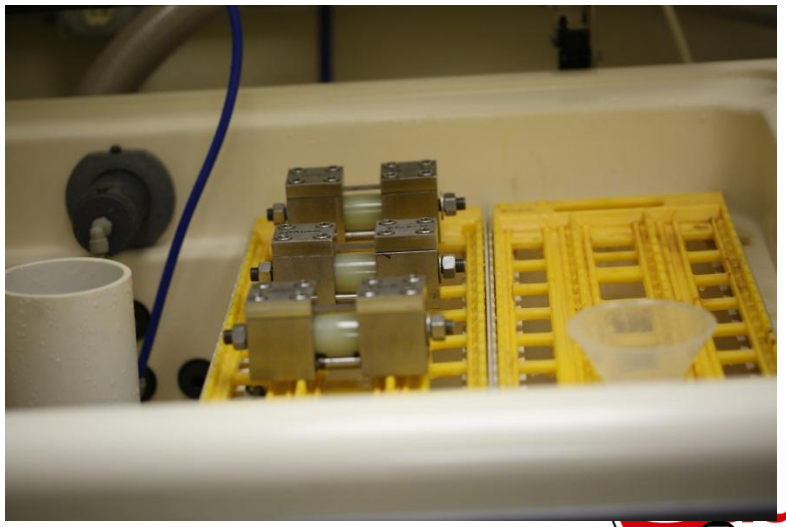
Environmental Chambers

Stress Corrosion Cracking of Al-6061

- Wrought Al 6061-T6
- As Cold Sprayed
- Tensile tests for baseline following ASTM E8
- SCC following ASTM G49-85
- In process

Eden

SCC Test Fixture



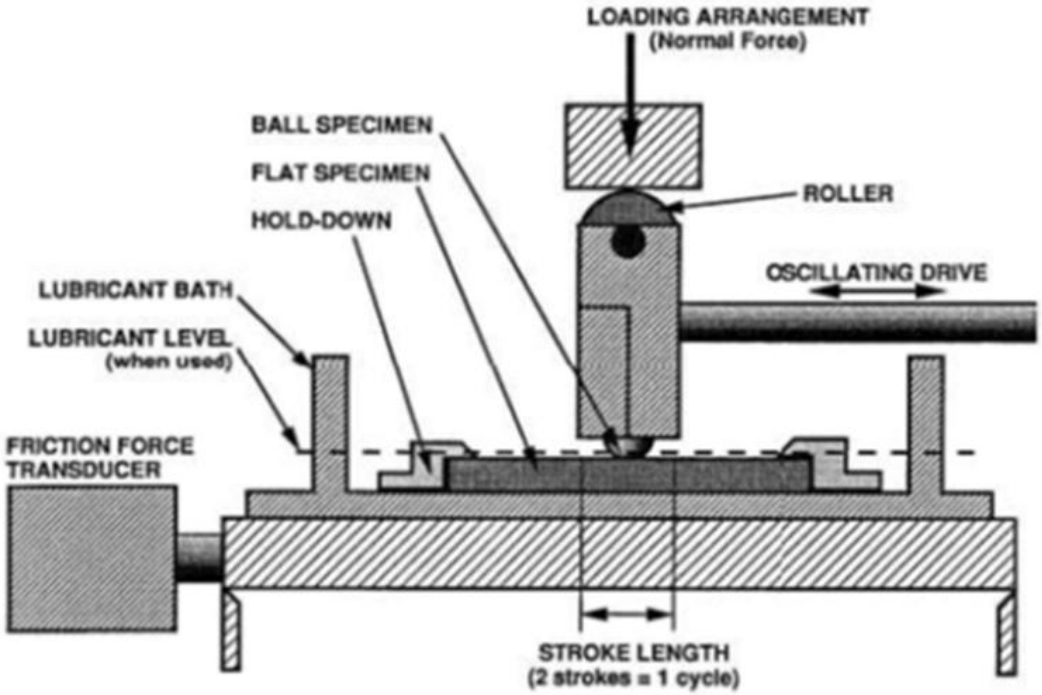
Testing Issues

- Wear
  - Does a “baseline” exist??
  - Many testing methods to choose from
  - Very sensitive to testing methodology

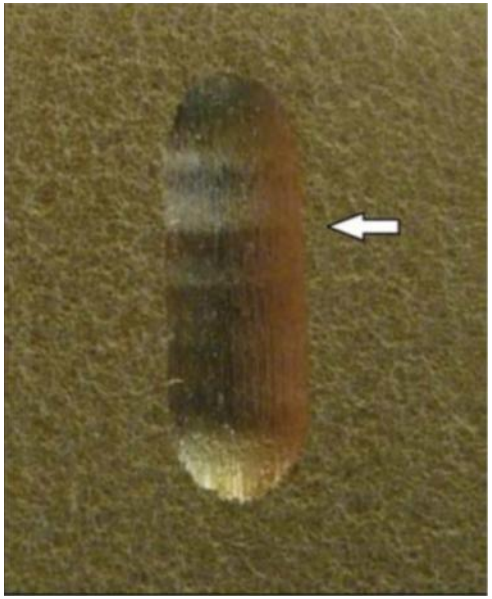
Evaluation of Coated Surfaces

- Friction Test

Eden



Reciprocating wear test
(ASTM International 2008)



wear scar after the test

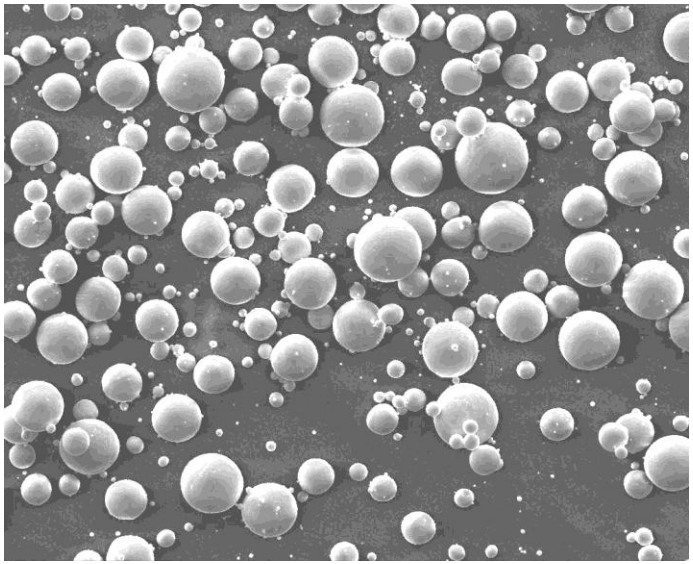
Testing Issues

- Powder Issues
  - Manufacturing method
  - Sizing
  - Morphology

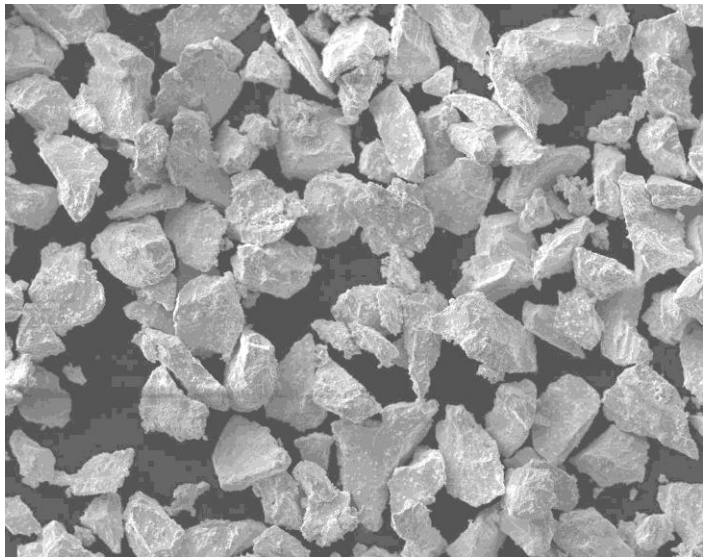
Powder Shape

Helfritch

Irregular particles spray better than spherical

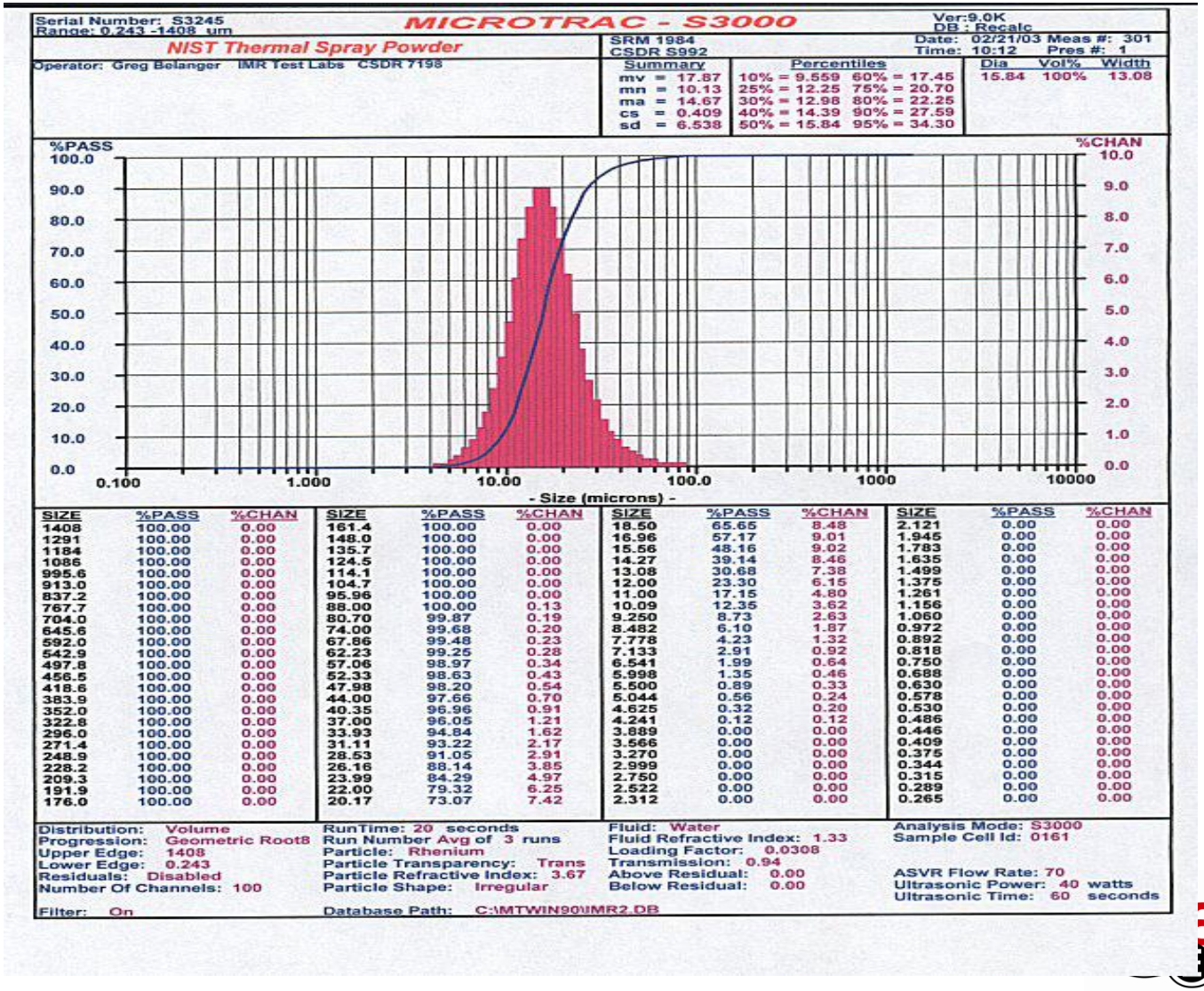


MMD = 20 microns
Shape factor = 1
DE = 23.4



MMD = 20 microns
Shape factor = 1.4
DE = 38.3

Powder - Particle Size Analysis



Serial Number: S3245
Range: 0.243 -1408 um

MICROTRAC - S3000

Ver:9.0K
DB : Recalc

NIST Thermal Spray Powder

SRM 1984
CSDR S992

Date: 02/21/03 Meas #: 301
Time: 10:12 Pres #: 1

Operator: Greg Belanger IMR Test Labs CSDR 7198

Summary		Percentiles				Dia	Vol%	Width
mv	= 17.87	10% = 9.559	60% = 17.45			15.84	100%	13.08
mn	= 10.13	25% = 12.25	75% = 20.70					
ma	= 14.67	30% = 12.98	80% = 22.25					
cs	= 0.409	40% = 14.39	90% = 27.59					
sd	= 6.538	50% = 15.84	95% = 34.30					

%PASS 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0.0

%CHAN 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

0.100 1.000 10.00 100.0 1000 10000

- Size (microns) -

SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN	SIZE	%PASS	%CHAN
1408	100.00	0.00	161.4	100.00	0.00	18.50	65.65	8.48	2.121	0.00	0.00
1291	100.00	0.00	148.0	100.00	0.00	16.96	57.17	9.01	1.945	0.00	0.00
1184	100.00	0.00	135.7	100.00	0.00	15.56	48.16	9.02	1.783	0.00	0.00
1086	100.00	0.00	124.5	100.00	0.00	14.27	39.14	8.48	1.635	0.00	0.00
995.6	100.00	0.00	114.1	100.00	0.00	13.08	30.68	7.38	1.499	0.00	0.00
913.0	100.00	0.00	104.7	100.00	0.00	12.00	23.30	6.15	1.375	0.00	0.00
837.2	100.00	0.00	95.96	100.00	0.00	11.00	17.15	4.80	1.261	0.00	0.00
767.7	100.00	0.00	88.00	100.00	0.13	10.09	12.35	3.62	1.156	0.00	0.00
704.0	100.00	0.00	80.70	99.87	0.19	9.250	8.73	2.63	1.060	0.00	0.00
645.6	100.00	0.00	74.00	99.68	0.20	8.482	6.10	1.87	0.972	0.00	0.00
592.0	100.00	0.00	67.86	99.48	0.23	7.778	4.23	1.32	0.892	0.00	0.00
542.9	100.00	0.00	62.23	99.25	0.28	7.133	2.91	0.92	0.818	0.00	0.00
497.8	100.00	0.00	57.06	98.97	0.34	6.541	1.99	0.64	0.750	0.00	0.00
456.5	100.00	0.00	52.33	98.63	0.43	5.998	1.35	0.46	0.688	0.00	0.00
418.6	100.00	0.00	47.98	98.20	0.54	5.500	0.89	0.33	0.630	0.00	0.00
383.9	100.00	0.00	44.00	97.66	0.70	5.044	0.56	0.24	0.578	0.00	0.00
352.0	100.00	0.00	40.35	96.96	0.91	4.625	0.32	0.20	0.530	0.00	0.00
322.8	100.00	0.00	37.00	96.05	1.21	4.241	0.12	0.12	0.486	0.00	0.00
296.0	100.00	0.00	33.93	94.84	1.62	3.889	0.00	0.00	0.446	0.00	0.00
271.4	100.00	0.00	31.11	93.22	2.17	3.566	0.00	0.00	0.409	0.00	0.00
248.9	100.00	0.00	28.53	91.05	2.91	3.270	0.00	0.00	0.375	0.00	0.00
228.2	100.00	0.00	26.16	88.14	3.85	2.999	0.00	0.00	0.344	0.00	0.00
209.3	100.00	0.00	23.99	84.29	4.97	2.750	0.00	0.00	0.315	0.00	0.00
191.9	100.00	0.00	22.00	79.32	6.25	2.522	0.00	0.00	0.289	0.00	0.00
176.0	100.00	0.00	20.17	73.07	7.42	2.312	0.00	0.00	0.265	0.00	0.00

Distribution: Volume
Progression: Geometric Root8
Upper Edge: 1408
Lower Edge: 0.243
Residuals: Disabled
Number Of Channels: 100

Filter: On

RunTime: 20 seconds
Run Number Avg of 3 runs
Particle: Rhenium
Particle Transparency: Trans
Particle Refractive Index: 3.67
Particle Shape: Irregular

Database Path: C:\MTWIN90\IMR2.DB

Fluid: Water
Fluid Refractive Index: 1.33
Loading Factor: 0.0308
Transmission: 0.94
Above Residual: 0.00
Below Residual: 0.00

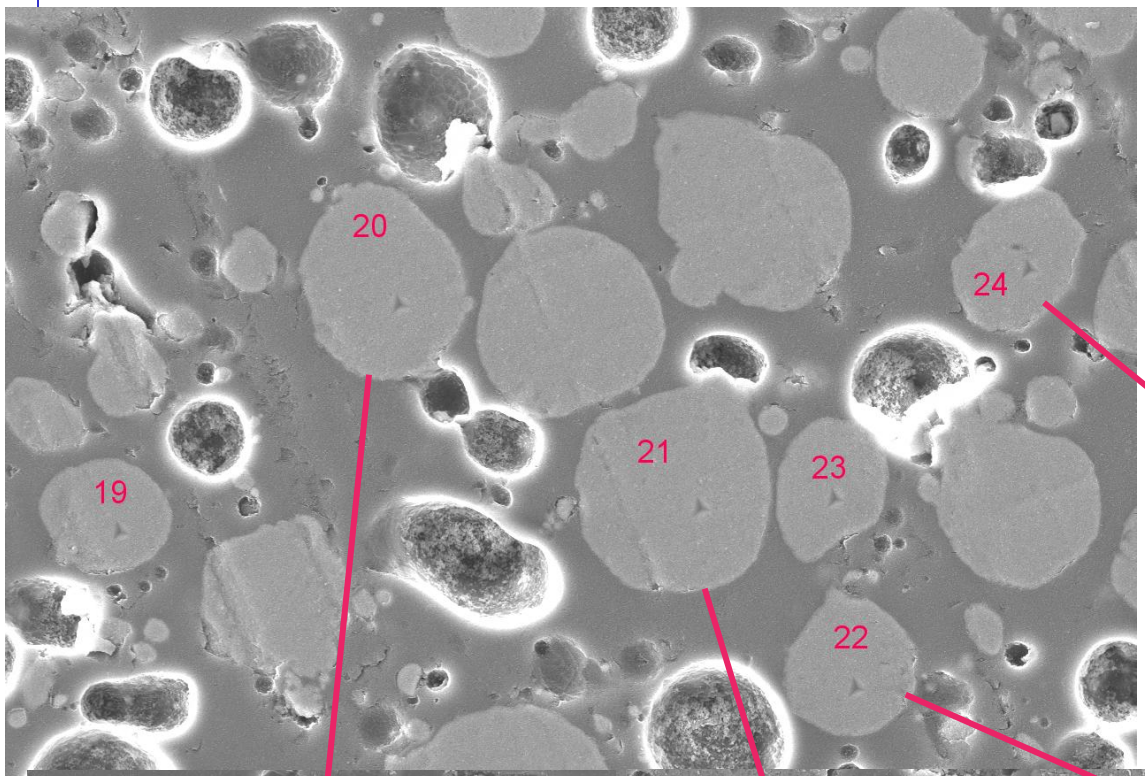
Analysis Mode: S3000
Sample Cell Id: 0161

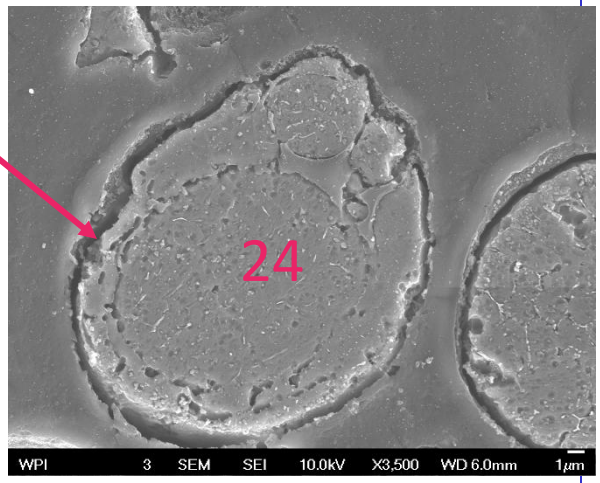
ASVR Flow Rate: 70
Ultrasonic Power: 40 watts
Ultrasonic Time: 60 seconds

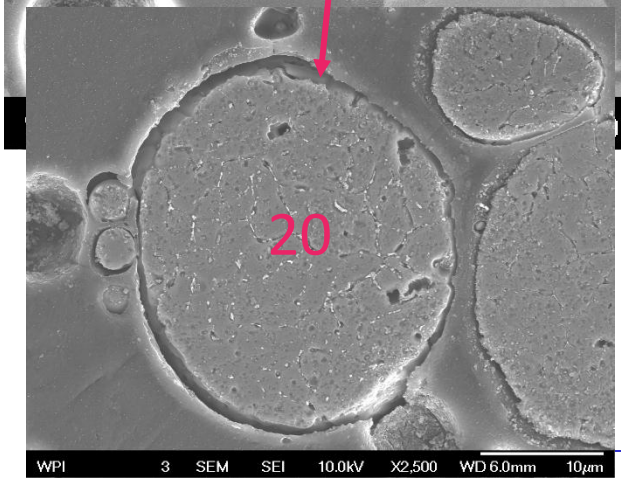
Experimental Hardness Measurements

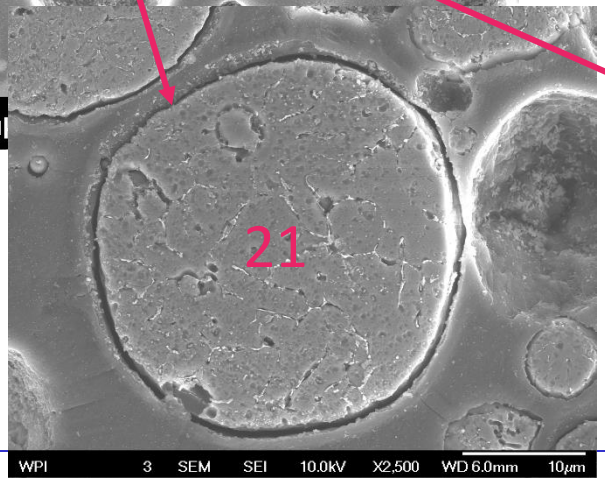
BIRT

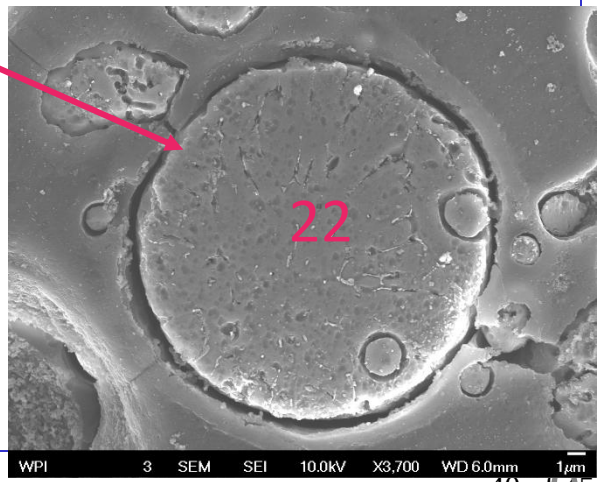
Nanohardness measurements of Al 6061 powder





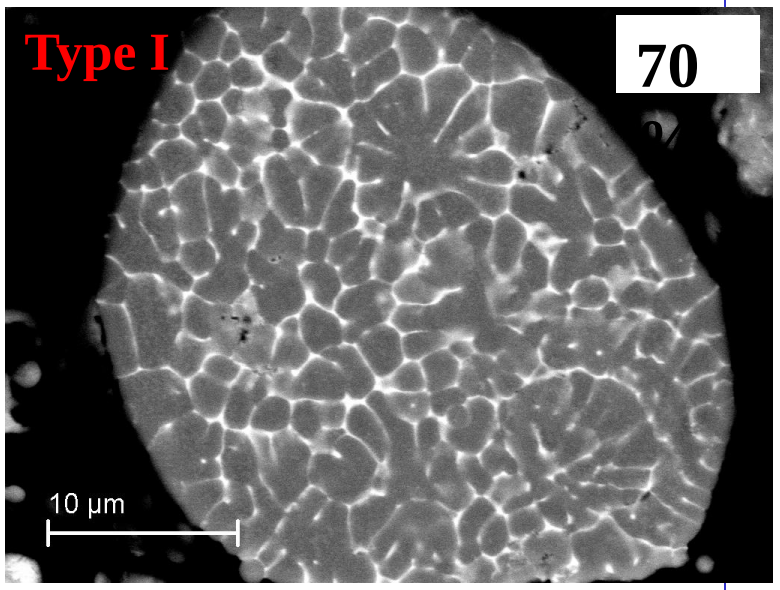


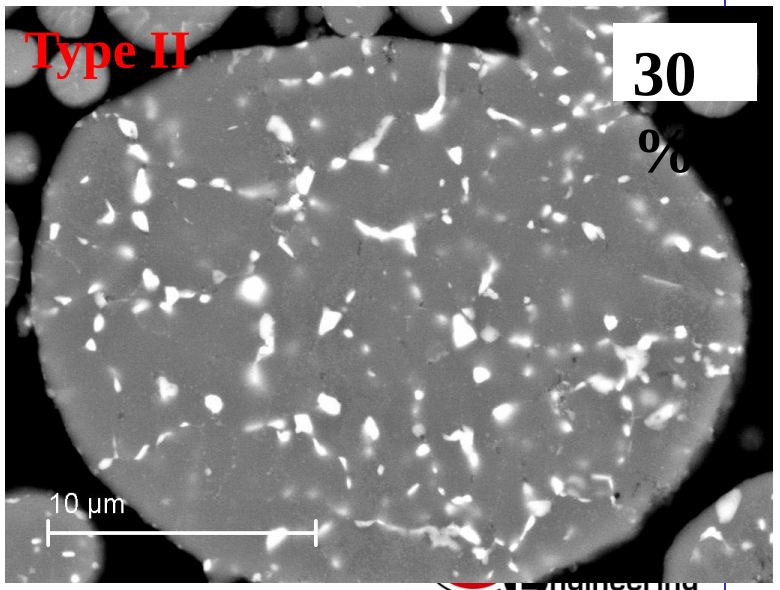




Powder Microstructure

- ✓ Two different internal grain and GB structure in the powder particles
- ✓ Type I: the same internal grain structure as that of the surface and with GB solutes segregation
- ✓ Type II: larger grain size with some precipitates at the GBs, due to lower solidification rate than that of the type I





Decision Making

- What do we want from a mature CS process??

COATING

OR

STRUCTURAL REPAIR??

This can and will dictate the qualification process going forward…..☺

Decision Making

- Shapes the next major philosophical steps going forward
- Needs to be a CS community decision and united philosophy going forward

Next Steps

- Co-ordinated efforts in sharing qualification efforts and not re-inventing the wheel
- If sharing is limited, at least collaborate on lessons learned when mistakes are made

Next Steps

- Process consistency TOTALLY ANOTHER TALK
  - Can we repeat the process
  - Powder consistency
  - Hardware consistency
  - Elimination of “spray and pray”
  - Use of plume sensor technology