Optimization of High Temperature Silicon Nitride For Microturbine Applications

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Hot Section Materials Development For Advanced Microturbines Program

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- Dave Richerson, Dave Carruthers

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Objective

Hot Section Si_3N_4 for DG Advanced Microturbine Program

- Develop and improve a cost-effective, reliable monolithic silicon nitride material for Hot Section Components
- Develop and improve a cost-effective, reliable complex shape forming capability
- Through surface engineering, demonstrate sufficient environmental stability

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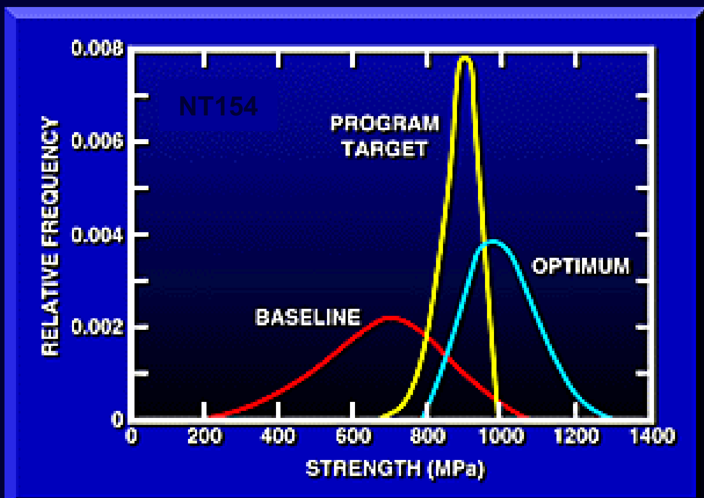
High Temperature Si_3N_4 (NT154)

Process Flow

ROBUST CLOSED-LOOP PROCESSING



DOE/ORNL Improved Processing Program (1993)

Process step	Purpose
Raw Materials	High purity
Aqueous Milling	Chemistry and particle size control
Magnetic Separation	Remove metallic impurities
Filtration	Remove potential flaws
Drying	
Forming	Dimensional and surface finish control
Densification (Glass HIPing)	Glass HIPing for consistently high density
Post-HIP Treatment	Improve high temperature properties

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Ceramic Microturbine Technology

Ceramic Microturbine Technology

Material Development

- Re-establish NT154/NT164
- As-Processed Surface Improvement
- Recession Resistance

Net Shape Forming Development

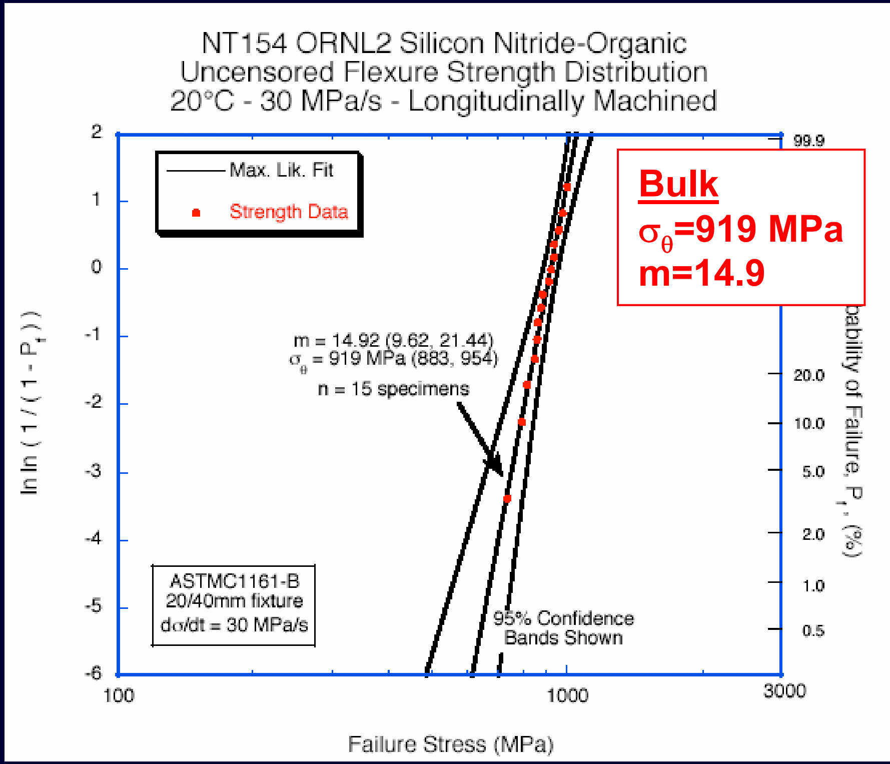
- Green CNC Machining
- Direct Casting

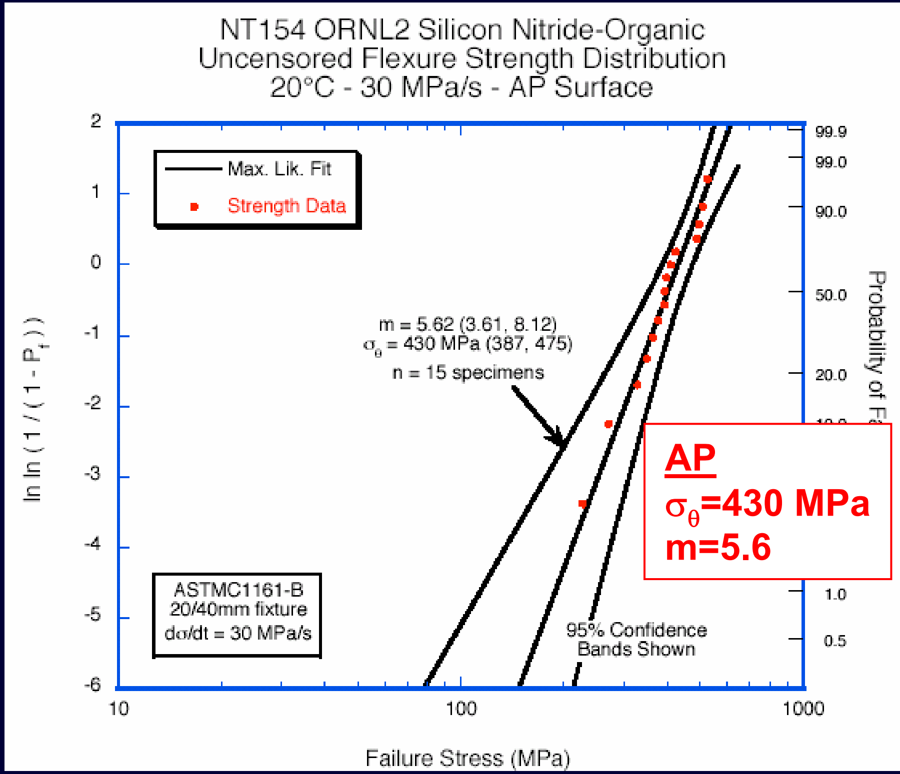
Testing at ORNL

NT154 Task II

➢Room Temperature Fast Fracture Results

- Green Machining Composition





Courtesy of HT Lin, ORNL

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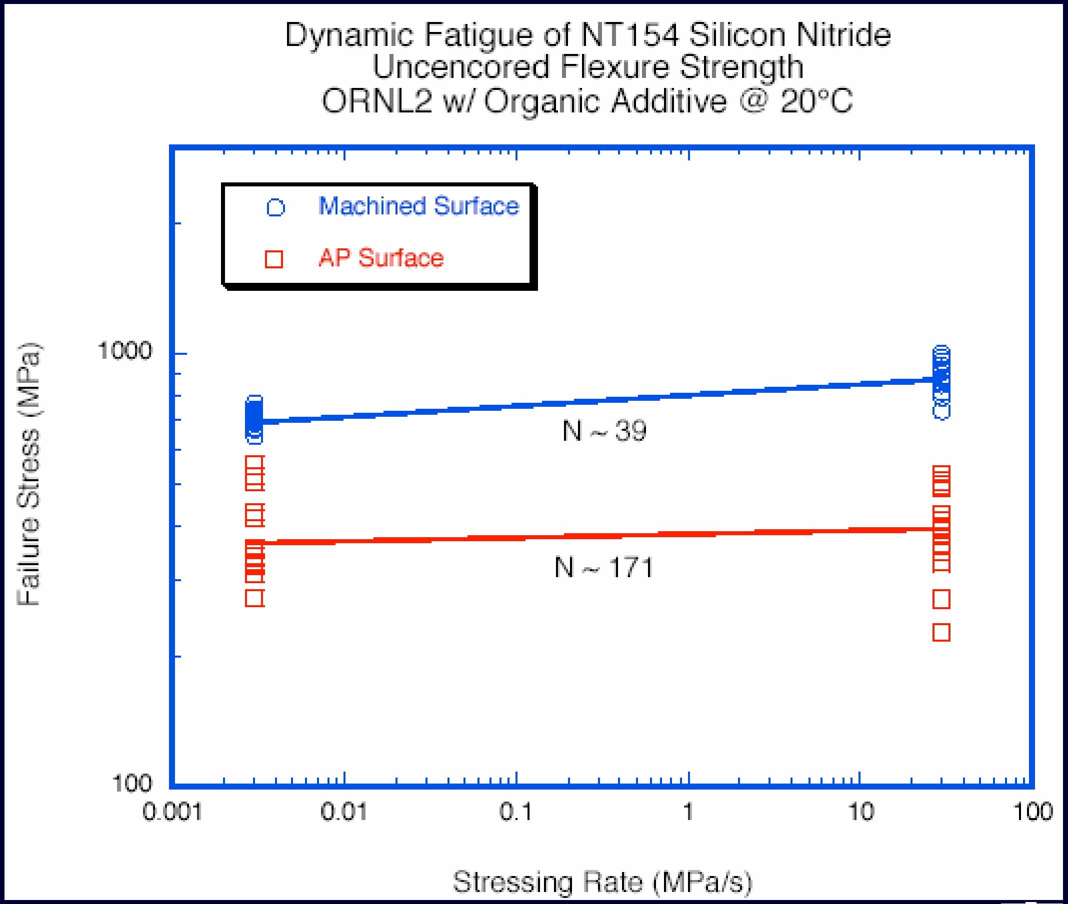
Testing at ORNL

NT154 Task II

- ➢Slow Crack Growth Testing



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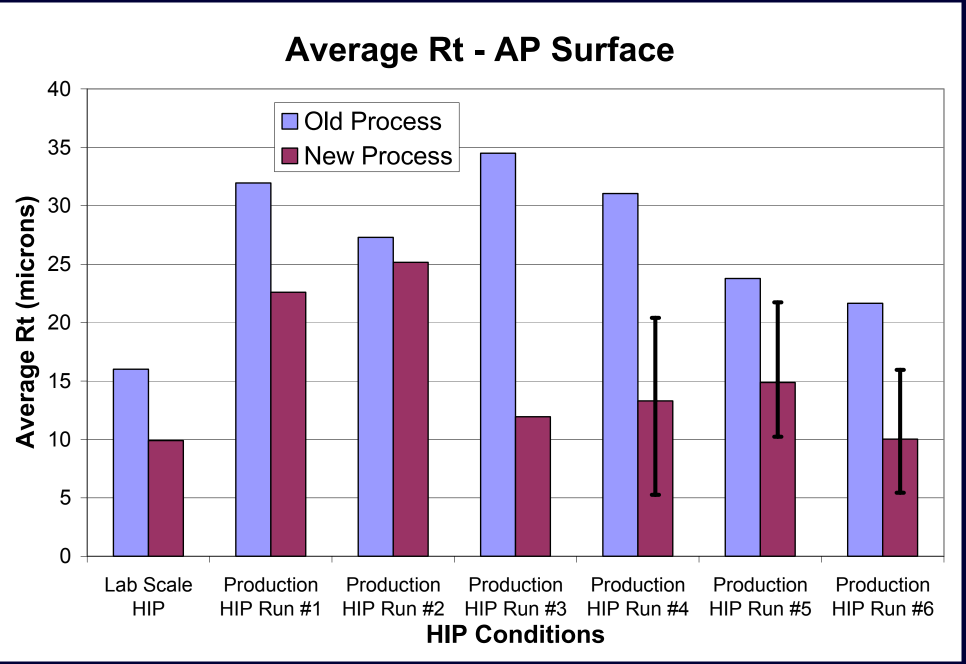
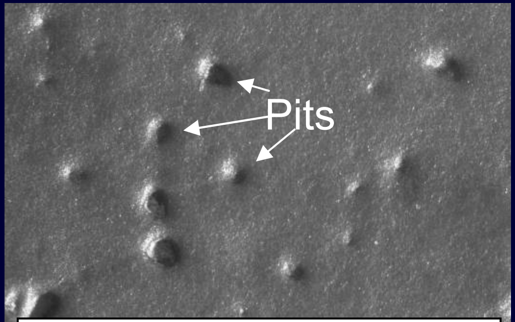
As-Processed Surface Improvement

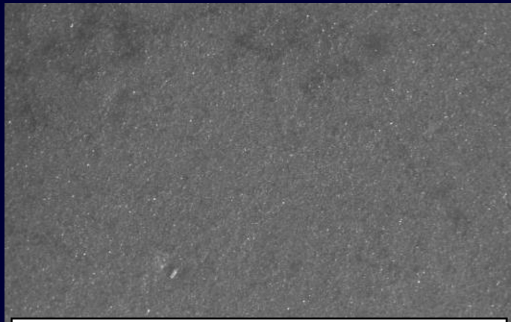
Proprietary New HIP Process

- ➢Minimize interaction between NT154 and HIP glass
- ➢Improved AP surface properties
- ➢Green surface quality has a direct impact towards the dense surface roughness





"Old Process" Si3N4 Surface



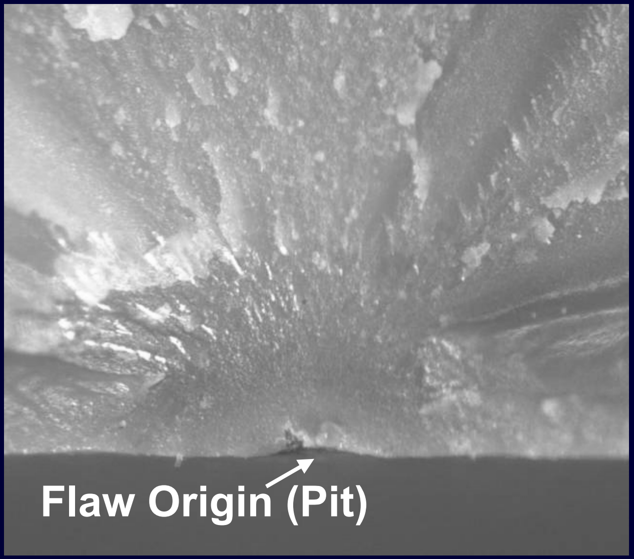
"New Process" Si3N4 Surface

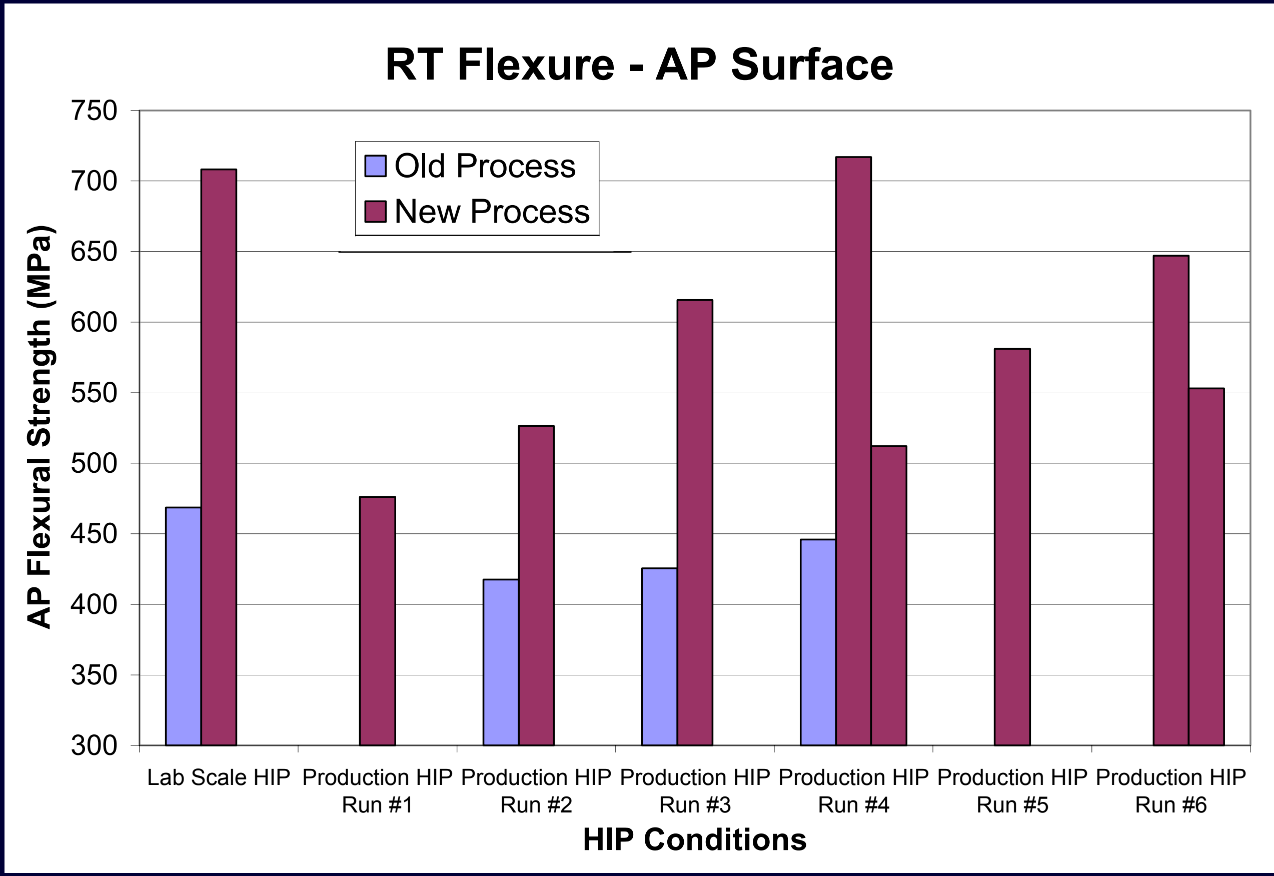
As-Processed Surface Improvement

Proprietary New HIP Process

- **AP strength controlled by:**
  - **Toughness**
  - **Flaw Size**

$$\sigma = C \frac{KIC}{(a)^{1/2}}$$

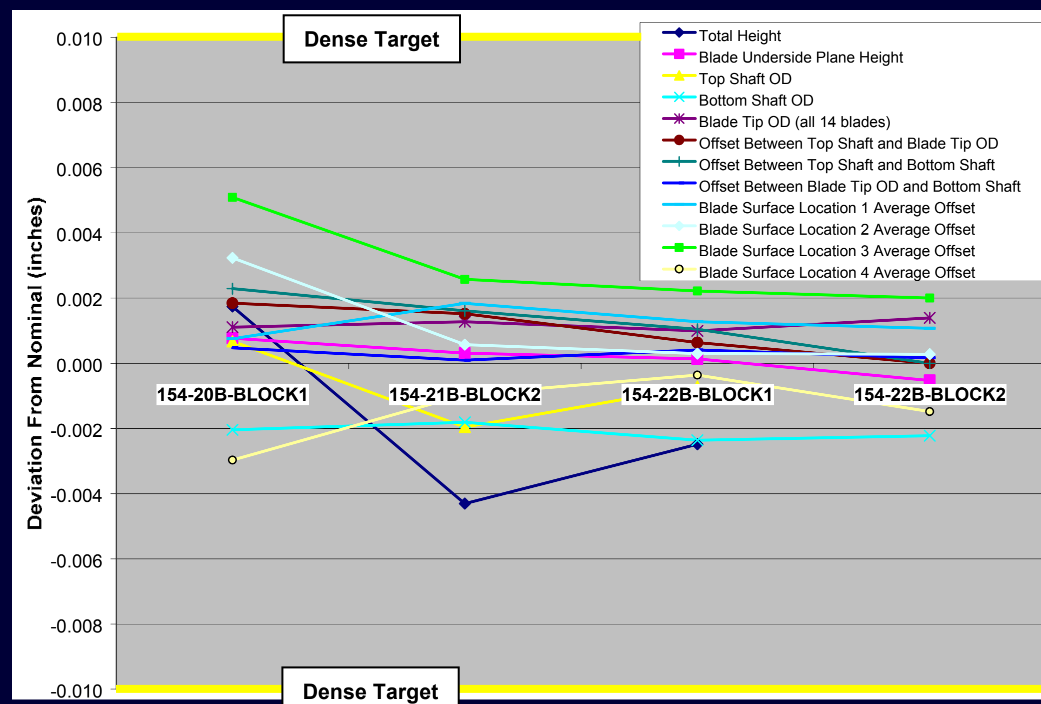




Net Shape Forming

Green Machining

- Developed green machining process through in-house experimentation
- Green machined four radial rotor prototypes
- Green dimensions within 0.004” of nominal



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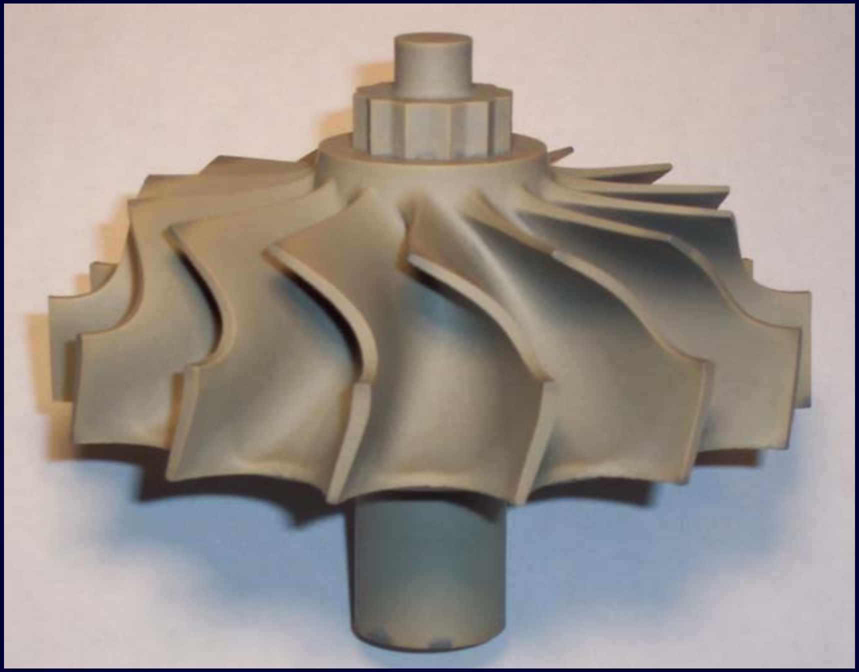
Net Shape Forming

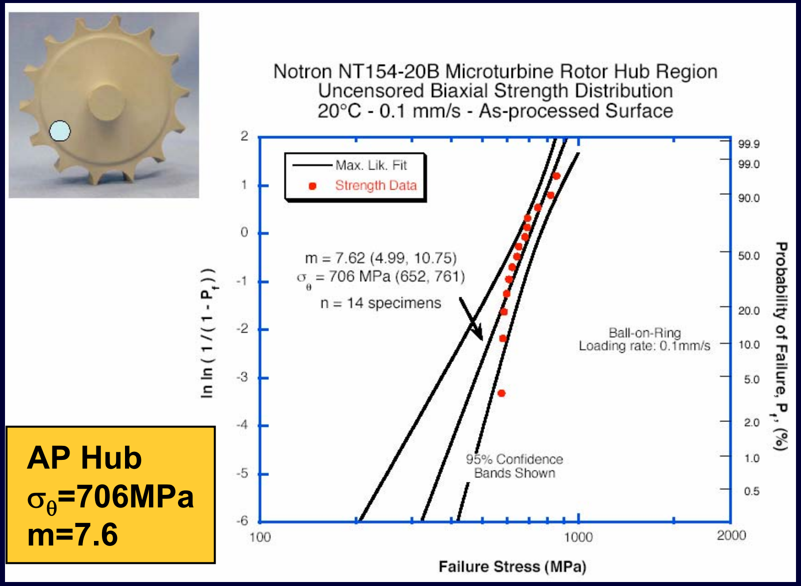
Green Machining

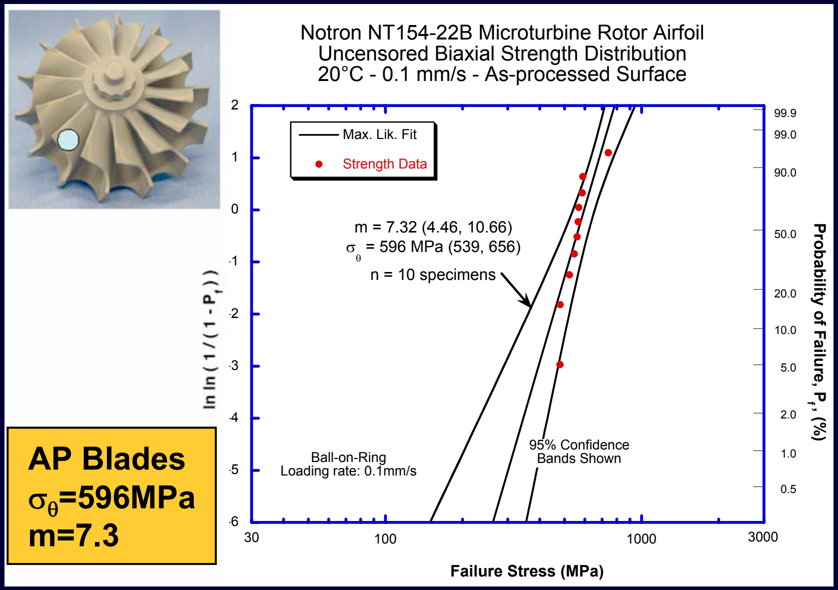
- HIPed All Rotors Utilizing New HIP Process for Improved AP
  - Average surface roughness of 30-38μin (Ave. Rt of 6-8μm)
  - Average part to part variation of 0.003”
  - Concentricity within 0.004”
  - Two rotors sent to ORNL for mechanical property evaluation

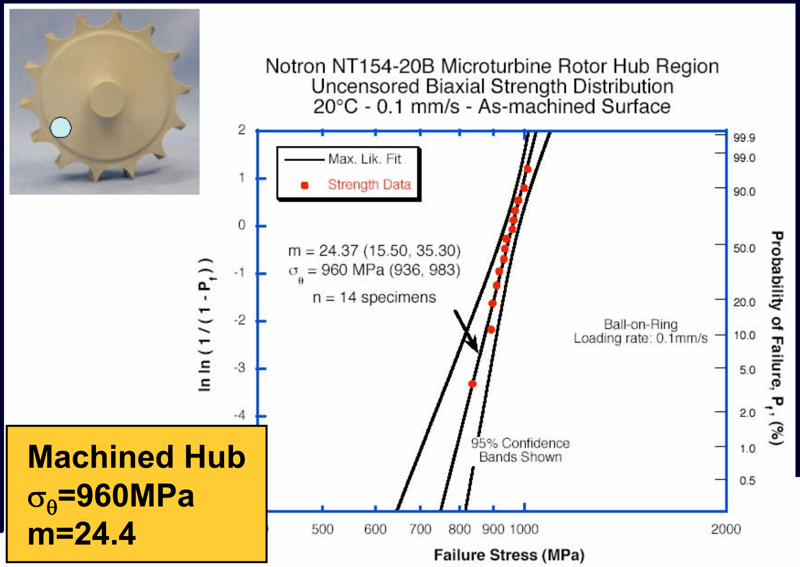


Net Shape Forming

Rotor Mechanical Property Evaluation at ORNL







Tile Properties Reproduced in Net-Shape Component

Courtesy of HT Lin, ORNL (11/12/04)

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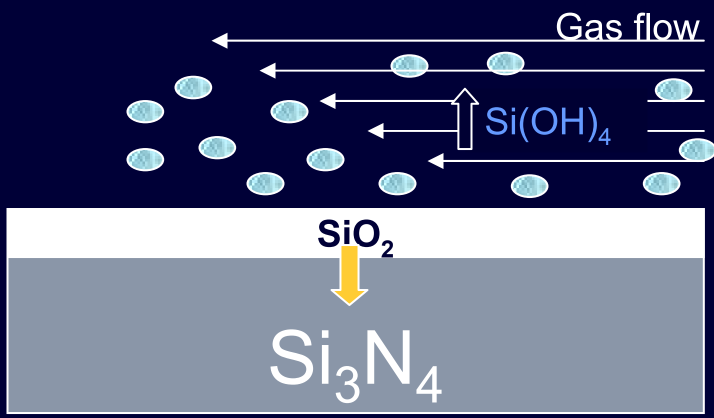
Recession Control

Mechanism

Volatilization of SiO_2 leads to recession of Si-Based Advanced Ceramics

$$SiO_2 + 2H_2O = Si(OH)_4$$



$$\text{Volatility} \approx \exp(-\Delta G/RT)\frac{V^{1/2}(P_{H2O})^2}{(P_{TOTAL})^{1/2}}$$

T: Temperature
V: Gas velocity
P_{H2O}: Water vapor pressure
P_{TOTAL}: Total pressure

(Opilia, Smialek, Robinson, Fox, and Jacobson, JACS, 1999, 82(7))

Si3N4 Weight Loss Rate ~ 0.05mg/cm²h (1200°C) → ~0.15μm/hour

- **For a 100μm EBC coating, a ~15X reduction in the recession rate is required**

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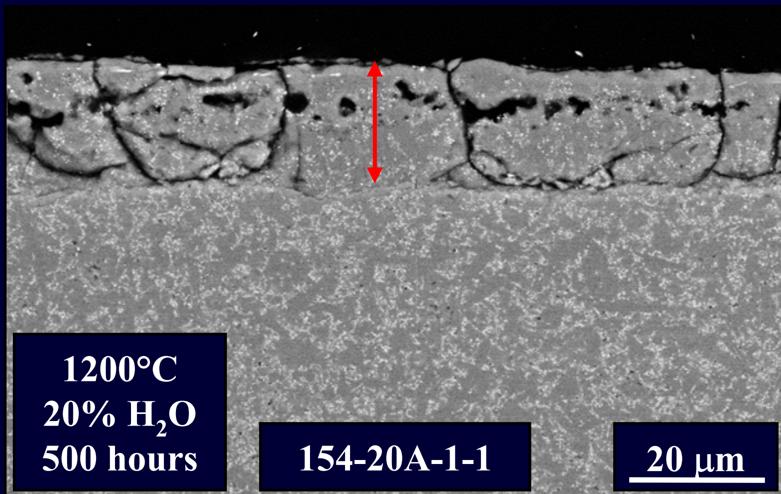
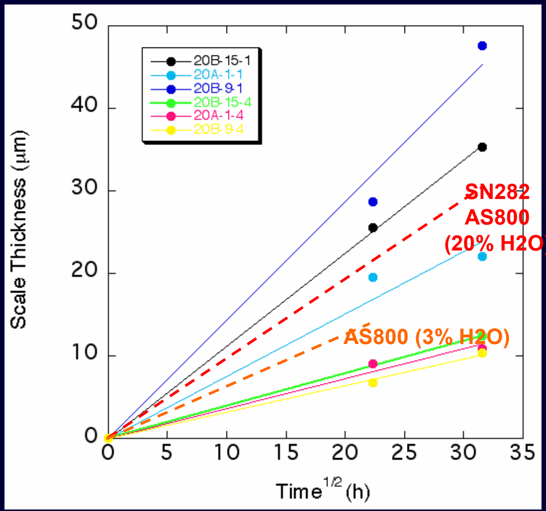
Recession Control

Keiser Rig Testing



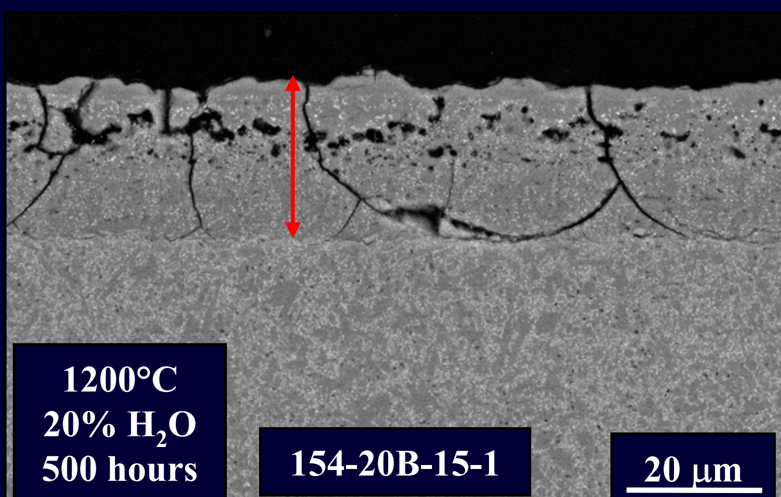


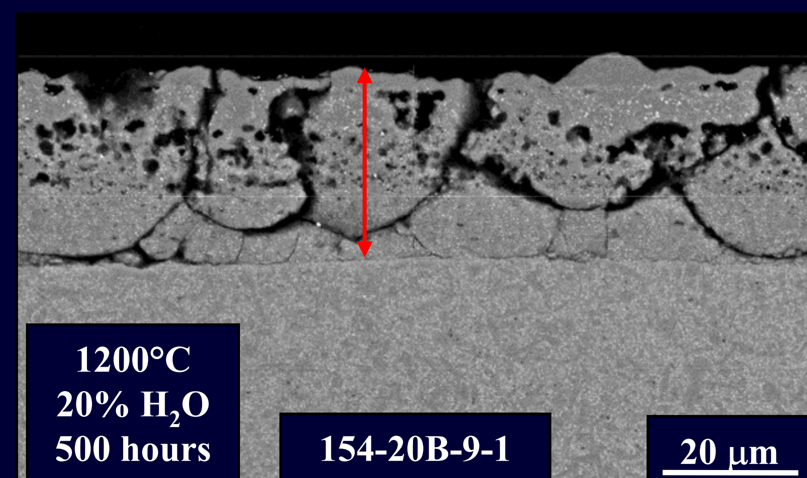
Exposed at 20% H_2O

- 20B-9 (Green mach. binder system + new HIP process)
- 20B-15 (Green mach. binder system)
- 20A-1 (No organics)

Exposed at 3% H_2O

- Differences here are harder to distinguish





Courtesy of Karren More, ORNL

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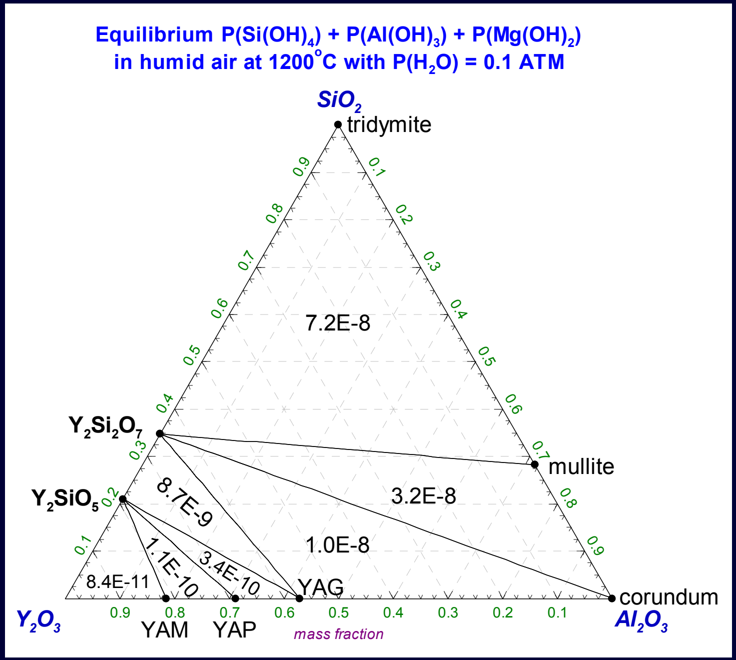
Recession Control

Candidate Materials

- Rare-earth Silicates (SiO2-Al2O3-Y2O3)
- Geomimetic with Ceramatec
- Other Alumino Silicates

Candidate materials chosen based on literature data, thermodynamic calculations and available CTE data



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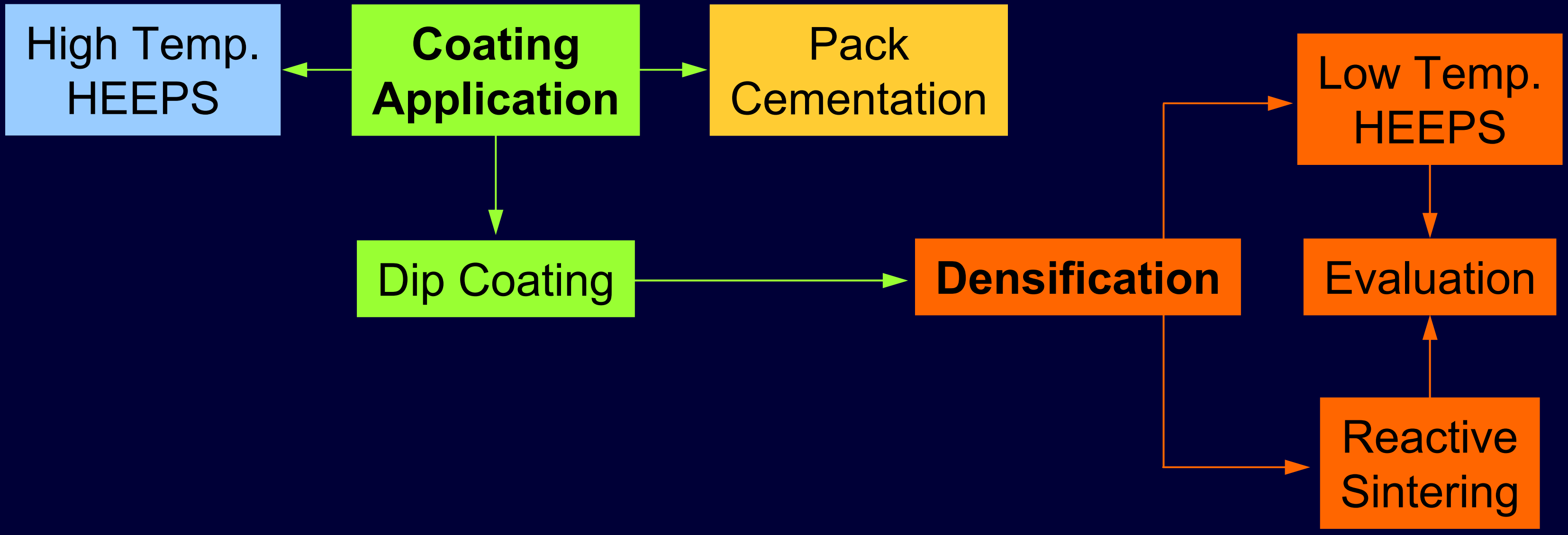
Recession Control

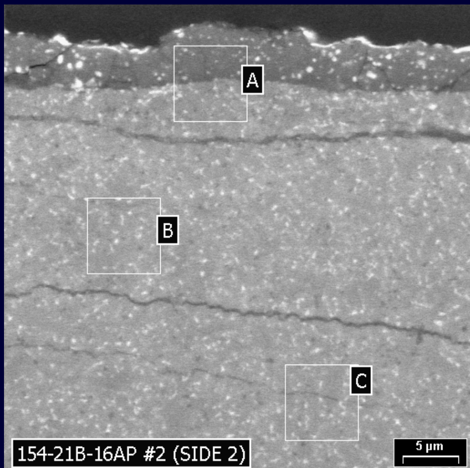
Coating Application and Densification

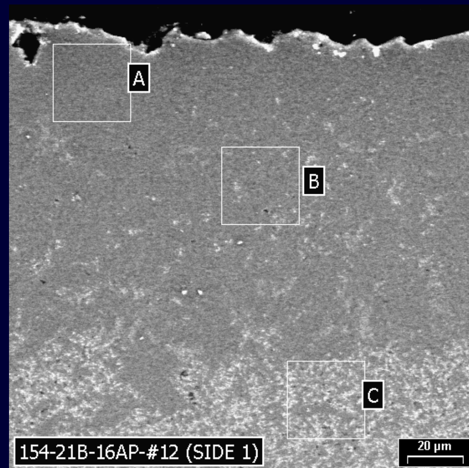
Courtesy of Ceramatec, Inc.

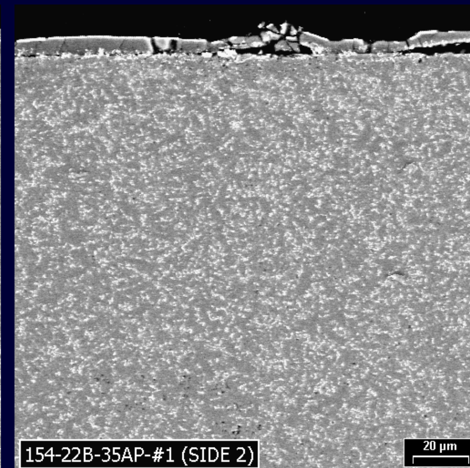


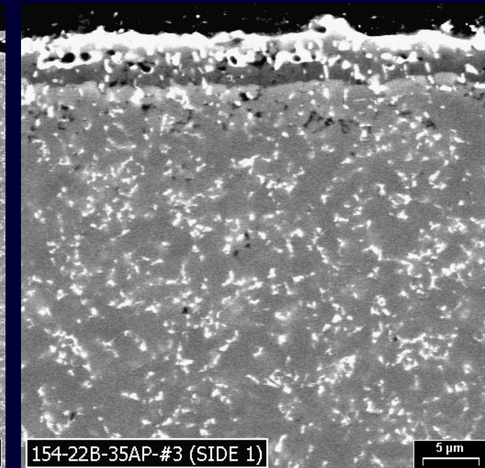
EBC Development

Pack Cementation









Experimental Matrix

- Dense NT154 imbedded in various oxide powder beds
- Conditions
  - 1200-1600°C
  - Environments of air and vacuum explored
  - Up to 48 hour soak times
- Analysis
  - SEM
  - XRD
  - Internal recession test

Issues

- Incomplete transformation of surface
- Low reaction depths
- Near surface cracking
- Si3N4 creep deformation at 1600°C
- Internal recession test shows only modest improvements

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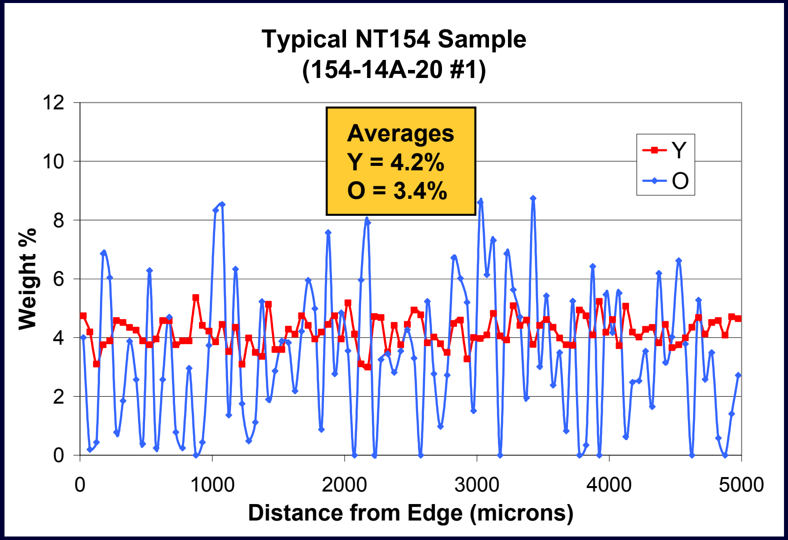
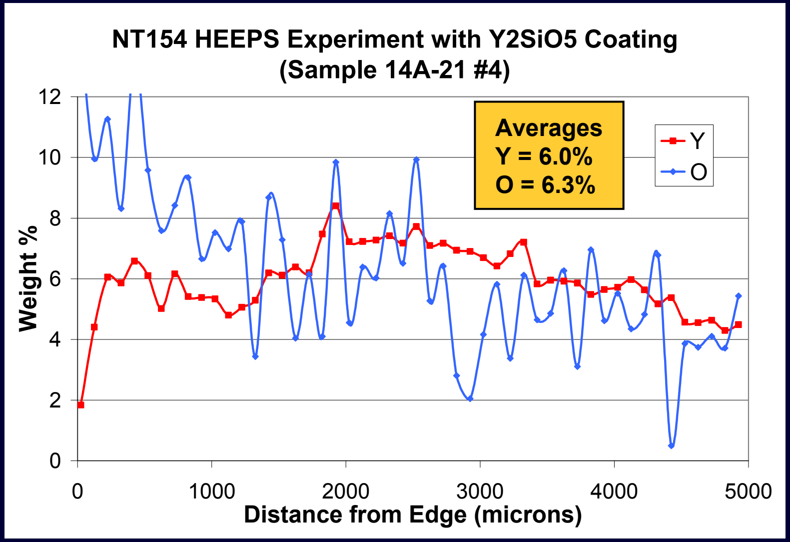
EBC Development
High Temperature HEEPS





Experimental Matrix
- Co-densification of NT154 and EBC
- Various rare earth oxide and silicate dip coatings
- Analysis
  - •SEM
  - •XRD
  - •Internal recession test

Issues
- Coatings appear to diffuse throughout
- Reaction with HIP glass
- Rough AP surfaces
- Internal recession test shows no improvement over baseline NT154

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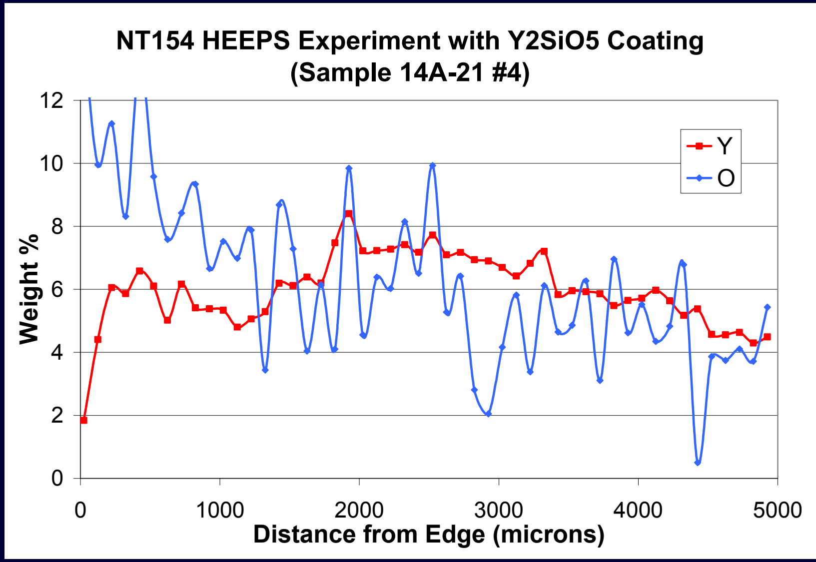
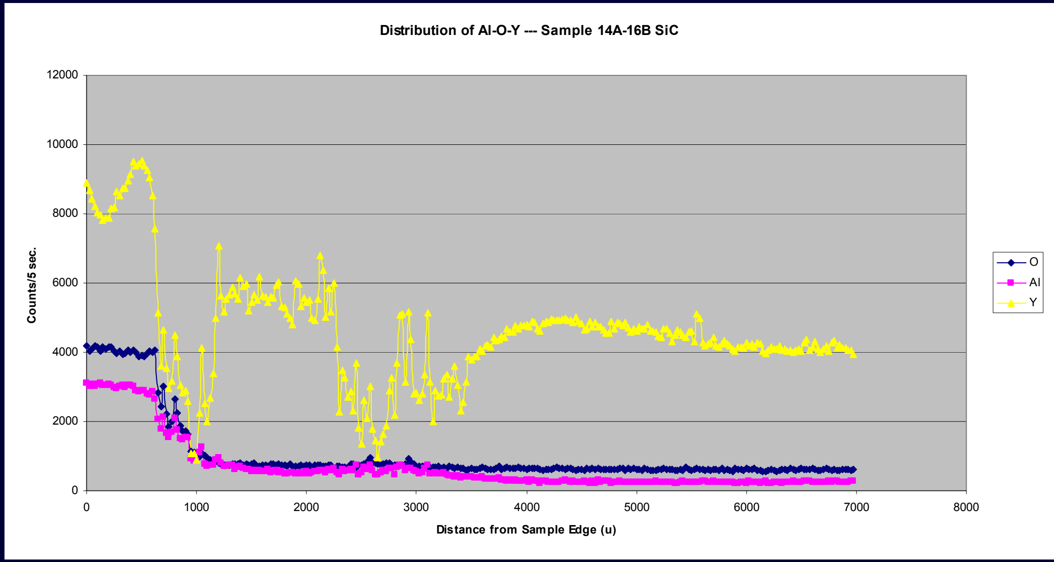
EBC Development
High Temperature HEEPS





Initial High Temperature HEEPS Trial Shows undesirable diffusion of Y_2O_3

Modified High Temperature HEEPS with Internal Barrier successfully retains Functionally graded surface with Y_2O_3

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Recession Control

Internal Recession Test

➢ **Screening tool**

- 1200°C
- ~50% H2O
- ~0.2 m/s flow rate
- 60 – 75 hours

Samples	Recession Run #3	
	Normalized mass change (mg/cm2/hour)	Recession Rate (micron/hour)
Yttria	**-4.9E-04**	**9.8E-04**
Alumina	-1.5E-03	3.8E-03
Yttria stabilized Zirconia	-2.6E-03	4.5E-03
NT154 Pack Cementation #12	-3.1E-03	9.6E-03
Baseline NT154	**-3.8E-03**	**1.2E-02**
NT154 HEEPS #1	-3.9E-03	1.2E-02
Fused Quartz	**-5.5E-03**	**2.5E-02**

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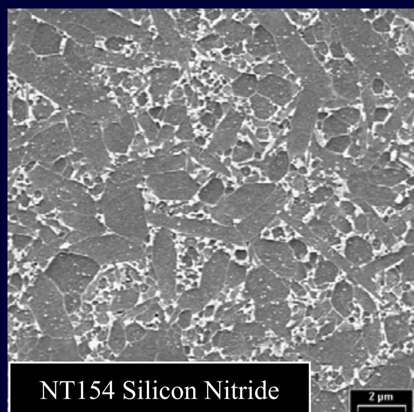
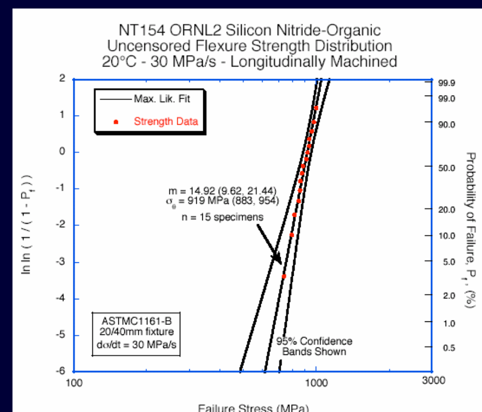
Microturbine Material & Process Technology Status

Material Development

NT154 Room Temp. Properties	NT154 High Temp. Properties	NT154 AP Strength	NT154 Slow Crack Growth	Recession Control
Strength > 950MPa Toughness > 6MPa-m$^{1/2}$	1200°C Strength > 700MPa	As-processed surface Strength > 700MPa	**N:** Bulk = 39 AP = 171	Possible materials and application methods identified





Shape Forming – Green Machining

Green Shape Control	Dimensional Control (Dense)	Surface Roughness	Strength
Dimensions of green rotors consistently within 0.004" of nominal	Part to part variation within 0.006" Concentricity within 0.003"	As-processed surface Ra = 30μin	Machined Strength = 960MPa AP Strength up to 706MPa

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