

# Hot Section Silicon Nitride Materials Development For Advanced Microturbines Applications

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*Vimal Pujari, Ara Vartabedian,  
Bill Collins, James Garrett  
Saint-Gobain Ceramics & Plastics, Inc.*



9 Goddard Road  
Northboro, MA 01532

  
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**CERAMICS & PLASTICS**

# Hot Section Materials Development For Advanced Microturbines Program

- > Co-Authors: Bill Collins, Bill Donahue, James Garrett, Oh-Hun Kwon, Bob Licht, Vimal Pujari, Ara Vartabedian

## Acknowledgements

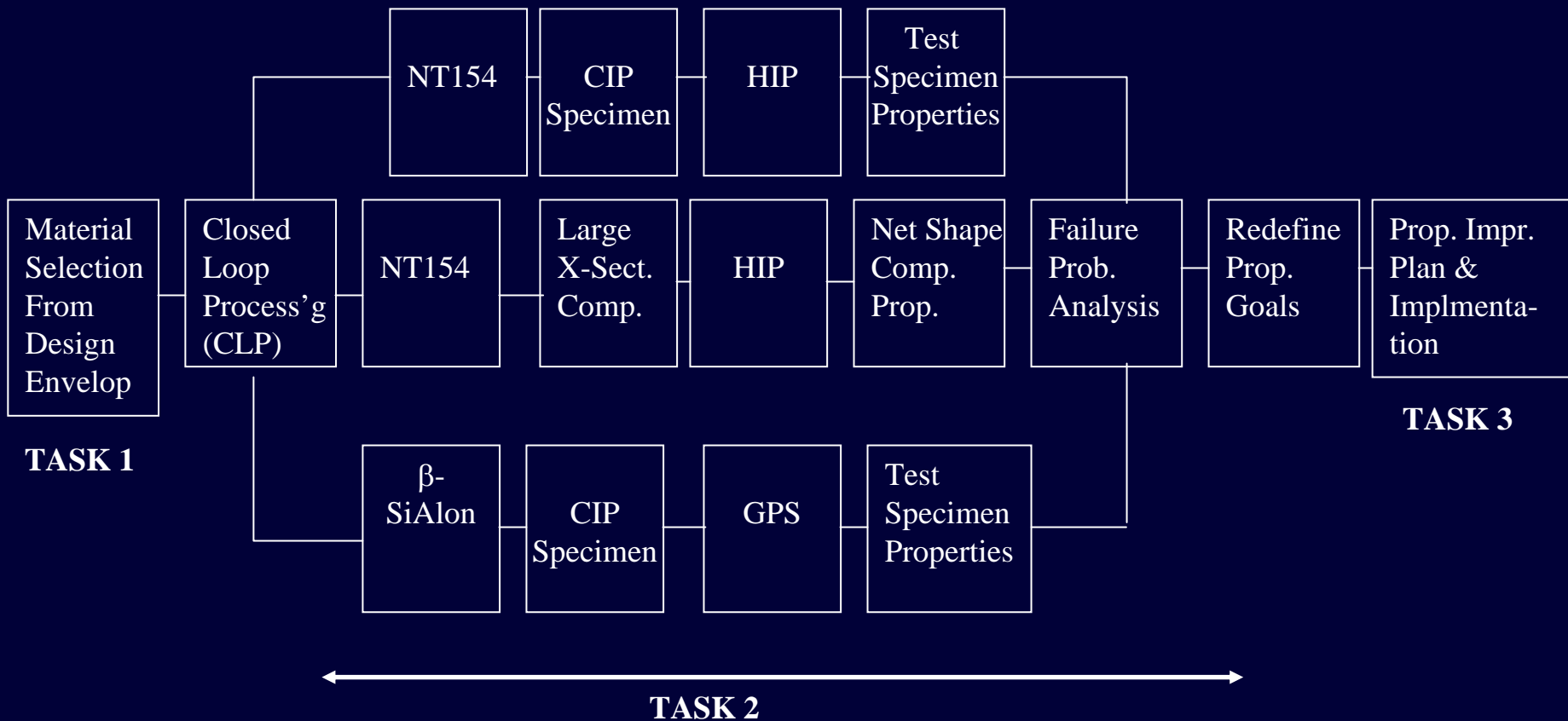
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# Objective

## Hot Section $\text{Si}_3\text{N}_4$ for Advanced Microturbine Program

- > Under DOE/ORNL Program, Develop and improve a cost-effective, reliable monolithic silicon nitride material for Hot Section Components in DER Advanced Microturbine Systems
- > Through surface engineering, demonstrate sufficient environmental stability for operation w/o EBC -- Or compatible with EBC

# Hot Section Materials Development For Advanced Turbines (Phase I)



# Material and Process Approach

## Ceramic Microturbine Technology

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graph TD; A[Ceramic Microturbine Technology] --> B[Material Development]; A --> C[Net Shape Forming Development];
```

### Material Development

- Re-establish NT154
- Improve NT154
- Recession Control
- Alternate Composition

### Net Shape Forming Development

- Green CNC Machining
- Casting / Molding

# Testing at ORNL

## NT154 Material Qualification

### ➤ Delivered task I tiles

- Testing has been completed

### ➤ Delivered task II tiles

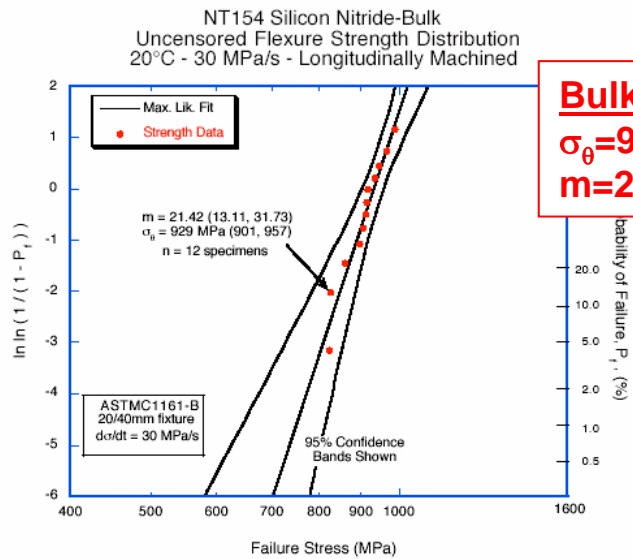
- Optimized HIP conditions for bulk material
- Two sets of tiles
- Room temperature fast fracture testing completed

# Testing at ORNL

## NT154 Task I

### ➤ Room Temperature Fast Fracture Results

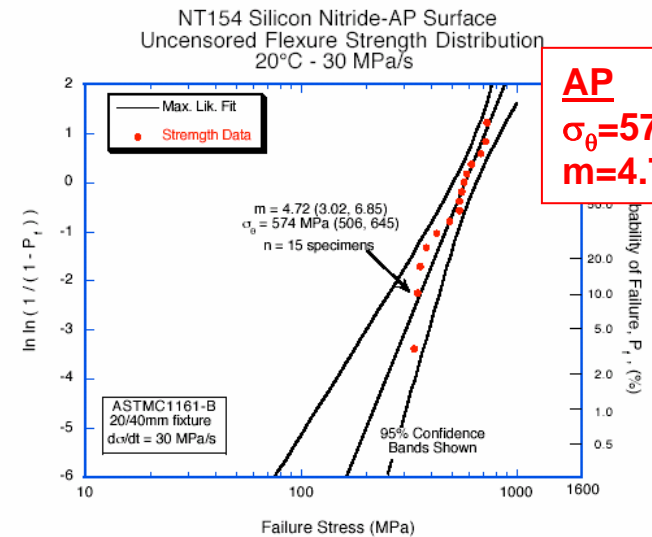
Results Obtained Consistent with the Reported Data for NT154-11C, but with a Higher Weibull Modulus



**Bulk**  
 $\sigma_\theta = 929$  MPa  
 $m = 21.4$

NT154-11C:  $\sigma_{\text{mean}}$ : 948 MPa,  $m = 8.6$

As-Processed NT154 Exhibited 62% Characteristic Strength Obtained for Bulk Material



**AP**  
 $\sigma_\theta = 574$  MPa  
 $m = 4.7$

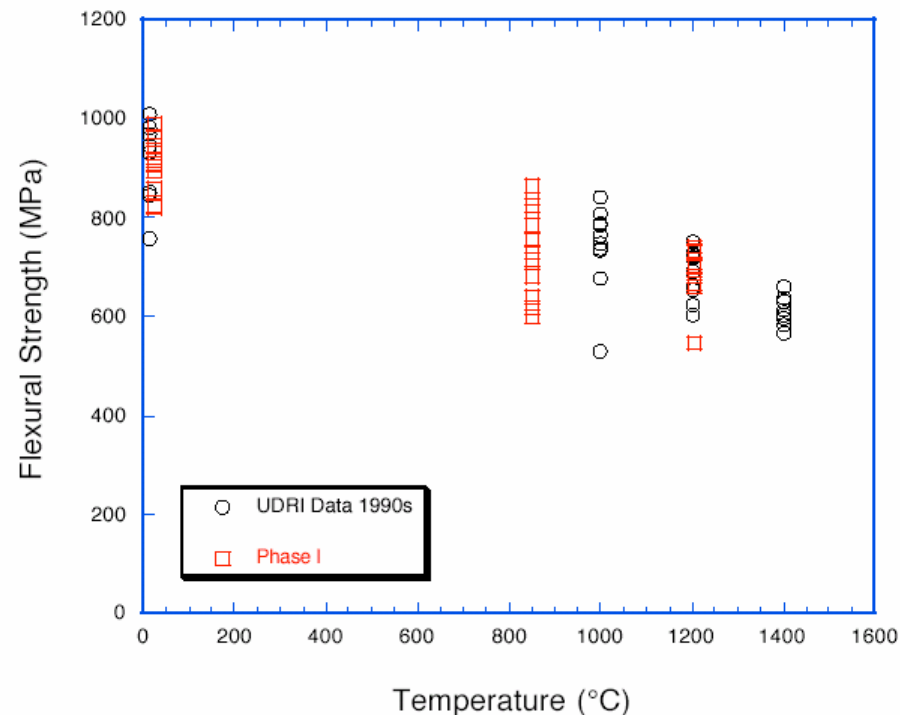
The as-processed NT154 also exhibited a low Weibull modulus ( $m = 4.7$ )

➤ Need to improve as-processed (AP) properties

# Testing at ORNL

## NT154 Task I

*The Mechanical Performance of NT154 Phase I Materials is Consistent to Those Manufactured in 1990s*



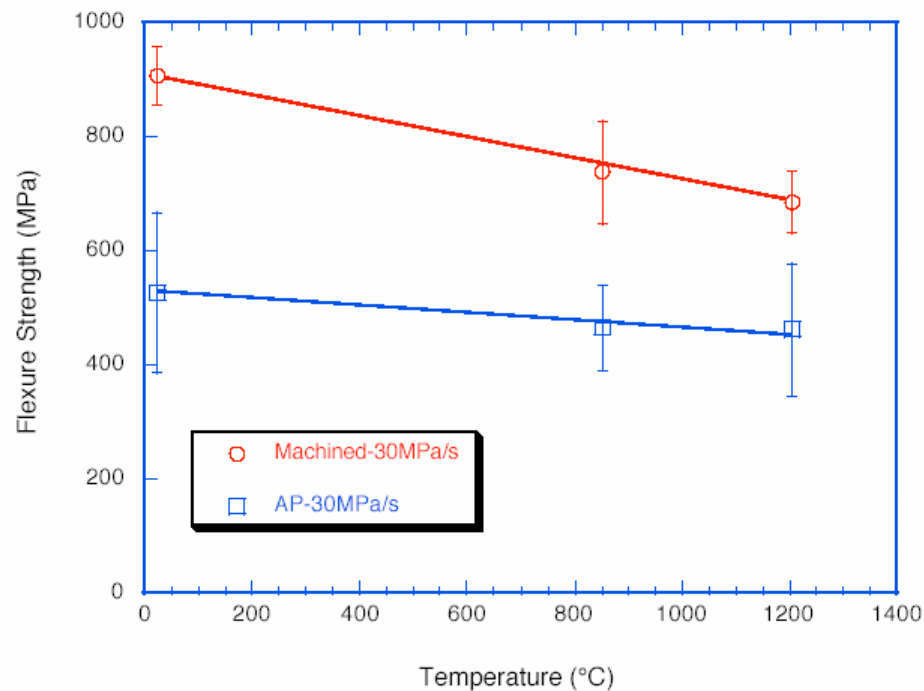
➤ **Fast fracture material properties re-established**



# Testing at ORNL

## NT154 Task I

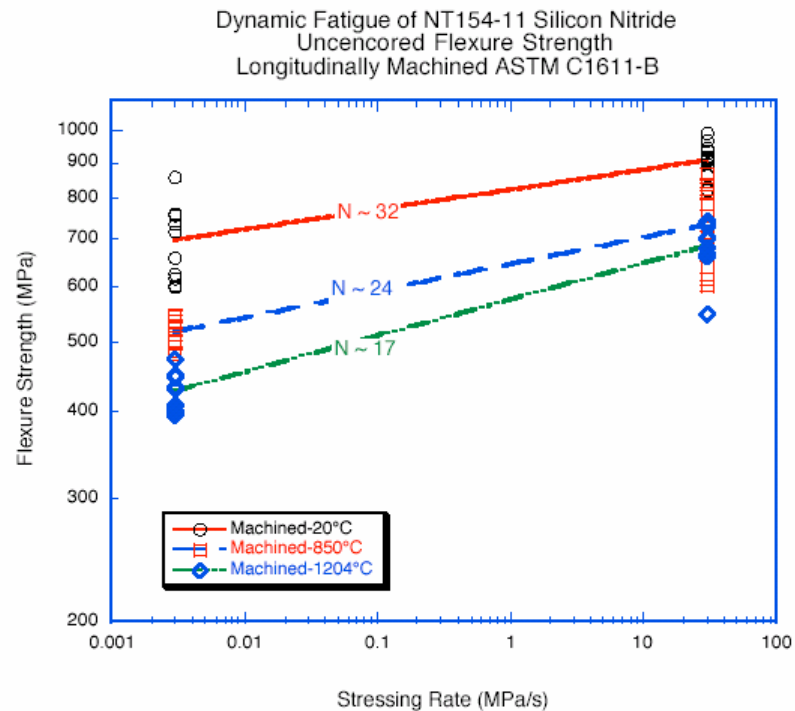
*The NT154-11 w/ As-Processed Surface Exhibits Less Temperature-Dependence of Strength Than the Bulk*



# Testing at ORNL

## NT154 Task I

*The NT154 Task I Materials Exhibited Low Dynamic Fatigue Exponents Between 20 and 1204°C*

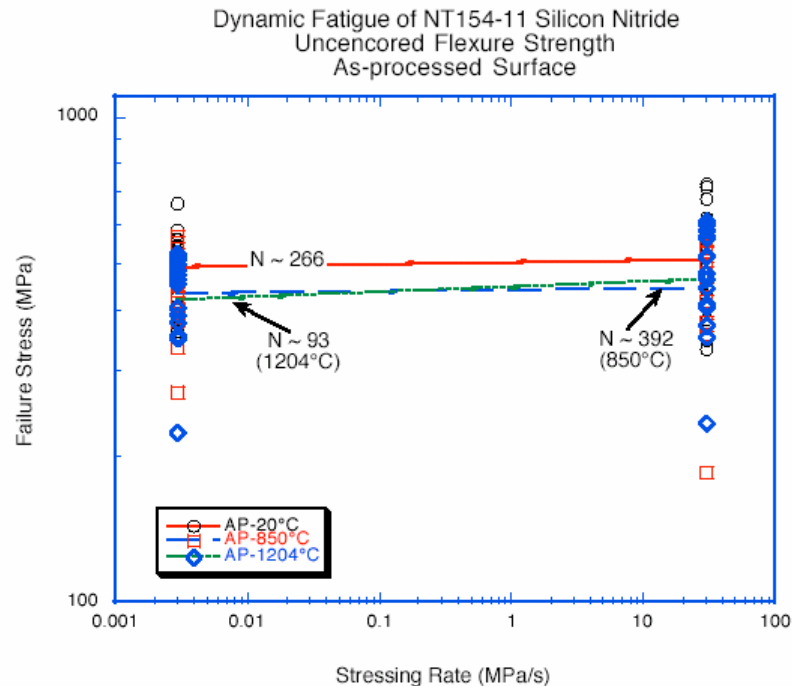


➤ **Fatigue exponents similar to historical data**

# Testing at ORNL

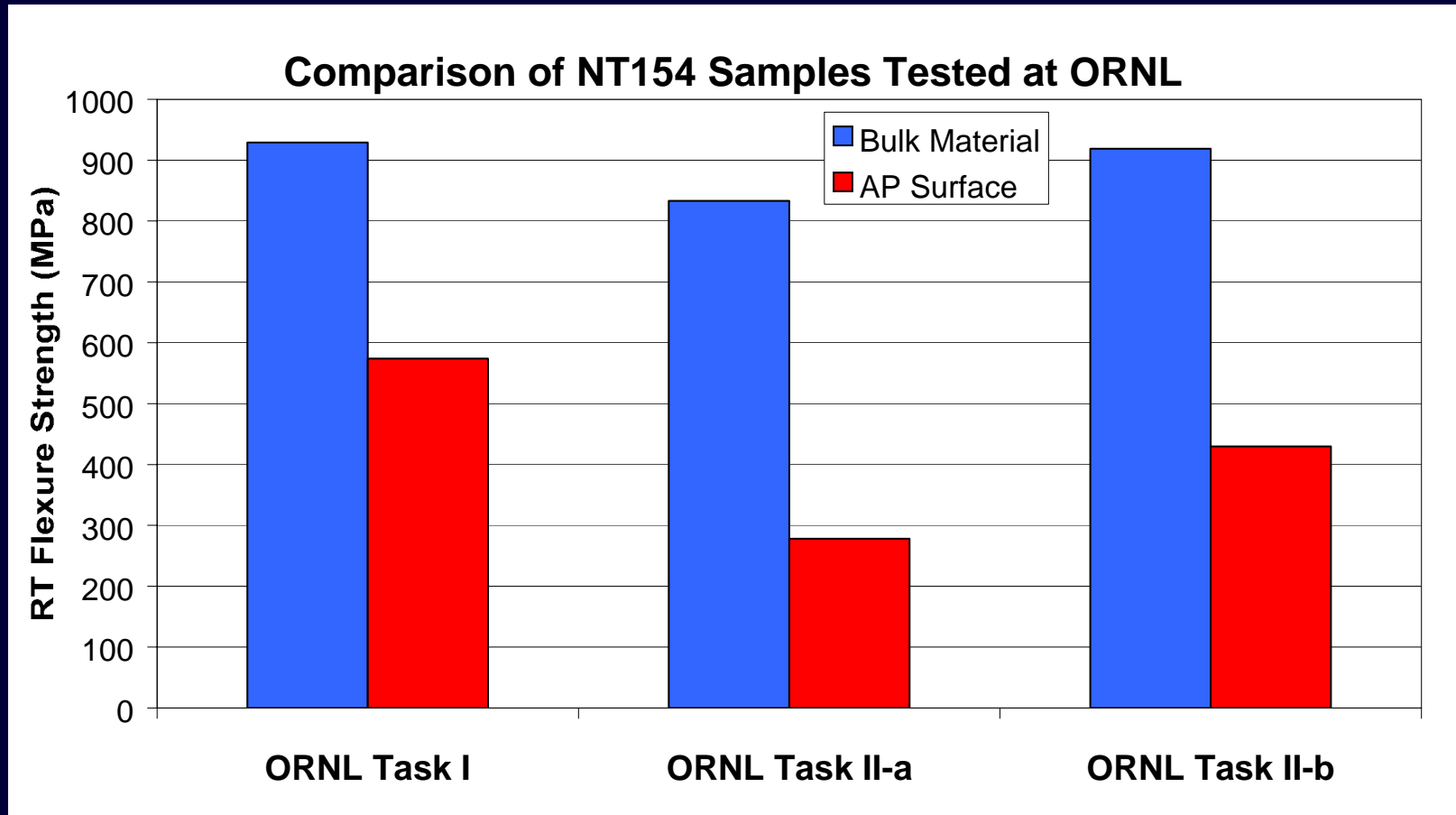
## NT154 Task I

*However, the NT154-11 with As-processed Surface Exhibited High Dynamic Fatigue Exponents up to 1204°C*



# Testing at ORNL

## NT154 Data

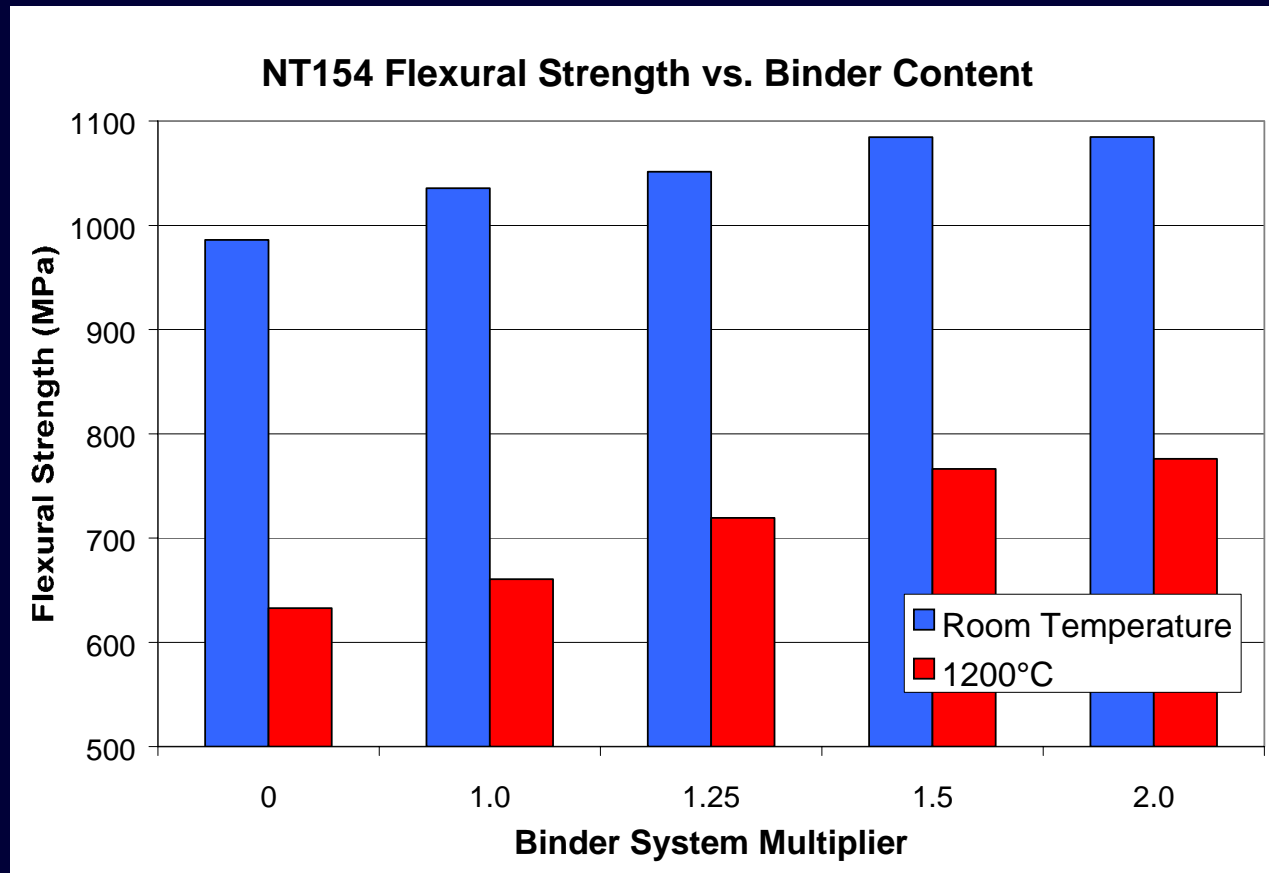


➤ **Continued need to improve as-processed (AP) properties**

# High Temperature Strength

## Influence of Binder Level on Properties

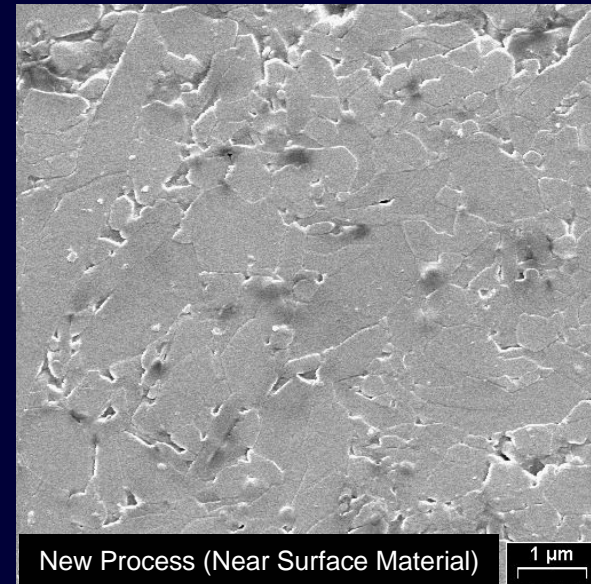
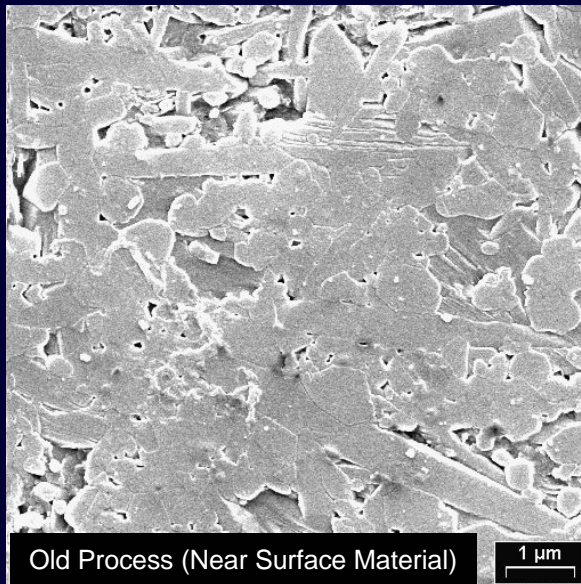
### ➤ Mechanical Properties (Bulk Material)



# As-Processed Surface

## Proprietary New HIP Process

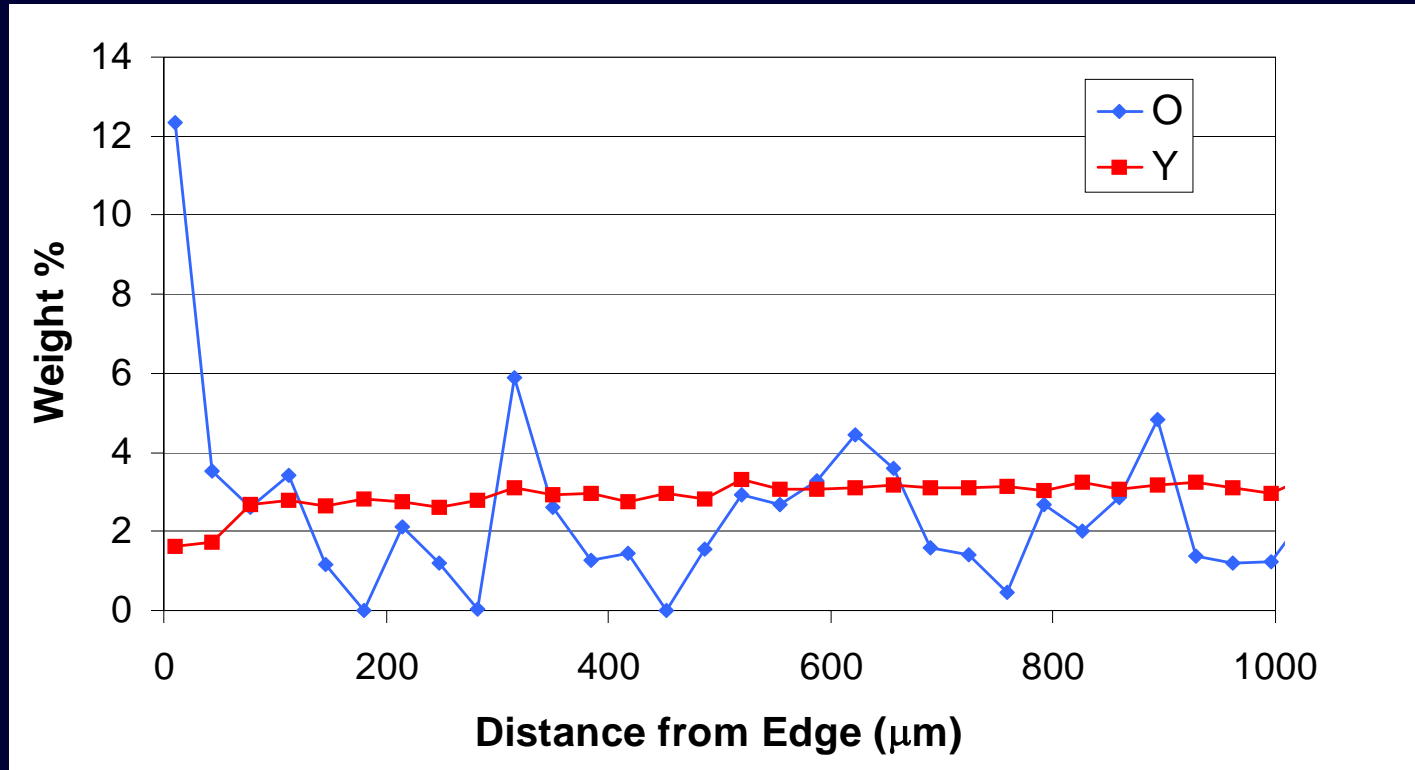
- Looking to reduce the  $\text{Si}_3\text{N}_4$  interaction with the HIP glass
  - Influence on the near surface material
- Initial experiments done in a laboratory scale HIP
- Etched microstructure of the near surface material



# As-Processed Surface

## Proprietary New HIP Process

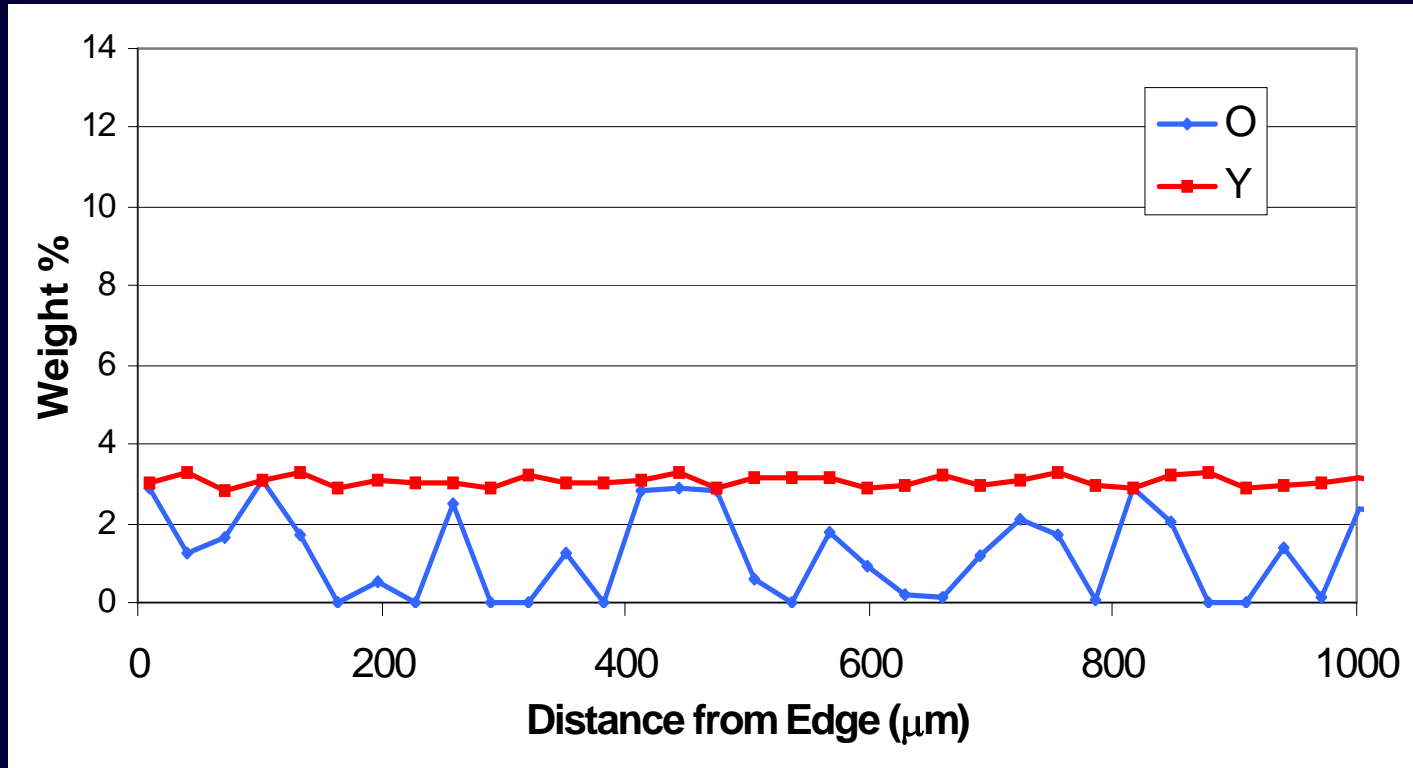
### ➤ Microprobe (Old Process)



# As-Processed Surface

## Proprietary New HIP Process

### ➤ Microprobe (New Process)



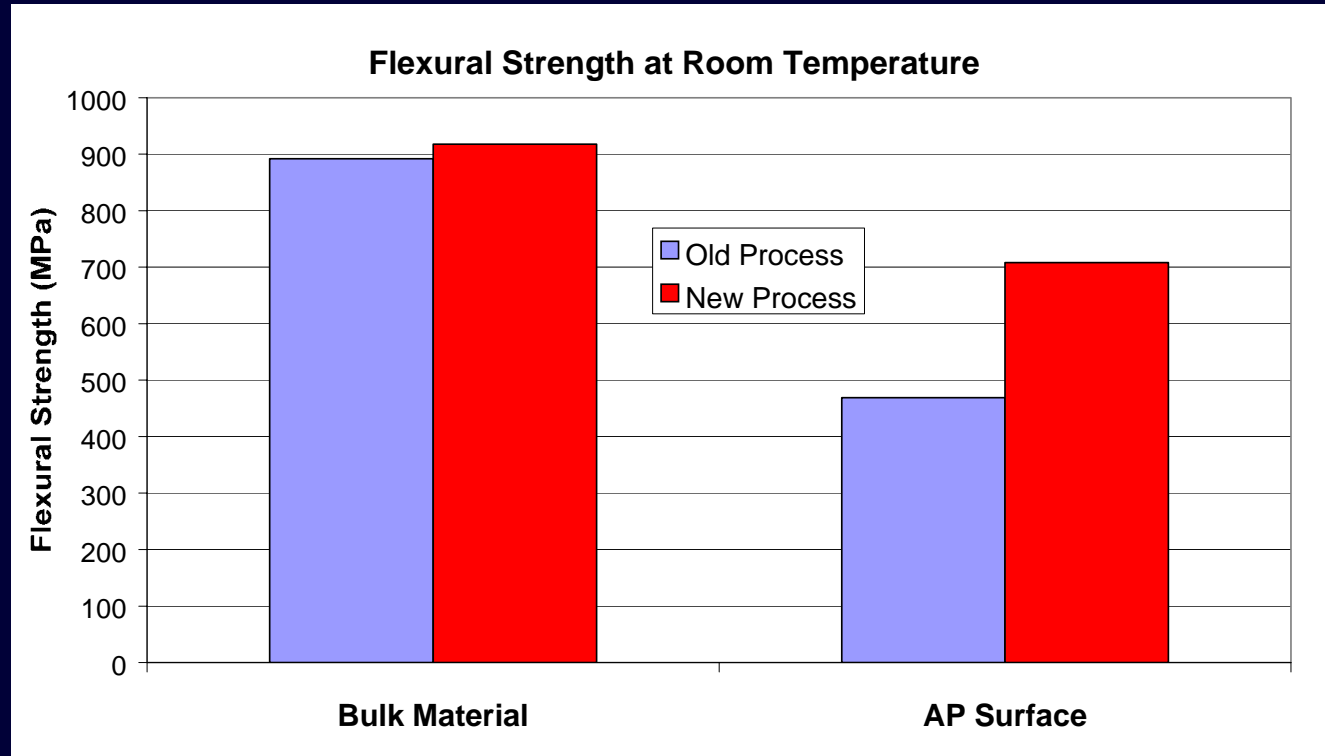


# As-Processed Surface

## Proprietary New HIP Process

➤ Significant improvement in AP strength

- Important for net-shape turbine components



➤ **50% improvement in AP strength!**

# As-Processed Surface

## Conclusions

- **Interaction between NT154 and the HIP glass**
- **New HIP process minimizes interaction with the HIP glass**
- **In process of scaling up new process to production HIP**
  - Initial experiments result in a decrease in AP properties for both the old and new processes
  - The new process still provides up to a 50% improvement compared to the old process
  - Issue appears to be with the near surface microstructure

# Net Shape Forming

## Green Machining

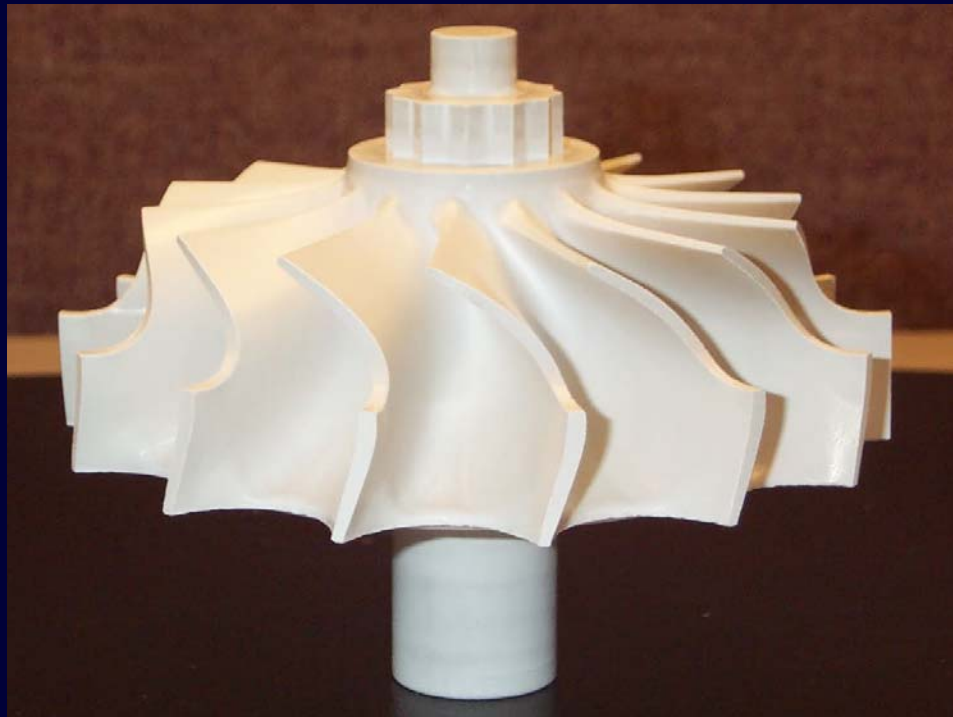


- Developed green-machining process
- Completed two demonstration axial rotors
- Green dimensions within 0.003" of nominal
- Dense surface roughness
  - Hub: 35-40  $\mu\text{in}$
  - Blades: 55-75  $\mu\text{in}$
- Uniform/isotropic shrinkage
- High yield
- Flexible prototyping process

# Net Shape Forming

## Green Machining

- Ingersoll-Rand design
- Machining completed on four prototypes
  - Machining time reduced by ~20%



# Net Shape Forming

## Green Machining

### ➤ Surface roughness on green rotors

- Blades: 25  $\mu\text{in}$
- Bottom face: 30  $\mu\text{in}$

### ➤ CMM on green rotors

- Dimensions within 0.004" of nominal
- Concentricity within 0.002"
- Blade surface locations within 0.003"

### ➤ Rotors to be HIPed

- Use new process for improved AP
- Shrinkage uniformity
- Surface roughness change
- CMM

### ➤ Material property evaluation at ORNL

### ➤ Test in Ingersoll-Rand microturbine?



# Net Shape Forming

## Direct Starch Casting

### ➤ Objective

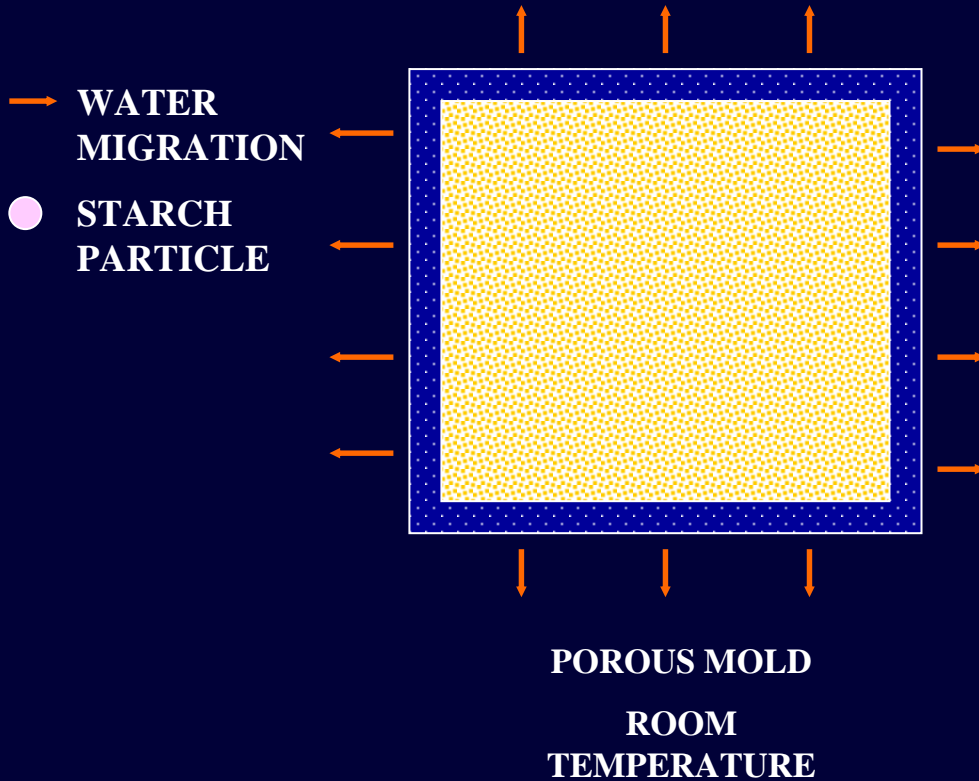
Develop a complex shape forming method using highly loaded starch containing ceramic suspension

### ➤ Optimization

- Improve density and minimize internal porosity
- Increase green strength
- Improve surface finish

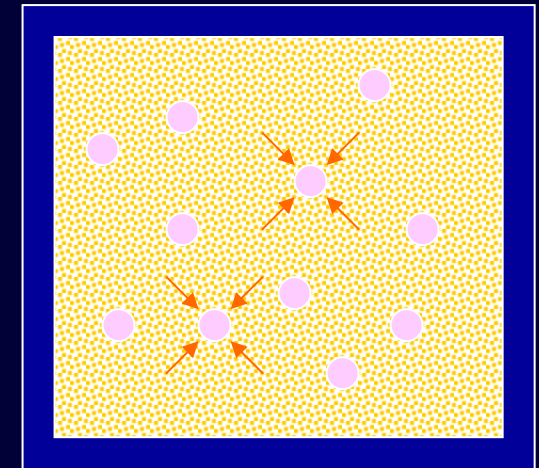
# Net Shape Forming

## Direct Starch Casting



**SLIP CASTING (DRAINED)**

→ DRYING SHRINKAGE



**DIRECT STARCH CASTING (UNDRAINED)**

→ NO DRYING SHRINKAGE

# Net Shape Forming

## Direct Starch Casting

Properties of HIPed Material			
	CIPed NT154	Standard Starch	Starch B
Average Surface Roughness ( $\mu\text{in}$ )	40-50	92	46
Flexural Strength (MPa)	955	791	1002
Weibull Modulus	13	9	17

- Improvements seen with standard starch compared to past results
- Further improvements seen with Starch B
  - However, shrinkage is present
- Surface roughness improvement demonstrated using new AP process
  - 34% decrease for standard starch (not shown in table)
- Further work would study the casting of complex shapes



# Recession Control

## ➤ Keiser Rig testing at ORNL

- Baseline NT154 samples (uncoated) being tested
- Preliminary test conditions
  - 1200°C
  - 3% or 20% H<sub>2</sub>O
  - 10atm total pressure, 1.5atm water vapor pressure
  - Up to 2500 hours

## ➤ Initial HEEPS coating analyzed

- In-situ process to modify surface during HIP
- Y<sub>2</sub>SiO<sub>5</sub> dip-coat
- Occasional cracking is evident by optical microscope
- Spot XRD suggests Y<sub>2</sub>SiO<sub>5</sub> layer no longer exists

## ➤ Initial Ceramatec coatings analyzed

- Proprietary coatings on dense NT154 tiles
- Cracks are evident by SEM
- Further process improvement necessary

# Conclusions

- **NT154 has been re-established**
- **Improvements in high temperature strength**
- **Improved AP strength through new HIP process**
  - Need to reproduce results in production HIP
- **Prototyping of microturbine rotors through green machining**
- **Improvements seen in starch casting**
  - Properties comparable to baseline NT154
- **Key microturbine OEM's contacted**
- **Three invention disclosures filed**

# Microturbine Plan For FY2004

## ➤ Phase II - Develop Novel Recession Control Technique

- ORNL program continuation to develop an innovative protective layer
- “HEEPS” (*In situ*) and EBC approaches

## ➤ Continue Material and Forming Advancements

- Continue to evaluate AP properties
- Continue to evaluate green-machining approach
- Direct casting improvements

## ➤ Prototypes in Support of OEMs

- Mechanical testing of a component at ORNL
- Plans to work with Microturbine manufacturers
- Cost analysis



NT154 Radial Microturbine Rotor  
Ingersoll-Rand Design