

# Hot Section Materials Development For Advanced Microturbines

**Environmental Barrier Coatings Workshop**  
**November 6-7, 2002**  
**Nashville, Tennessee**



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Northboro, MA 01532*

  
**SAINT-GOBAIN**  

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**CERAMICS & PLASTICS**

# Outline

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- Perspective
- Objective
- Technology Development
- Summary

# OPT DER Advanced Microturbine Systems

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Small combustion turbines, 25 kW to 500 kW  
(some say 1 MW)

## Goals:

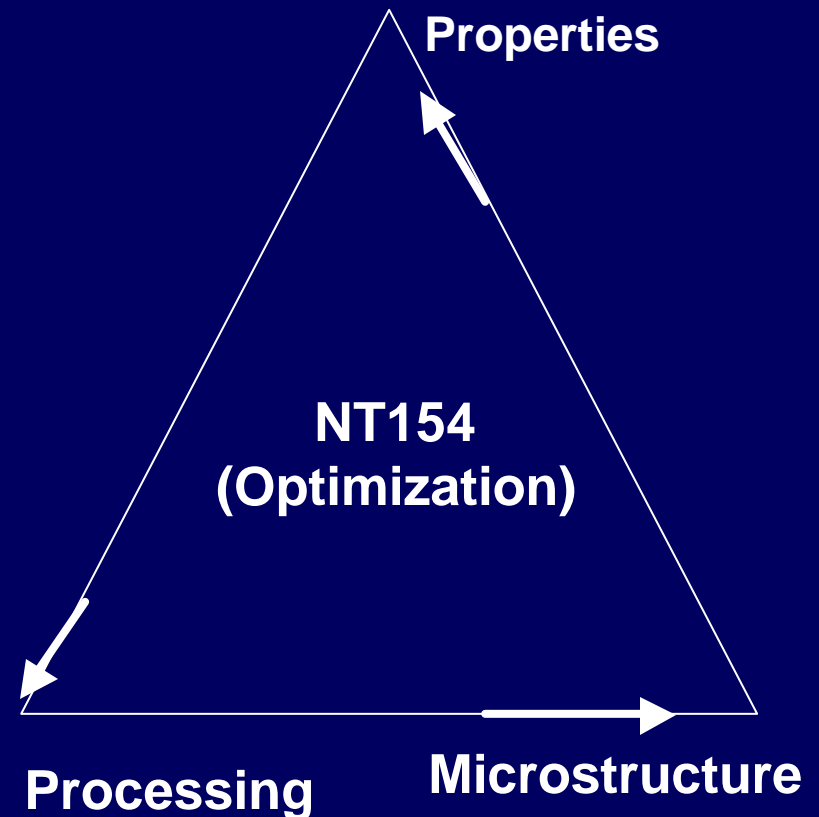
- Increase efficiency to  $> 40\%$
- Enabling technology: ceramics and EBC
- Less than 7 ppm NO<sub>x</sub>
- Durability -- 11,000 hours bet major overhaul, 45,000 hour service life
- Cost of Power, \$500/kW (now ~\$1,000)
- Fuel Flexible
- DOE Funding \$60 M FY 2000 - 2006

# Objective

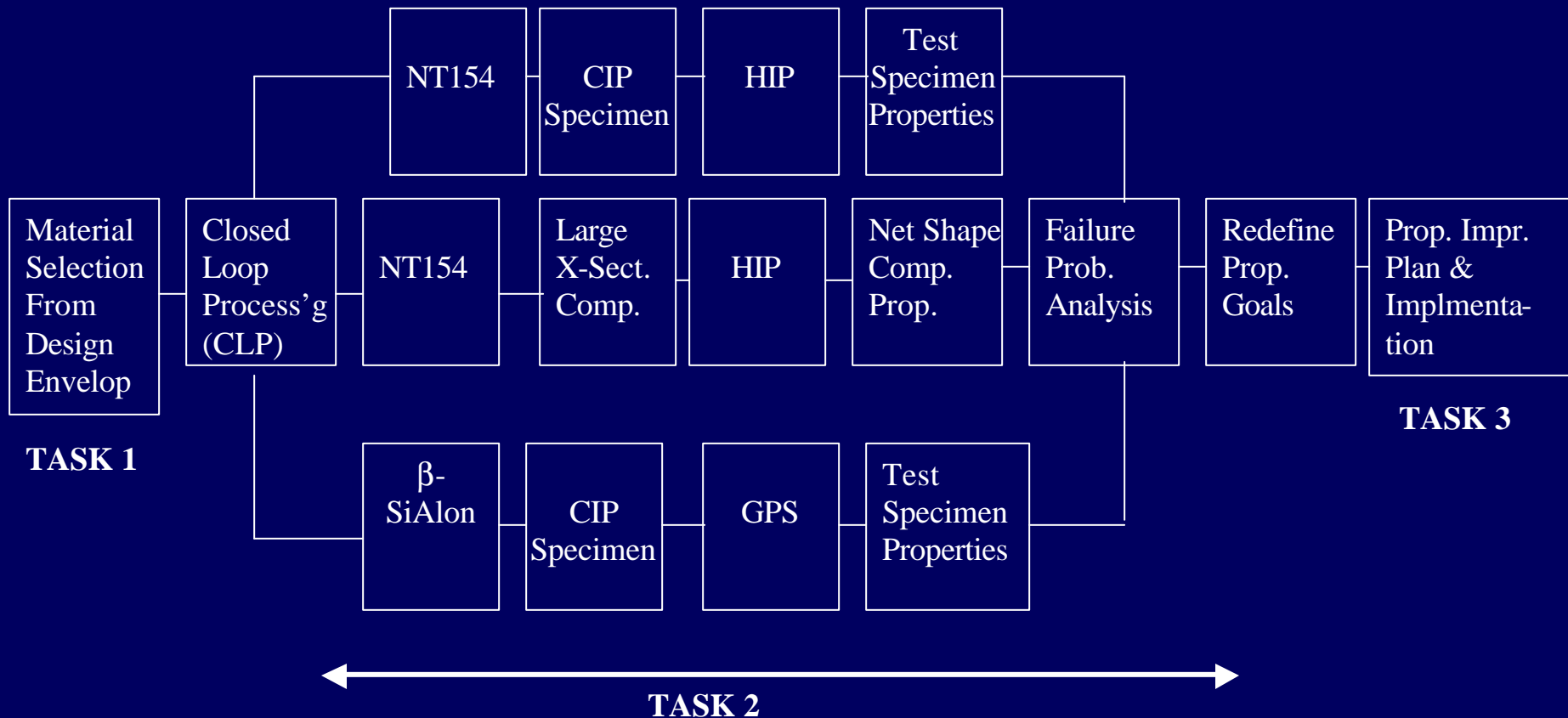
Develop and optimize a high temperature ceramic material and process suitable for microturbine applications up to 1300°C.

## Specific Properties

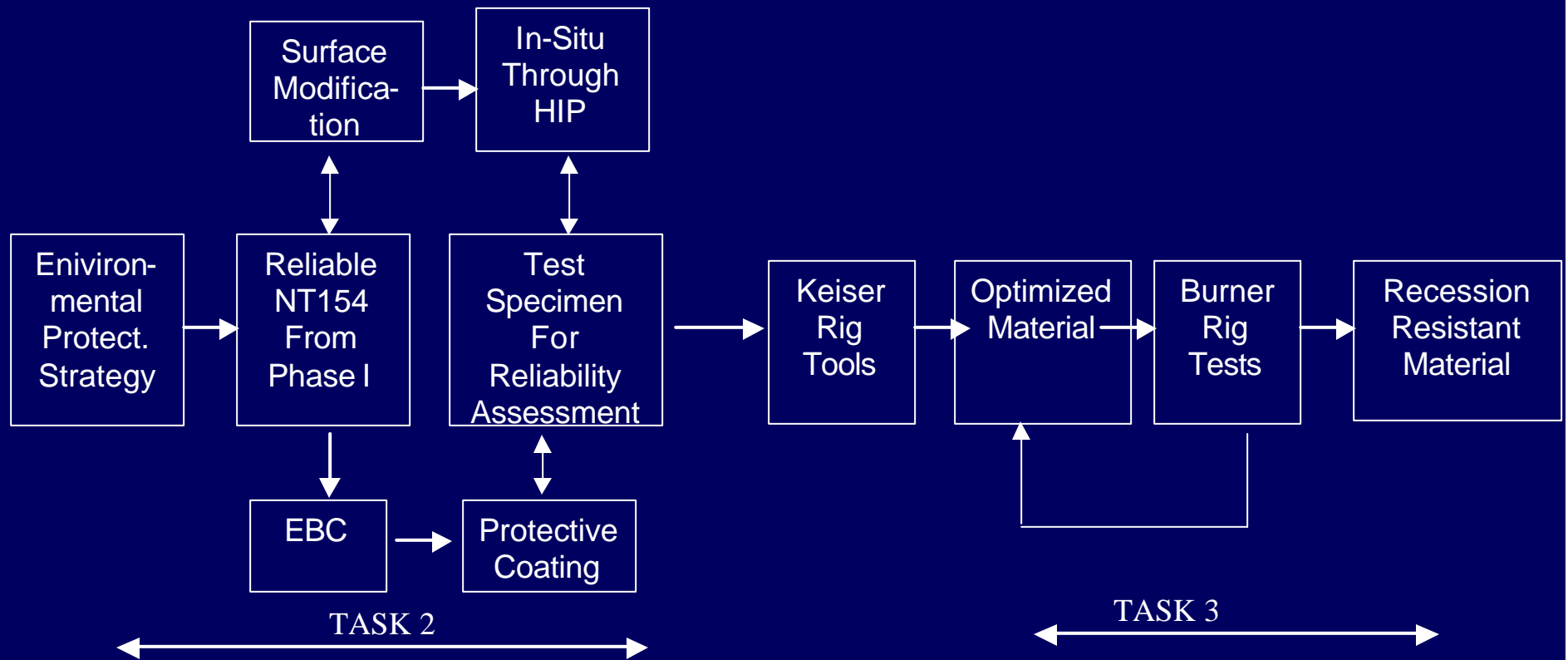
- **Fast Fracture**
  - RT –  $\sigma \geq 950$  MPa
  - 1300°C  $\sigma \geq 600$  MPa
- **Fracture Toughness**  $\approx 6.5$  MPa $\sqrt{\text{m}}$
- **Weibull Modulus**  $\approx 12$
- **High Temperature Creep Rate**
  - »  $1.9 \times 10^{-8}$  @ 1250°C/130 MPa
- **Oxidation Resistance up to 1250°C**
- **Recession Resistance in humid environment up to 1250°C**



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



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



# Ceramic Microturbine Technology

## Ceramic Microturbine Technology

### Material Development

- Re-establish NT154/NT164
- b-SiAlON Development

### Net Shape Forming Development

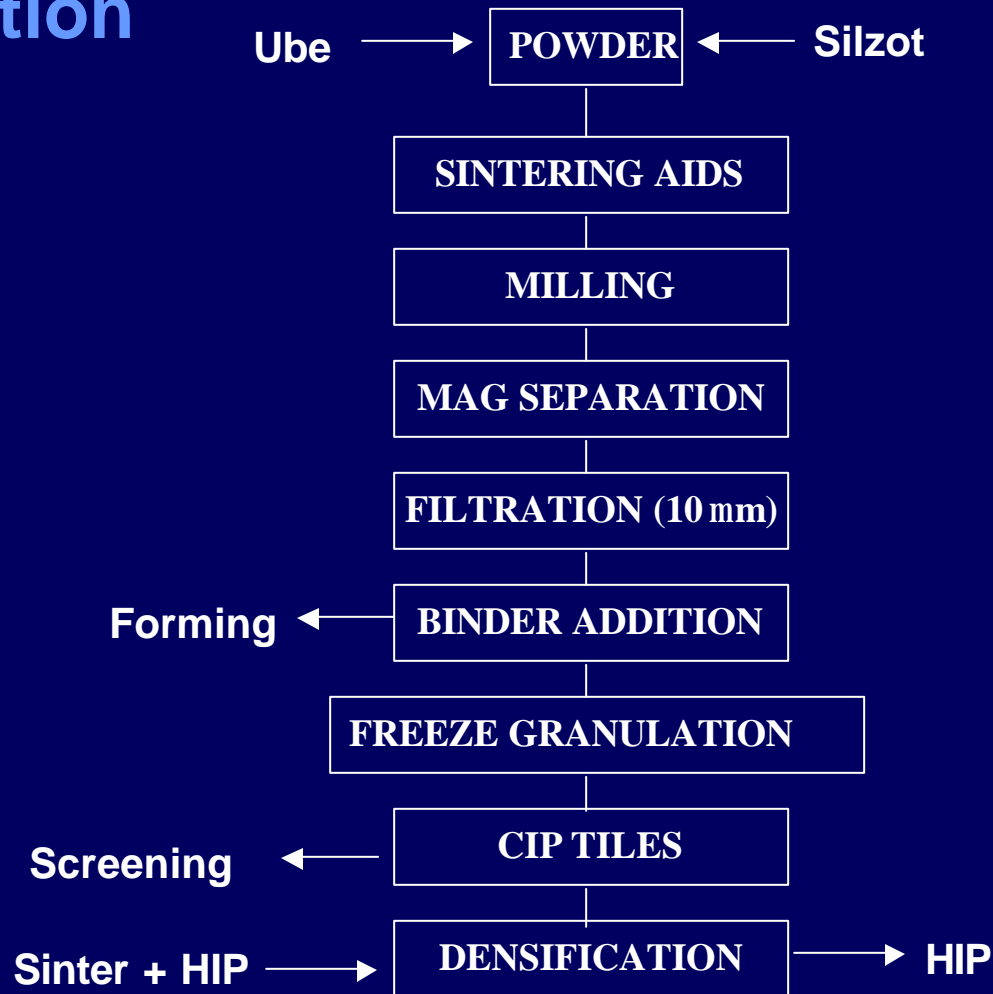
- Green CNC Machining
- Direct Casting

# Material Screening/Selection

## COMPOSITIONS

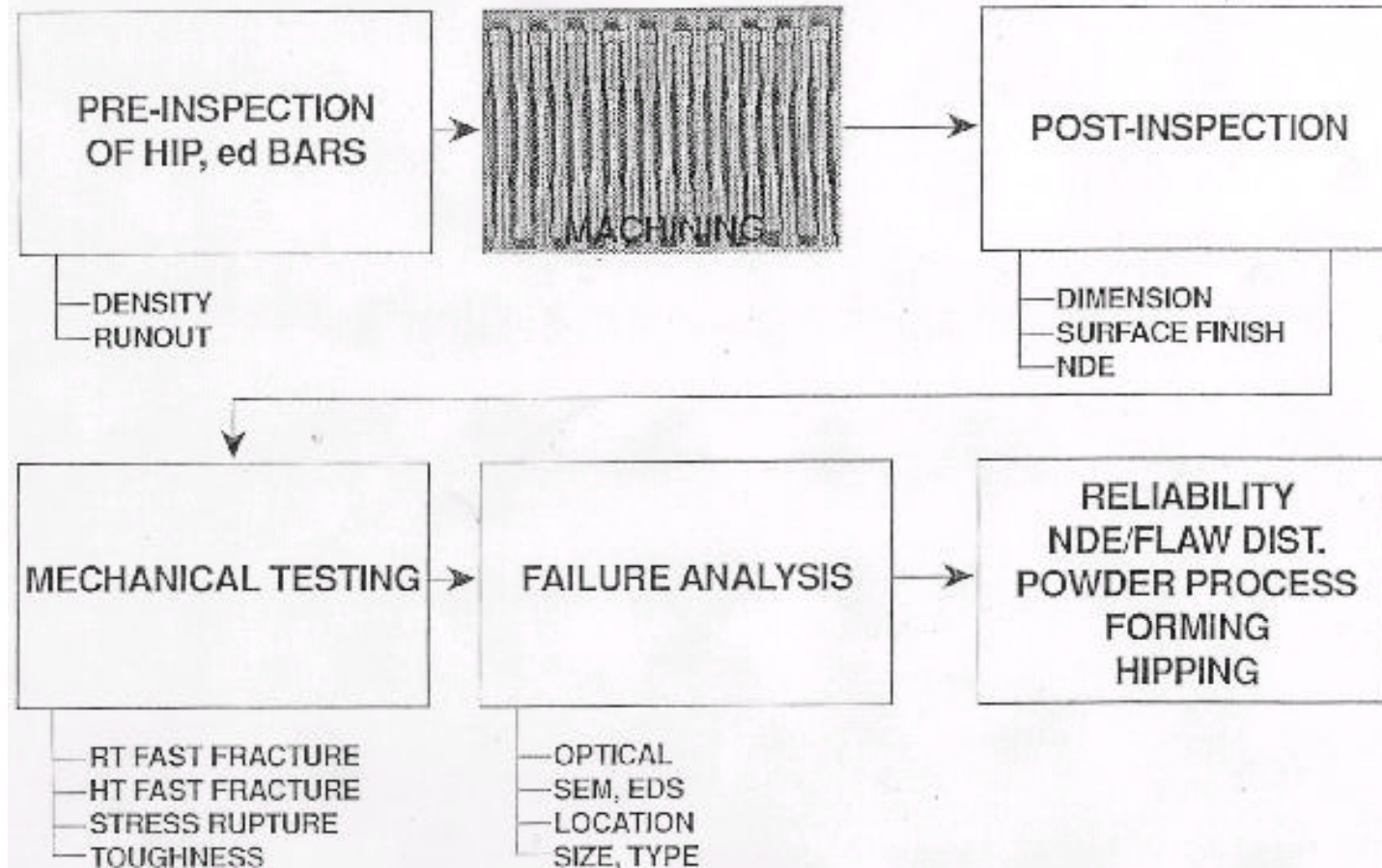
- NT154
- NT164
- b- SiAlON

## CLOSED LOOP PROCESSING



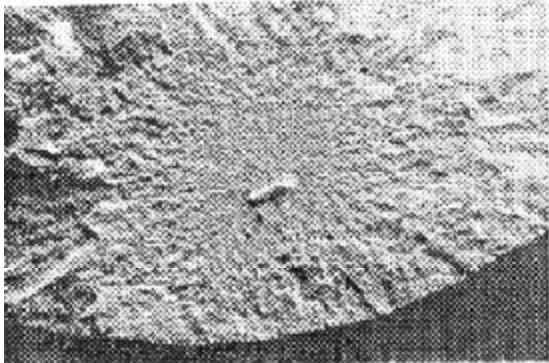


# Testing and Failure Analysis

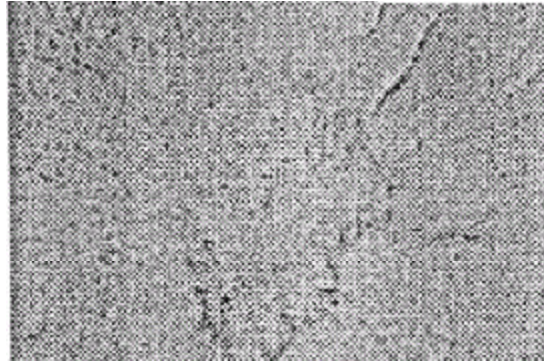


# Chronology of Process Improvement

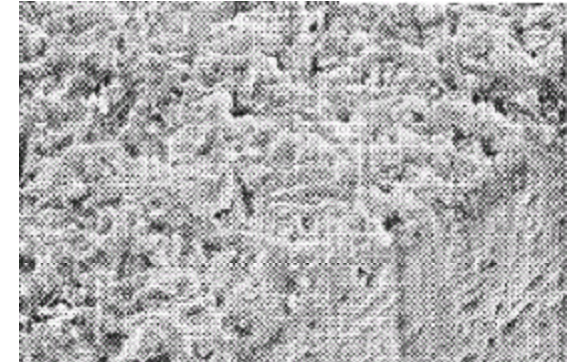
## Silicon Nitride Net Shape Formed Buttonhead Tensile Test Bars



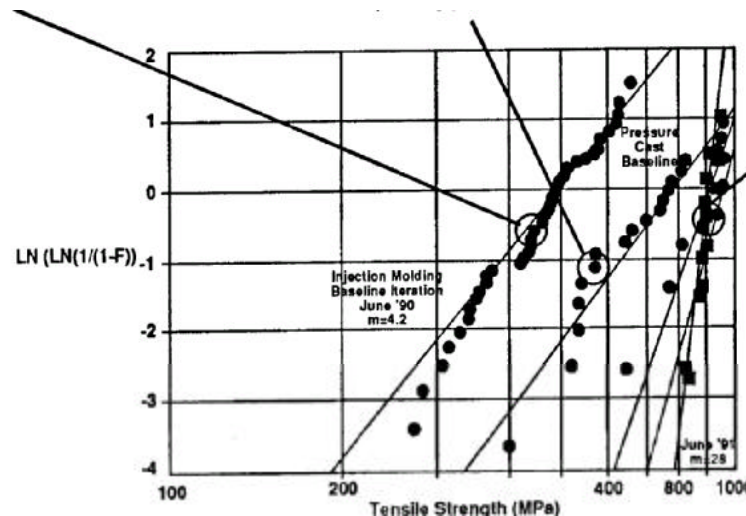
Injection molded tensile bar failed at 444 MPa due to 200mm metallic inclusion



Fracture surface of pressure cast Tensile bar which fractured at 570 MPa from 65mm agglomerate

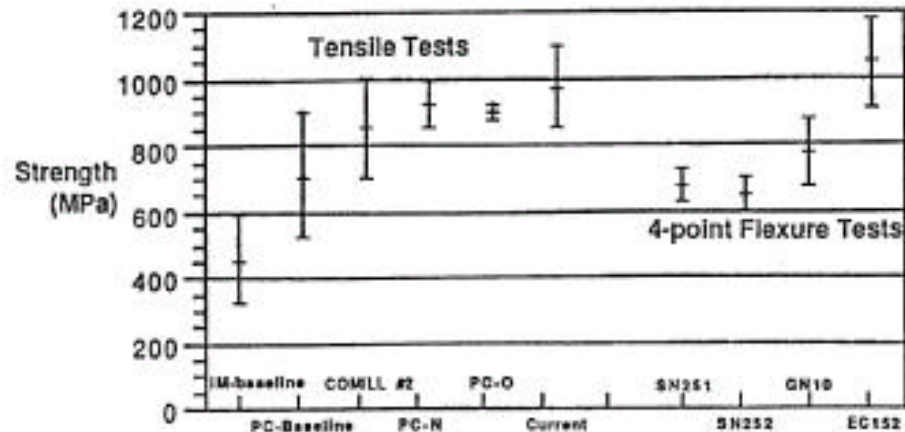


Failure origin at surface of 884 MPa Strength tensile bar centered about a 5mm wide machining groove.

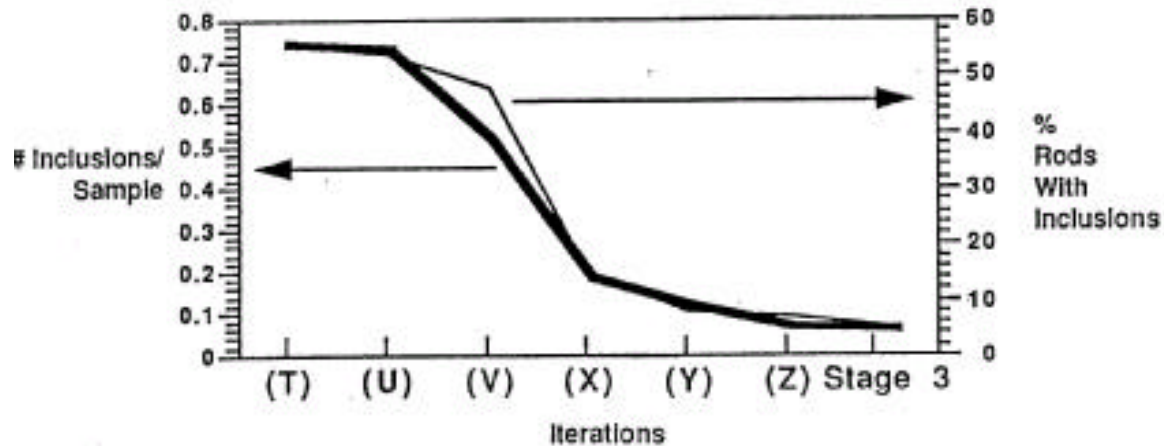


Tensile Strength data illustrating Improvements in materials processing and reliability.

# Chronology of Process Improvements

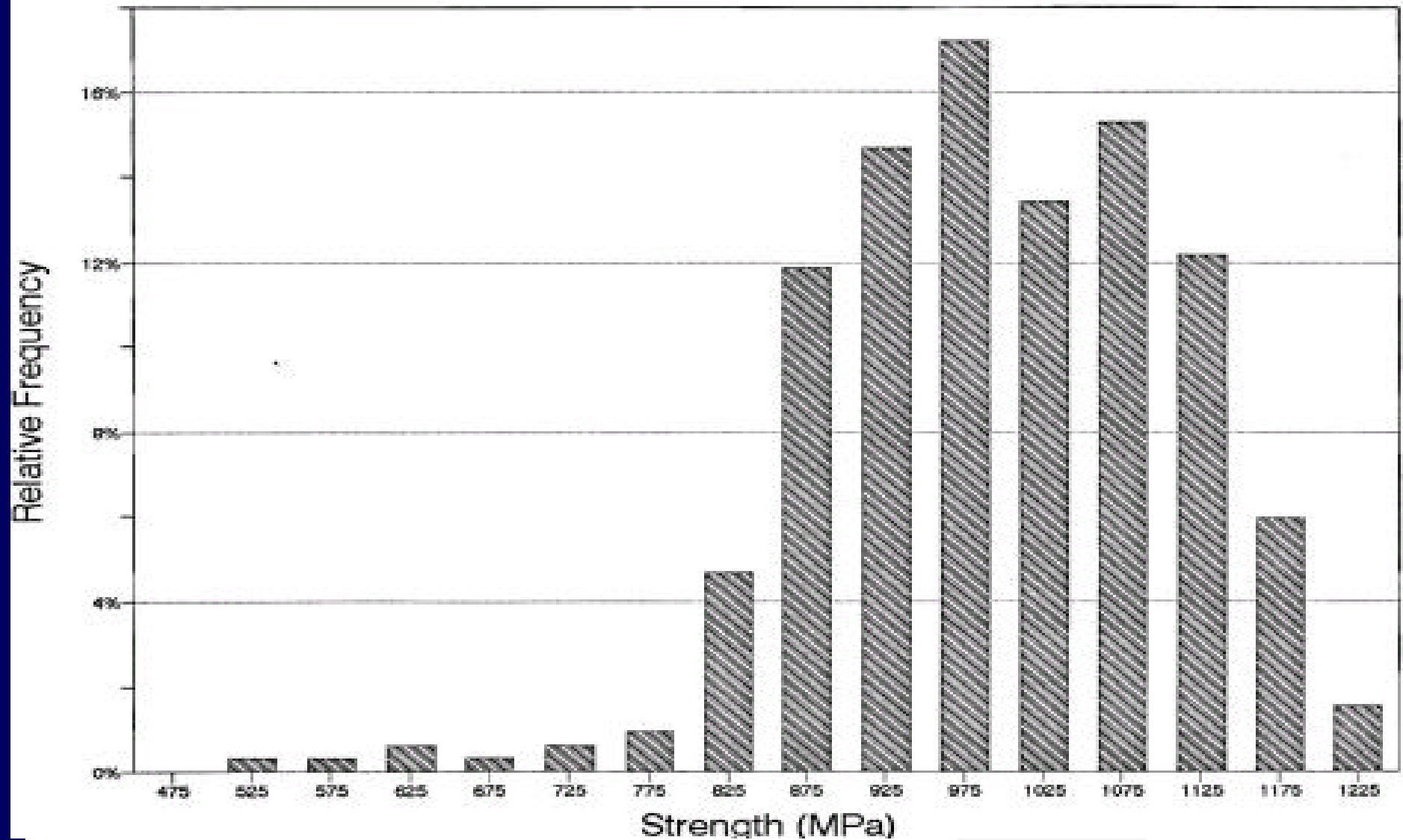


a) Tensile Strength Improvement - NCX5102

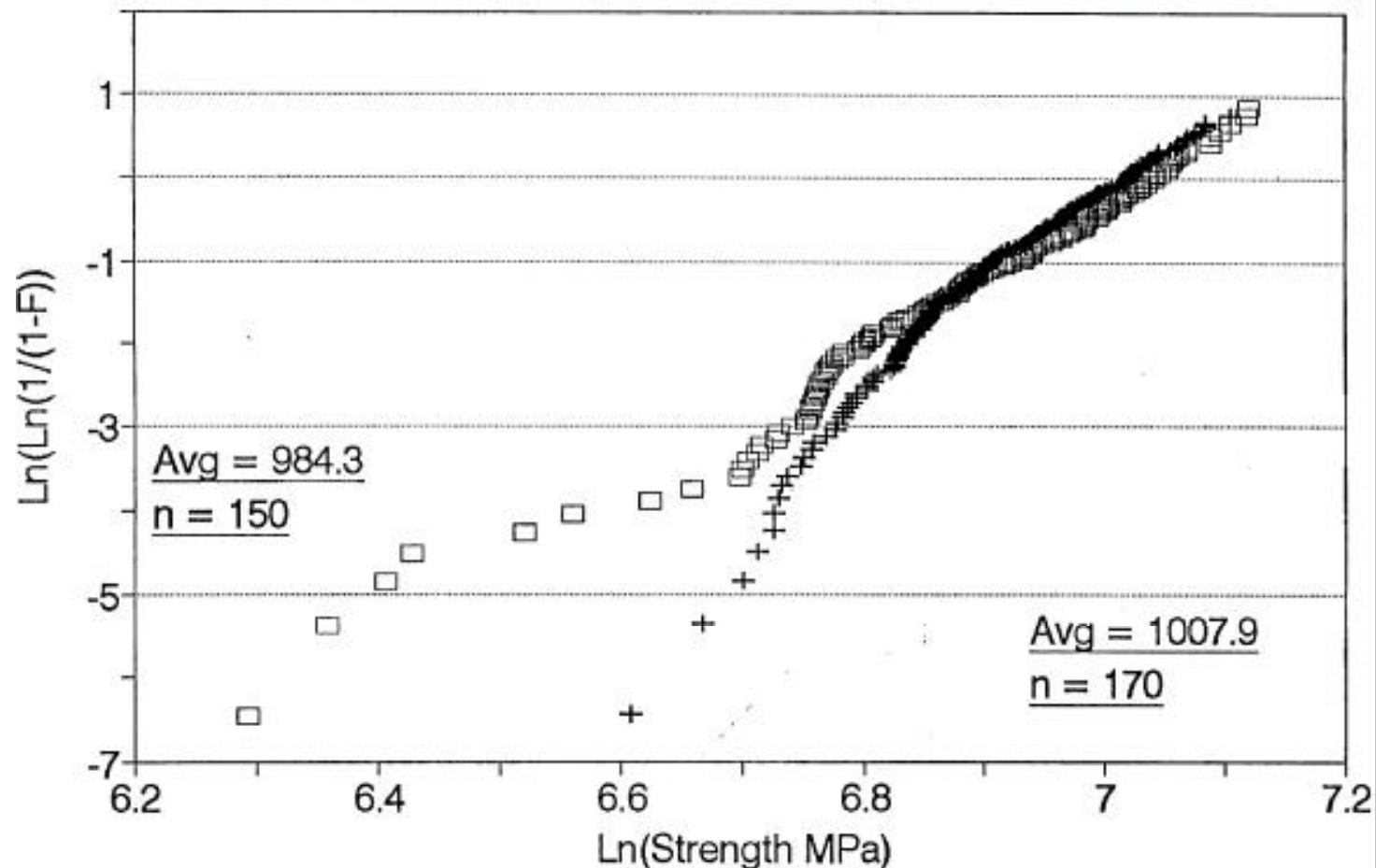


b) Flaw Reduction

# Frequency of Failures vs. Strength

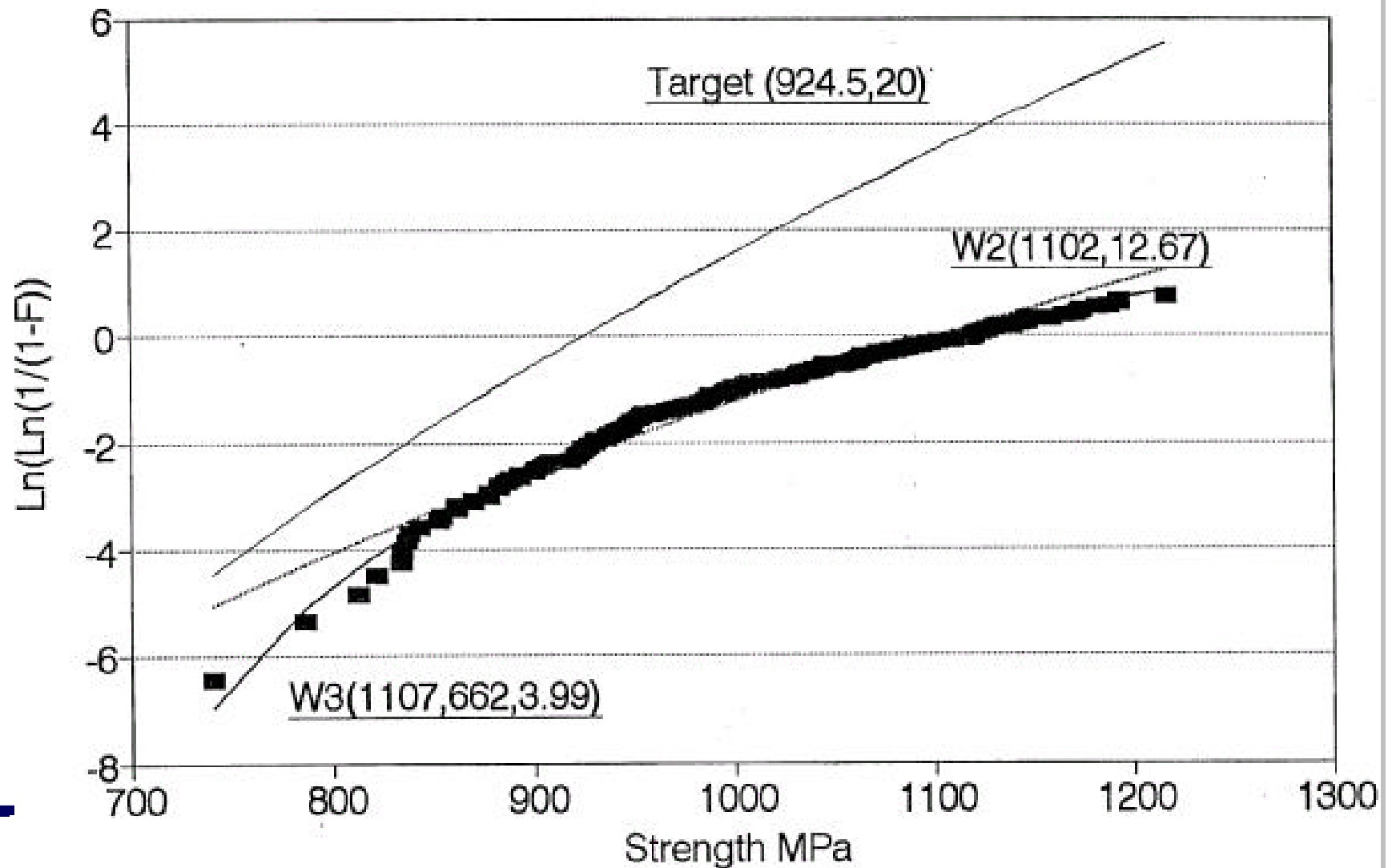


# Competing risk Weibull Analysis Ti & Machining Damage vs Rest.



# Competing-risk Weibull Analysis

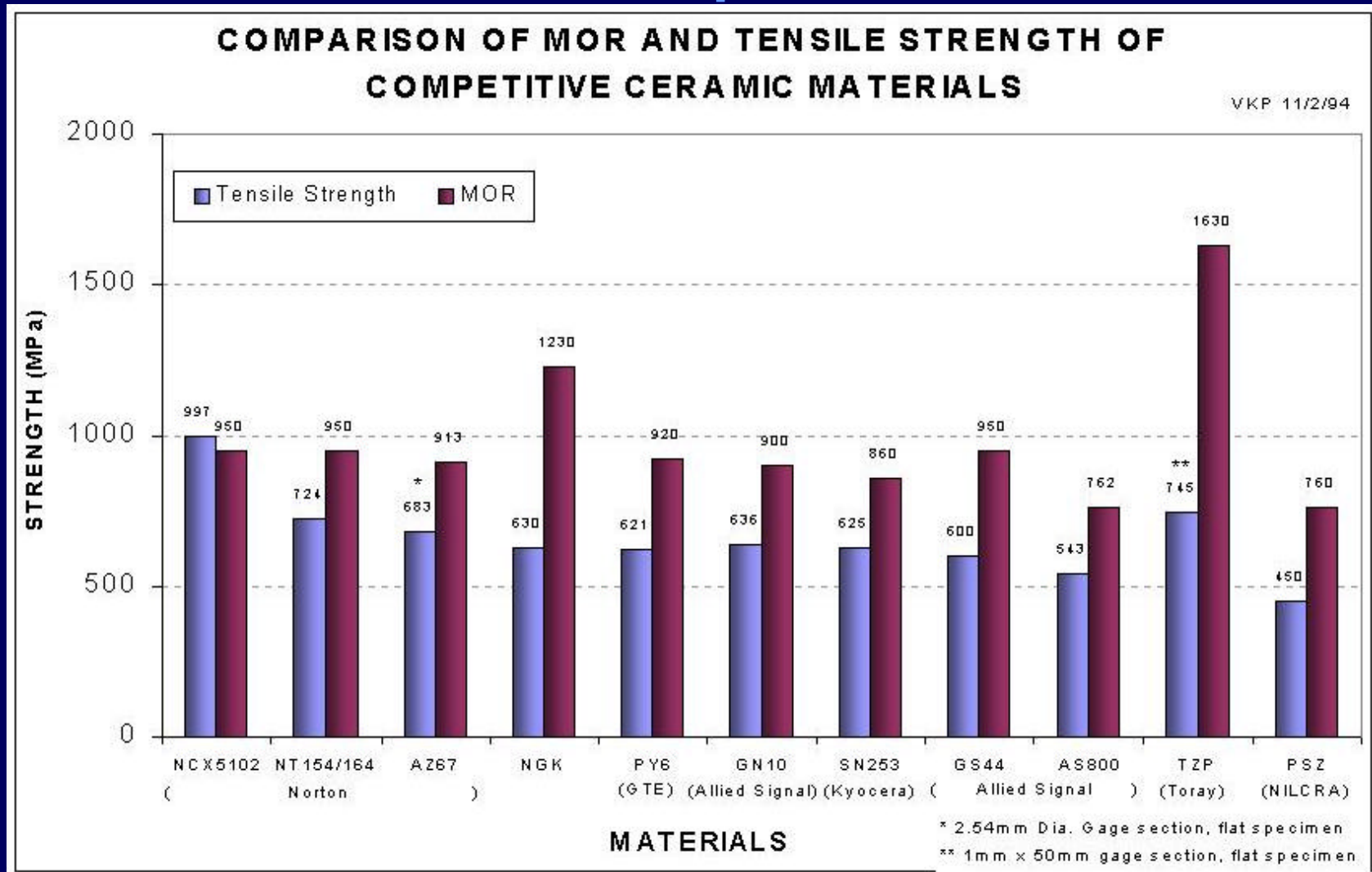
Target, Weibull 2p and 3p fit to data.



# Closed Loop Qualification Flexure Testing

CLM#	R.T. FLEX. Avg. (PSI)	H.T. FLEX. Avg. (PSI)	A.P. R.T. Avg. (PSI)	$K_{IC}$ MPa X M 1/2
X01	157,217	104,428	57,350	6.25
X02	161,110	109,253	84,970	6.33
X03	156,042	103,587	68,300	6.32
X04	154,974	92,748	51,110	6.46
X05	147,785	110,272	56,580	7.11
X06	147,350	107,609		6.91
X07	167,895	104,690	80,240	6.42
X08	152,136	106,646	94,073	5.76

# Comparison of MOR and Tensile Strength Database for Competitive Materials





# GREEN/SHAPE FORMING

## Green Machining

### Colloidal Isopressing (CI)

MILLING  
(Tri-Modal)

FILTER/  
MAG. SEP.

COAGULATION

FILTER  
PRESS

CI  
BLANK

### Direct CIP

MILLING  
(Tri-Modal)

FILTER/  
MAG. SEP.

BINDERIZE

SPRAY  
DRY

CIP  
BLANK

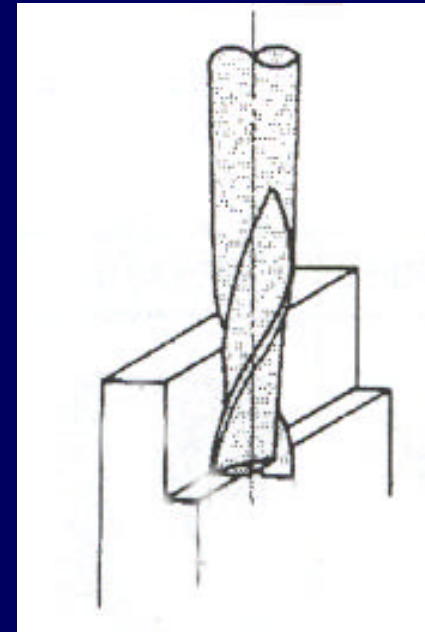
Green Machine  
5 Axis CNC

# Test Setup



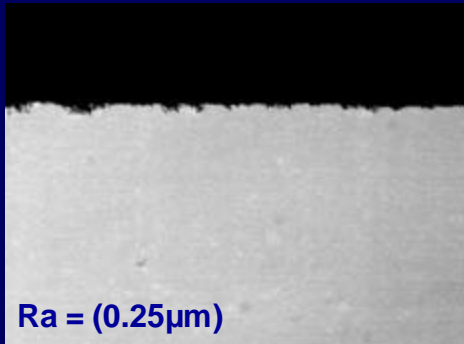
# Test Conditions

- 4-Fluted end milling tools
  - High Speed Steel
  - Carbide
  - Carbide coated with TiN
  - Carbide coated with thin diamond film
- Surface speed: 120 m/min
- Table speed: 0.127 m/min
- Depth of cut: 0.6mm (rough)  
0.2mm (finish)
- Length of cut: 25mm

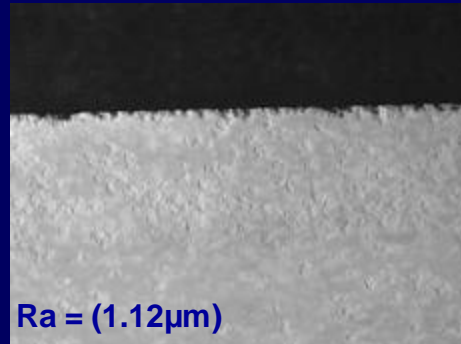


# Machined Surface (NT154-6B )

New

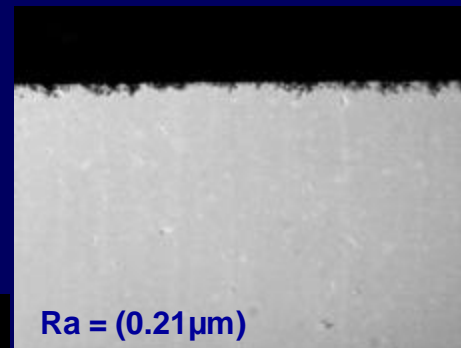


After 1.8m

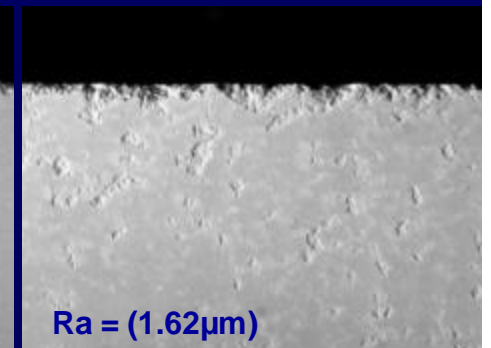


Carbide

New

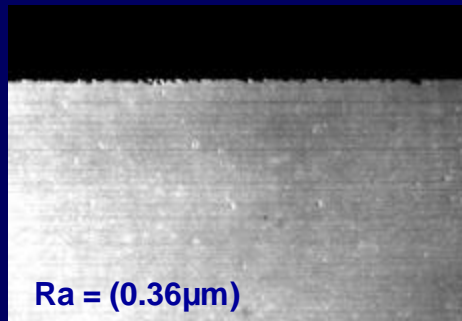


After 1.8m

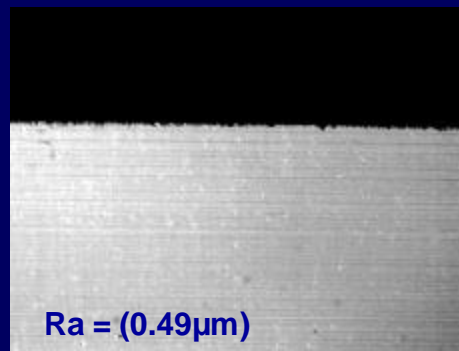


TiN Coated

New



After 1.8m



Diamond Coated

- Green Machining Optimized
- CNC Machining Trial of Rotor

# Direct (Starch) Casting

Develop a high solids shear thinning aqueous suspension

- Solids loading (52-58 v%)
- Pourable (shear thinning,  $<1$  Pa/s @ shear rate 10/5)
- De-airable (no trapped air bubbles)
- Adequate wet green strength (1-2 MPa)
- Minimal shrinkable after drying ( $<2\%$ )

# Direct (Starch) Casting

- **Net shape mold fabrication**
  - a) Pattern
  - b) Net shape mold fabrication with RTV
- **Slurry De-airing at 25 Hg. Of vacuum**
- **Casting**
  - a) Mold Filling by pouring
  - b) Mold Filling under pressure
- **Automated Mold Set Up**

## Direct Casting in RTV Mold



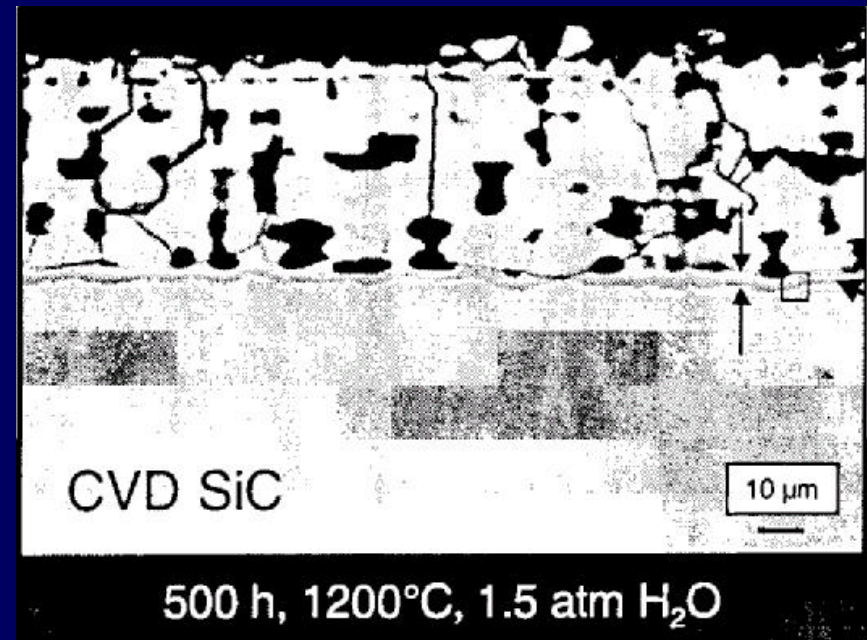
## Phase II - Objective:

Investigate various approaches to recession resistance improvements:

### APPROACHES

- Composition Adjustment
- Surface Engineering
- Environmental barrier coatings (EBC)

### Recession of Silicon Nitride



# Phase II: Recession Control

## *Material Selection*

### Criteria

- Low oxygen permeability
- CTE match with NT154
- Low Young's Modulus
- Good temperature stability
- Does not sacrifice fast fracture and creep properties
- Low partial pressure for high temperature Si gases species for silicate based solutions (ex.  $\text{Si(OH)}_4$ ,  $\text{SiO}$ )
  - **$\text{SiO}_2$  (high tridymite),  $P_{\text{SiO}} = 1.29\text{E-}10$  ATM**
  - **$\text{Al}_6\text{Si}_2\text{O}_{13}$ ,  $P_{\text{SiO}} = 1.93\text{E-}14$  ATM**

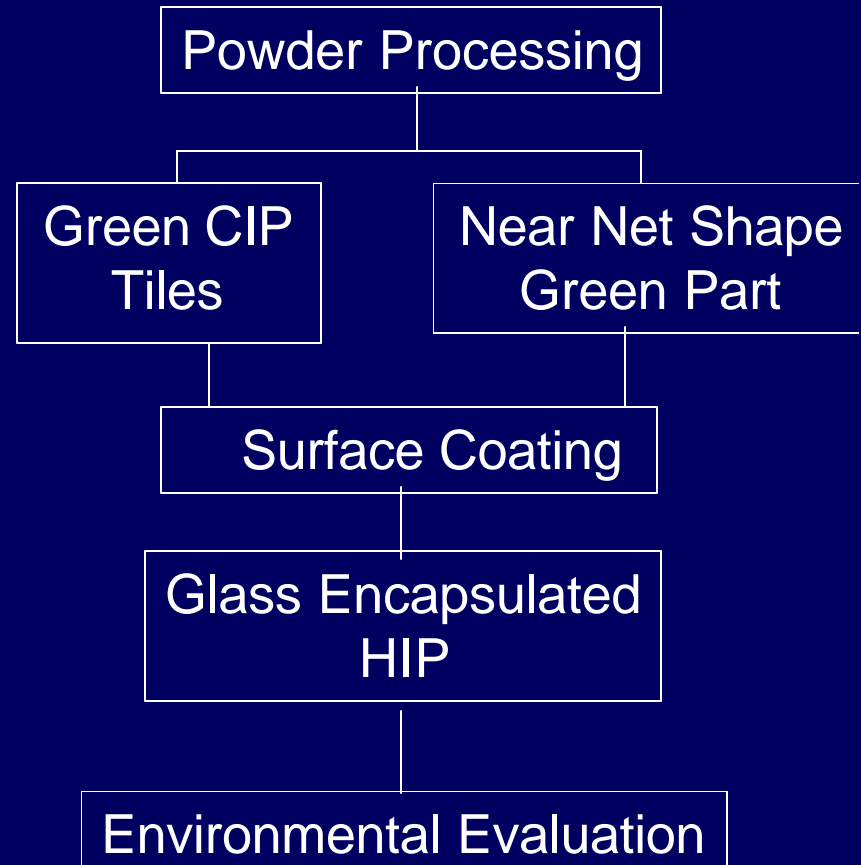


# Phase II – Recession Control

## Surface Coating

- Reactivity with Substrate
- Oxidation Resistance
- Coefficient of Thermal Expansion vs Substrate
- Influence on Fast Fracture and Creep Properties

## Surface Engineering



# Summary

- **NT154 process established and sample delivered to ORNL.**
- **Material development plan includes:**
  - a. Test tiles
  - b. Test coupons from net shape formed components
- **Recession control strategy involved surface modification**