Evaluation of Mechanical Reliability of Si_3N_4 Nozzles After Exposure in an Industrial Gas Turbine

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7th International Symposium on Ceramic Materials and Components for Engines

Goslar, Germany, June 19-21, 2000

Research sponsored by the U.S. Department of Energy, Office of Power Technologies, Microturbine Technologies Program, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC

Presentation Outline

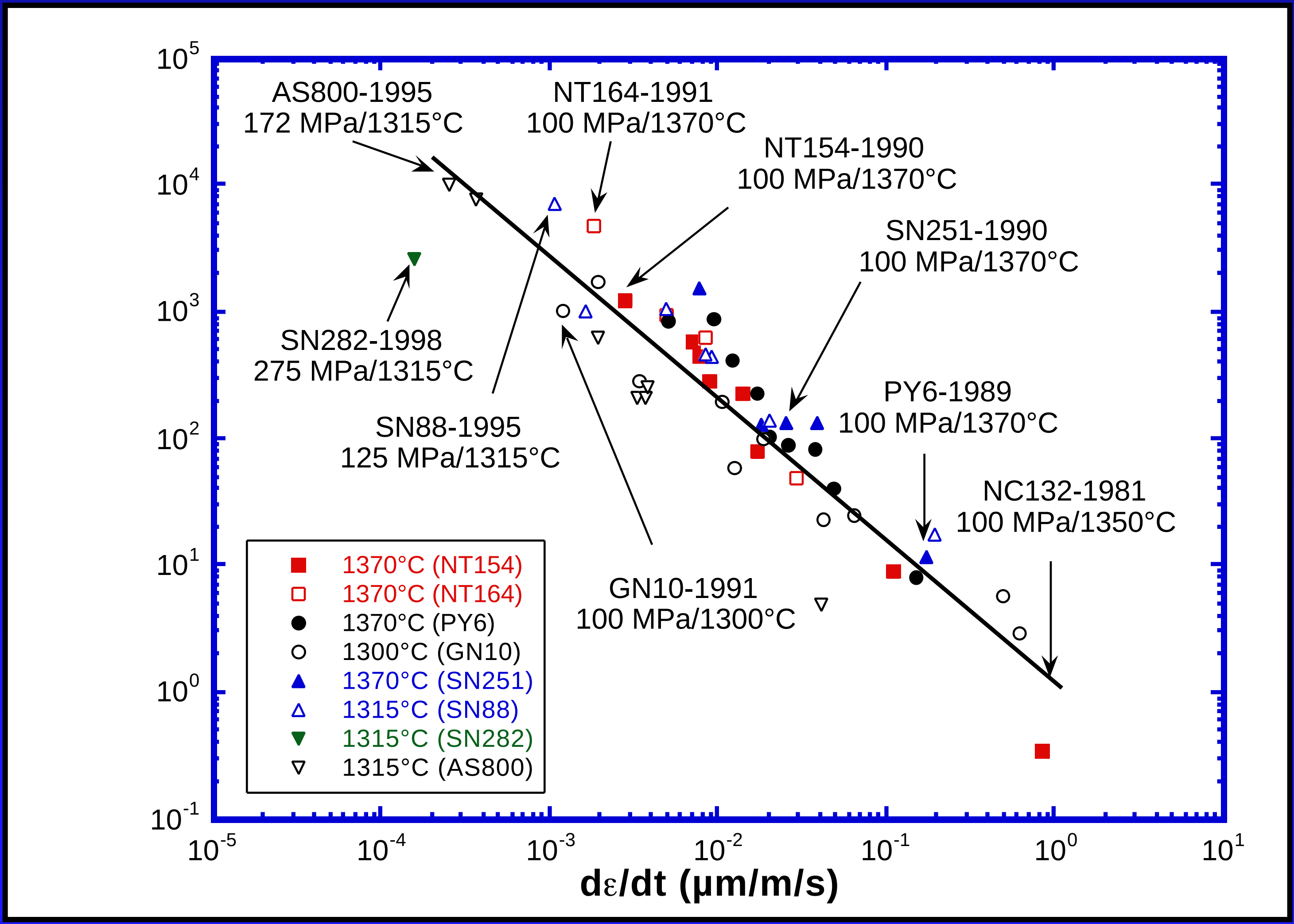
- Background
  Motivation of Present Research Effort

- Case Study of Solar Turbines Ceramic Turbine Components
  - Microstructure characterization
  - Mechanical property evaluation
  - Hypothesis of findings

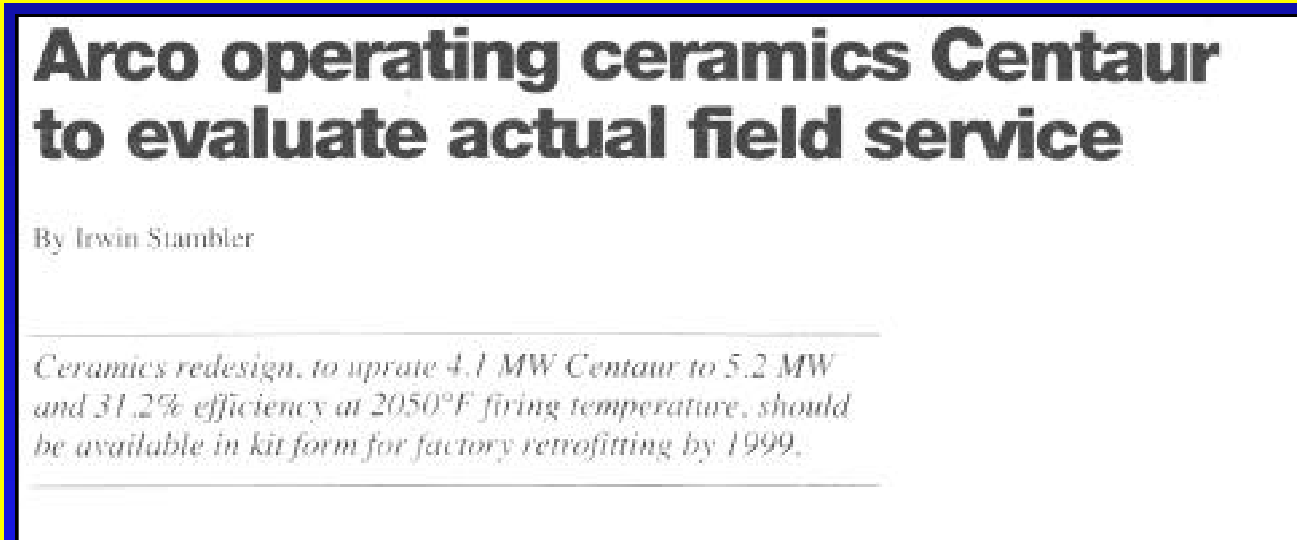
- Mechanical Testing to support hypothesis
  - Dynamic fatigue test
  - Stress rupture test
  - Microstructural analysis

- Summary

High-Temperature Mechanical Performance and Reliability of Si_3N_4 Ceramics Significantly Improved



Solar Turbines Efforts in DOE Ceramic Stationary Gas Turbine (CSGT) Program



Arco operating ceramics Centaur to evaluate actual field service

By Irwin Stambler

Ceramics redesign, to uprate 4.1 MW Centaur to 5.2 MW and 31.2% efficiency at 2050°F firing temperature, should be available in kit form for factory retrofitting by 1999.

Earlier this year, in May, Arco Western Energy started in-service field tests of a Solar gas turbine fitted with ceramic hot section components. The test project, with over 800 operating hours as of June 25th, is located at Arco's oil field site near Bakersfield, California.

Test engine is a Centaur 50S operating on natural gas fuel. This is a stan-

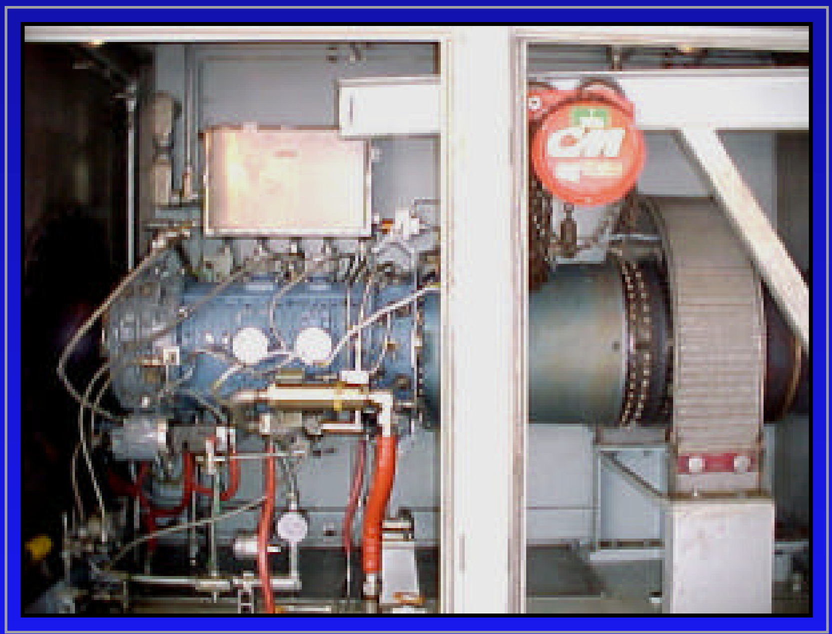
1120°C (2050°F) for ISO base output increase to 5217 kW and over 5.5% points increase in thermal efficiency. To run at this rating for 4000 hours to prove durability.

The Arco project is a key milestone in the U.S. Dept. of Energy supported Ceramic Stationary Gas Turbine program. CSGT goal is to develop and

1st stage turbine blade

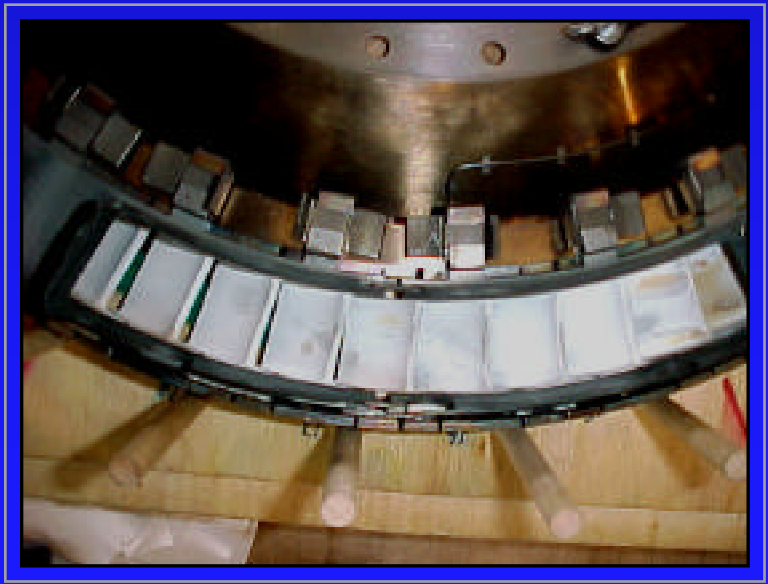
1st stage turbine nozzle

Rolls-Royce Allison Efforts in DOE Ceramic Stationary Gas Turbine (CSGT) Program



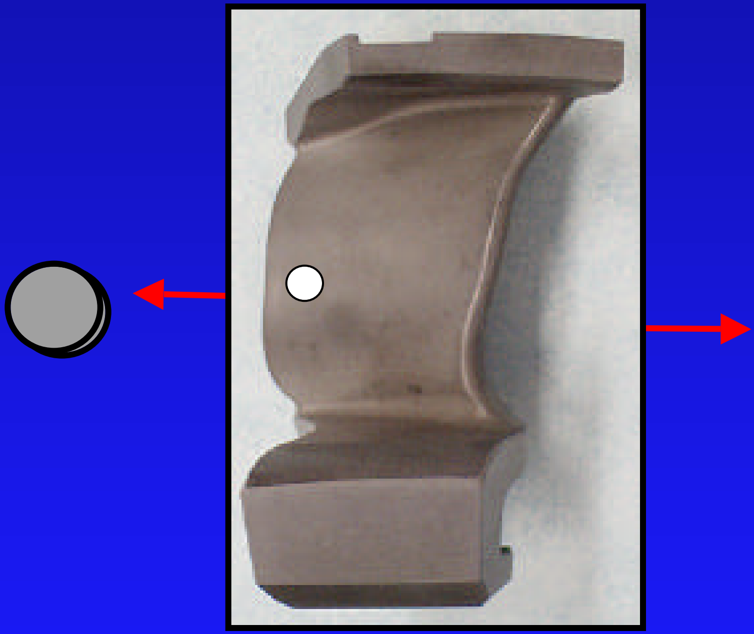
501-KB5 Turbine Engine

1st stage turbine vane

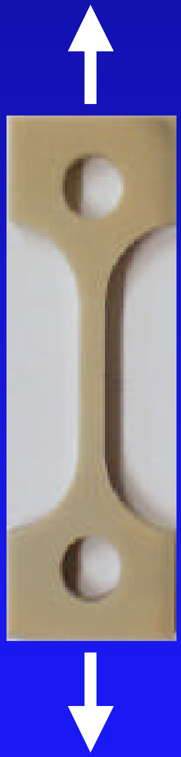


"Component Verification" Provides Key Supports to Facilitate the Development and Implementation of Complex-Shaped Ceramic Components

Evaluation of
As-processed
Surface Properties



Phase identification
Residual Stress
NDE
Microstructure Characterization



Evaluation of
Bulk Properties

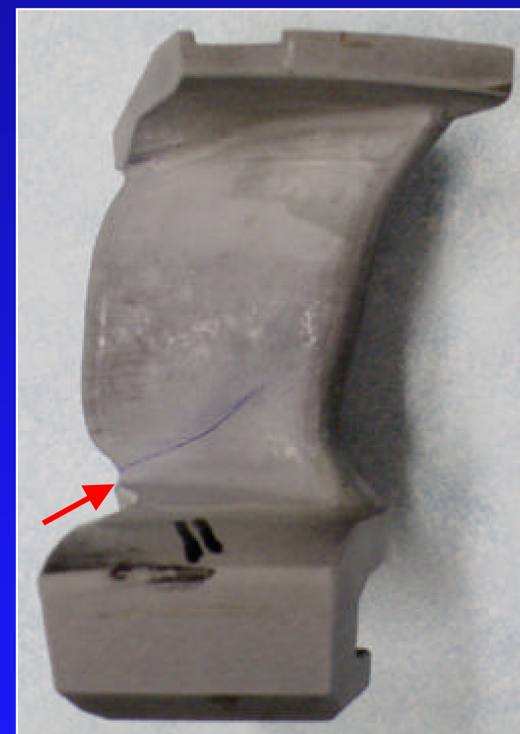
Objective

Characterization of Microstructure and Mechanical Properties of SN88 Si_3N_4 Nozzles After Exposure in Solar Centaur 50S Turbine

NGK SN88 Silicon Nitride Nozzles Designed for Solar Centaur 50S Turbine

As-received

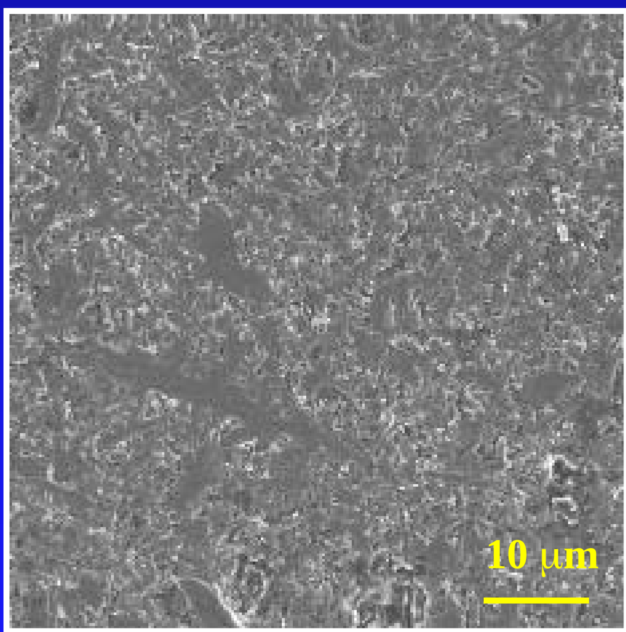
10 h test



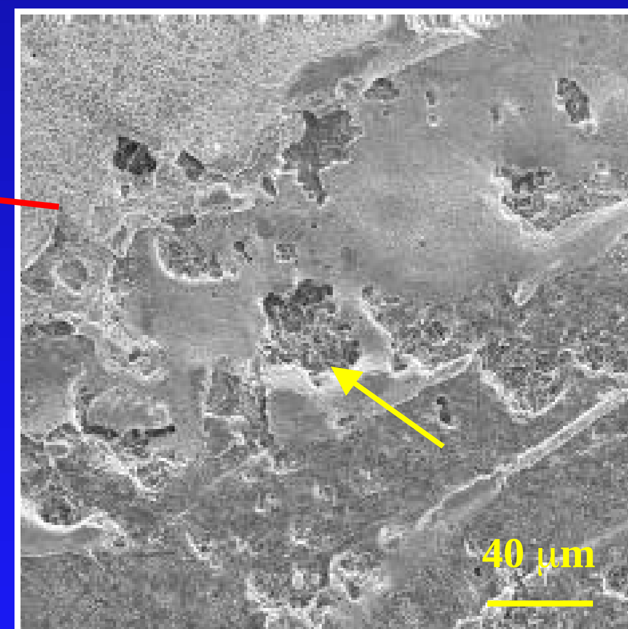
68 h test

Crack observed after 68 h engine operation with 15 start/stop cycles

Minor Surface Pitting Observed on Airfoil Surface of SN88 Nozzles After 10 h Engine Exposure



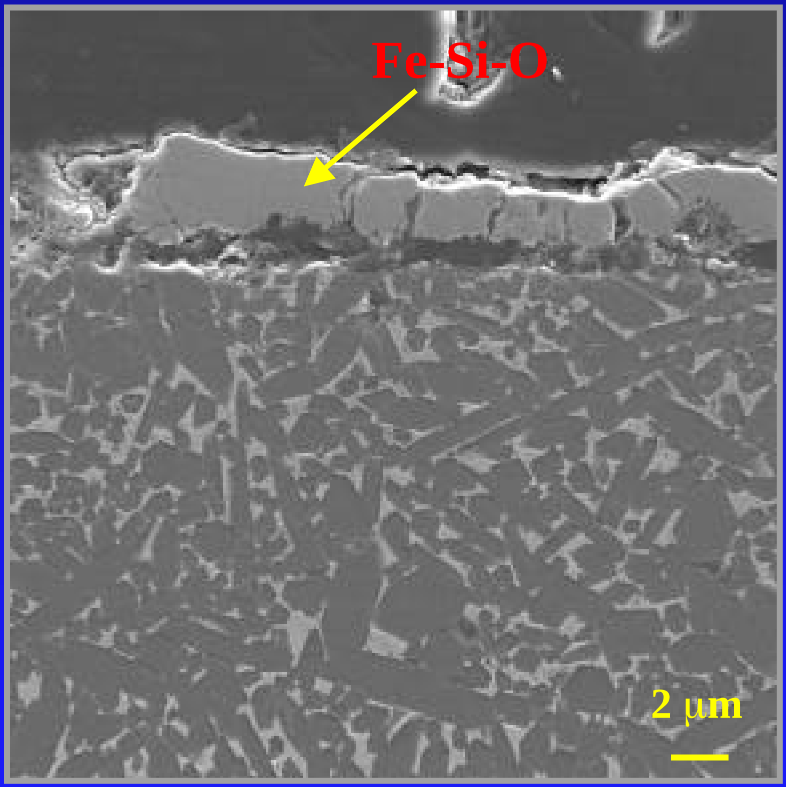
Non-pressured side

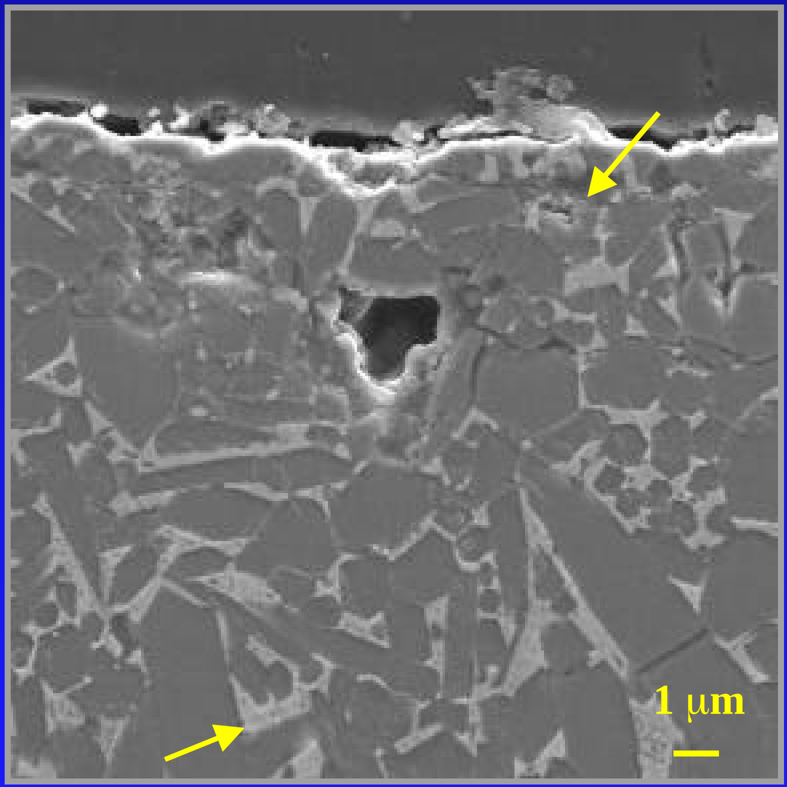


Pressured side

N768_16 after 10 h field test

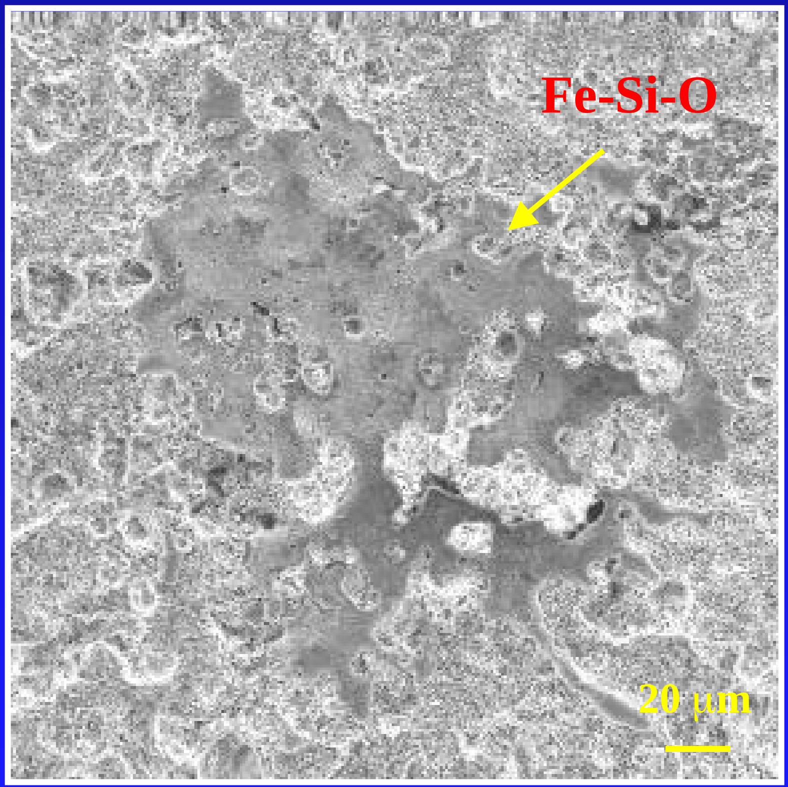
Fe-oxide Deposit and Limited Subsurface Damage Observed in Nozzle Airfoil

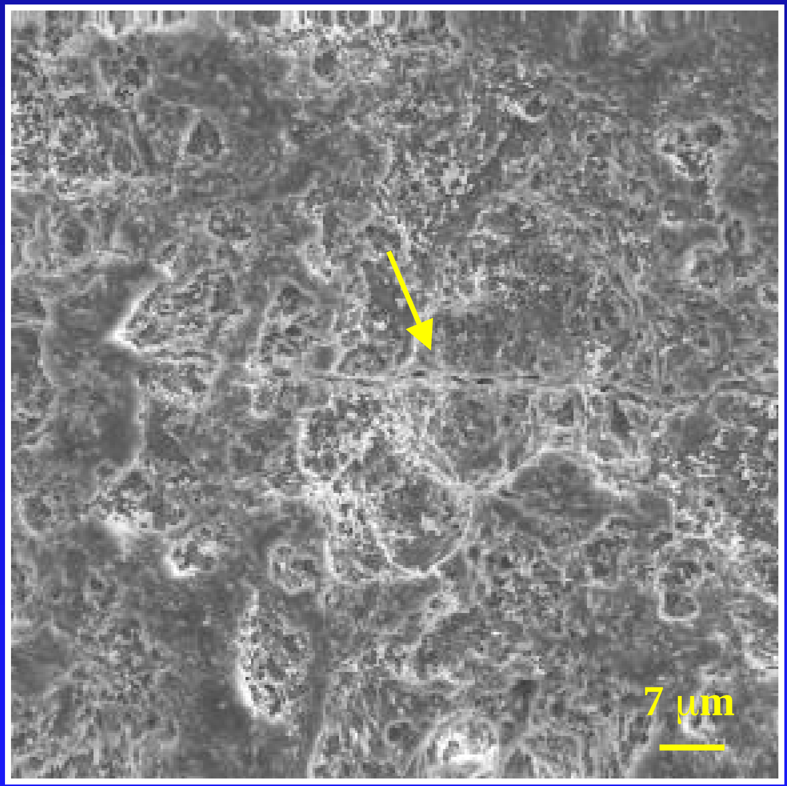




N768_16 after 10 h field test

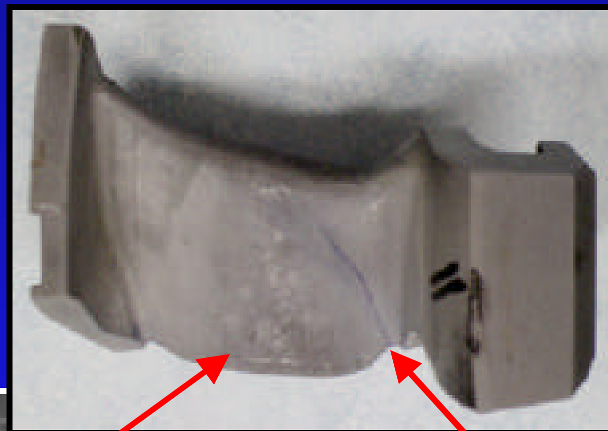
Metal Oxide Deposits and Sever Pitting Observed on Airfoil Surface of SN88 Gas Turbine Nozzles



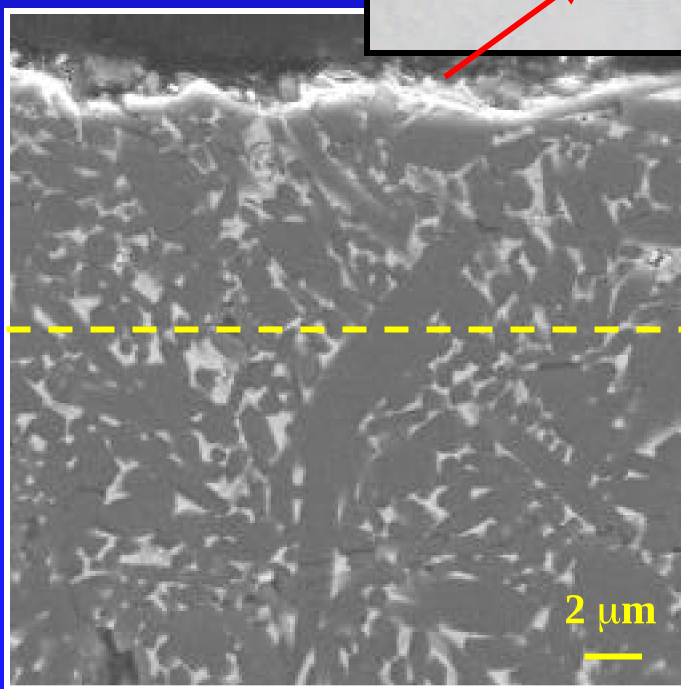


N761_15 nozzle after 68 h field test

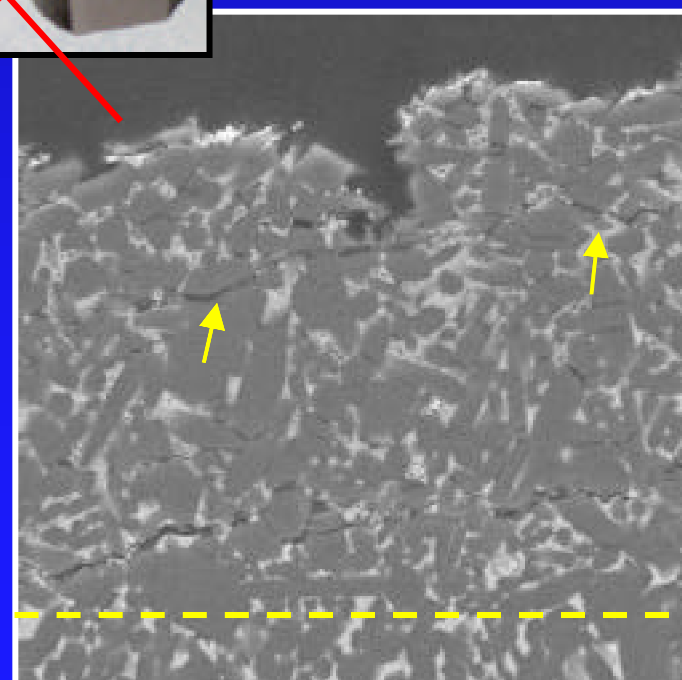
Extensive Crack Generation and Pores in Secondary Phase Observed in Airfoil/Platform Region



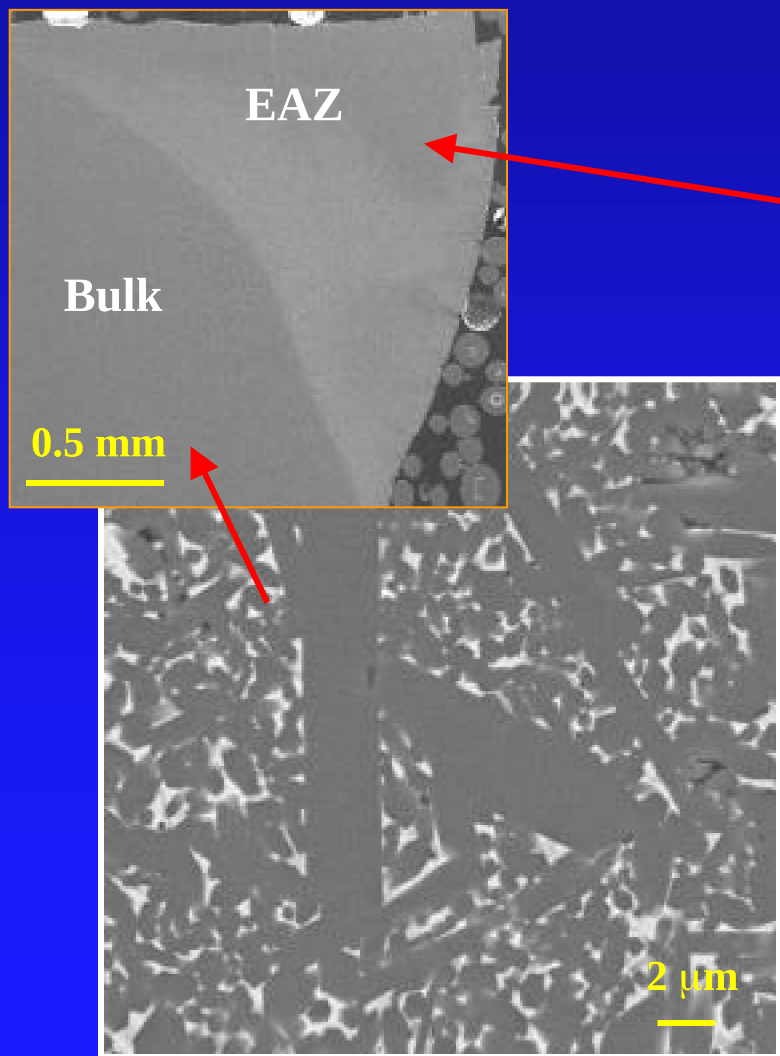
Middle Surface Region

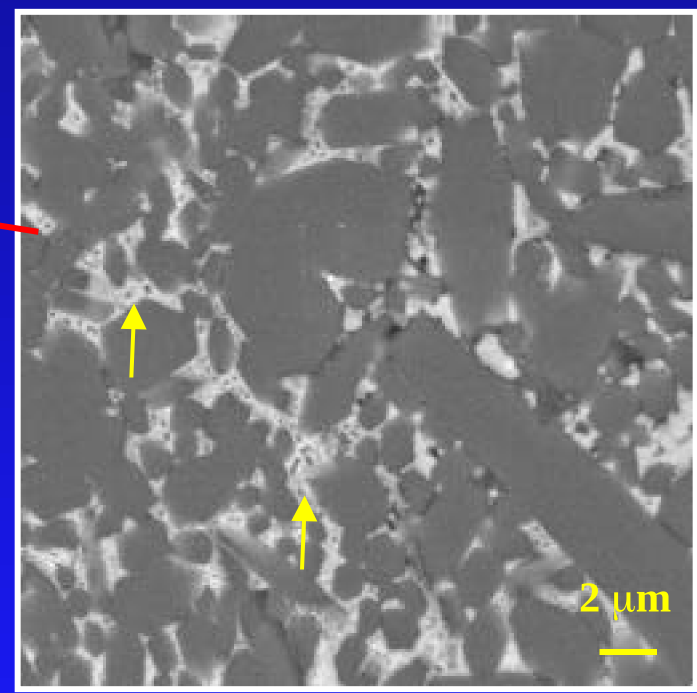


Near Airfoil/Platform Transition Region (800-950°C)



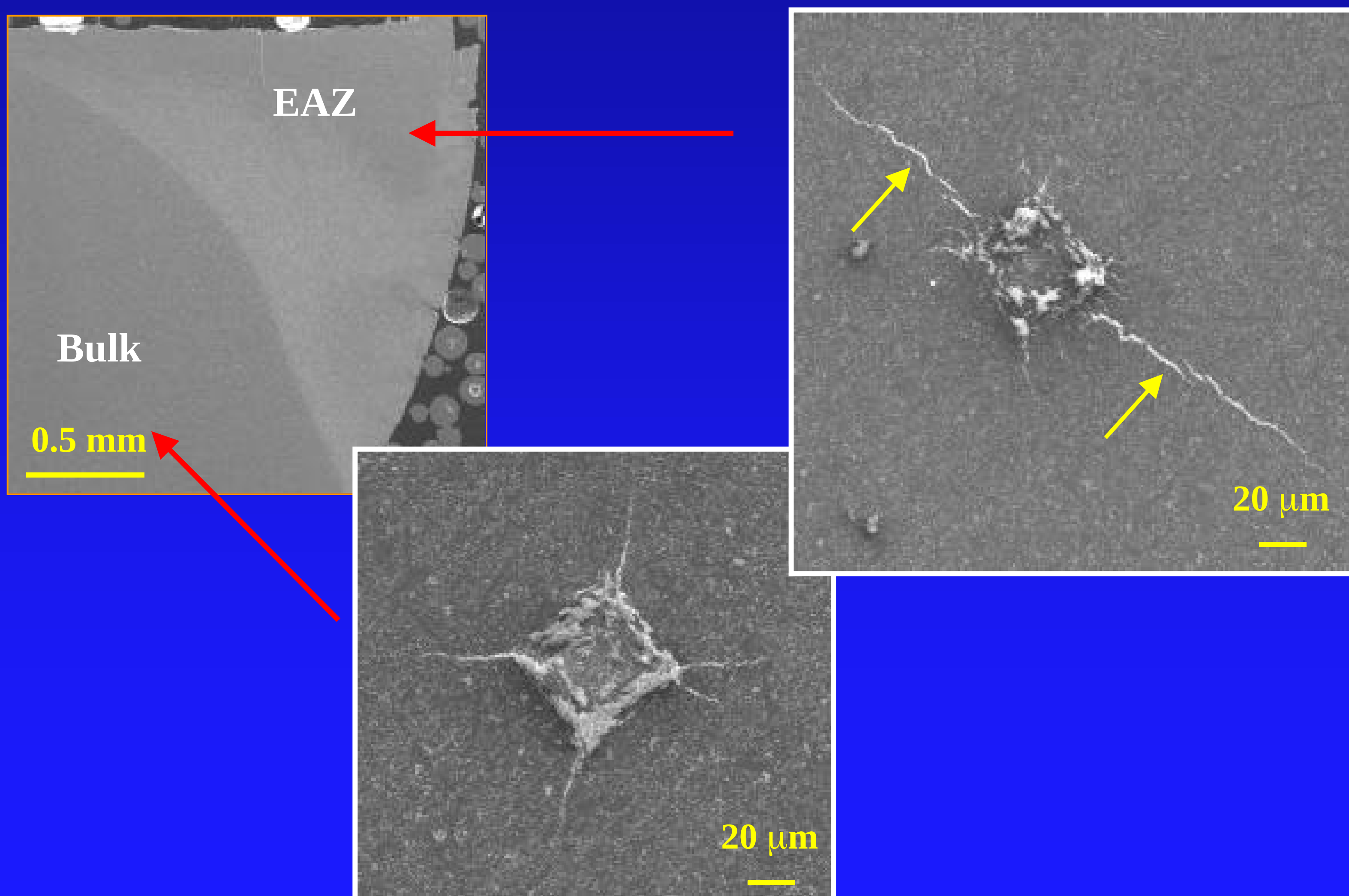
Phase Change Occurred in Intergranular Phase in Environment-Affected Zone (EAZ)



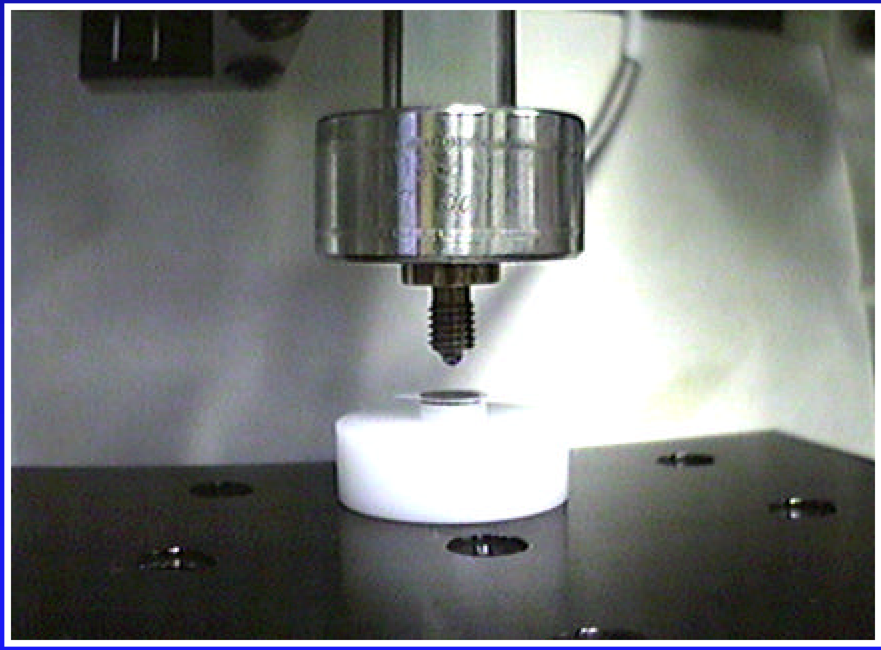


$Yb_4Si_2O_7N_2$ (J-phase)
∅ $Yb_2Si_2O_7 + Yb_2SiO_5$

Vickers Indentation Indicated a High Residual Stress Developed in Environment-Affected Zone (EAZ)



Biaxial Tests Used to Evaluate Disk Specimens Extracted From Airfoils in SN88 Nozzles



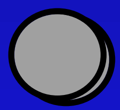
Biaxial Test Stage

9 h

68 h

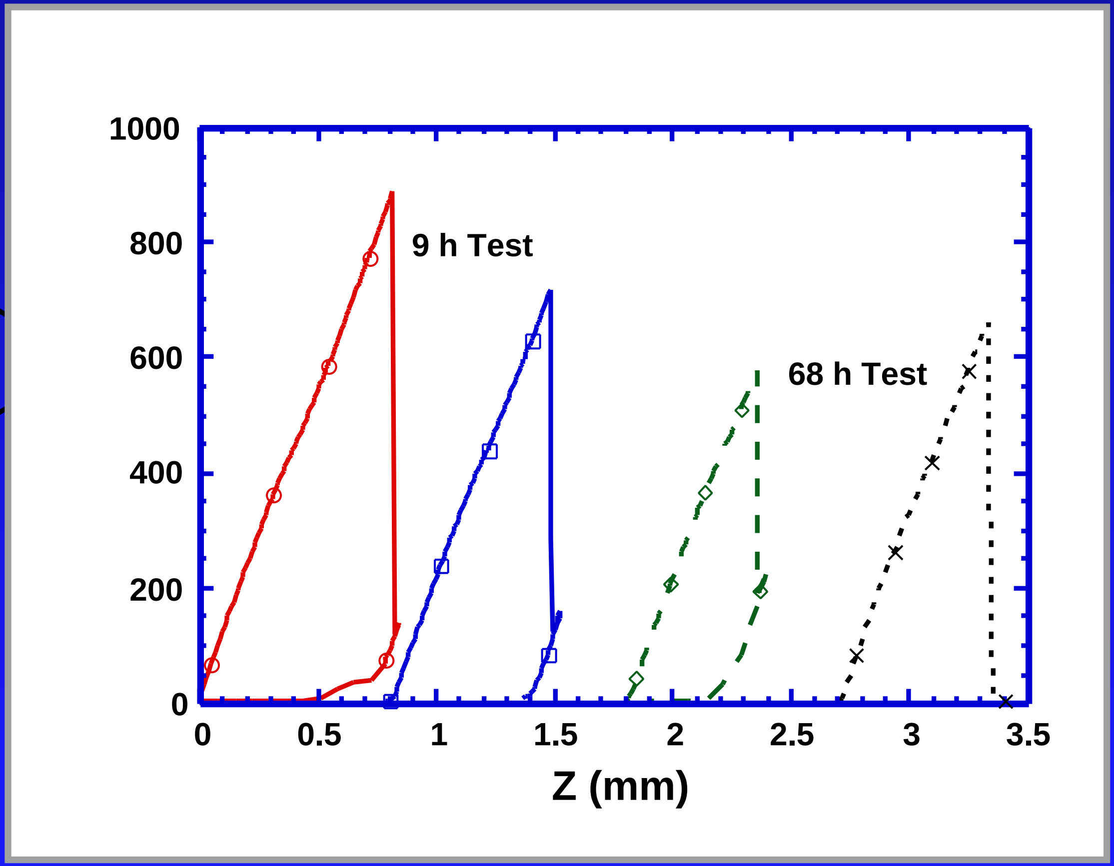
Solar NGK SN88 Gas Turbine Nozzles After Engine Test

Strength Degradation Observed in Specimens Extracted From Nozzle Airfoils After 68 h Engine Test



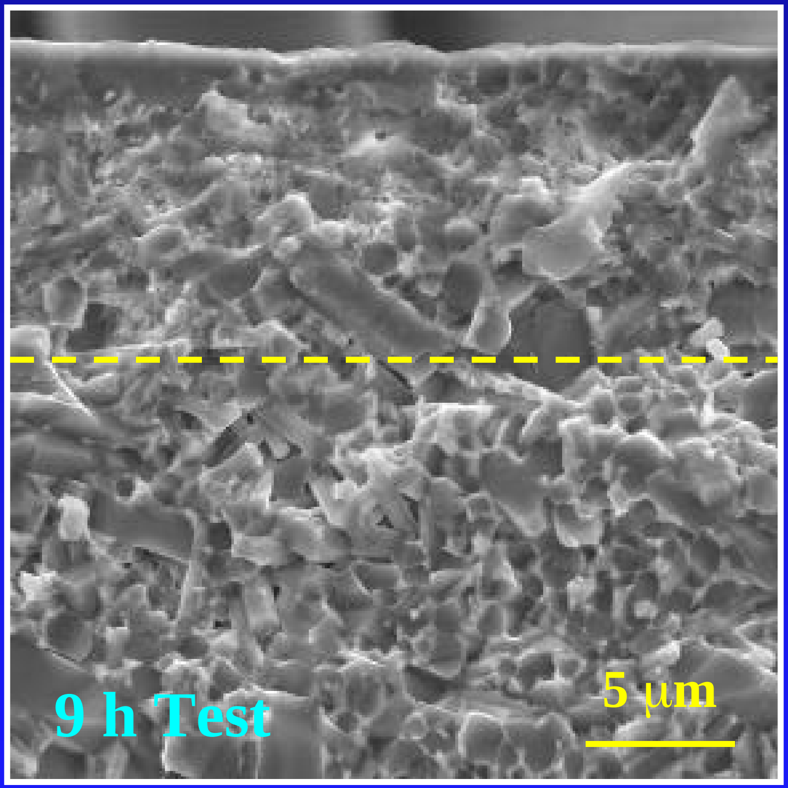
D = 5 mm
t = 0.3 mm

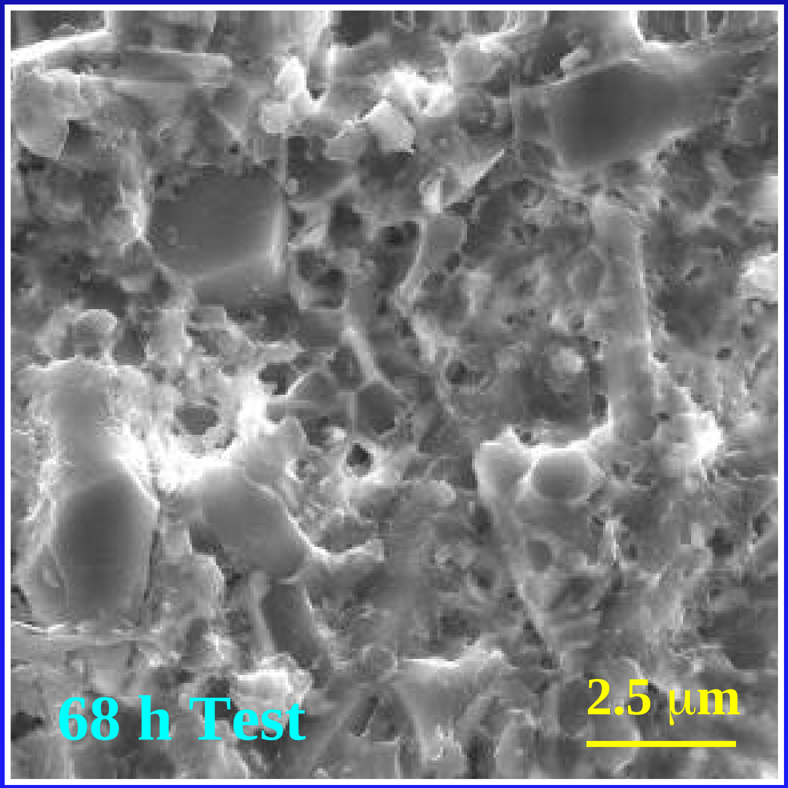
@ 20°C



Exposed airfoil surfaces tested as tensile surfaces

Larger Environmentally-Affected Zone (EAZ) Observed in 68 h Test Airfoil Specimens

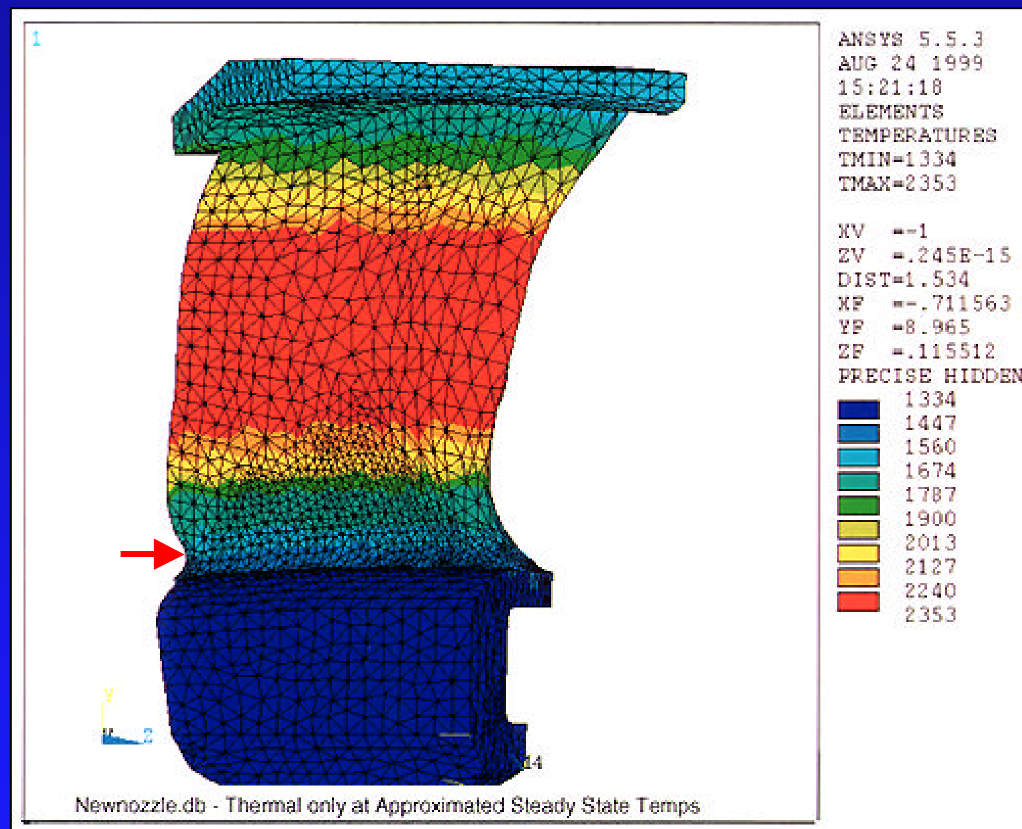




EAZ ~ 10 μm from airfoil surface

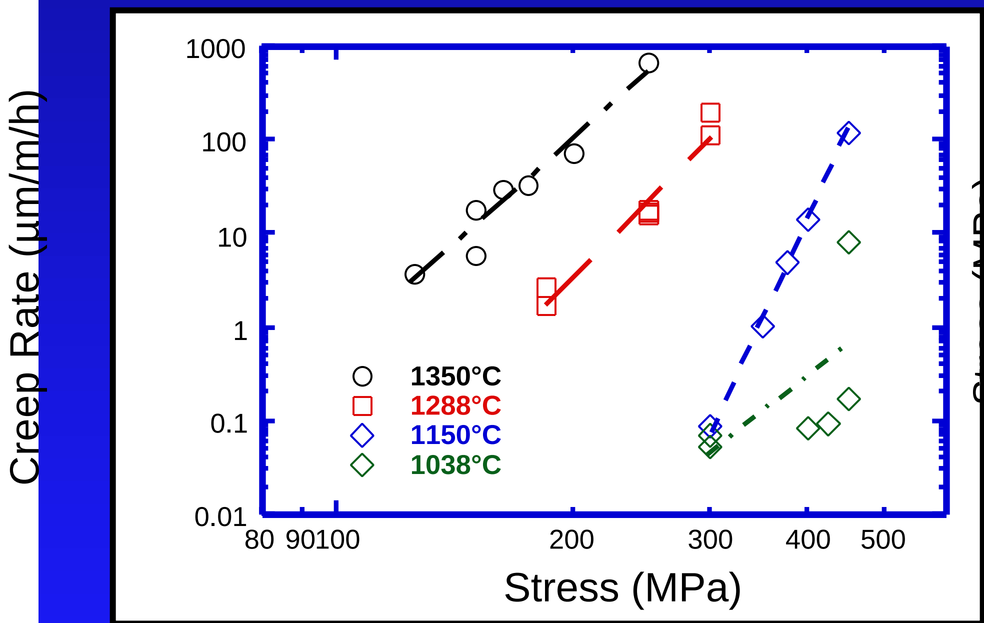
EAZ ~ 70 μm from airfoil surface

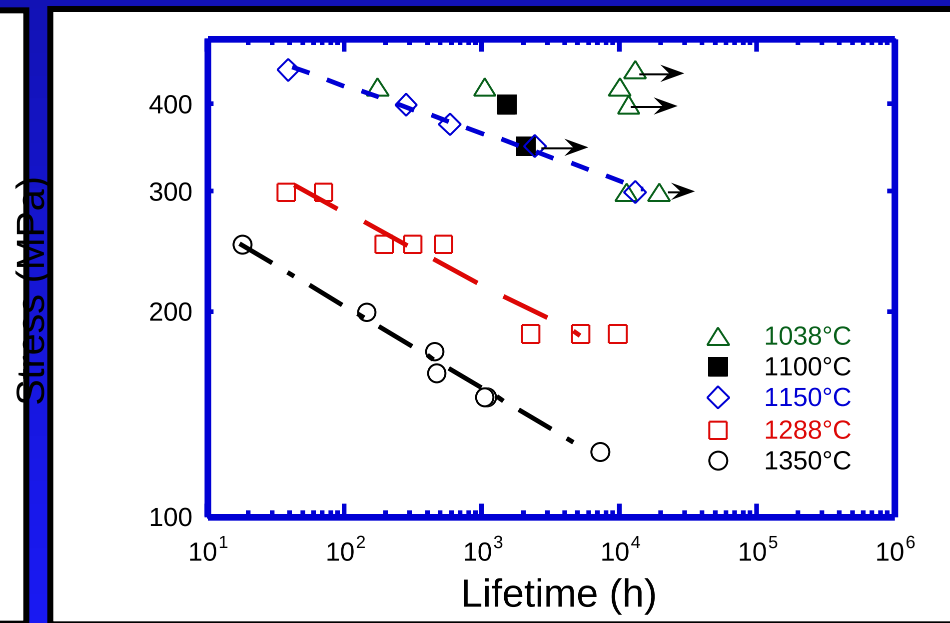
Critical Crack Initiated at Low Temperature (< 920°C) Airfoil Trailing Edge Region



Temperature distribution based on steady state temperature of 1120°C

SN88 Si_3N_4 Exhibited Adequate Long-Term High-Temperature Creep Performance and Lifetime





Hypothesis: SN88 Si_3N_4 may exhibits a mechanical instability at intermediate temperatures in air

Summary of SN88 Si_3N_4 Nozzle Evaluation

- Oxidation and volatilization of Si_3N_4 leads to accumulation of secondary phase on airfoil surface and formation of surface defects.

- Dominant crack developed in the low temperature airfoil/platform transition region (800°-920°C) after 68 h engine exposure .

- An environment-affected zone (EAZ) with extensive cracking and pores developed near the crack initiated region. A large residual stress plus changes in secondary phase observed in the EAZ.

- Mechanical properties of SN88 airfoils degraded after exposure in gas turbine environment.

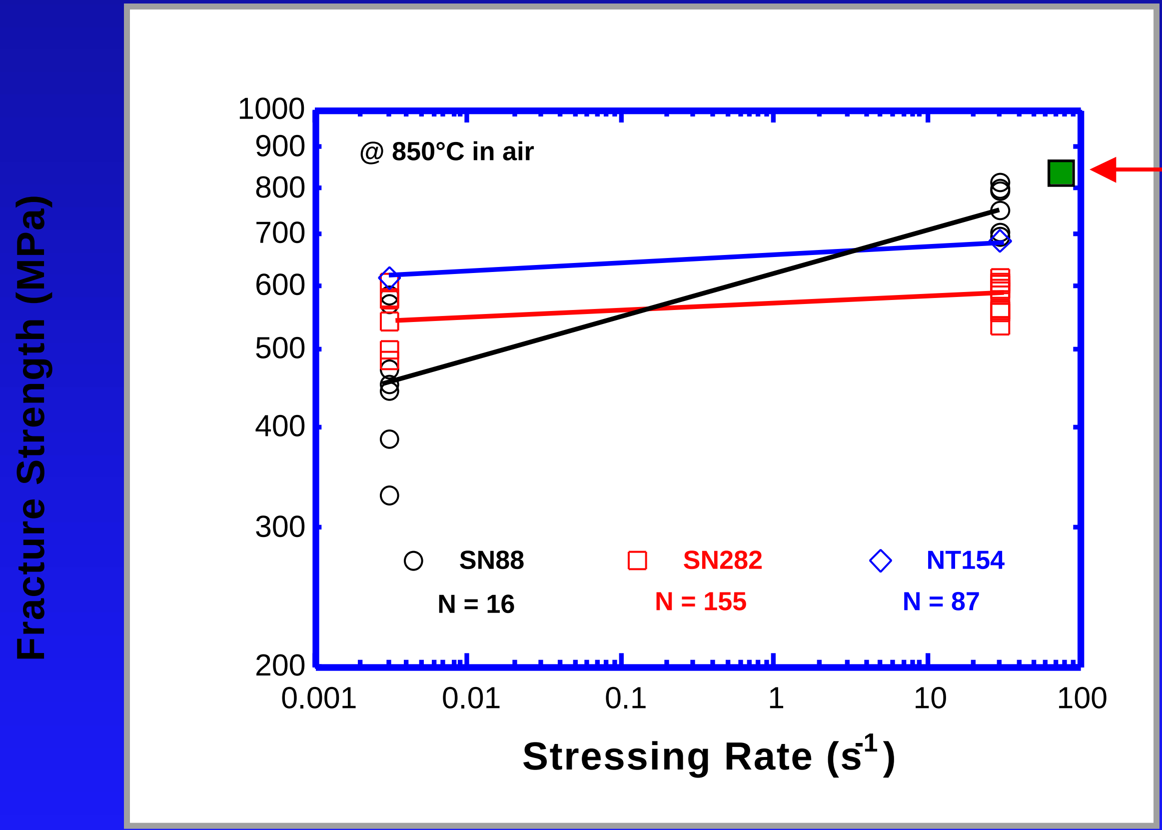
Evaluation of Mechanical Properties for SN88 Silicon Nitride At 850°C in Air

1. Dynamic Fatigue Test
    - 4-Point Bend Fixture: 20 mm/40 mm Spans
    - Stressing Rate: 30 MPa/s and 0.003 MPa/s
    - @ 850°C in air

2. Stress-Rupture Test
    - 4-Point Bend Fixture: 20 mm/40 mm Spans
    - Stress Level: 200 to 600 MPa
    - @ 850°C in air (and N_2)

Objective: to provide insight into the material degradation/failure mechanisms of SN88 silicon nitride ceramic nozzles

Fracture Strength of SN88 Silicon Nitride at 850°C in Air Significantly Influenced by the Stressing Rate

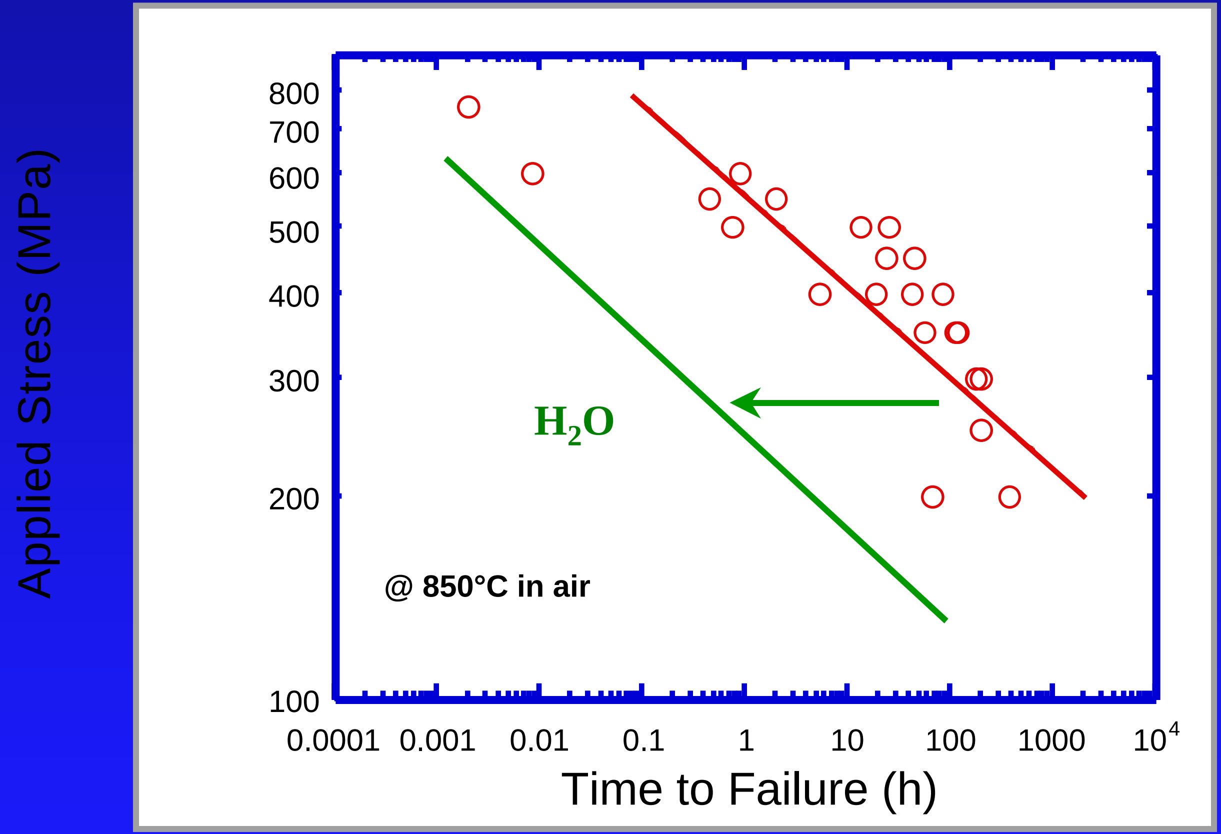


RT fracture strength of SN88

$\ln(\sigma_f) = 1/(N+2)\ln(\dot{\sigma}) + C$

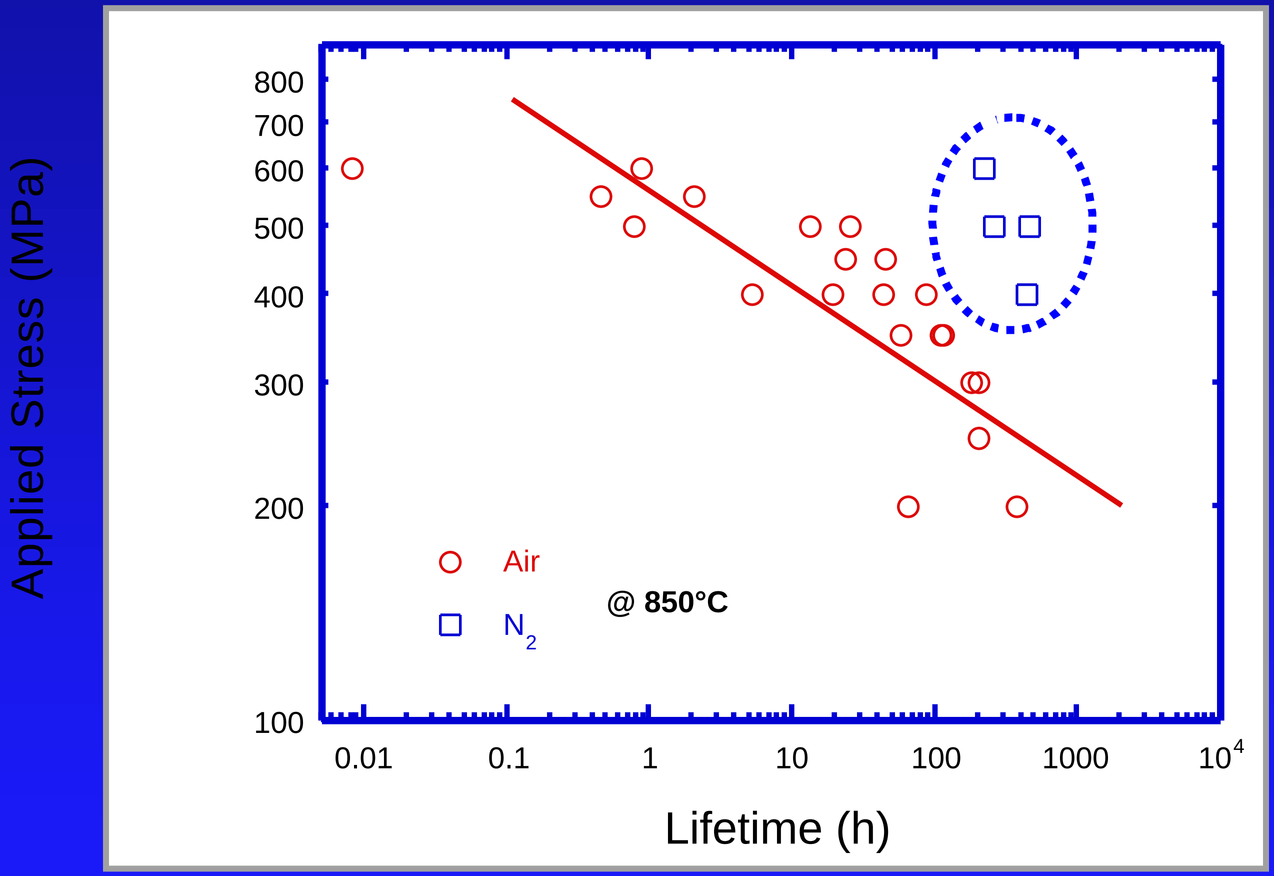
Low fatigue exponent (N) of SN88 indicated a high susceptibility to slow crack growth (SCG) process

Also, Lifetime of SN88 Silicon Nitride @ 850°C in Air Strongly Depends Upon Applied Stress Levels



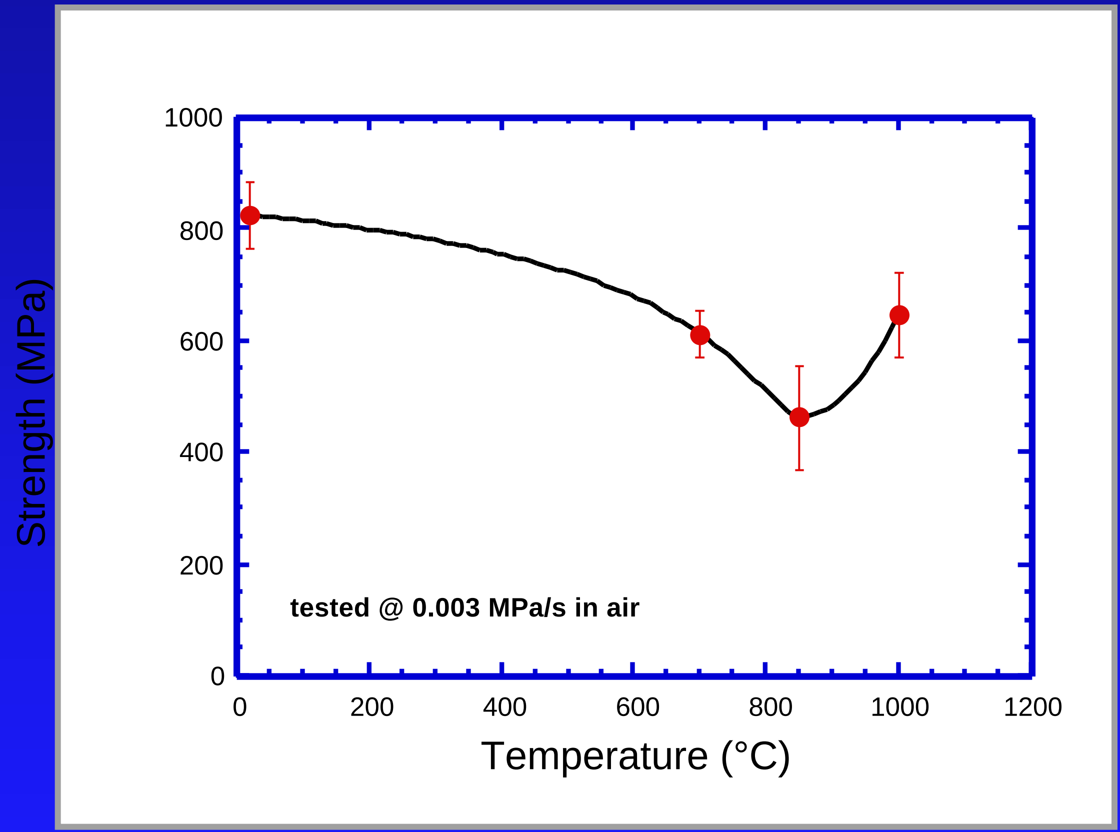
Presence of high pressure water vapor might further shorten the lifetime

Lifetime of NGK SN88 Si_3N_4 Material Substantially Extended in N_2 Environment



Strength degradation of SN88 Si_3N_4 is simply an oxidation-related issue

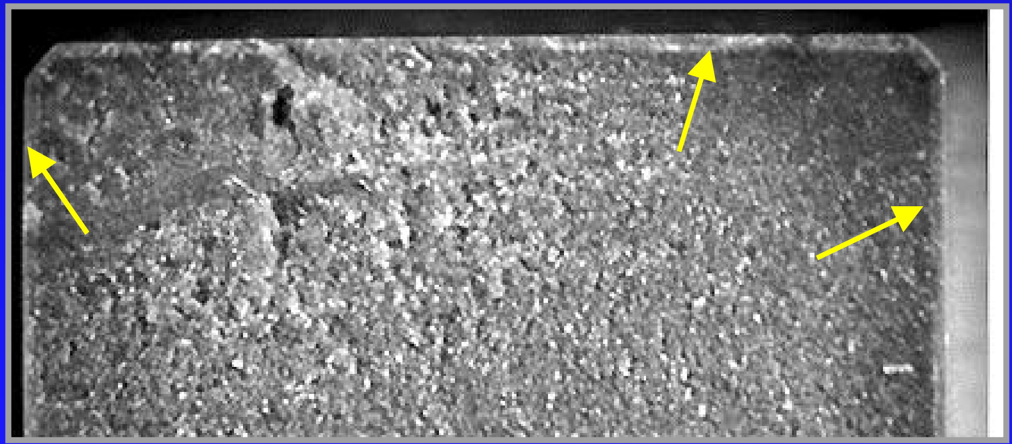
SN88 Silicon Nitride Material Exhibited the Greatest Strength Degradation @ 850°C in Air



Strength degradation is also a temperature-dependent phenomenon

Fracture Surfaces of SN88 Silicon Nitride Exhibited an Environment-Affected Zone (EAZ)

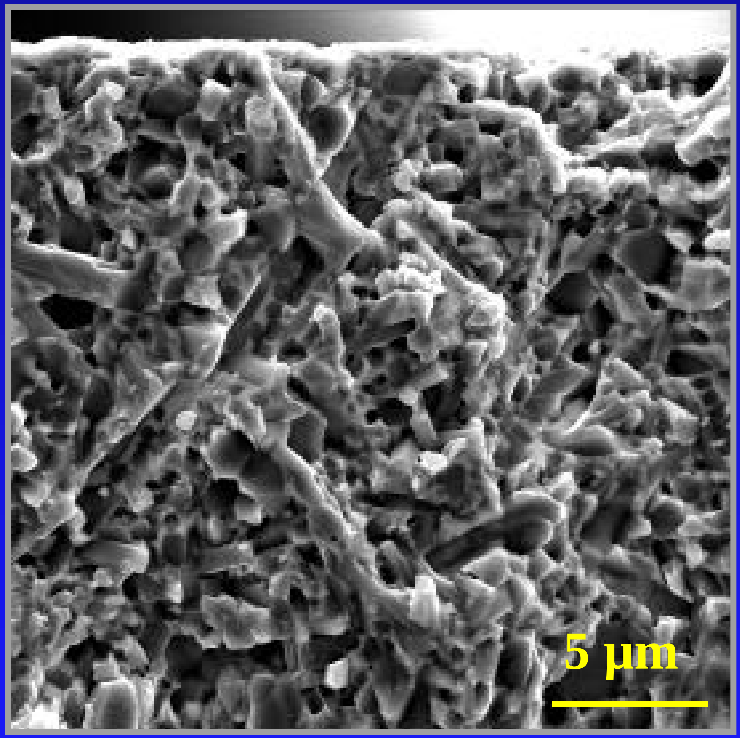
30 MPa/s
Arrow denotes fracture origin

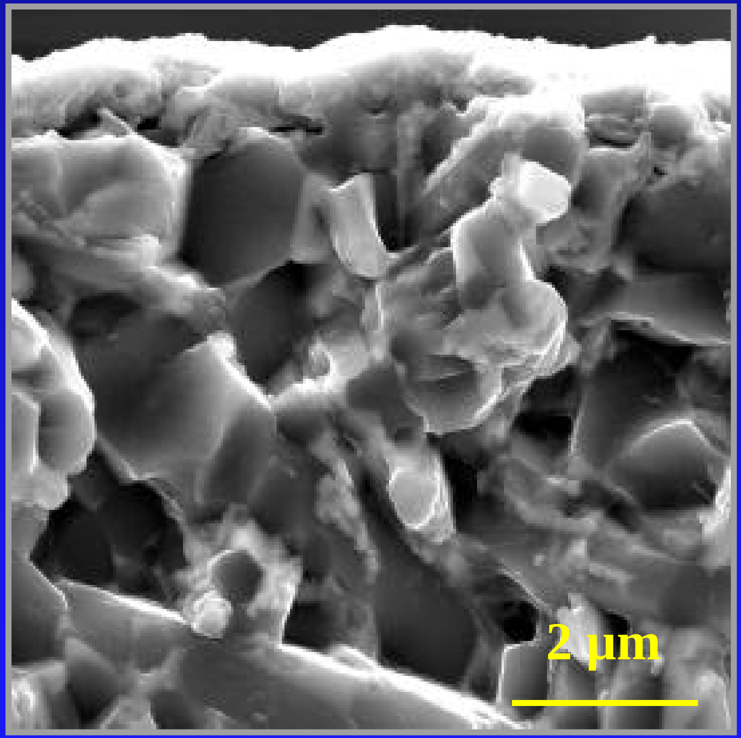


0.005 MPa/s
Arrow denotes environment-affected zone (EAZ)

Tested at 850°C in air

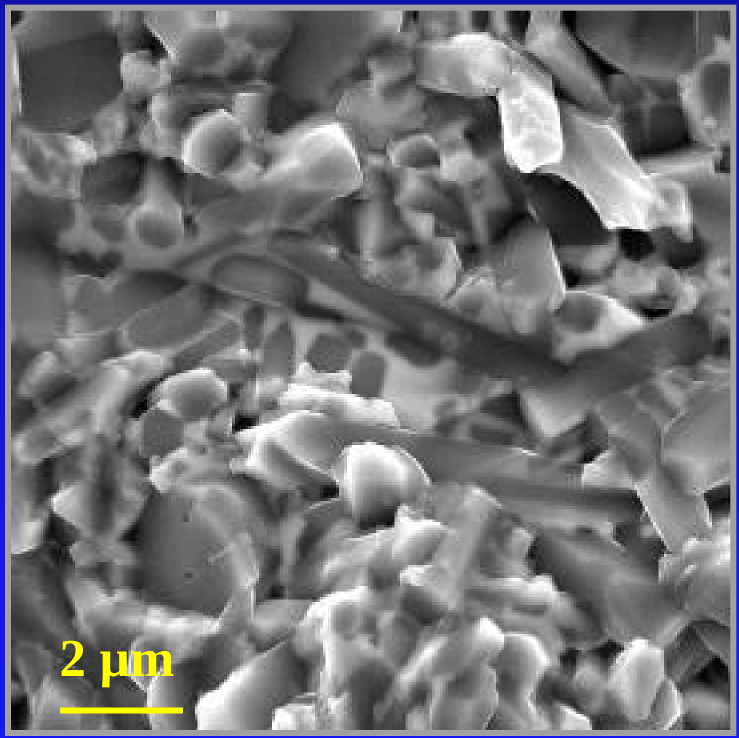
Little Changes in Microstructure Observed in Samples Tested at 850°C/30 MPa•s^{-1}



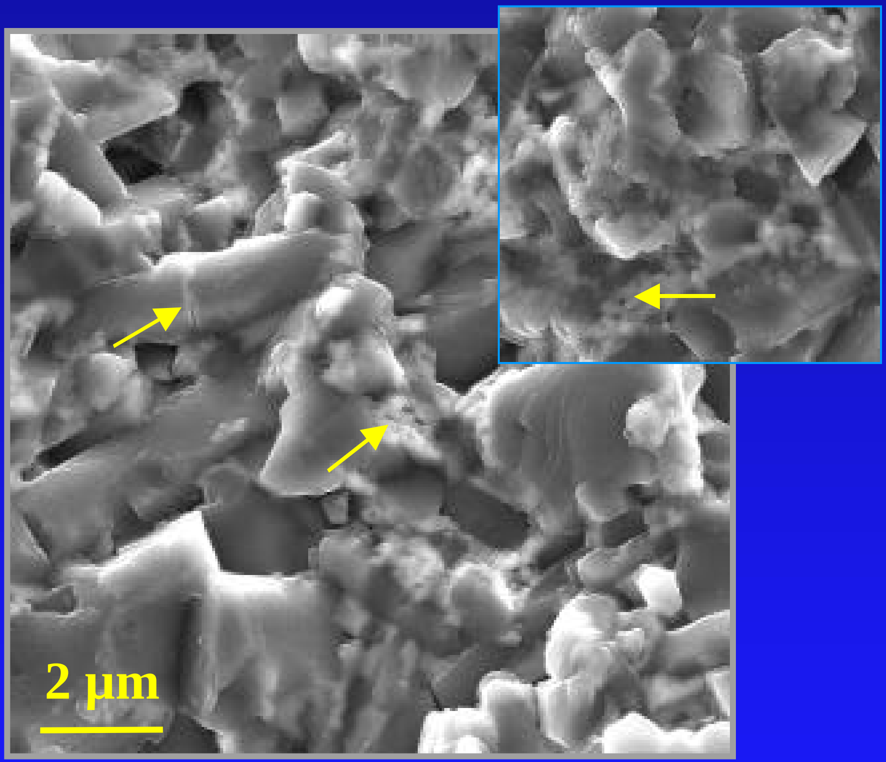


SEM micrograph of fracture surface feature in tensile surface region

However, Substantial Changes in Microstructure Observed in EAZ of Samples Tested at 0.003 MPa•s^{-1}



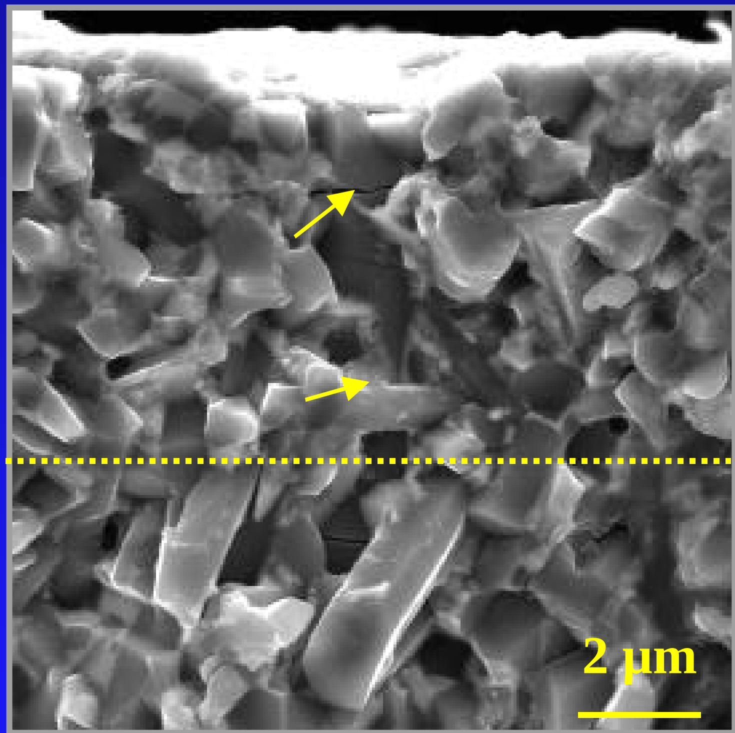
Outside EAZ



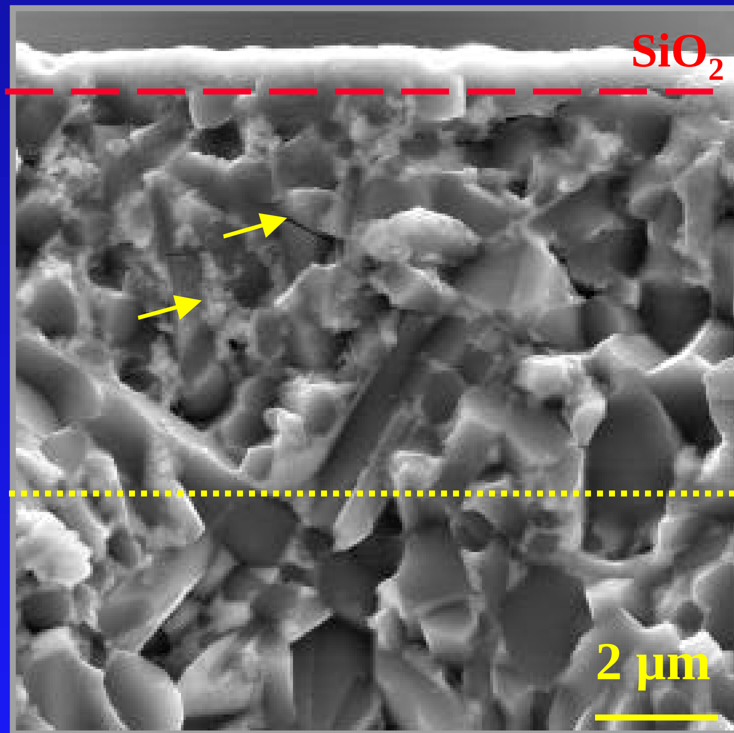
Inside EAZ (25-30 µm)

No changes in microstructure characteristics outside the EAZ

Similar Changes in Microstructure Observed in EAZ of Samples Tested at 700° and 1000°C in Air



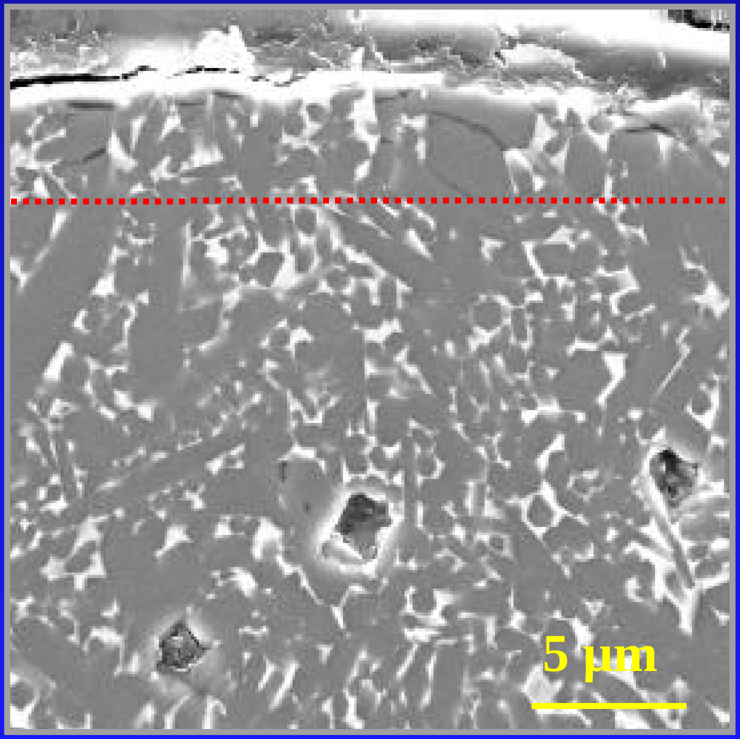
700°C

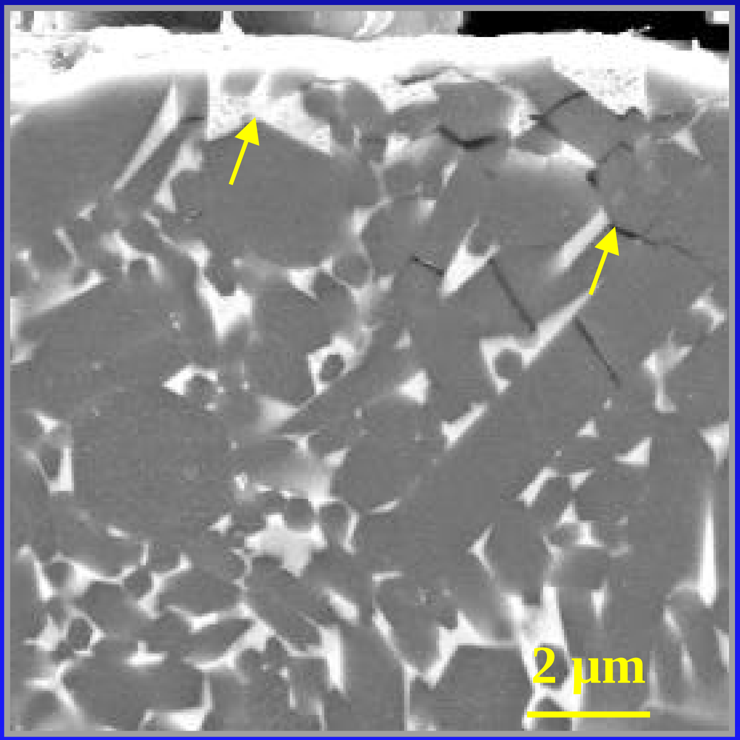


1000°C

SiO_2 formation at 1000°C slowed down the oxidation reaction and thus phase transformation processes

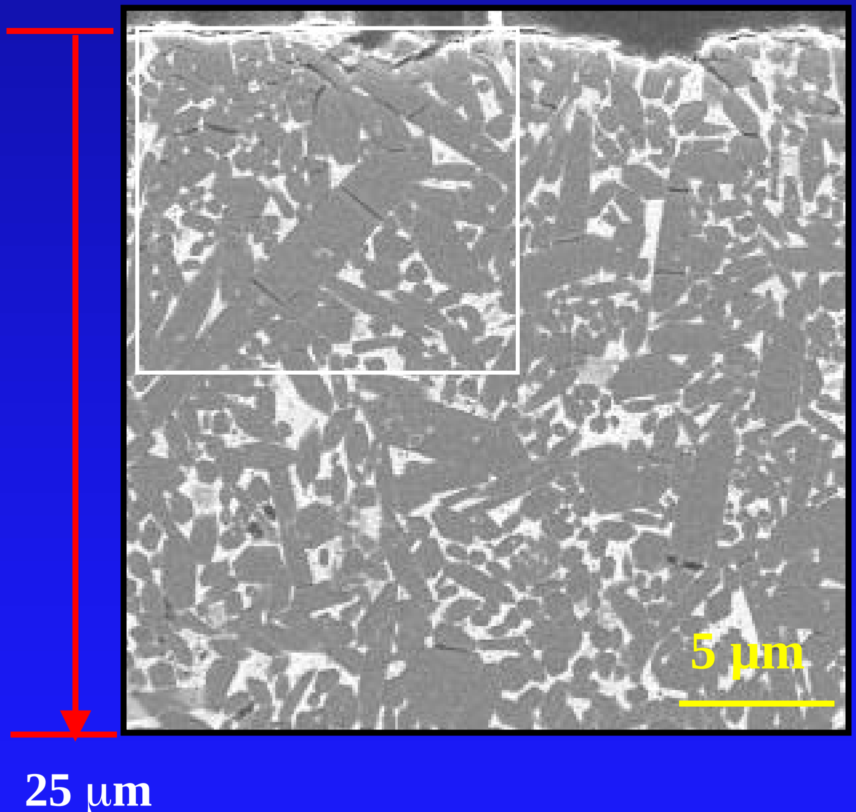
Very Limited Microstructure Changes Observed in Specimens Tested @ 850°C/30 MPa•s^{-1}

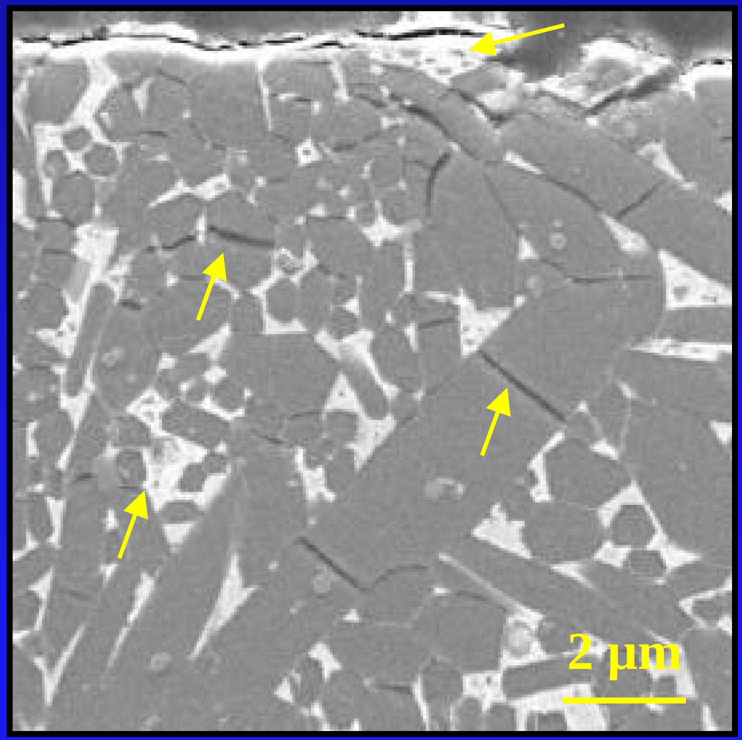




Polished cross-section in tensile surface region

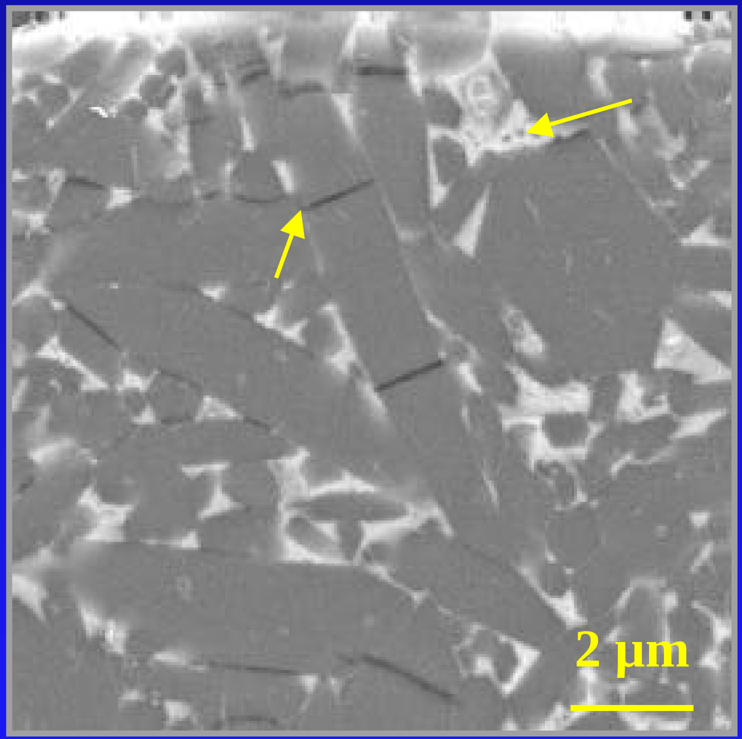
Substantial Cracking Associated With Pores in Secondary Phases Observed in EAZ Region



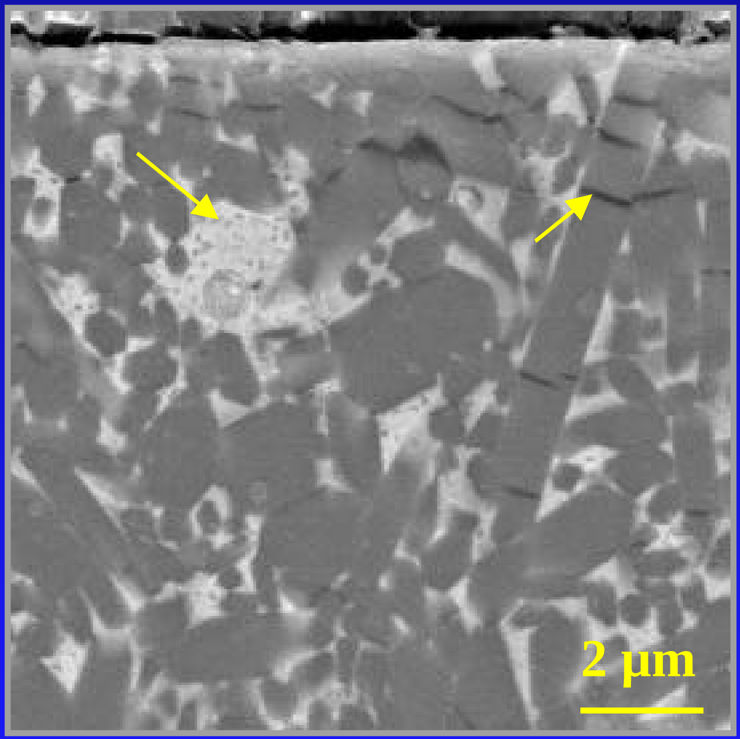


Tested @ 850°C/0.003 MPa•s^{-1} in air

Similar Feature of Cracking and Pores in Secondary Phases Observed on All Four Side Surfaces



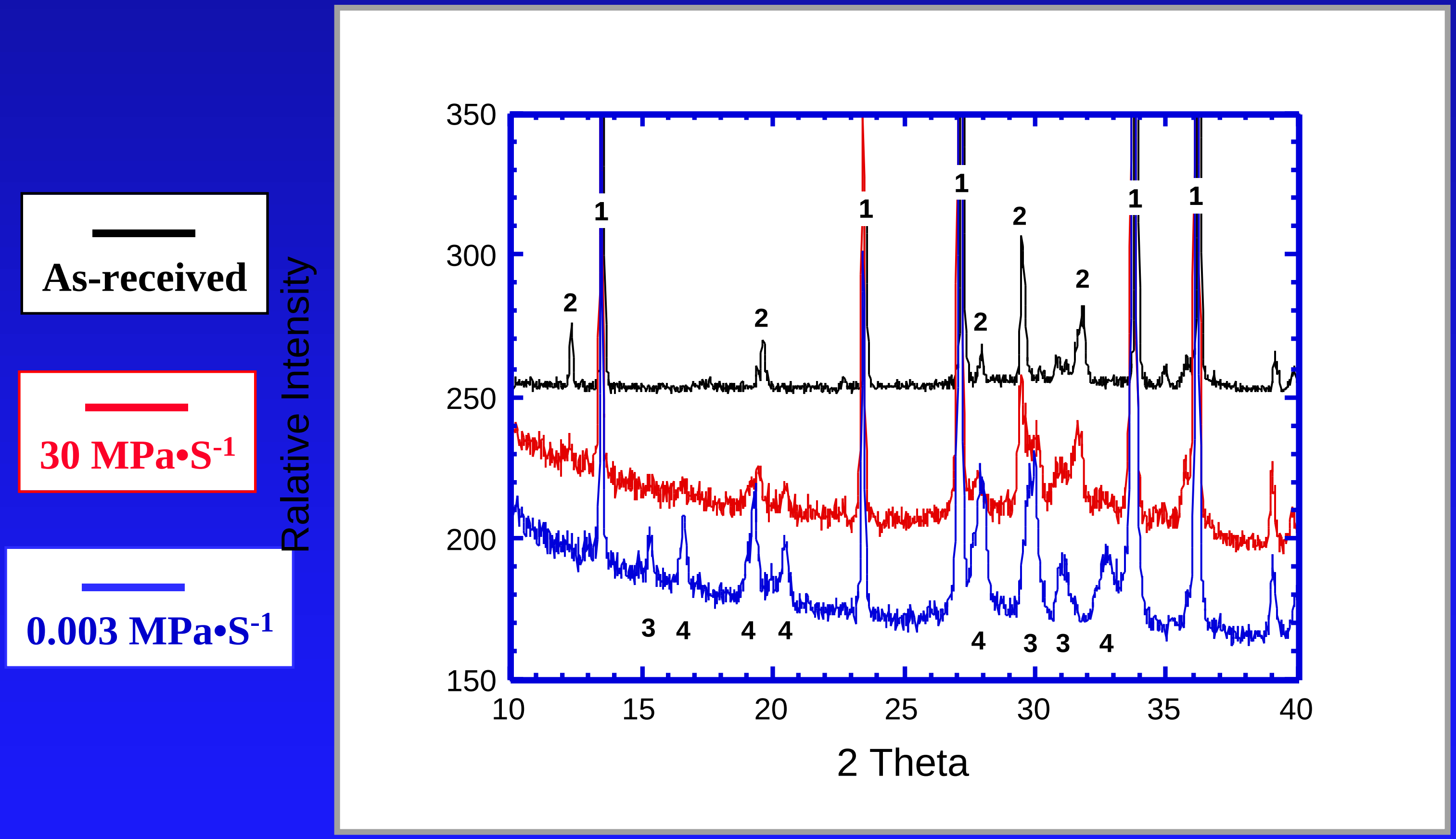
Thickness Side



Compressive Side

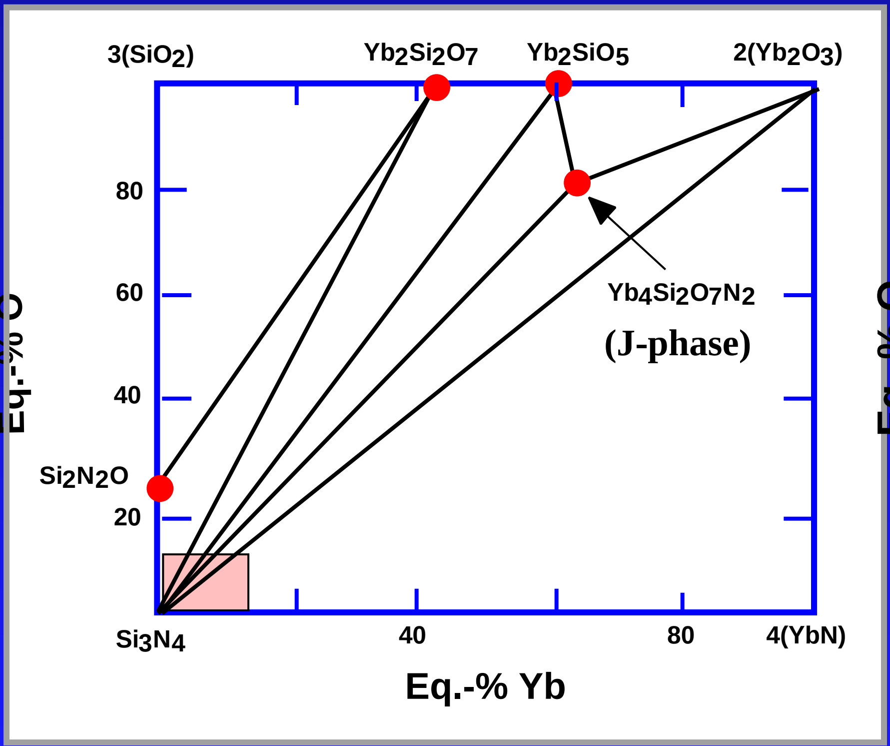
Features of cracks and pores are not stress-promoted phenomena

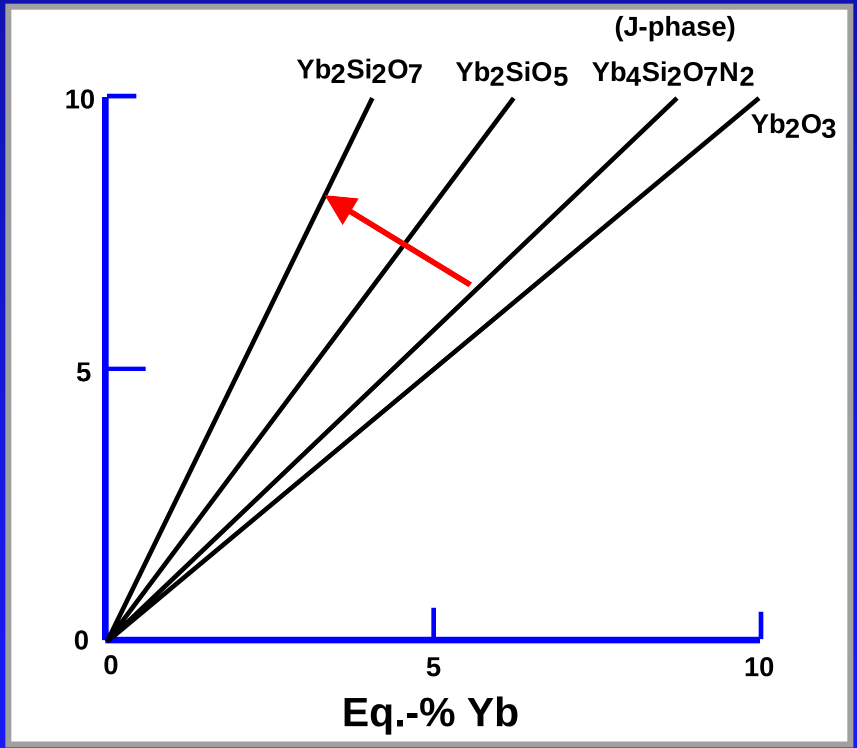
Substantial Changes in Secondary Phase(s) Observed in Samples Tested at 0.003 MPa•s^{-1}



1: Si_3N_4 2: $Yb_4Si_2O_7N_4$ 3: Yb_2SiO_5 4: $Yb_2Si_2O_7$

Phase Relationships in the System of Si_3N_4 - Yb_2O_3 -SiO_2 -YbN





Ingress of oxygen resulted in changes in secondary phase of SN88 Si_3N_4

Substantial Volume Change Occurred due to Phase Transformation at Temperatures in air

$$Yb_4Si_2O_7N_2 + SiO_2 + 2/3\ O_2 \text{ ----> } Yb_2Si_2O_7 + Yb_2SiO_5 + N_2$$

$Yb_4Si_2O_7N_2$ ----> $Yb_2Si_2O_7$ (-ΔV = 64.2%)

$V_J = 790 \times 10^{-24}\ cm^3$ $V_7 = 283 \times 10^{-24}\ cm^3$

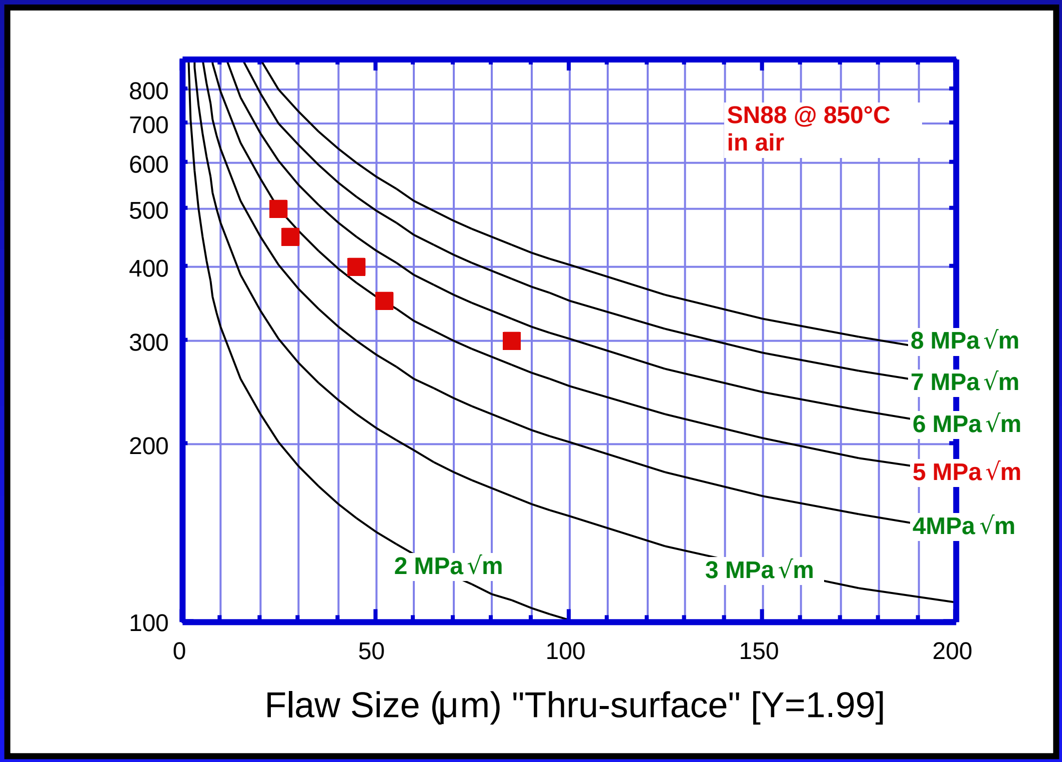
$Yb_4Si_2O_7N_2$ ----> Yb_2SiO_5 (+ΔV = 5%)

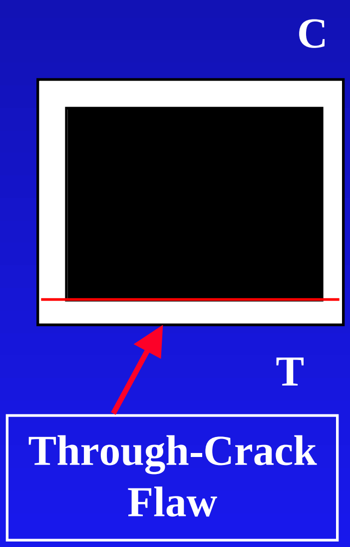
$V_J = 790 \times 10^{-24}\ cm^3$ $V_5 = 828.8 \times 10^{-24}\ cm^3$

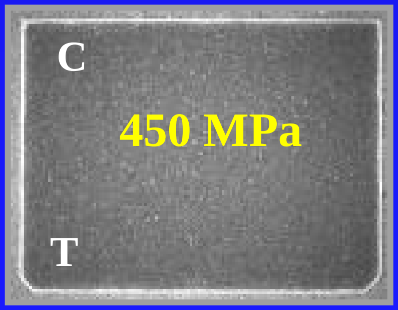
$$\sigma_r \sim [\Delta V/V]x[E/(1-\nu)]$$

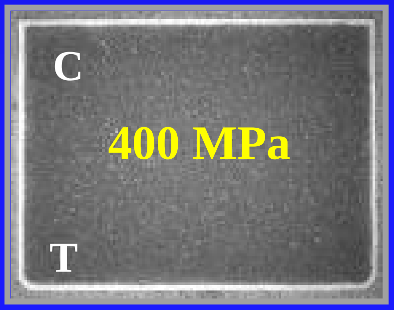
A large surface tensile stress developed, resulting extensive crack formation (similar to the case observed in earlier Si_3N_4 ceramics with Y_2O_3)

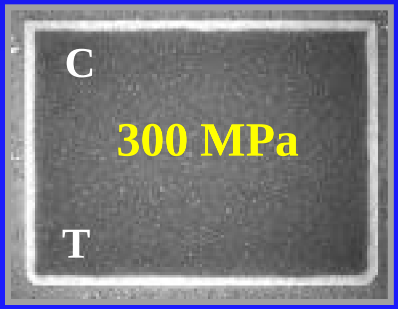
Stress/Flaw Size Dependence on Fracture Toughness (Through-Surface Flaw)











Summary of Supporting Studies

• Degradation in strength of SN88 Si_3N_4 is strongly influenced by the testing environment and is time- and (also temperature-) dependent.

• Development of an environment-affected zone (EAZ) is an oxidation-related issue. It's not a stress-promoted phenomenon.

• Oxidation (and phase change) of secondary phase(s) results in development of a high residual tensile stress, leading to the generation of subsurface damage zone with substantial cracking and pores.

• Formation of through-surface flaw would result in significant degradation of mechanical reliability of SN88 Si_3N_4 .

Failure of SN88 Si_3N_4 Turbine Nozzles Appears to Involve Several Processes

- Oxidation/volatilization processes removed the protective silica layer present in the as-received SN88 nozzles.

- An environment-affected zone (EAZ) developed in the airfoil/platform transition region (800°-920°C).

- The generation of extensive multiple cracking in EAZ would significantly reduce the mechanical reliability and increase the SCG susceptibility of nozzles.

- A critical crack would readily initiate at the airfoil transition region and lead to the failure of SN88 nozzles.