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# **Erosion, Corrosion and Foreign Object Damage Effects in Gas Turbines**

(Les conséquences de l'endommagement des turbines à gaz par érosion, corrosion et objets étrangers)

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## **RESISTANCE OF SILICON NITRIDE TURBINE COMPONENTS TO EROSION AND HOT CORROSION/OXIDATION ATTACK**

Thomas E. Strangman AlliedSignal Engines Phoenix, Arizona, USA

Dennis S. Fox NASA Lewis Research Center Cleveland, Ohio, USA

## **1 SUMMARY**

Silicon nitride turbine components are under intensive development by AlliedSignal to enable a new generation of higher power density auxiliary power systems. In order to be viable in the intended applications, silicon nitride turbine airfoils must be designed for survival in aggressive oxidizing combustion gas environments. Erosive and corrosive damage to ceramic airfoils from ingested sand and sea salt must be avoided.

Recent engine test experience demonstrated that NT154 silicon nitride turbine vanes have exceptional resistance to sand erosion, relative to superalloys used in production engines. Similarly, NT154 silicon nitride has excellent resistance to oxidation in the temperature range of interest - up to 1400C.

Hot corrosion attack of superalloy gas turbine components is well documented. While hot corrosion from ingested sea salt will attack silicon nitride substantially less than the superalloys being replaced in initial engine applications, this degradation has the potential to limit component lives in advanced engine applications. Hot corrosion adversely affects the strength of silicon nitride in the 850 to 1300C range.

Since unacceptable reductions in strength must be rapidly identified and avoided, AlliedSignal and the NASA Lewis Research Center have pioneered the development of an environmental life prediction model for silicon nitride turbine components. Strength retention in flexure specimens following 1 to 3300 hour exposures to high temperature oxidation and hot corrosion has been measured and used to calibrate the life prediction model. Predicted component life is dependent upon engine design (stress, temperature, pressure, fuel/air ratio, gas velocity, and inlet air filtration), mission usage (fuel sulfur content, location [salt in air], and times at duty cycle power points), and material parameters.

Preliminary analyses indicate that the hot corrosion resistance of NT154 silicon nitride is adequate for AlliedSignal's initial engine applications. Protective coatings and/or inlet air filtration may be required to achieve required ceramic component lives in more aggressive environments.

## 2 Introduction

Silicon nitride ceramics are under intensive development for advanced gas turbine applications. In order to be viable, silicon nitride turbine airfoils must be designed for survival in aggressive erosive, oxidizing combustion gas environments. This paper reviews the stability of silicon nitride in aggressive environments and describes the status of environmental life prediction for gas turbine applications.

## **3** Experimental Procedure

The NT154 silicon nitride used for this study was manufactured by Norton Advanced Ceramics (Northboro, MA). This commercially available hot isostatically pressed (HIPped) silicon nitride material contains 4 weight percent yttria added as the densification aid.

Billets for flexure specimens were cold isostatically pressed from the powder and then HIPed to full density. Turbine vanes used in the erosion tests were produced by slip casting the vane to near-net-shape and HIPing to

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Machined flexure specimens were used to assess the effects of environmental (furnace oxidation and burner rig hot corrosion) exposure conditions on room temperature strength. Three specimens geometries were used. Oxidation effects were evaluated with 3 x 4 x 50 mm specimens, which had the tensile surface machined in the transverse (lowest strength) direction. Thick (6.35 x 12.7 x 101.6 mm), specimens machined in the longitudinal (highest strength) direction were used for 1 atmosphere burner rig hot corrosion tests. Thin, longitudinally machined specimens (2.4 x 4.8 x 26.4 mm) were used in the 5 atmosphere burner rig.

## 3.1 Furnace Oxidation

A matrix of 180 transverse machined NT154 specimens (shown in Figure 1) was exposed in an electric furnace in air for logarithmically distributed times (1 to 1000 hours) and temperatures in the range of 982 to 1400C. This distribution of specimens permitted effects of oxidation on strength to be analyzed as a function of exposure time and temperature.

## 3.2 <u>One Atmosphere Burner Rig Hot</u> Corrosion Test

NT154 flexure specimens were exposed to the series of cyclic burner rig hot corrosion environments indicated in Table 1 for logarithmically distributed times up to about 3287 hours.

The following nominal conditions were used for each test:

## Fuel: Jet A (0.035% sulfur [nominal])

Air: 5ppm synthetic sea salt (ASTM specification D1141-52)

#### Fuel/air weight ratio: 0.0606

Combustion gas velocity: 267 meters/second

Operation of the burner rig was controlled with the aid of a personal computer. Combustion products from the burner were ducted through a rotating (800 rpm) carousel that contained eight vertically mounted specimens. One of the specimens contained a thermocouple embedded below the surface in the 3 cm long hot zone test section. This thermocouple was monitored continuously by computer during the test. Due to the thickness of the specimens used in the 1 atmosphere burner rig tests, interior temperatures lagged surface temperatures by several seconds. In order to avoid overtemperature of the surface during rapid heating, an optical pyrometer continuously measured surface temperature. Optical surface temperatures were used to facilitate computer adjustments to the burner's fuel flow during heating. Testing was periodically interrupted for removal and replacement of individual specimens (to achieve a logarithmic distribution of exposure times) and for daily visual inspection. Following test interruptions, a steady state thermocouple measurement of hot zone temperature was obtained and used to calibrate the pyrometer's emissivity.



Figure 1. Air Furnace Oxidation Exposures Initially Improved Strength of Transverse Machined NT154 Silicon Nitride Flexure Specimens.

#### Table 1. Atmospheric Burner Rig Cyclic Hot Corrosion Environments.

Test	Cycle	Test Duration
1	860C/27 minutes + air cool/3 minutes	3287 hours
2	890C/27 minutes + air cool/3 minutes	600 hours
3	954C/27 minutes + air cool/3 minutes	600 hours
4	982C/27 minutes + air cool/3 minutes	400 hours
5	1052C/27 minutes + air cool/3 minutes	300 hours
6	1121C/27 minutes + air cool/3 minutes	300 hours
7	1218C/27 minutes + air cool/3 minutes	300 hours
8	968C/3min + 1218C/24min + air cool/3min	600 hours
9	968C/3min + 1288C/24min + air cool/3min	300 hours

ASTM synthetic sea salt was added to the combustion air at a concentration of 5ppm (weight) by injecting an air atomized salt spray solution through a hole in the combustor's liner. Chemical analysis of the ASTM sea salt is provided in Table 2.

## Table 2. Composition of Synthetic Sea Salt Used in Burner Rig Hot Corrosion Tests.

Salt Constituent	Weight %
NaCl	58.49
MgCl2-6H2O	26.46
Na2SO4	9.75
CaCl2	2.765
KCI	1.45
NaHCO2	0.477
KBr	0.238
H3BO3	0.071
SrCl2-6H2O	0.095
NaF	0.007

Following exposure to the above environments, flexure specimens were water washed to remove any unreacted salt and photographed to document the extent of corrosion as a function of exposure time in the test.

## 3.3 Five atmosphere burner rig test

Gas turbine engines operate at internal pressures greater than one atmosphere. For this reason, a NASA Lewis Research Center burner rig that can be pressurized up to five atmospheres was also used in this study. Hot corrosion occurs in a temperature range between the melting point of the salt and the dew point.[1] An increase in the rig pressure increases the dew point, thereby widening the temperature range in which corrosion will occur. The 5-atmosphere burner rig has been used previously to study the sodium sulfate hot corrosion of silicon nitride and silicon carbide.[2-4] In those studies, which were focused on identification of corrosion mechanisms and effect of corrosion on strength, the four experimental variables (exposure time, exposure temperature, rig pressure, and salt concentration) were fixed.

The current study is different in that another salt (ASTM D1141-52 synthetic sea salt) is used, and measured effects of pressure (1, 3 and 5 atmospheres), salt concentration in the combustion air (2, 5 and 10 ppm), and exposure time are obtained to facilitate development and validation of a life prediction model. A series of short (2 hour) 5 atmosphere burner rig tests was conducted at higher temperatures with 5 ppm sea salt to experimentally establish the dew point for salt deposition.

The NASA rig consists of a standard Mach 0.3 burner [2] with the samples enclosed in a flame tunnel. Pressure is increased by closing an exhaust control valve.[3] Groups of specimens were exposed for up to 75 hours in a 980C five-atmosphere burner rig (and subsequently flexure tested at room temperature). In the 5 atmosphere tests Jet A fuel was burned at a fuel/air weight ratio of 0.025, and the combustion gas velocity was 53 m/sec. A total of eight samples were tested at each of the five exposure times. Sample size was 2.64 x 0.48 x 0.24 cm. The samples (four per run) were positioned horizontally with their thicknesses facing the flow. The sample ends were held in aluminosilicate (grade A lava) holders. The temperature was monitored with two type R thermocouples positioned between the specimens in the gas stream.[3] The salt was added by aspirating a salt water solution into the flame.

## 3.4 Flexure test

Exposed specimens were broken at room temperature in four-point flexure to quantify the effect of the environment on the surface strength of the NT154 silicon nitride. To facilitate testing of specimens exposed to hot corrosion conditions, silicate reaction products were removed from the loading pin locations. Specimens exposed for long times in the one-atmosphere burner rig required a combination of abrasive grinding (240 grit) and dilute HF acid etching. Cleaning of the tensile surface of the specimens was limited to water washing. Loading pin locations on specimens exposed in the five-atmosphere burner rig were cleaned by abrasive grinding with 180 grit. Specimens exposed in a furnace air oxidation environment received no post-exposure cleaning prior to testing.

Specimen geometries and room-temperature fourpoint bend flexure test conditions used to characterize effects of prior oxidation and hot corrosion environments are provided in Table 3.

## 3.5 Erosion test

Sand erosion tests were conducted using a GTCP85 auxiliary power unit. The test was conducted with a hybrid turbine nozzle consisting of 17 silicon nitride vanes and 6 superalloy vanes. The test was conducted for 9 hours at the maximum power setting. During this 9 hour period, AC coarse silica sand (0 to 200  $\mu$ m particle diameter) was injected into the combustor at a rate of 0.34 kilograms/hour.

#### 3.6 Post-test analysis

All flexure specimens were examined visually at a minimum 10X magnification in order to identify the fracture origin. Selected specimens were also examined under the SEM to further characterize the fracture origin and oxidation/hot corrosion microstructures. EDX and WDX spectroscopy were used during the SEM analyses to characterize elements present in the corrosion products.

Engine tested vanes were examined visually and with fluorescent penetrant for evidence of erosion and cracking.

## 4 Results and Discussion

## 4.1 Oxidation

#### Effect of oxidation on flexure strength

Following exposure in the static air furnace, the specimens were tested in four-point bending at room temperature. Test results are shown as lines of relative strength in Figure 1. Test data and failure origin locations for individual specimens are documented in Reference 5.

Unexpectedly, exposure to the oxidation environment increased strength, relative to as-received specimens, which had the standard Norton pre-oxidation heat treatment. Exposures in the 980 to 1200C range provided the largest improvement (up to a 38% increase) in strength. For longer exposure times (up to 100 hours) in the 1100C to 1400C temperature range, specimen strength decreased, but still remained above as-received values.

Figure 1 also illustrates unusual behavior for exposures longer than 100 hours in the 1100 to 1400C temperature range; the average specimen strength increased with additional exposure time. This behavior was unexpected since it was not observed in an earlier evaluation of NT154 specimens oxidized in a cyclic 1260C Mach 0.3 burner rig environment.[6] Following exposure to this cyclic burner rig test environment, which more closely simulates conditions within gas turbines, measured room temperature flexure strength continued to decline for exposures up to 1000 hours. The rate of strength reduction during 1000 hours in the burner rig environment was consistent with that of furnace exposed specimens for times up to 100 hours. Table 3. Four-Point Flexure Tests Were Used to Quantify the Effects of Environment on the Surface Strength of NT154 Silicon Nitride.

Type of Test	Specimen Geometry
Oxidation	3 x 4 x 50mm
1 atm burner rig	6.35x12.7x101.6mm
5 atm burner rig	2.4 x 4.8x 26.4mm

Post-exposure room temperature flexure strength was modeled as a function of exposure time and temperature, using data with exposures less than or equal to 100 hours. In this time range, constant strength lines in Figure 1 have constant values of T + 33.33\*ln[t], where T is the temperature in degrees C and t is the time in hours. Strengths for the 145 specimens with exposure times up to 100 hours was described with the following equation:

average flexure = 1690 - 0.614\* (T + 33.33\*Ln[t]) > 925 MPa strength G4700-74

Fractures in the as-received specimens, which were pre-oxidation heat treated by Norton, typically initiated in the chamfered edge of the specimen. Only seven percent of the as-received transverse machined specimens failed on the tensile surface. This result implies that machining damage was more severe on the chamfer than on the transverse machined surface.

Exposure to the high-temperature oxidizing environment reduced the severity of chamfer damage; i.e., over eighty percent of the environmentally exposed specimens failed on the tensile surface. When specimens associated with chamfer failure are eliminated from the database, scatter in the data is reduced, as shown in Figure 2.

#### **Oxidation Affected Surface Layer**

Although specimen weights were measured before and after exposure to the furnace air environment, weight changes were trivial -- within measurement error. Therefore, fracture surfaces were examined on the SEM to determine the thickness of the oxidation-affected layer. In this analysis, it was found that growth of the oxidation-affected layer of these specimens appeared to follow logarithmic instead of expected parabolic growth behavior. Wavelength dispersive X-ray (WDX) maps from fracture surfaces indicated that an oxygen gradient

Flexure	Test Parameters
Inner/outer Spans	Crosshead Speed
20/40 mm	0.5 mm/minute
19/38 mm	0.25 mm/minute
9.5/19 mm	0.5 mm/minute

existed in the surface layer of the silicon nitride. WDX analysis also indicated that the concentration of nitrogen in the oxygen-affected zone remained high -- almost to the level present below the oxygen-affected zone.

Applicability of logarithmic or parabolic growth kinetics in NT154 is the subject of an on-going investigation. At this time the kinetics remain ambiguous in a 1300C air environment. Parabolic growth was documented in a 1300C oxygen environment.

#### Discussion

Oxidation at temperatures below 1400C strengthened the surface of NT154 and reduced the severity of machining induced surface flaws. It is speculated that the increase in flexure strength is a consequence of oxidation induced compressive stresses. Examination of fracture surfaces indicated that oxidation in this temperature range did not occur as a plane front process; i.e., an oxygen gradient exists below the surface, which is thought to imply preferential oxidation within grain boundaries (and machining microcracks). Compressive stresses are expected when the volume expansion associ-



Figure 2. Oxidation Increased Flexure Strength of Transverse Machined Specimens and Reduced Incidence of Chamfer Initiated Failures.

ated with oxidation (conversion of silicon nitride to silicon oxynitride) in grain boundaries and microcracks is constrained by unoxidized silicon nitride grains. Maximum strength is anticipated when oxidation induced stresses aren't relaxed by deformation in the oxynitride grain boundary phase. At higher temperatures, stress relaxation in the oxynitride grain boundaries (e.g., by extrusion of grain boundary material to the free surface) may be responsible for the decreasing benefit of oxidation at temperatures in the 1200 to 1400C range. (Following exposure to 1371C oxidation, yttrium disilicate crystals were observed on specimen surfaces. Yttrium in these crystals was initially present in the grain boundaries.) At this time the above explation for strengthening remains speculative; i.e., compressive surface stresses in oxidized specimens has not been confirmed.

## 4.2 Hot Corrosion

In contrast with oxidation behavior, reaction between salt deposits and NT154 resulted in significant reduction in room temperature flexure strength. For tests conducted at 893C and above, the most severe loss in strength occurred during the initial hour(s) of exposure (Figure 3). The rate of strength loss then slowed significantly as exposure time increased.

During exposure to 860C burner rig combustion environment, salt deposited on the specimen, but remained virtually unmelted. EDX examination of salt deposits removed from specimens indicated that Na, K, Mg, Ca, and S were present. With the exception of isolated areas, salt deposits were easily removed by water washing. Visual examination confirmed that surface reaction with salt deposits was minimal. In contrast with other tests discussed below, strength reduction was minimal following exposure to the 860C hot corrosion.

Room temperature flexure strength data obtained following exposure of specimens to a range of 982C burner rig environments are illustrated in Figure 4. This figure shows that the surface strength is reduced by 30 to 40 percent during the first few hours of exposure to the hot corrosion environment. Following the initial 50 hours of exposure, room temperature strength decreased gradually -- even though the surface became roughened. Reducing the concentration of salt ingested into the burner results in a predictable increase in strength retention. Similar strength retention was predicted and observed for tests conducted at 1, 3 and 5 atmospheres; this result occurred because the product of salt deposition (increased by pressure) times salt basicity (reduced by pressure) remained constant.

The initial reduction in strength coincides with the initial formation of a glassy silicate layer by reaction between deposited salt and the silicon nitride. Glassy silicate reaction products on several specimens were qualitatively analyzed using the EDX and WDX spectroscopy capabilities of the SEM; results of these analyses indicated that the glassy silicates on the surface contain Na, K, Mg, Ca, Si, Y, O and N. Co, Ni, and Fe were also present in small concentrations in some spectrographs; these elements are present in the burner rig system.

It is speculated that the initial rapid reduction in strength may be attributed to relaxation of pre-oxidation strengthening (e.g., by elimination of compressive preoxidation stresses). Although the average depth of reaction during the initial hour(s) of exposure is minimal (less than 1 micron), reaction with the salt can transform the refractory yttrium silicon oxynitride grain boundary phases at the surface to a viscous (Na, K, Mg, Ca, Y, Si, O, N) glass.

Longer exposures result in surface roughening, which is associated with small variations in the corrosion rate of the NT154 silicon nitride and large variations in the distribution of the viscous silicate scale, which flows under the influence of impinging high-velocity combustion gases. Flow induced roughening of the surface of specimens exposed at 982C is shown in Figure 5. Despite the size and thickness of the silicate deposits, there was no obvious correlation between deposit thickness and the fracture origin. This result is attributable to the high level of gas porosity within the silicate scale, which reduces its strength and effectiveness as a stress raiser.

Surface recession was minimal (less than 25 microns) during exposure to 1 atmosphere burner rig hot corrosion at or below 982C. Above this temperature, surface recession was more rapid and may be life limiting. Surface recession (thickness loss) rates were approximately linear with time. Recession rate as a function of temperature are illustrated in Figure 6. Corrosion penetration is sufficiently rapid above 1000C to be life limiting for thin-walled (e.g., 625 microns [0.025 inch] thick) components.



Figure 3. Exposure to a Hot Corrosion Environment Reduced the Surface Strength of NT154 Silicon Nitride.



Figure 4. Surface Strength Reduction is Sensitive to Exposure Time and the Severity of the Corrosive 982C Burner Rig Environment.

Calculated dew points for the evaporation of sodium sulfate are approximately 950 and 1030C, respectively, for the 1 and 5 atmosphere test conditions used in this investigation. Consequently, a narrow temperature range for hot corrosion was initially expected. However, corrosive sodium rich silicate scales were unexpectedly present in the hot zones of specimens exposed 1050 and 1121C in the 1 atmosphere burner rig. Inspection of the specimens exposed in the 1218C test indicated that the actual dew point for a sodium-free surface was near 1200C at 1 atmosphere. In a series of short (2 hour) 5 atmosphere burner rig tests conducted with 5 ppm sea salt, the dew point was near 1290C. Reasons for the unexpectedly high dew point for sodium deposits are under investigation.

Multi-temperature 1 atmosphere hot corrosion exposure conditions (tests 8 and 9 in Table 1) more closely simulate idle and max power conditions anticipated for advanced gas turbines. In the multi-temperature tests, salt deposition occurred when the specimen was held at 968C for 3 minutes. Glassy silicates and severe surface recession were observed in the test with a 1218C maximum temperature. In contrast, exposure in the cycle



- 982C (1800F) Mach 0.3, 1 atm Burner Rig
- 5 ppm Sea Salt in Combustion Air
- Jet A Fuel

Cycle: 27 min Hot + min Forced Air Cool

G4032-5

## Figure 5. Glassy Silicate Layer Roughened the Surface of NT154 Specimens During Exposure to 982C Burner Rig Hot Corrosion Environment.

with a higher maximum temperature (1288C) promoted sodium-salt evaporation and inhibited formation of sodium silicate.

Surface recession ceased when the maximum test temperature inhibited sodium-salt deposition. A thin layer rich in magnesia, calcia, and silica was present on the hot zone surface of specimens exposed in tests 7 and 9 (Table 1). Reaction between the silica rich surface of NT154 and deposited Ca and Mg oxides roughened the interface. Small pits (about 25 microns deep), which containing trace amounts of sodium oxide, were observed on the fracture surfaces of a few specimens.

#### 4.3 Erosion

Engine tested NT154 silicon nitride exhibited superior resistance to sand erosion. As illustrated in Figure 7, ceramic vanes exhibited negligible damage in this erosion test. In contrast, all of the nickel-base superalloy vanes exhibited severe erosion of trailing edges. Based on these test results, particulate erosion will not be a significant damage mode for silicon nitride airfoils.

## 5 Life Prediction

While oxidation and hot corrosion from ingested sea salt attack silicon nitride substantially less than the superalloys being replaced in initial engine applications, this mode of degradation has the potential to limit component lives in advanced engine applications. Hot corrosion adversely affects the strength of silicon nitride in the 850 to 1300C range. Since unacceptable reductions in strength must be rapidly identified and avoided, we have pursued the development of an environmental life prediction model for silicon nitride turbine components. The following paragraphs provide a summary of model development.

The approach to modeling is similar to that used for predicting environmental lives of turbine airfoil coatings [7]. Overall, the rate of environmental (hot corrosion) degradation is modeled as the product of the severity of the environment (salt deposition times salt corrosiveness [basicity]) times stress dependent degradation mechanism (surface strength reduction and thickness loss in-



Figure 6. Surface Recession Rates Were High in NT154 Flexure Specimens Exposed to Hot Corrosion at Temperatures Above 982C.



Figure 7. Erosion Testing Demonstrated Superior Ceramic Capability.

duced rupture) functions. Operative temperature ranges for the degradation modes are illustrated in Figure 8.

In order to be a useful design tool, the environmental life prediction model has been formulated to utilize parameters recognized by those designing and operating the engine. Therefore, the model predicts the time for surface strength retention as functions of engine design (stress, temperature, pressure, fuel/air ratio, gas velocity, and inlet air filtration), mission usage (fuel sulfur content, location [salt in air], and times at duty cycle power points), and material parameters. Effects of design stress and inlet air filtration on the predicted life of NT154 silicon nitride components in a 10 atmosphere turbine

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Figure 8. A Mechanistic Model Predicts the Environmental (Strength Retention) Life of NT154 Silicon Nitride.

operating at a coastal location are indicated in Figure 9 and Figure 10 illustrate the current capabilities of the model. These preliminary results indicate a need for protective (salt resistant) coatings to facilitate use of silicon nitride turbine components in high temperature, corrosive combustion environments.

## Conclusions

- Silicon nitride has superior resistance to sand erosion in a gas turbine environment.
- Oxidation of NT154 in the 1000 to 1400C range is very slow and significantly increases room temperature flexure strength.

- Hot corrosion attack significantly reduces the strength of NT154 silicon nitride in the 850 to 1300C range.
- A mechanistic model was developed to predict the strength retention life of silicon nitride turbine components.
- The predicted environmental life of a component is dependent on engine design (stress, temperature, pressure, fuel/air ratio, gas velocity, air filtration efficiency), mission usage (fuel[sulfur content], location[salt in air], time at duty cycle power points), and material system parameters.



Figure 9. Predicted Environmental Lives of NT154 Silicon Nitride Components are Dependent on Design Stress and Temperature.

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Pressure at Max Power: 10 atm Time at Max Power: 90% Pressure at Idle: 5 atm Time at Idle: 10% Idle Temp = Max Power Temp <900C Idle Temp = 900C for Max Power Temp >900C Fuel: Jet A (0.035% S) Fuel/Air: 0.02 Gas Velocity: 850 ft/sec Location: Coast Altitude: Sea Level



Figure 10. Air Filtration Increases Predicted Environmental Lives of NT154 Silicon Nitride Components.

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