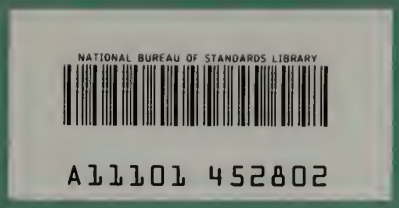

NBSIR 83-2664

Structural Reliability of Yttria-Doped, Hot-Pressed Silicon Nitride at Elevated Temperatures

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
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NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director*

STRUCTURAL RELIABILITY OF YTTRIA-DOPED, HOT-PRESSED SILICON NITRIDE AT ELEVATED TEMPERATURES

S. M. Wiederhorn and N. J. Tighe

ABSTRACT

The strength of yttria-doped, hot-pressed silicon nitride was investigated as a function of temperature and applied load. Data collected at 1200 °C are presented in the form of a strength degradation diagram for an applied stress of 350 MPa. At this temperature, the behavior of the yttria-doped material is found to be superior to that of magnesia-doped silicon nitride, in which creep results in the formation of microcracks that lead to strength degradation. By contrast, the yttria-doped material does not suffer from microcrack formation, or strength degradation at 1200 °C. At higher temperatures strength degradation does occur, and as a consequence, an upper limit of 1200 °C is recommended for yttria-doped, hot-pressed silicon nitride in structural applications.

1. INTRODUCTION

Modern structural ceramics are intended for use in applications such as heat engines and heat exchangers, in which mechanical and thermal loading can degrade the mechanical strength of these materials. The strength can also be degraded by exposure to hostile environments at elevated temperatures, which cause both localized and generalized chemical corrosion of ceramics. Although materials scientists and engineers are aware of the long term susceptibility of ceramic materials to strength degradation, the engineering techniques for addressing time-dependent strength degradation in ceramic materials are limited. The usual approaches that are used to improve lifetime employ empirically derived safety factors, or fracture mechanics based crack growth theories. Whereas both of these approaches are of value to predict structural reliability, experience in high temperature heat engine programs suggests that more effective lifetime prediction techniques are needed to assure the structural reliability of ceramics.

In an earlier paper, a new approach was suggested to improve component reliability.[1] The approach is more probabilistic in nature than either the fracture mechanics, or the safety factor approach, and is based on the assumption that both the applied stress and the strength of a set of components can be described by their own probability density function. The failure rate of a set of components is determined by the time evolution of the two probability density functions. Concepts of fracture mechanics can be used as part of this probabilistic approach to structural reliability to provide a mechanistic understanding of the time evolution of the probability density function for strength.

To characterize the mechanical behavior of a structural ceramic, both the strength and the time evolution of strength must be determined. As noted in reference 1, it is possible to represent this time evolution of strength graphically on a diagram that relates strength, exposure time and cumulative failure probability. Information on crack propagation and flaw generation can be incorporated into the method to give a picture of the time-evolution of component strength. Strength degradation diagrams (or maps) developed by application of the method give a general picture of the strength behavior of ceramics, and as such, are similar to the deformation maps and fracture maps first suggested by Ashby.[2]

An example of a strength degradation map is given in figure 1 for magnesia-doped, hot-pressed silicon nitride (NC 132)*. Details of the methods used to develop this map are given in references 1 and 5. The map represents the strength evolution of four-point, bend specimens subjected to an initial maximum fiber stress of 250 MPa at 1200 °C.** As can be seen from the figure, specimen strength is plotted as a function of exposure time. The curves in

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* The commercial designation of these materials (NC 132 and NCX 34) is presented for purposes of identification only, and does not imply endorsement by the National Bureau of Standards.

** The fiber stress is calculated from the simple elastic beam formula, which does not take into account the possibility of stress relaxation due to creep, or the possibility that creep in tension may differ from creep in compression.[3] A creep exponent N ~1 has been reported in the literature[4], for NCX 34 which supports the use of the simple elastic beam formula in the present study.

the figure represent the cumulative failure probability of specimens that were broken at temperature after specified times of exposure. In figure 1, 12 to 18 specimens were broken at each time increment to obtain sufficient statistical data to locate the failure probability curves. Also included in figure 1 are lines representing the applied stress, and the static fatigue limit. Failure of a component at a given probability level occurs when the probability curve representing the strength of a component, intersects the line for applied stress. Subcritical crack growth occurs when the strength of the component is less than the stress that corresponds to the static fatigue limit. Thus, in figure 1 subcritical crack growth is expected for specimens that have strengths between 250 and 350 MPa.

Figure 1 also suggests that there are several regions to the strength behavior. For periods less than 0.5 hours exposure, stress relaxation and crack healing in the vicinity of machining damage result in a strength increase. Between 0.5 hours and 2 hours of exposure the change in strength is slight, suggesting that relaxation in the vicinity of the machining flaws has been completed. Between 2 and 4 hours of exposure, pit generation in the ceramic surface caused by oxidation results in a radical change in the distribution of strength for the test specimens. Pit formation occurs quite rapidly and in a random way between 2 and 4 hours of exposure, so that there is no relation between the initial set of flaws caused by machining and the pits resulting from localized oxidation. Between 4 and 16 hours of exposure, the pits become less severe as sources of failure and the probability curves plateau suggesting that new pits are not generated after this time. After approximately 1 week of exposure, creep of the specimens results in the formation of cavitation cracks which gradually degrade the strength. Thus, for magnesia-doped, hot-pressed silicon nitride, there are at least 4 regions

of strength degradation: (1) subcritical crack growth; (2) machining flaws; (3) pits; and (4) cavitation cracks. An advantage of a diagram such as that shown in figure 1 is that it gives perspective to the strength behavior vis-a-vis potential strength degradation mechanisms. The diagram delineates those regimes of stress where peak overloads should be avoided and the time frame over which strength degradation is to be expected.

The objective of the present study is to present a similar set of strength data obtained on yttria-doped, hot-pressed silicon nitride (NCX 34). This material was selected for study because of its excellent resistance to creep at elevated temperatures.[5] Furthermore, the material does not suffer strength degradation as a result of pit formation at elevated temperatures. Some billets of yttria-doped, hot-pressed silicon nitride, however, do suffer from strength loss at an intermediate temperature ~700-900 °C as a result of grain boundary cracking.[6-8] As will be shown, the billets used in the present set of studies did not suffer from this type of strength degradation. Strength data for the yttria doped material will be presented in the form of a strength degradation diagram, which highlights the difference in behavior of the two materials at 1200 °C.

2. EXPERIMENTAL PROCEDURE

Investigations were conducted on two billets of yttria-doped, hot-pressed silicon nitride, which were cut into both strength test-bars (3 X 4 X 50 mm) and fracture mechanics, double-torsion, test plates (2 X 25 X 75 mm). The double torsion specimens were grooved (2 mm wide by 1 mm deep) to control the direction of crack growth during fracture. The bars were tested in four point bending using a silicon carbide bend fixture with an upper and lower span of 10 mm and 40 mm respectively. All test bars were randomly selected from their

respective billets to avoid systematic errors resulting from point-to-point variation in the billets. Thirty double torsion test plates and 80 strength test bars were cut from one billet, herein designated as billet A, whereas, 150 strength test bars were cut from the second billet, billet B.

The data for the strength degradation maps were obtained primarily from billet B. Strength studies were conducted using a test facility developed for this purpose.[5] The facility uses pneumatic, "frictionless" bellows to apply the load to each specimen, and an electronic load cell to measure the load. Because the loading system is compact, three such systems were used in each furnace to obtain strength data. Using four furnaces, 12 specimens could be tested simultaneously. By attaching linear differential transformers to each silicon carbide loading ram, the displacement of each specimen could be monitored during exposure.

The data for the strength degradation map were obtained by subjecting each specimen to a sustained load, at a given temperature for a fixed period of time. Most of the strength data were obtained at 1200 °C using an initial applied stress of 350 MPa. However, data were also obtained as a function of temperature and applied load. Strength data at elevated temperatures were obtained by exposing and breaking the specimens at temperature without first cooling them to room temperature.

Billet A was used for several different studies. The double torsion specimens were used to obtain measurements of the critical stress intensity factor, K_{IC}, at room temperature; specimens were broken at a constant rate of loading[9]. Based on 7 measurements, values obtained for K_{IC} were 5.4 MPa-$m^{1/2}$ with a standard deviation of 0.3 MPa-$m^{1/2}$.

Strength test bars from billet A were used to evaluate the effect of mechanical damage on the strength. Specimens were indented with a Knoop

indenter using a load of 20 N before exposing them to elevated temperatures. The crack that forms beneath the Knoop indentation simulates the type of damage that normally occurs when ceramics are machined. By studying the effect of load, temperature, and exposure time on the strength of specimens that contain indentation cracks, the effect of high temperature exposure on machining damage could be elucidated. The technique was also used to establish the static fatigue limit for this material at 1200 °C.

3. RESULTS

Catastrophic failure of yttria-doped, hot-pressed silicon nitride due to chemical instability has been reported[6-8] in the temperature range, 700-900 °C. To investigate this possibility in the present study, exposure tests were conducted in the temperature range 600 °C through 1300 °C. After 168 hours of exposure, specimen strength was measured at elevated temperature. At temperatures of 600 °C through 1000 °C, and at 1300 °C, three specimens were used for each strength measurement. At room temperature, ten specimens were used, while at 1200 °C six were used (the six at 1200 °C were subject to an applied load of 350 MPa for 100 hr). As can be seen from figure 2, there is no evidence of strength loss in the intermediate temperature range of interest. In fact, the strength appears to be higher in this temperature range than at room temperature. The drop in strength at ~1300 °C is typical of hot-pressed silicon nitride tested at elevated temperatures.[10]

Examples of the probability distribution curves used to plot the strength degradation map are given in figures 3 and 4. At room temperature a bimodal distribution curve is obtained; the slope of the curve at low levels of probability is less than the slope at high levels of probability. A similar bimodal probability distribution for NCX 34 was reported recently by Easler et al.[11] After the specimens are exposed at 1200 °C a unimodal strength

distribution is obtained (figure 4). The weak specimens from the low probability end of the distribution in figure 3 are eliminated from the distribution in two ways: (1) crack growth, in which case the weak specimens break while subjected to load at elevated temperatures; (2) crack "healing", in which case the strength of specimens increase as cracks get shorter or blunter. Of these two possibilities, the second appears to have dominated the present study.

Crack healing can be demonstrated by testing specimens that have been intentionally weakened with a Knoop indentation (using a load of 20 N) before testing. Data from a set of specimens that has been tested in this way are illustrated in Table 1. As can be seen from the table, specimens that were exposed to 1200 °C in air for at least 100 hours, were stronger by ~50 percent (~490 MPa → ~740 MPa) after exposure to high temperatures. The increase of strength occurs regardless of the level of the load applied to the specimens during the annealing treatment. In some cases, healing was so complete that the fracture did not initiate from the indentation, but from some other flaw at the surface. The fact that an initial applied stress of 450 MPa could be sustained by the specimens without failure during the 100 hour annealing period, suggests that at 1200 °C subcritical crack growth in the yttria doped material is minimal, i.e. the $v\text{-}K_I$ curve probably has a steep slope at this temperature.

Despite the above observation of crack healing, crack growth must have occurred in some of the specimens tested in the "as-ground" condition because of the breakage that occurred during the annealing period. Of the 78 specimens that were subjected to 350 MPa at 1200 °C, two broke within the first 5 minutes of the test, probably as a result of subcritical crack growth from surface flaws; a third broke after ~43 hours of exposure. In total,

approximately 4 percent of the specimens broke in the course of the test. This small number of failures supports the suggestion that crack growth in yttria-doped silicon nitride is minimal at 1200 °C.

The strength degradation map for the yttria-doped, hot-pressed silicon nitride is given in figure 5. The static fatigue limit in figure 5 was determined from the data in Table 1. Based on the fact that specimens with a mean strength of ~486 MPa were able to sustain a load of 450 MPa for 100 hours, we estimate that the static fatigue limit had to be less than ~1.08 (486/450) times the applied stress, i.e. ~380 MPa in figure 5. Thus, the fatigue limit and the applied stress are nearly the same for NCX 34, suggesting that at 1200 °C subcritical crack growth plays a minor role in failure process. This conclusion is supported by stress rupture data obtained on NCX 34 by Quinn[12], who showed that rupture of this material occurred over a very narrow range of applied stresses at 1200 °C.

By comparing figure 5 with figure 1, we see that the fracture behavior of the yttria doped, hot-pressed silicon nitride differs considerably from that of the magnesia-doped material. After a slight decrease in strength for short exposure times (<2 hr), the strength of the yttria doped material remains relatively constant with exposure time. Between 100 and 1000 hours of exposure, the strength increases significantly. This long-term behavior is in sharp contrast to the results obtained for the magnesia doped material, which exhibits a serious drop in strength after ~100 hours of exposure. By comparing the diagrams for the two materials, the mechanical superiority of the yttria doped material at 1200 °C is apparent.

The effect of temperature on the strength degradation of NCX 34 was studied by collecting strength data as a function of temperature for a fixed sustained load of 350 MPa and an exposure time of 100 hours. Figure 6

illustrates the dramatic effect of temperature on strength. Below 1200 °C, the shape of the curve is very similar to that obtained for specimens that were exposed to elevated temperatures, but which were not forced to support an applied stress (figure 2). The strength at 1250 °C is about the same as at 1200 °C for all probability levels. However, between 1250 °C and 1300 °C, the strength drops precipitously. Of the six specimens tested at 1300 °C, five broke in the course of the test; only one survived the full 100 hours at 350 MPa. The one survivor was well on its way to failure since its strength, 395 MPa, was only ~45 MPa above the applied stress. For the specimens that failed at 1300 °C, the failure times were: 0.25, 1, 1.5, 3 and 6 hours; there was no prior warning from time-displacement curves of any of these specimens that failure was imminent.

Failure of the silicon nitride at 1300 °C appears to be related to the relative ease of creep deformation of this material at this temperature. Measurements of specimen displacement as a function of time and temperature suggest that the displacement is a power function of the exposure time, figure 7, and that the creep rate increases as the temperature is increased. This parabolic creep behavior occurred over the entire period of measurement (~100 hours). In agreement with other studies on hot pressed silicon nitride,[13-15] steady state creep was not observed within this time frame. A plot of the logarithm of the displacement rate, $\ln \dot{\delta}$, as a function of $(RT)^{-1}$ indicates an Arrhenius behavior with an apparent activation energy for creep of ~110 kcal/mol figure 8. Both the absence of steady state creep, and the apparent activation energy are consistent with earlier measurements on other grades of hot-pressed silicon nitride.[4,13-15] With regard to the failure that occurred at 1300 °C (fig. 6), the higher creep rate shown in figures 7 and 8 correlates with the shorter lifetime of the specimens at higher temperatures. However,

as noted below, a quantitative comparison of component lifetime with creep rate was not achieved, so it appears that lifetime and creep rate are not simply related for NCX 34.

4. DISCUSSION OF RESULTS

The mechanical behavior of ceramic materials is considerably more complicated at elevated temperatures than at low temperatures.[16] Failure occurs by diffusive creep or by viscous flow processes that control crack nucleation and crack growth at elevated temperatures. As noted by Evans,[16] the processes that affect failure include the nucleation and growth of cavities, the linking up of cavities to form small cracks, and the growth of small cracks (or pre-existing flaws) to form critical size cracks for spontaneous failure. Provided these processes occur sequentially, the slowest one constitutes the rate limiting step in the failure process.

Extensive theoretical investigations on the subject of high temperature failure suggest that the failure of structural ceramics is in many ways similar to that in metals.[16] Diffusion at elevated temperatures appears to control cavitation creep, which eventually results in creep rupture in both metallic and ceramic materials. Therefore, rupture by cavitation creep and crack growth is expected to be common to both types of materials. Extensive experimental studies on metals have shown that creep rupture in tension can be described by parametric relationships between failure-time, t_f, temperature, T, and steady-state creep rate, $\dot{\varepsilon}$. Two such relations are the Monkman-Grant relation,[17]

$$t_f \dot{\varepsilon} = \text{constant} \qquad (1)$$

and the Orr-Sherby-Dorn relation,[18]

$$t_f \exp(-Q/RT) = f(\sigma) \qquad (2)$$

The term, Q, in equation (2) is the activation energy for the rate limiting step in the failure process.

Recent theoretical investigations suggest that equations similar to 1 and 2 also apply to creep rupture of ceramic materials at elevated temperatures.[16,19] Since all of the proposed failure mechanisms are activated processes, the form of the equation for failure is identical to equation 2 with regard to temperature. However, the equations may be quite different with regard to applied stress, σ, because $f(\sigma)$ depends on the failure mechanism. Thus, for steady state viscous creep, $\dot{\varepsilon} \propto \sigma$ and $t_f \propto \sigma^{-1}$. By contrast, $t_f \propto \sigma^{-14}$ if crack growth is involved in the rate limiting step[20].

In a qualitative way, equation 2 describes the failure results of the present study. As the temperature is increased, the exponential term in equation 2 also increases, requiring a decrease in the failure time for the equality to hold. The relative failure times at 1200 °C and 1300 °C can be estimated from the present set of data. At 1300 °C, the median failure time of the yttria-doped, hot-pressed silicon nitride was ~ 2 hours. Assuming that equations 1 and 2 apply, and that Q is given by the activation energy for creep (110 kcal/mol), a failure time of ~20 hours is predicted at 1200 °C. Although this prediction is in the right direction, it does not agree with our finding that the failure time at 1200 °C exceeded ~1000 hours. Thus, the theoretical analyses embodied in equations 1 and 2 are not adequate to explain the data obtained in the present study. We suspect that the failure mechanism changed as the temperature was increased from 1200 to 1300 °C. This change in

mechanism accounts for the lack of agreement between our stress rupture data and equation 2. Further studies will be needed to identify the mechanism of failure in NCX 34 at 1300 °C. This finding is in marked contrast to the results on magnesia-doped, hot-pressed silicon nitride (NC 132) published recently by Quinn and Quinn.[21] Using an activation energy of ~168 kcal/mol, these authors showed that stress rupture data from this material fit equation 2 over the temperature range 1100 to 1300 °C.

The difference in stress-rupture behavior of the magnesia and yttria doped material results from differences in the microstructure and chemical composition of the two materials.[8,22-24] High-resolution, transmission electron microscopy studies have shown that magnesia-doped, hot-pressed silicon nitride has a thin layer of vitreous silicate at grain boundaries and packets of vitreous silicate at triple junctions of the material. At elevated temperatures, the vitreous phase softens resulting in a reduction of the mechanical load capacity of the material due to creep. By contrast, the structure of the yttria-doped materials is more complex. Second phase, crystalline yttrium silicon-oxynitride is usually observed at the triple grain junctions. The crystalline structure is, however, very sensitive to the exact composition of the material because of the complex phase diagram that controls the Y_2O_3-SiO_2-Si_3N_4 structures. In some yttria-doped, materials an amorphous phase has been detected at the grain boundaries; in others (NCX 34), no amorphous phase is apparent. The consequence of adding yttria to silicon nitride is to produce a material that is resistant to creep and creep fracture at elevated temperatures. Of the two materials discussed, the yttria doped material is far more resistant to creep and creep-cracking at 1200 °C. The microstructures of the two materials after similar amounts of deformation at 1200 °C are illustrated in figure 9. In the magnesia doped material (fig. 9a) microcracks are common at grain boundaries that are perpendicular to the

applied tensile stress. In some cases, large voids open at triple point junctions, and microcracks link up to form cracks that extend over many grains of the material. By contrast, the yttria doped material (figure 9b) maintains its integrity despite the deformation. There is no evidence for void, or microcrack formation in this material, which suggests that the yttria doped, hot-pressed silicon nitride exhibits sufficient ductility (via dislocation motion, diffusive creep, or grain boundary sliding) at 1200 °C to avoid the microcrack formation that is the main cause of long-term strength degradation in the magnesia doped material.

From a practical point of view, the results in figure 6 suggest a sharp limit to the allowable temperature for the use of the yttria doped material in structural applications. At temperatures below 1250 °C, the strength of this material is virtually identical to that of specimens that were not subjected to external loads during exposure (figure 2). Furthermore, the strength does not appear to degrade with time. Above 1250 °C, however, the drop in strength was so precipitous at an applied stress of 350 MPa, that for practical purposes the strength should be considered to be zero for long term structural applications. This finding is consistent with the results reported by Quinn,[10] who used a stepped temperature, stress rupture test to study the mechanical response of NCX 34 to temperature. In order to use NCX 34 at temperatures in excess of 1300 °C, the applied stress had to be reduced substantially (<200 MPa for 1300 °C). Considering these two sets of data, it may be concluded that 1200 °C is a safe upper limit for the temperature at which NCX 34 can be used in structural applications.

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REFERENCES

1. S. M. Wiederhorn, "A Probabilistic Framework for Structural Design", in Fracture Mechanics of Ceramics, Vol. 5 and 6, R. C. Bradt, A. G. Evans, D.P.H. Hasselman, and F. F. Lange eds., Plenum Publishing Corp., New York (1983).
2. M. F. Ashby, "First Report on Deformation-Mechanism Maps," Acta Met. 20, 887 (1972).
3. G. W. Hollenberg, G. R. Terwilliger and R. S. Gordon, "Calculation of Stresses and Strains in Four-Point Bending Creep Tests," J. Am. Ceram. Soc. 54, 196-9 (1971).
4. D. C. Larsen and J. W. Adams, "Property Screening and Evaluation of Ceramic Turbine Materials," Semi Annual Technical Report No. 9, Contract F33615-79-C-5100, prepared for U.S. Air Force, Wright Aeronautical Laboratories, Wright Patterson AFB, Dayton, Ohio 45433, by: IIT Research Institute, Materials Technology Division, Chicago Illinois 60616, December 1980.
5. N. J. Tighe and S. M. Wiederhorn, "Effects of Exposure on the Reliability of Silicon Nitride", in Fracture Mechanics of Ceramics, Vol. 5 and 6, R.C. Bradt, A.G. Evans, D.P.H. Hasselman and F.F. Lange, eds., Plenum Publishing Corp., New York (1983).
6. G. E. Gazza, ""Hot Pressed Si_3N_4," J. Amer. Ceram. Soc. 56, 662 (1973).
7. F. F. Lange, S. C. Singhal and R. C. Kuznicki, "Phase Relations and Stability Studies in the Si_3N_4-SiO_2-Y_2O_3 Pseudoternary System," J. Am. Ceram. Soc. 60, 249-52 (1977).

8. N. J. Tighe, K. Kuroda, T. E. Mitchell and A. H. Heuer, "In-Situ Oxidation of Y_2O_3-Doped Si_3N_4," pp. 310-13, Electron Microscopy 1980, Vol. 4, High Voltage, P. Brederoo and J. Van Landuyt, eds., North Holland Publishing Co., New York (1980).

9. A. G. Evans and S. M. Wiederhorn, "Crack Propagation and Failure Prediction in Silicon Nitride at Elevated Temperatures," J. Mat. Sci., 9, 270 (1974).

10. A. F. McLean, E. A. Fisher, R. J. Bratton and D. G. Miller, in Brittle Materials Design, High Temperature Gas Turbine, Technical Report AMMRC CTR 75-28 to Army Materials and Mechanics Research Center, Watertown, MA, Oct. 1975.

11. T. E. Easler, R. C. Bradt, and R. E. Tressler, "Effects of Oxidation and Oxidation under Load on Strength Distributions of Si_3N_4," J. Amer. Ceram. Soc. 65, 317-319 (1982).

12. G. Quinn, "Characterization of Turbine Ceramics after Long Term Environment Exposure", Army Materials and Mechanics Research Center, AMMRC TR 80-15, April 1980.

13. R. Kossowsky, D. G. Miller, E. S. Diaz, "Tensile and Creep Strengths of Hot-Pressed Si_3N_4," J. Mat. Sci. 10, 983-997 (1975).

14. M. S. Seltzer, "High Temperature Creep of Silicon-Base Compounds," Bull. Am. Ceram. Soc., 56, 418-423 (1977).

15. B. S. B. Karanaratne and M. H. Lewis, "High Temperature Fracture and Diffusional Mechanisms in Si-Al-O-N Ceramics," J. Mat. Sci. 15, 449-462 (1980).

16. A. G. Evans, "Elevated Temperature Fracture," in Fracture Mechanics of Ceramics, Vol. 5 and 6, R.C. Bradt, A.G. Evans, D.P.H. Hasselman and F.F. Lange, eds., Plenum Publishing Corp., New York (1983).

17. F. C. Monkman and N. J. Grant, "An Empirical Relationship between Rupture Life and Minimum Creep Rate in Creep-Rupture Tests," Proc. ASTM, 56, 593-620 (1956).

18. R. L. Orr, O. D. Sherby, and J. E. Dorn, "Correlations of Rupture Data for Metals at Elevated Temperatures," Trans., ASM, 46, 113-128 (1954).

19. A. G. Evans and C. H. Hsueh, "Creep Fracture in Ceramic Polycrystals," in Creep and Fracture of Engineering Materials and Structures, Pineridge Press, Ltd., Swansea, U.K., 1981, pp. 409-431.

20. T.-J. Chuang, K. I. Hagawa, J. R. Rice and L. B. Sills, "Non-Equlibrium Models for Diffusive Cavitation of Grain Interfaces," Acta Met. 27, 265-284 (1979).

21. G. D. Quinn and J. B. Quinn, "Slow Crack Growth in Hot-Pressed Si_3N_4," in Fracture Mechanics of Ceramics, Vol. 5 and 6, R.C. Bradt, A.G. Evans, D.P.H. Hasselman and F.F. Lange, eds., Plenum Publishing Corp., New York, (1983).

22. D. R. Clarke and G. Thomas, "Grain Boundary Phases in a Hot-Pressed MgO Fluxed Silicon Nitride," J. Amer. Ceram. Soc. 60, 491-5 (1977).

23. D. R. Clarke and G. Thomas, "Microstructure of Y_2O_3 Fluxed Hot-Pressed Silicon Nitride," J. Amer. Ceram. Soc. 61, 114-118 (1978).

24. N. J. Tighe, "Analysis of Oxide and Oxide/Matrix Interfaces in Silicon Nitride," NBSIR 82-2574, July 1982, to be published in The Proceedings of the American Ceramic Society, 1983.

Table 1: Indentation Study

Test Condition	Strength (MPa)	Fracture Origin
Room Temperature	491 (16)*	Indentation
1200°C (1/2 hr.)	486 (70)	Indentation
1200°C (100 hr.)	743 (25)	2 at Indentation 1 not at Indentation
1200°C (163 hr. 450 MPa Load)	699 (52)	6 at Indentation 3 not at Indentation

*Number in parentheses gives the standard deviation of the measurement.

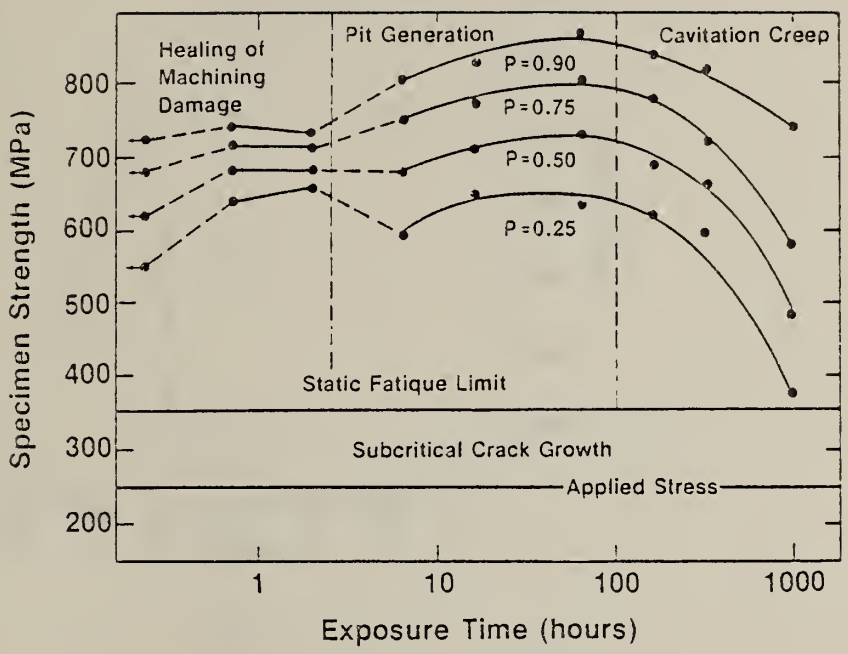

Fig. 1. Strength degradation diagram for magnesia-doped, hot-pressed silicon nitride (NC 132). Temperature of exposure 1200 °C; applied load 250 MPa.

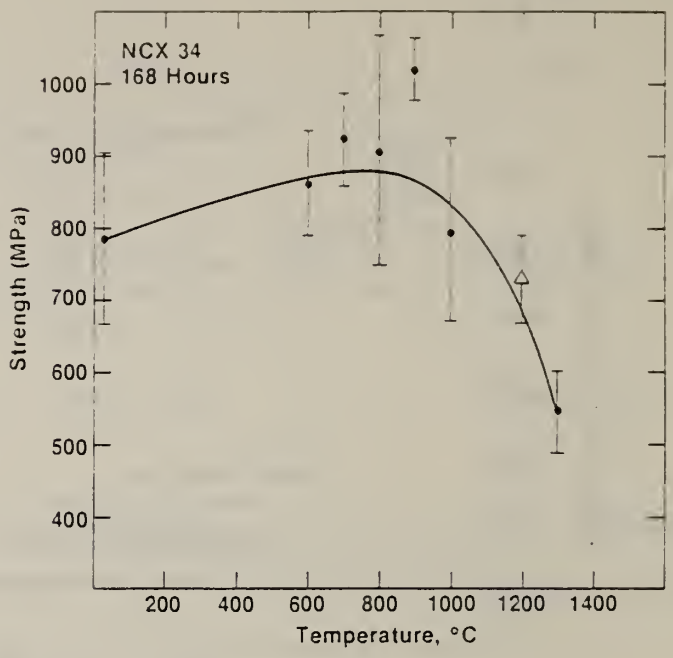

Fig. 2. Strength of NCX 34 after exposure to test temperature for 168 hours. Specimens were exposed and tested at temperature without cooling to room temperature.

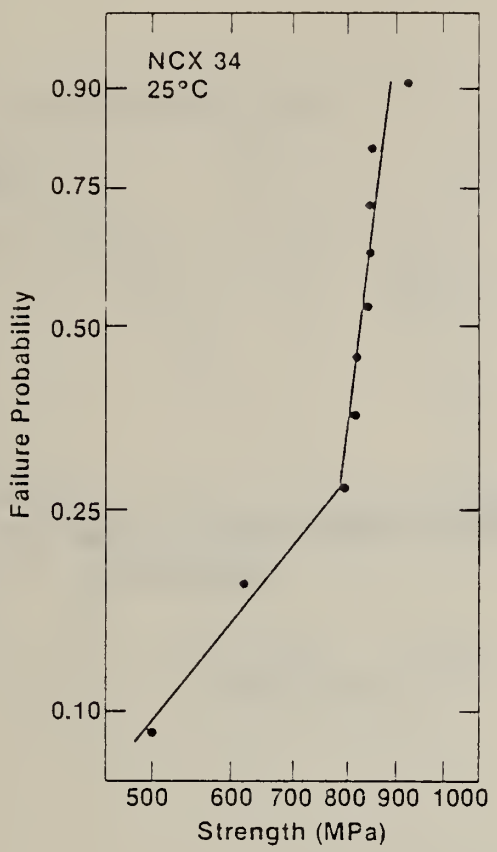

Fig. 3. Initial room temperature strength distribution of yttria-doped, hot-pressed nitride.

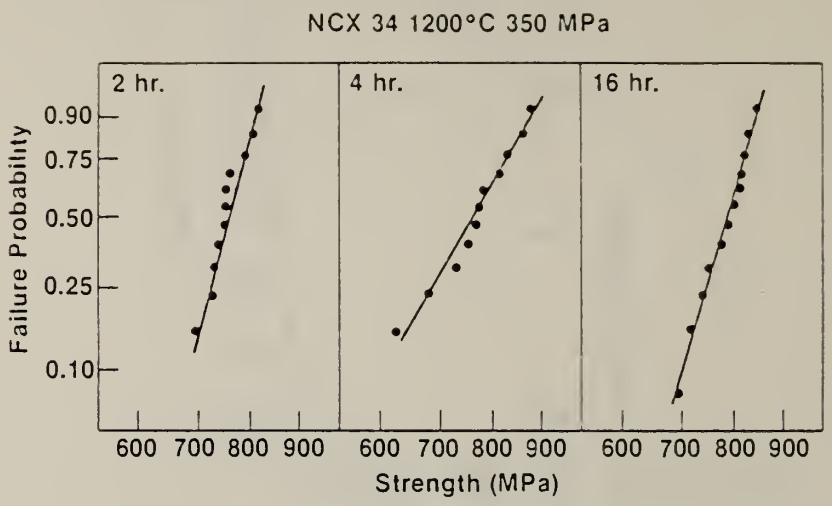

Fig. 4. Strength distribution after 2, 4, and 16 hours of exposure at 1200 °C at a sustained load of 350 MPa.

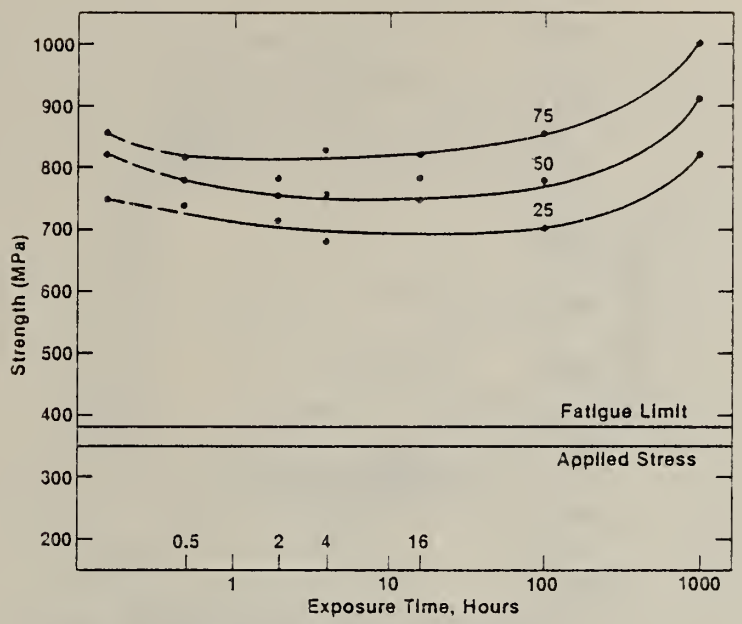

Fig. 5. Strength degradation diagram for yttria-doped, hot-pressed silicon nitride (NCX 34). Temperature of exposure 1200 °C; applied stress 350 MPa.

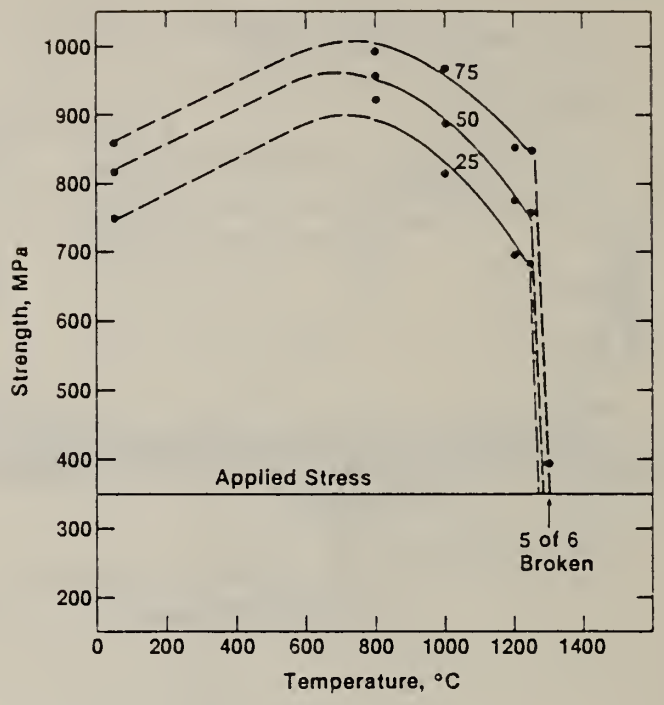

Fig. 6. Effect of temperature on the strength of NCX 34 which is subject to a sustained load of 350 MPa for 100 hours.

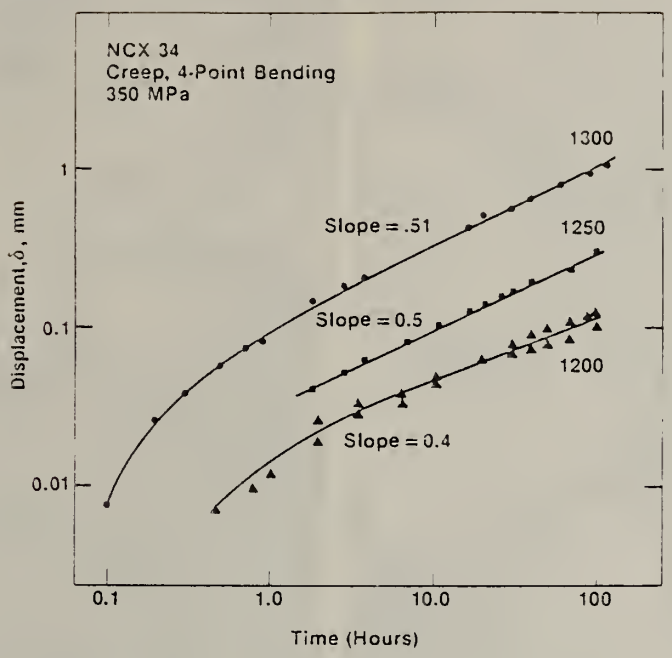

Fig. 7. Creep behavior of NCX 34 at 350 MPa maximum fiber stress. Center point displacement as a function of time.

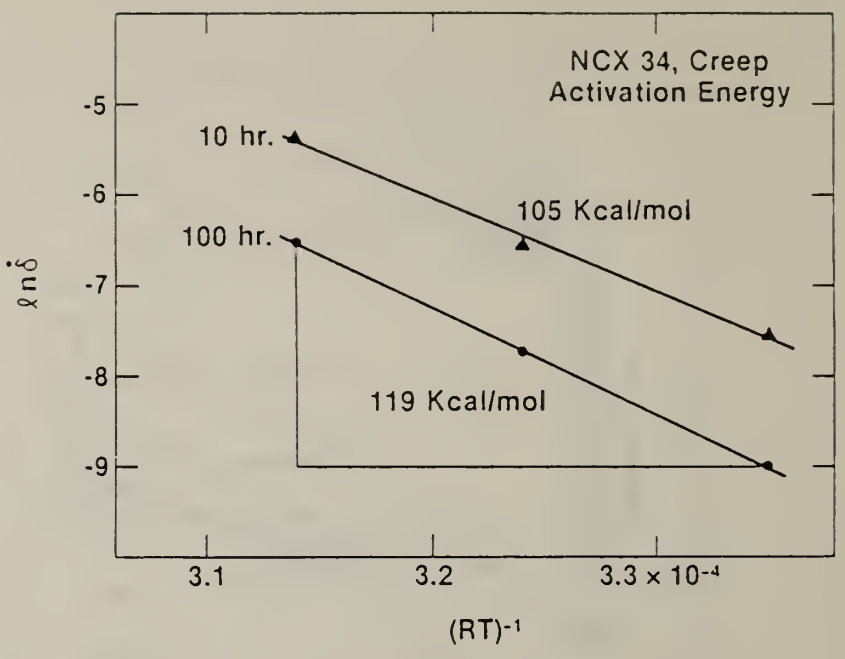

Fig. 8. Estimate of the activation energy for creep for NCX 34.

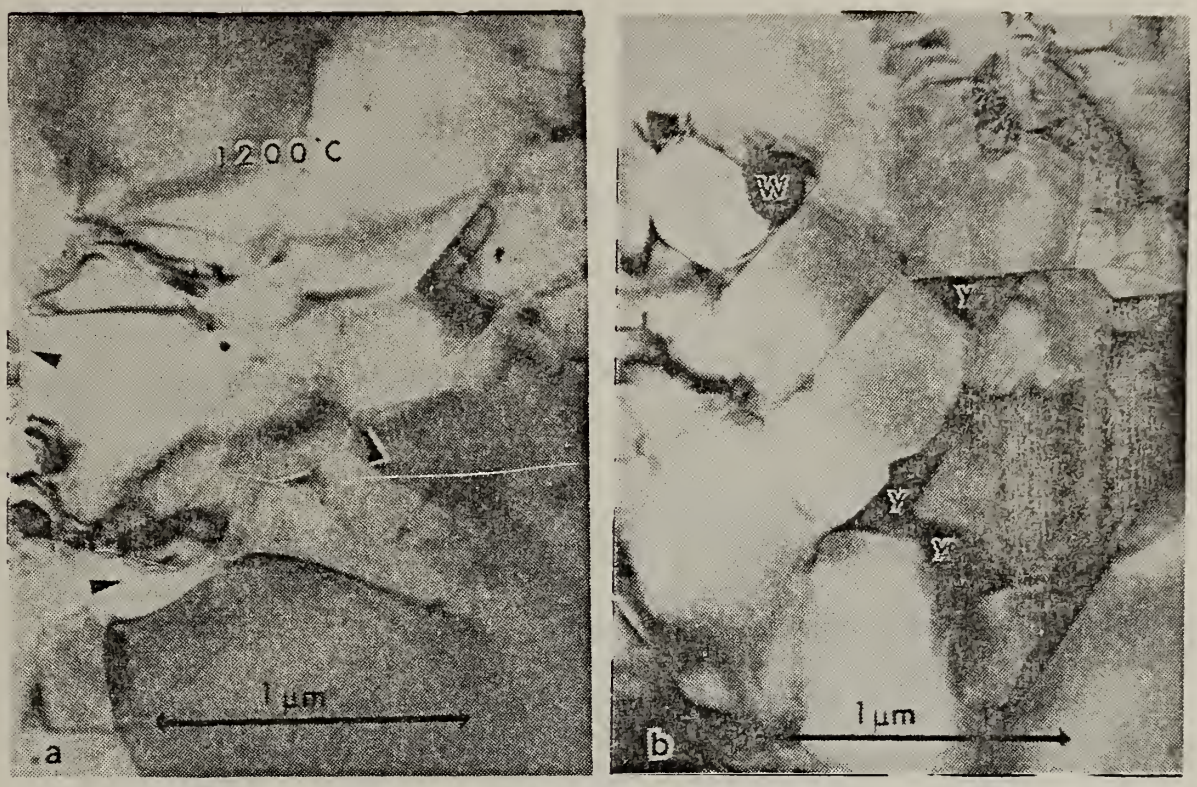

Fig. 9. Transmission Electron Micrographs of hot-pressed silicon nitride: (a) NC 132, (b) NCX 34. The arrows in (a) indicate boundries within the foil that have cracked as a result of creep. The yttria doped material (b) gives no evidence for crack formation. Y indicates the yttria rich phase; W indicates the tungsten rich phase.

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☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)

The strength of yttria-doped, hot-pressed silicon nitride was investigated as a function of temperature and applied load. Data collected at 1200 °C are presented in the form of a strength degradation diagram for an applied load of 350 MPa. At this temperature, the behavior of the yttria-doped material is found to be superior to that of magnesia-doped silicon nitride, in which creep results in the formation of microcracks that lead to strength degradation. By contrast, the yttria-doped material does not suffer from microcrack formation, or strength degradation at 1200 °C. At higher temperatures strength degradation does occur, and as a consequence, an upper limit of 1200 °C is recommended for yttria-doped, hot-pressed silicon nitride in structural applications.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

ceramics; creep; creep rupture; fracture; silicon nitride; strength

13. AVAILABILITY

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