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Effect of Flaw Generation on Proof-Testing

S. M. Wiederhorn and N. J. Tighe

Inorganic Materials Research
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National Bureau of Standards
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Interim Report

Prepared for
United States Air Force
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ABSTRACT

Proof testing is investigated as a method of assuring the structural reliability of components made of hot pressed and reaction bonded silicon nitride. At 25°C, proof testing is shown to be a good procedure for truncating the strength distributions of these materials. At 1200°C, however, the effect of oxidation on the flaw structure of silicon nitride must be taken into account before proof testing can be applied to structural design. An upper limit to the proof test stress is recommended for silicon nitride because of flaw generation and flaw modification at elevated temperatures.

Introduction

Proof-testing is one of the methods being seriously considered as a means of improving the reliability of ceramic components for gas turbines. Proof-testing is used to eliminate weak components before they are placed in service, since it is these components that fail in service. In order to break the weak components, a proof-test stress that is higher than that expected in service is applied to every component. By breaking components that contain flaws greater than a certain maximum size the proof-test stress truncates the strength distribution leaving only the strong ones to be put into service. The theory of proof-testing provides a unique relation between the proof-test stress, the service stress and the predicted time-to-failure. This relation enables engineers to establish minimum conditions for component lifetime in service (1,2).

The theory of proof-testing is based on the assumption that (1) the population of flaws remains invarient after the proof-test has been conducted and (2) the flaw population tested is the same one that causes failure under service conditions (2). There are, however, several reasons why this condition might not be fulfilled: the component might suffer mechanical damage after the proof-test; the stress distribution during the proof test might not duplicate that which occurs during service; and finally, environmental conditions might alter the flaw distribution. In silicon nitride, which is to be used in high temperature oxidizing environments, the effect of oxidation on the flaw population should be considered before proof-testing is accepted as a screening procedure. This paper presents the results of a study on the effect of elevated temperatures on the strength of silicon nitride. The results of this study are used to evaluate proof testing as a method of lifetime assurance.

Experimental Procedure

Test specimens were made from both reaction bonded (NC350) and hot-pressed (NC132) silicon nitride. Two billets of the hot pressed material were used in these studies. Although they were manufactured one-year apart, the two billets had the same nominal composition. However, the earlier billet (billet A) was made from a powder lot that had twice as much calcium and aluminum as the later billet (billet B).

Test specimens were bars (approximately 4x5x50 mm) that were cut from the billets and ground along their length with a 180 grit diamond wheel. Strength measurements and proof tests were conducted in air at 25°C and 1200°C using four-bending. Specimens

that passed the proof test without breaking were left on the test fixture without altering their position, so that the stress distribution during proof testing was duplicated during the strength measurements. To determine the effect of temperature on strength, billets were tested at 25°C and at 1200°C after 100 hours of exposure in air at 1200°C. High temperature measurements were made without first cooling the test specimens to room temperature. Finally, in order to determine the effect of temperature on large flaws, artificial cracks were introduced into the specimens by means of a Knoop hardness indenter.

Experimental Results

Proof Testing

The effect of proof testing on the strength distribution at 25°C is demonstrated in figure 1a for hot-pressed silicon nitride and in figure 1b for reaction bonded silicon nitride. As can be seen in these figures, proof-testing truncates the strength distribution for both types of silicon nitride. In all proof-test studies conducted at 25°C, the breaking strengths of components were greater than the proof-test stress. This finding indicates that proof-testing can be used for room temperature applications to eliminate weak components and thus improve the structural reliability of silicon nitride.

For turbine applications, proof-testing must also be able to truncate the high temperature strength distribution to assure component reliability. In principle, the easiest way to obtain such a distribution is to proof-test the components at room temperature before using them at high temperature. Then provided the high temperatures do not effect the flaw distribution, the truncated population formed at room temperature will also be truncated at high temperatures (3). To test this possibility, sets of components from both hot pressed silicon nitride (billet A) and reaction bonded silicon nitride were proof tested at room temperature and broken at 1200°C after 1/2 hour of high temperature exposure. The results of this study are shown in figure 2. Included in figure 2 are the theoretically predicted values of the 1200°C strength (the curved lines) after proof testing. We note from this figure that the strength values determined after proof testing did not agree with the theoretical curves. These results suggest that the high temperatures used in these tests effect the population of flaws that cause failure at elevated temperatures. In the light of these results the applicability of proof testing (or nondestructive evaluation techniques) to high temperature structural reliability must be reexamined.

Proof-testing improves the reliability of ceramic components by eliminating components that contain large flaws. In effect, the proof-test load determines the maximum size flaw that can be present in a specimen that passes the proof test. As long as this flaw is still an effective nucleus for fracture after exposure, the minimum time to failure can be estimated by proof testing procedures. If, however, the flaw is modified by the high temperature exposure then the predicted lifetime will differ from the measured lifetime, and proof testing might not be a viable screening procedure. The results of the tests shown in figure 2 indicate that specimens are weaker than expected theoretically, which means that the initial flaw structure of the material has, in fact, been modified by the high temperature exposure. Thus, proof testing will be of doubtful value at the level of strength used to obtain the data shown in figure 2. However, at lower values of strength, (larger flaws), proof testing may still be of value for improving the reliability of structural ceramics. To ascertain the value of proof testing, it must be shown, that after exposure large flaws are still effective nucleation sites for fracture.

To determine if proof testing is effective in eliminating large flaws strength measurements were made on specimens that contained flaws that were introduced using a

Knoop hardness indenter. Strength measurements at room temperature (Table 1) indicated that the indented specimens were considerably weaker than any of the specimens that contained only machining flaws. For proof-testing to be effective in eliminating flaws of this magnitude it must be demonstrated that these flaws are still fracture nucleation sites after exposure to high temperatures. Strength measurements on indented specimens after exposure to 1200°C for 100 hours indicate that both billet B and the reaction bonded material fractured from the site of the indentation (table 1). Since the artifically introduced flaws were still effective nucleation sites in these materials the results indicate that proof testing can be used to eliminate large flaws and therefore truncate the strength distribution. By contrast, specimens from billet A failed from origins that were not located at the site of the indentation, which indicates that proof-testing would be of little value for billet A.

The findings obtained in the indentation studies were confirmed in strength studies conducted on specimens that were not indented. Strength measurements on billet A (after 100 hours of exposure at 1200°C) indicate that the strength at 25°C and 1200°C has decreased to values that are considerably below the initial values (figure 3). Thus, flaws generated by high temperature oxidation are far more severe than the machining flaws originally present in the specimens. The flaws are also more severe than the large cracks introduced by indentation. The nature of these flaws will be discussed in a later section of this paper.

The strength behavior of billet B differs from that obtained on billet A. Strength measurements at 25°C after 100 hours of exposure to 1200°C (figure 4a) indicate that the mean strength decreases from ∿640 MPa to 580 MPa, whereas the Weibull slope increases from ∿5.9 to ∿14.4. The net effect of the increase in the Weibull slope is to increase the strength of specimens that fall at low levels of failure probability, which means that large strength impairing flaws are not generated in billet B by high temperature exposure. As a consequence of this behavior, components that contain large flaws can be eliminated by proof testing without fear that new, more serious flaws will be generated by high temperature oxidation. This conclusion is supported by the data

TABLE 1. STRENGTH OF INDENTED SPECIMENS: 2kg LOAD

Material	Test-Temp. °C	Exposure Conditions	Strength MPa	Fracture At indentation
NC132	25	as indented	397 ± 12	Yes
Billet A	25	16 hr 1200°C	432 ±	Yes
	25	100 hr 1200°C	461 ± 42	No
	1200	1/2 hr 1200°C	438 ± 15	Yes
	1200	16 hr 1200°C	396 ± 42	Yes
	1200	100 hr 1200°C	402 ± 12	No
NC132	25	as indented	408 ± 17	Yes
	25	100 hr 1200°C	524 ± 47	Yes
	1200	1/2 hr 1200°C	441 ± 10	Yes
	1200	100 hr 1200°C	488 ± 11	Yes
NC350	25	as indented	115 ± 24	Yes
	25	100 hr 1200°C	181 ± 22	Yes
	25	33 hr 1200°C	225 ± 38	Yes
	1200	100 hr 1200°C	188 ± 64	Yes

obtained at 1200°C after 100 hours of exposure at 1200°C. For this condition of testing, the measured strengths (figure 4b) were greater than those obtained after 1/2 hour of exposure at 1200°C. Therefore the natural flaws generated by high temperature exposure are less severe than those that were introduced by indentation, and as a consequence, proof testing can be used to truncate the strength distribution by eliminating large flaws that might be present in the specimens. We note from figure 5 and table 1 that the data on the reaction bonded billets were qualitatively the same as those obtained on billet B. Therefore, it is concluded that proof testing can also be used to improve the performance of components made from reaction bonded material.

Flaw Generation

To determine the reason for strength degradation in hot pressed silicon nitride, a study of the oxidized surfaces and of the fracture surfaces was conducted using light microscopy, scanning electron microscopy and transmission electron microscopy. Light microscopy studies shown in figure 6 present a time sequence of surface oxidation of hot-pressed silicon nitride. As can be seen from this figure, the surface texture changes gradually with time, so that after 16 hours of exposure at 1200°C the surface is entirely covered with a layer of oxide, and the grinding marks are no longer visible on the surface. Platelets of oxide are first observed after 2 hours of oxidation and by the time the components have been oxidized for 100 hours, the surface is covered with a thick polycrystalline layer of oxide. X-ray diffraction studies indicate that silicon oxynitride (Si_2N_2O) forms within the coat after only 1/2 hour of exposure. While after 100 hours of exposure, enstatite β-cristobalite (β-SiO_2) as well as silicon-oxynitride are found in the coat. Transmission electron microscopy studies of the oxide coat demonstrate the presence of two amorphous phases (probably silicate glasses) within the oxide coat. Transmission electron microscopy studies also show that the silicon oxynitride is located at the silicon nitride, oxide coat interface, where-as the glasses and other crystalline materials are located towards the oxide coat, air interface. One concludes from these microscopy studies that the oxidation of silicon nitride is a relatively complex process involving both mass transport and the nucleation and growth of new phases within the oxide coat.

With regard to the strength of hot-pressed silicon nitride, the most pertinent finding of this study is the formation of pits in the surface of silicon nitride after long term oxidation. By examining fracture origins of bend specimens that were broken after long term exposure to 1200°C, it could be shown that fracture originated from these surface pits (figure 7). This observation was especially true for billet A which was very susceptible to pit formation. The pits appear to form under isolated mounds that are observed in the oxide coat. These mounds have a glassy appearance, and as shown in figure 8, sometimes contain a hole in them. By focusing through these holes (using light microscopy), pits ∿50 μm in diameter are observed beneath the mounds. The appearance and structure of the mounds suggest that a liquid phase was present at the site of the pit during oxidation. By removing the oxide coat with an etchant (HF-H_2SO_4), pits ∿50 μm in diameter were shown to exist beneath all of the mounds.

The suggestion that pits are the main site for crack nucleation after high temperature exposure is consistent with observations made by a number of other investigators (4-7). Although there is no doubt that pit formation causes strength degradation, there is some question as to the mechanism of pit formation. Freiman et al., (5,6) attribute pit formation to locally severe corrosion, but present no details of the corrosion process. Singhal (4), suggests that pore formation results from localized nitrogen generation at the silicon nitride interface, which results in a pressure build up that locally ruptures the oxide coat forming cracks and pits. Wiederhorn and Tighe (8) suggest that pit formation may involve tungsten compound inclusions that are known to be present in these materials. As these compounds oxidize, the tungsten oxide gases

that form escape from the surface leaving mounds at the point of gas eruption and small pits behind where the inclusions had been located. Most of the suggestions of mechanisms of pit formation are tentative in nature, so that the real mechanism is not yet known with certainty. Since the elimination of surface pits is a crucial step in improving the performance of this material, ascertaining the correct mechanism of pit formation is an important topic for future research.

Discussion of Results

The results of this paper indicate quite clearly that there are limits to the application of proof-testing (or non-destructive evaluation techniques) as a method of assuring the long term reliability of silicon nitride as a turbine material. Proof-testing is of value if the flaws eliminated during the proof test are in fact the flaws that will cause failure during service. Modification of the flaw population by oxidation can invalidate a proof test procedure by either eliminating the machining flaws that cause failure at room temperature, or by changing their severity. In the present study, it has been shown that high temperatures (1200°C) modify the flaw population sufficiently that truncation of the normal flaw population (due to machining) is no longer observed after exposure times as short as 1/2 hour. Therefore, although the small flaws that result from machining operations can be truncated by proof testing, truncation will serve little practical value since these flaws are probably not the ones that cause failure after high temperature exposure. One can show from these results that, depending on the material and the condition of use, there is an upper proof-test load above which no positive benefits can be gained by proof testing (7). Subjecting components to loads in excess of the upper proof test level will merely break them without limiting the maximum size of a crack that can be present during service.

The maximum proof test load for a given application can be determined by the introduction of artificial flaws of varying sizes into a test specimen in order to determine if these flaws are still effective as crack origins after high temperature exposure. In the present study, these flaws were introduced by hardness indentations which simulate accidental surface damage in silicon nitride. The results of the surface indentation studies indicate that proof testing is a viable procedure for the reaction bonded silicon nitride for which bend specimens broke at the indentations after 100 hours of exposure. Since these specimens would have been broken by a load of 115 MPa prior to high temperature exposure, the results indicate that a proof load of 115 MPa would have truncated the strength distribution of the population. In the proof test conducted on reaction bonded silicon nitride, however, it was shown that a load of 225 MPa did not result in truncation. From these two results we conclude that the proof test load for the reaction bonded material should be between 115 MPa and 225 MPa.

The strength studies on the hot pressed silicon nitride can also be used to obtain an estimate of the maximum proof test strength for this material. Indentations in billet B, which reduced the strength to ~400 MPa, were effective flaw nucleation sites after 100 hours of exposure at 1200°C. By contrast, a 630 MPa proof test on this billet did not result in a truncated population at high temperatures. From these results we conclude that the maximum proof-test load for billet B lies somewhere between 400 MPa and 630 MPa. By contrast to this finding on billet B, results on billet A indicate that the maximum effective load for proof testing must be less than ~400 MPa, since the indentations in specimens from this billet were not effective nucleation sites after high temperature exposure. Therefore, proof testing would be more effective in truncating the strength of a population from billet B than one from billet A.

It can be shown that a proof-test stress of 400 MPa for hot-pressed silicon nitride is not restrictive with regard to current development programs on ceramic turbine engines. Referring to figure 9, which relates the proof test stress to the applied stress for various times at load, the applicability of a maximum proof stress of 400 MPa can be ascertained. In one vehicular turbine that is currently under development for example, a maximum stress of 220 MPa is expected in the rotors of this turbine for steady state operation under full-power. To assure an operational lifetime of 25 hours (the current program goal) under these conditions, a proof-stress of 390 MPa is necessary, figure 9. Because this stress is less than the suggested maximum proof-stress, this proof-stress should assure satisfactory operation of the turbine rotors for the expected exposure time. As a second example, a turbine being designed for electric power generation is expected to experience extreme stress conditions during emergency shutdown. Thermal transients during shutdown will expose the trailing edges of the stator blades to a stress of 350 MPa for a period of approximately 1 second. If a proof-test could be devised for a thermal shock situation, then a proof-stress of approximately 400 MPa would be needed for this application, figure 9. Since this value lies at the proof-test limit shown in figure 9, it too would be acceptable as a proof-stress. As a final example, a turbine being developed for Naval applications is expected to experience a 98 MPa stress in the first stage rotor at a temperature of 1040°c. If we assume that the 1200°C data presented in this paper is applicable, then, for a design lifetime of 50 hours, a proof stress of 170 MPa will be needed.* This stress is considerably less than the maximum indicated in figure 9. Thus, for the three examples noted, the proof-stresses are less than the maximum recommended stress and one may use proof-testing with the assurance that surface oxidation will not inerfere with lifetime predictions.

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* Because crack growth at 1040°C is more difficult than at 1200°C, the proof test diagram at 1040°C will give a lower proof stress for the same operating conditions. Therefore, lifetimes resulting from a proof stress of 170 MPa will be greater than 50 hours.

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Figure Captions

1. Effect of proof testing on the strength distribution of silicon nitride at 25°C: (a) hot-pressed silicon nitride; (b) reaction bonded silicon nitride.

2. Effect of proof testing on the high temperature (1200°C) strength of silicon nitride: (a) hot-pressed silicon nitride; (b) reaction bonded silicon nitride.

3. Effect of high temperature exposure on the strength of hot-pressed silicon nitride (billet A). 100 hours of exposure in air at 1200°C: (a) strength at 25°C; (b) strength at 1200°C.

4. Effect of high temperature exposure on the strength of hot-pressed silicon nitride (billet B). 100 hours of exposure in air at 1200°C: (a) strength at 25°C; (b) strength at 1200°C.

5. Effect of high temperature exposure on the strength of reaction bonded silicon nitride. 100 hours of exposure at 1200°C: (a) strength at 25°C; (b) strengths at 1200°C.

6. Effect of temperature on the surface structure of hot-pressed silicon nitride: (a) 1/2 hour exposure; (b) 2 hour exposure; (c) 16 hour exposure; (d) 100 hour exposure.

7. Fracture source in hot-pressed silicon nitride. The test was conducted on a specimen from billet B at 1200°C.

8. Mound formed during high temperature oxidation of hot-pressed silicon nitride. The hole in the center of the mound suggests that gas had issued forth from the mound.

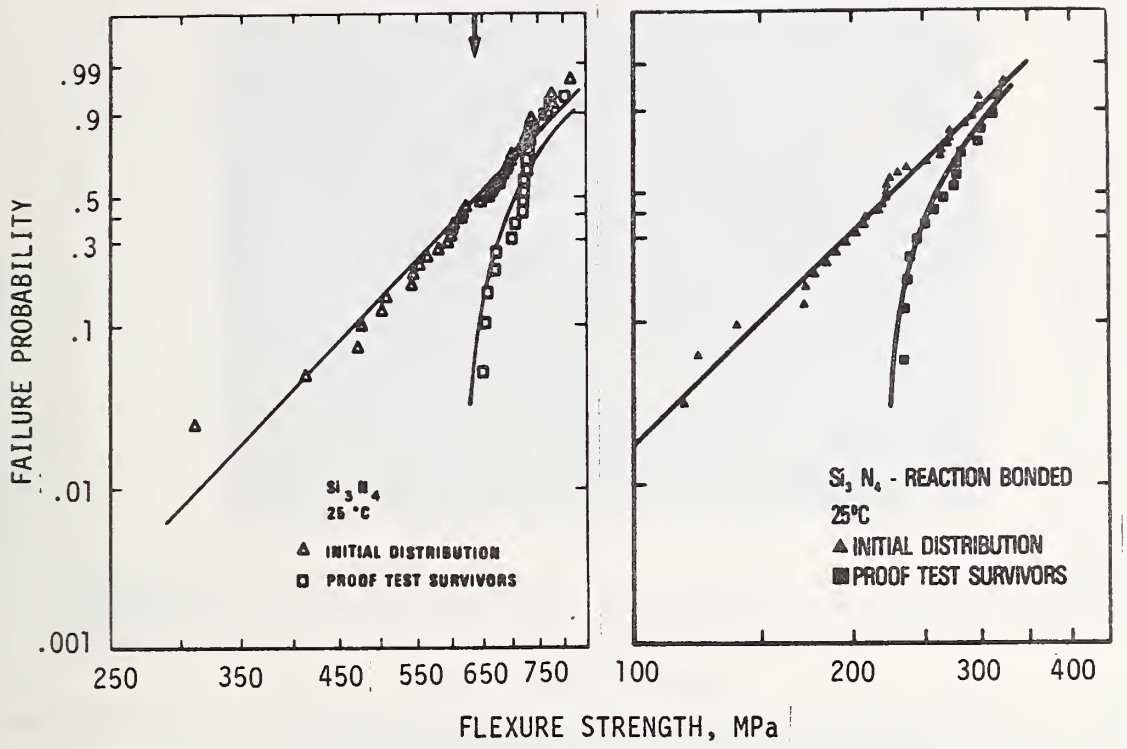

Figure 1. Effect of proof testing on the strength distribution of silicon nitride at 25°C: (a) hot-pressed silicon nitride; (b) reaction bonded silicon nitride.

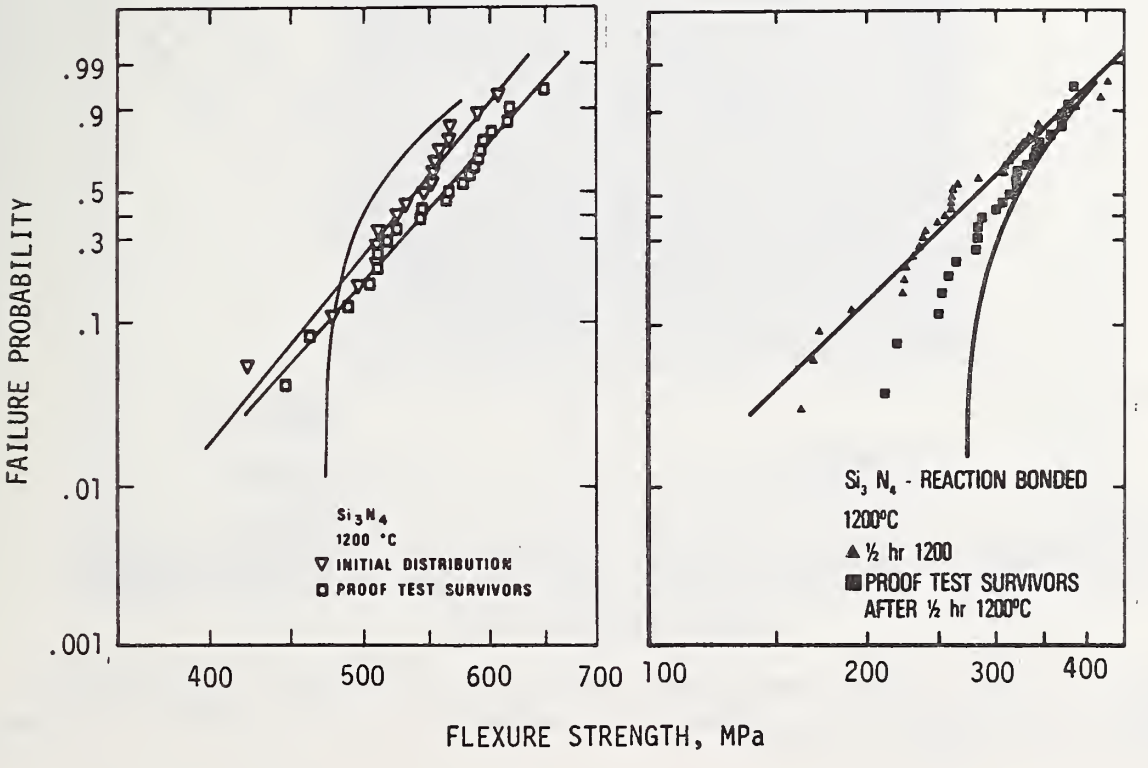

Figure 2. Effect of proof testing on the high temperature (1200°C) strength of silicon nitride: (a) hot-pressed silicon nitride; (b) reaction bonded silicon nitride.

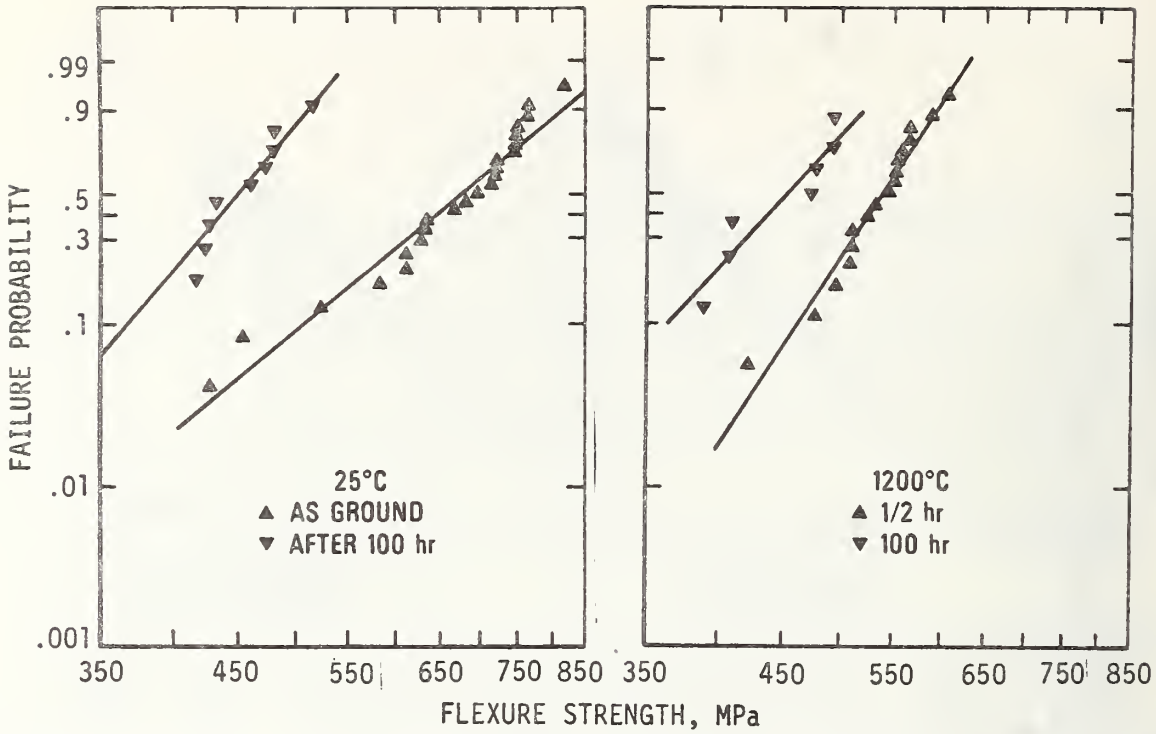

Figure 3. Effect of high temperature exposure on the strength of hot-pressed silicon nitride (billet A). 100 hours of exposure in air at 1200°C: (2) strength at 25°C; (b) strength at 1200°C.

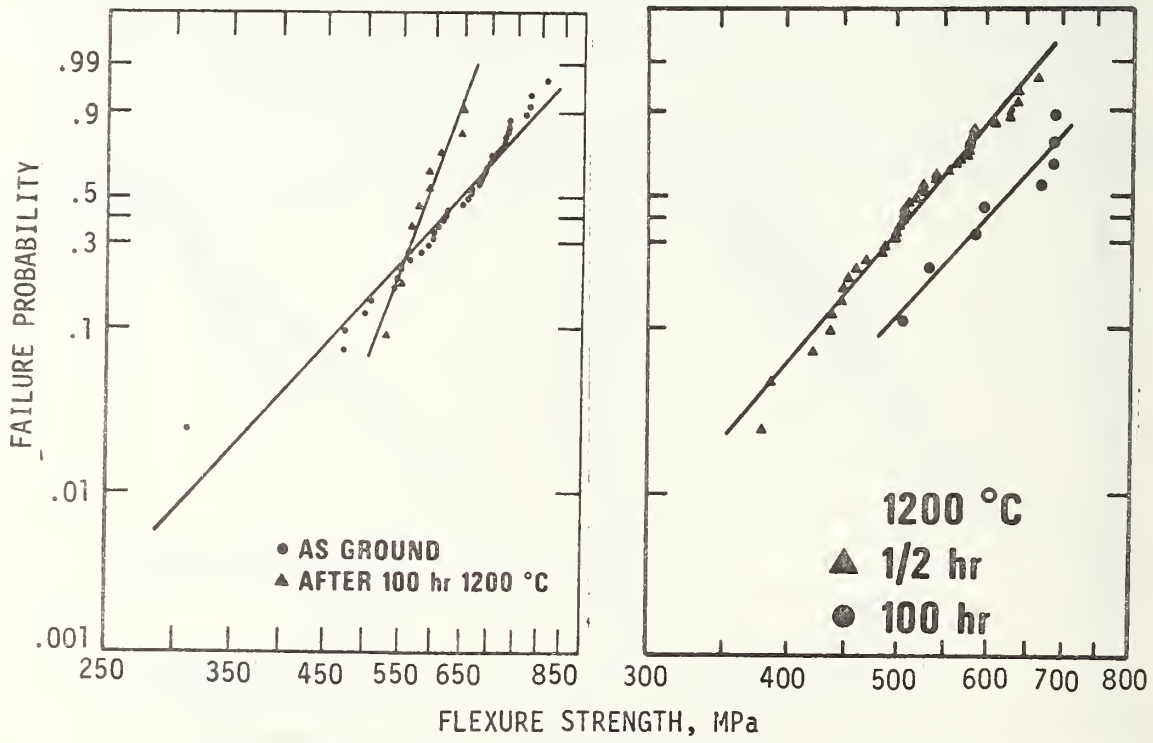

Figure 4. Effect of high temperature exposure on the strength of hot-pressed silicon nitride (billet B). 100 hours of exposure in air at 1200°C: (a) strength at 25°C; (b) strength at 1200°C.

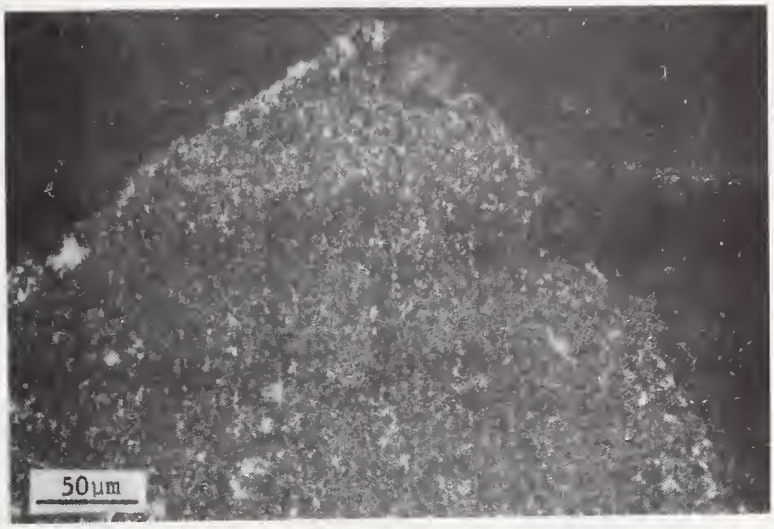

Figure 7. Fracture source in hot-pressed silicon nitride. The test was conducted on a specimen from billet B at 1200°C.

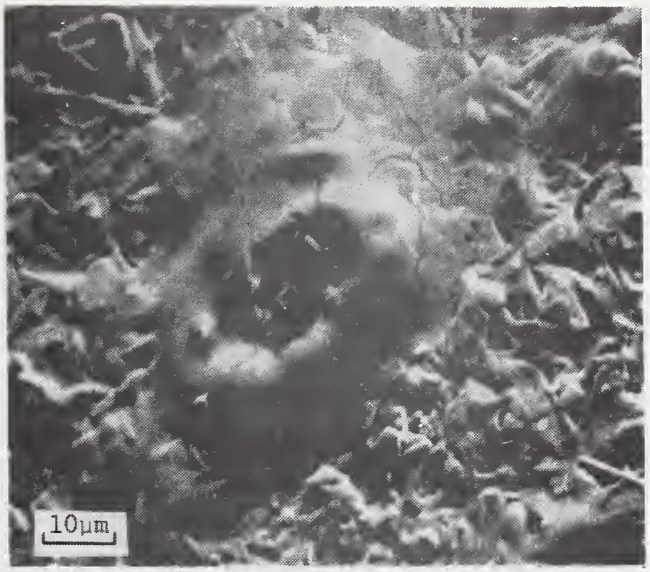

Figure 8. Mound formed during hihg temperature oxidation of hot-pressed silicon nitride. The hole in the center of the mound suggests that gas had issued forth from the mound.

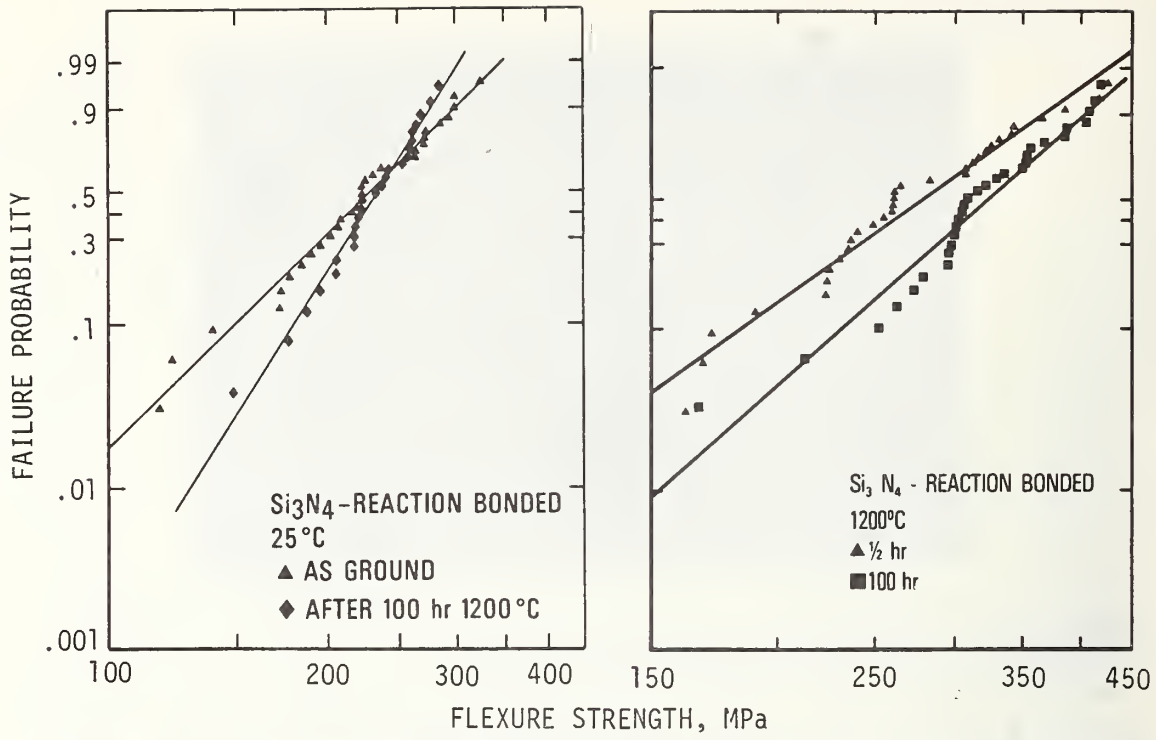

Figure 5. Effect of high temperature on the strength of reaction bonded silicon nitride. 100 hours of exposure at 1200°C: (a) strength at 25°C; (b) strengths at 1200°C.

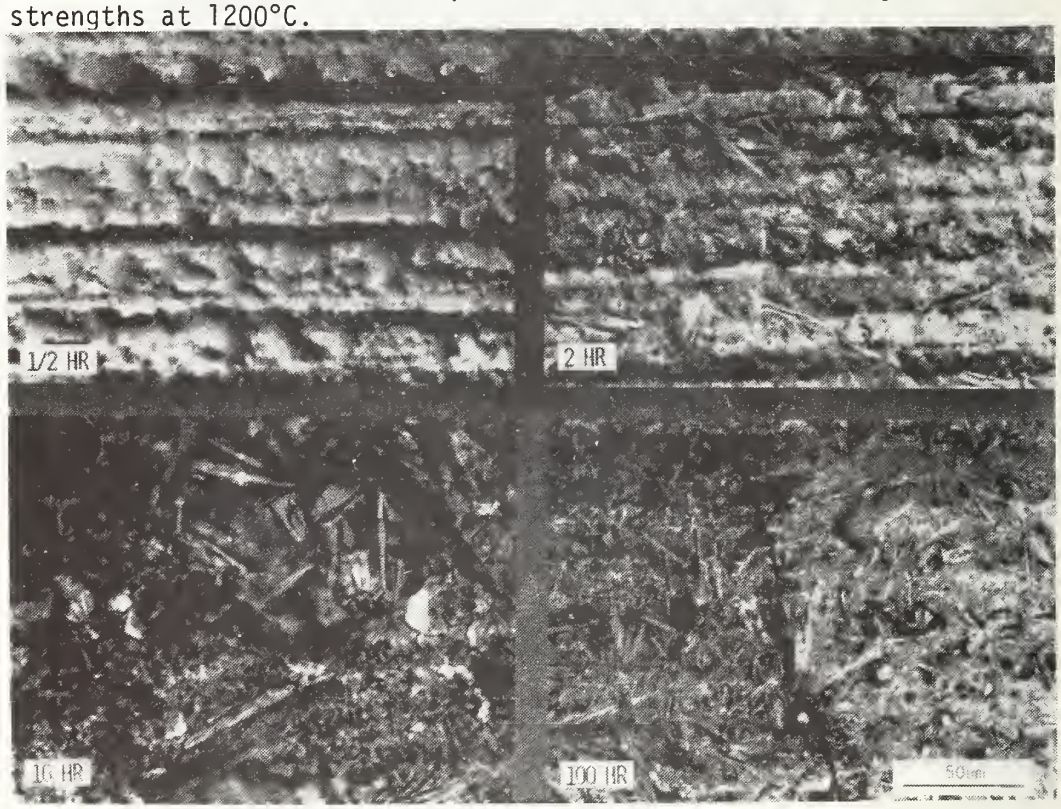

Figure 6. Effect of temperature on the surface structure of hot-pressed silicon nitride: (a) 1/2 hour exposure; (b) 2 hour exposure; (c) 16 hour exposure; (d) 100 hour exposure.

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Ceramic turbines; failure prediction; flaws; fracture; oxidation; silicon nitride; strength.

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