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Application of Proof Tests to Silicon Nitride

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ABSTRACT

In the ceramic turbine program, the high cost of turbine components and the disastrous effect of component failure require development of both destructive and nondestructive tests for detecting and rejecting defective components. Proof tests, in which a load is applied to break weak components, will ensure that the survivors will have a probability of failure that is acceptable for the design requirements. Thus, the development of a reliable proof test program requires testing a statistically significant number of specimens. Proof tests have been applied to assure the reliability of glass components and are being developed for polycrystalline ceramic components, such as silicon nitride. The initial proof tests are being done with 4 point bend specimens of hot-pressed and reaction-bonded silicon nitride. The tests are conducted at room temperature and at the maximum useful temperature of the materials.

1. Introduction

In order to assure the reliability of ceramic components for turbine engines it is necessary to use an evaluation program which will eliminate flawed and low strength components. Failure prediction techniques based on fracture mechanics data are being developed and evaluated for this testing[1-3]. The present paper describes results obtained as part of a program to evaluate the proof testing technique. In proof testing, weak components are eliminated by applying a load larger than the expected service load. The survivors of this test then should have predictable

lifetimes in service. This method was evaluated for glass components by Wiederhorn[4], and for silicon carbide tested at 800 and 1400°C by Evans and Lange[5].

The validity of the proof testing procedure must be established on laboratory specimens by testing significant numbers of specimens under conditions similar to service conditions. If the method is reliable then it can be extended to components. The proof test test method has not been evaluated for silicon nitride. In this paper we present results obtained on hot-pressed, magnesia-doped silicon nitride by proof-testing at 25°C and at 1200°C. Above 1200°C the material deforms plastically and has short predicted lifetimes.

2. Experimental Procedure

2.1 Materials

Specimens were made from two billets of hot-pressed magnesia-doped silicon nitride. These billets were manufactured at different times but had the same nominal composition. The typical impurities (wt %) are: 0.20Al, 0.05Ca, 0.40-0.64Fe, 0.70-0.74Mg, 2.6-2.7W. Tungsten particles were picked-up during powder preparation.

2.2 Test Procedure

Test bars 4x5x50 mm were cut from the billets and were ground along their length with a 180 grit diamond wheel. From the billet designated-A 120 bars were cut at different times. Specimens for crack propagation tests were cut from the remainder of the billet. Three hundred bars were cut from billet-B. These bars were numbered and were randomized for testing.

The bars were tested in 4-pt bending using a silicon carbide jig with upper and lower spans of 10 and 40 mm respectively. The proof test procedure was to load the specimen at constant stress rate (crosshead speed of 0.2mm/m) until the calculated proof stress was reached; and, then, to unload at the same rate. The specimens that survived the proof tests were left in the jig and were broken using the same loading conditions. The test temperatures were 25°C and 1200°C. For the high temperature tests the furnace was heated at ∿20°/min and the specimens and test rig were equilibrated for 1/2 hr at 1200°C before loading. Additional tests at times of 2, 16, 64 and 100 hrs were made to determine the effects of oxidation and of heating on the strength of the material.

The proof stress was chosen from the flexure strength data to break 35 to 50% of the specimens.

3. Results of Proof Testing

The proof test results are presented as Weibull plots of the strength distribution. At room temperature the application of a proof stress of 630 MPa broke ∿50% of the specimens. The Weibull plot in Fig. 1 shows the initial distribution (inert strength) and shows that the proof test survivors all broke above the proof stress. Thus the strength distribution is clearly truncated by a room temperature proof test. Similar results were obtained with specimens from billet A. Fracture initiated primarily at the surface.

In order to test the effectiveness of the proof test procedure for high temperature use, another group of 80 specimens was used to obtain the data in Fig. 2. The data in this plot were obtained by applying

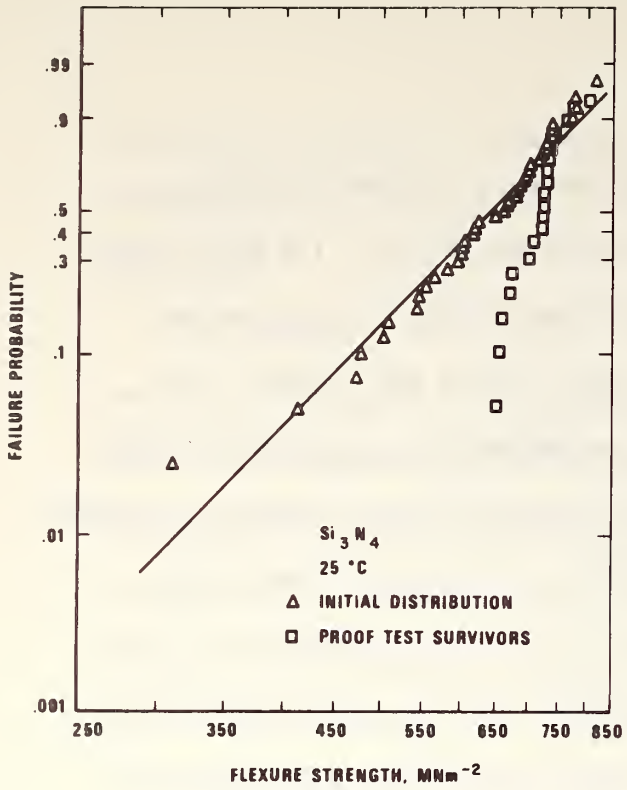

Fig. 1 Effect of proof testing on the strength distribution of magnesia-doped hot pressed silicon nitride, billet-B, proof stress 630 MN m^{-2}.

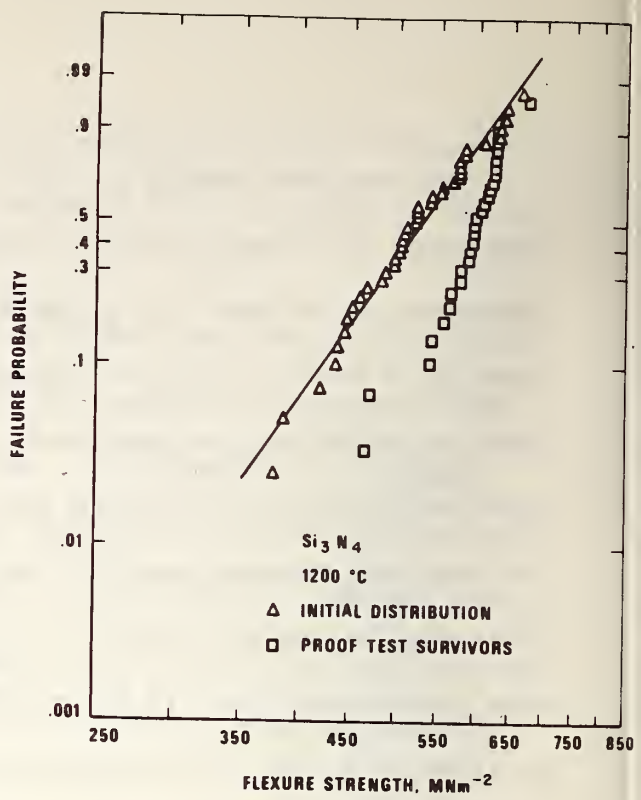

Fig. 2 Effect of proof testing at 1200°C on the strength distribution, billet-B, proof stress 500 MN m^{-2}.

proof stress of 500 MPa which was calculated to break 35% of the specimens at 1200°C. The strength distribution of the survivors shows some truncation and a slight increase in average strength. However 2 specimens broke below the proof stress after proof testing. The difference between the 25° and 1200°C results suggests that the flaw distribution which limits strength is different at the two temperatures. The specimens were held at 1200°C for 1/2 hour before applying the proof test. It may be that both oxidation and annealing affected surface flaws.

Since some degradation of strength can occur as a result of oxidation, the survivors of the 25°C proof test (630 MPa) were heated for 1/2 hr at 1200°C, cooled to 25°C and broken. The results of this test are shown in Fig. 3. It is seen that the strength distribution has changed but

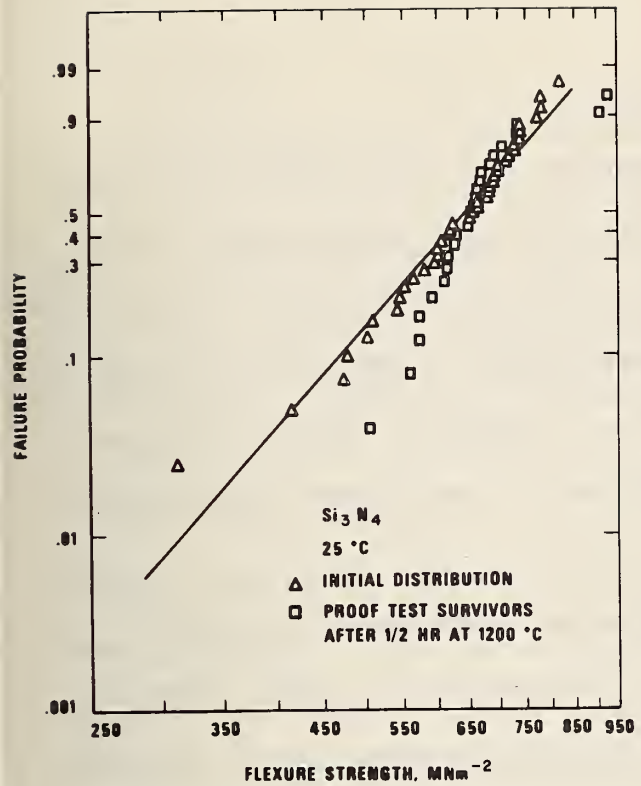

Fig. 3 Effect of oxidation on strength distribution, proof test survivors were heated 1/2 hr., at 1200°C, cooled and broken at 25°C, billet-B.

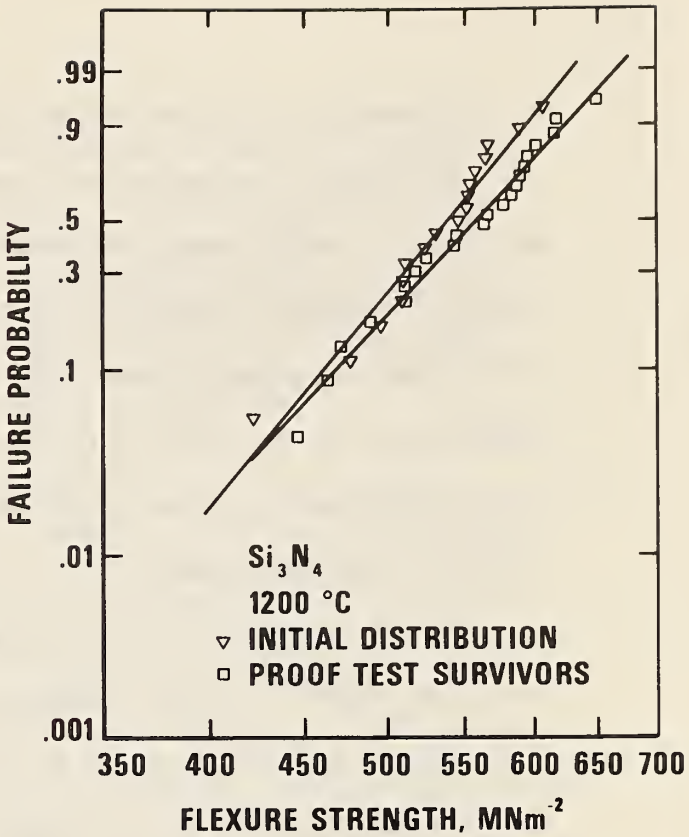

Fig. 4 Effect of proof testing on strength distribution, survivors of 25°C proof test were broken at 1200°C, proof test 650 MN m^{-2}.

∿50% of the survivors broke below the proof load of 630 Mpa. Comparing these results with those in Fig. 1, it is seen that a substantial change occurs in the strength distribution of the proof test survivors after 1/2 hour oxidation.

The data shown in Fig. 4 were obtained with specimens from billet A. These specimens were proof tested at 25°C and the survivors were broken at 1200°C. The strength distribution is not changed much between the proof test survivors and the initial distribution.

The series of 80 specimens oxidized for 1/2, 2, 16, 64 and 100 hrs., has not been completed. Initial results show a slight increase in strength after 1/2, 2, and 16 hrs., and a decrease after 64 and 100 hrs.

4. Discussion of the Proof Test Results

The results presented here demonstrate that the strength distribution at 1200°C is sufficiently different from the inert strength that some of the proof testing assumptions must be reassessed. The flaws that limit the flexure strength at room temperature appear to comprise a different population from the flaws that limit the strength at 1200°C. Where as, in the development of the proof test theory it was assumed that the flaw population did not change.

It is clear from our test program that considerable oxidation occurs at 1200°C. In our preliminary tests there is a slight change in the strength distribution and an improvement in strength but by the time the specimen has been heated 64-100 hrs., the strength distribution is again close to the initial distribution. The oxidation can be detrimental to the room temperature strength, because the oxides that form, namely cristobalite, enstatite, silicon oxynitride and glass[6] have a larger specific volume than the silicon nitride. These oxides fracture on cooling and the oxide film changes composition during longtime heating. The oxidation or annealing can be beneficial to the strength by healing the strength limiting flaws or by relieving the strain around such flaws.

If the proof test is required to eliminate weak specimens from the components that will be used at temperatures as high as 1200°C, then, these preliminary results suggest that specimens will have to be proof tested near their service temperatures. Clearly this is a difficult process for some components.

The results we have obtained are based on statistically significant sample sizes. We found that data from fewer than 10 specimens did not give a reliable distribution curve. The randomization of the specimens from billet B ensured that the distributions were free of bias related to preparation method or to inhomogenities in the billets.

5. Conclusions

The results demonstrate that proof testing is valid for hot-pressed silicon nitride when the testing is done at room temperature i.e. under inert conditions. When the testing is carried out at 1200°C, i.e. under corrosive conditions, the proof test approach to assuring reliability may not be valid. The strength distribution and hence the flaw distribution are changed by the heating and oxidation during the tests and the strength distribution was changed only slightly by the proof tests.

Acknowledgment

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

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