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# STRESS RELAXATION TESTING AS A BASIS FOR CREEP ANALYSIS AND DESIGN OF SILICON NITRIDE

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## ABSTRACT

A new approach to tensile creep testing and analysis based on stress relaxation is described for sintered silicon nitride. Creep rate data covering up to five orders of magnitude were generated in tests lasting less than one day. Tests from various initial stresses at temperatures from 1250C to 1350C were analyzed and compared with creep rates measured during conventional constant load testing. It was shown that at least 40% of the creep strain accumulated under all test conditions was recoverable, and that the deformation could properly be described as viscoelastic/plastic. Tests were conducted to establish the level of repeatability and the effects of various thermomechanical histories. It was shown that none of the prior exposures led to significant impairment in creep strength. The results were used for three different grades to establish the value of the accelerated test to compare creep strengths for acceptance and for optimization. Several useful correlations were obtained between stress and creep rate. The systematic creep rate dependence as a function of loading strain prior to relaxation provided a possible basis for design in terms of a secant modulus analysis.

## INTRODUCTION

Ceramics offer a number of potential advantages for high temperature components in gas turbines. Their low density, high stiffness, low thermal conductivity and, in most cases, higher creep strength at temperatures in excess of 1000C, make them good candidates for a number of components. The greatest payoff is for those components which require the use of turbine air cooling, such as first stage turbine blades and shrouds. Other components which could substantially benefit from the replacement of superalloys are the combustor, transition piece and first stage turbine nozzle. Tensile creep testing of ceramics can be difficult and expensive because of the need to use sophisticated specimen gripping, precision extensometry and stable temperature control at very high temperatures. However, a number of laboratories, worldwide, are now developing the capabilities, and beginning to report the results, of careful and accurate tensile creep tests on engineering ceramics (Carroll et al., 1989, Dyson et al., 1989, and Ferber et al., 1994)). This requires a major commitment in time and expense to generate the extensive data using traditional creep testing of the many specimens necessary to provide a basis for both statistical analysis and extrapolation to the required design life. Consequently, in recent years such studies have often been part of major collaborative programs (Hecht et al., 1997).

An alternative approach to generating creep data, which is far more efficient in terms of numbers of test specimens and test duration, involves the generation of stress vs. creep rate responses derived from stress relaxation tests (SRT). This methodology was strongly promoted several years ago by Hart (1970) as a basis for his plastic equation of state, and has been developed and applied subsequently to a wide variety of metallic materials (Hart and Solomon, 1973, Li, 1981, and Woodford, 1975)). Very recently, it has been applied as part of a new framework for materials development, design and remaining life assessment of operating components (Woodford, 1993)). This use of a high precision short time stress relaxation test has also been successfully applied to creep analysis for engineering polymers (Grwzwinski and Woodford, 1995).

Some preliminary work on stress relaxation testing of alumina and silicon nitride has been reported (Woodford et al., 1991, and Wereszczak et al., 1995)). This was sufficiently promising to justify a more detailed study of a ceramic from a design perspective using this methodology. Two of the most promising monolithic silicon nitride materials have been evaluated for stage one turbine stator shroud components for large utility gas turbines with turbine inlet temperatures in the range of 1250-

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1300C (Hecht, 1997)). SN-88, supplied by NGK Insulators, Ltd., was extensively tested and evaluated, and the results compared with those generated in conventional creep tests (Woodford, 1998)). The second material, AS-800, was supplied by AlliedSignal Ceramic Components and results on an earlier vintage of this material have also been reported (Woodford, 1996)).

The AS800 has now been improved significantly compared with previous vintages. The present work is a more comprehensive study of new (1997) AS800. In addition to generating basic data at temperatures between 1200C and 1350C and comparing with the previous results, tests were conducted to examine repeatability and the effects of prior thermomechanical exposures.

## EXPERIMENTAL PROCEDURE

Tests were performed at Oak Ridge National Laboratory on an Instron electromechanical series 1380 test system fitted with self-aligning grips, a 1500C short furnace, and contacting capacitive extensometry. Specimens, designed for use with the grips, were 165mm long, and featured a reduced section of 40mm and diameter of 6mm. Temperature calibration along a 25.4mm gage length was maintained and controlled to 1C at temperatures to 1350C. With the closed-loop strain control the set strain was held to $\pm 15\text{x}10^{-6}$.

Standard procedure involved loading at a rate of 10MPa/sec to a prescribed stress and switching to strain control on the specimen and monitoring the relaxation of stress. The stress vs. time response fitted to a fourth order polynomial was converted to a stress vs. creep strain-rate response by differentiating and dividing by the modulus measured on loading according to the following:

$$e_e + e_I = e_t = \text{constant}$$

$$\frac{de_I}{dt} = \frac{-de_e}{dt} = \frac{-1}{E} \cdot \frac{ds}{dt}$$

Where: $e_e$ = elastic strain
$e_I$ = inelastic creep strain
$e_t$ = total strain
$s$ = stress
$E$ = elastic modulus

This is, in effect, a self-programmed variable stress creep test. Typically, a test lasting less than one day may cover up to five decades in creep rate. The accumulated inelastic strain was usually less than 0.1%, so that several relaxation runs at different stresses could be made on a single specimen with minimal change in the mechanical state. Thus, an enormous amount of creep data was generated in a short time on a single specimen. Separate specimens were used for different temperatures of 1200C, 1250C, 1300C and 1350C.

The test sequence involved relaxation runs from progressively increasing set stresses. Each run was unloaded after approximately twenty hours and the residual strain was monitored for at least two hours to measure any anelastic creep recovery.

Basic tensile and creep data on AS800 were obtained from AlliedSignal Ceramic Components. These data were used to set stresses for the SRT tests. All tensile testing had been done at the University of Dayton, at a stressing rate of 150MPa/sec. Round bar creep testing was also done at the University of Dayton and flat specimen testing was done at NIST. These data were used for comparison and were not separately identified since there appeared to be no systematic effect of specimen geometry.

The test matrix is described below:

1 1200C, one-day tests at stress levels of 300MPa, 350MPa and 400MPa
2 1250C, one-day tests at stress levels of 250MPa, 300 MPa and 350 MPa
3 1300C, one-day tests at stress levels of 200MPa, 250MPa and 300 MPa
4 1350C, one-day tests at stress levels of 150MPa, 200MPa and 250MPa
5 1200C, 3 one-day tests from 300 MPa for repeatability
6 1350C, 3 one-day tests from 200MPa for repeatability
7 Testing in sequence 1200C at 350 MPa and 1350C at 200MPa
8 Testing in sequence 1350C at 200MPa and 1200C at 350 MPa
9 Preexpose 20 hr. at 1250C then test at 1200C at 300 MPa and 350 MPa.
10 Preexpose 20 hr. at 1300C then test at 1200C at 300 MPa and 350 MPa
11 Preexpose 20 hr. at 1350C then test at 1200C at 300 MPa and 350 MPa
12 1300C, run test for three days at 300 MPa

The objectives were:

Tests 1 to 4 were analyzed for basic design and comparison with creep data.
Tests 5 and 6 were to determine repeatability.
Tests 7 and 8 examined the effect of temperature sequence.
Tests 9 to 11 examined the effect of preexposure on creep strength.
Tests 12 was a longer test run.

## RESULTS

Examples of the tensile curves at 1200C and 1350C are shown in Figures 1 and 2. In addition to the relaxation at a fixed strain these figures also show the anelastic recovery which occurs on unloading. During the two hour hold at near zero stress up to 50% of the accumulated creep strain recovered. During relaxation the strain was held within a band of approximately $3\text{x}10^{-5}$ i.e. ±15 microstrain. For a modulus of about 300,000MPa this would lead to a stress uncertainty of ± 4.5MPa. An example of the stress vs. ln time curves at 1200C which reflect these stress variations is shown in Figure 3.

Examples of the derived log stress vs. creep rate curves at 1200C and 1350C are shown in Figures 4 and 5. These typically cover nearly four decades in rate and show systematic dependence on the initial stress. Figure 6 compares the derived data for three

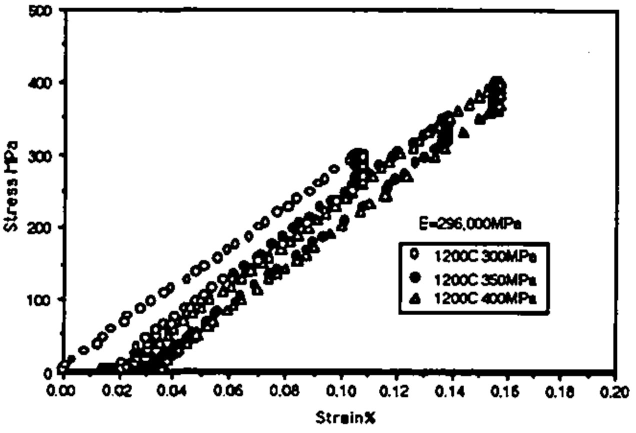

Figure 1 Stress-Strain for specimen #1 1200C

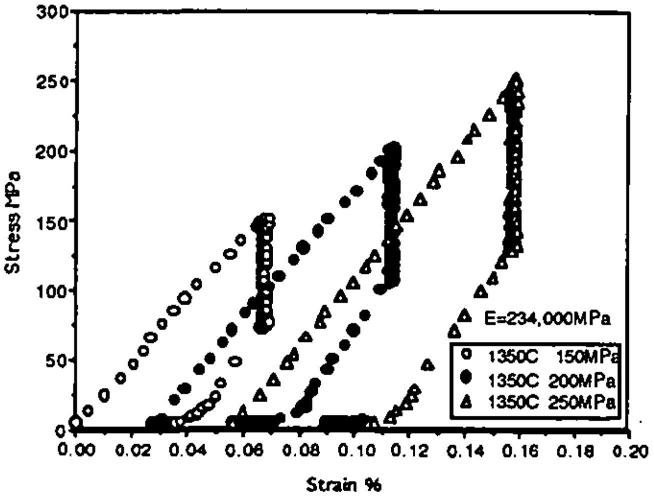

Figure 2 Stress-strain for specimen #4 at 1350C

successive tests from 300MPa and 1200C with that from the first run in a separate specimen. Repeatability between the separate specimens 5-1 and 1-1is good. However, subsequent repeat tests on the same specimen (5-2 and 5-3) indicate an appreciable effect of prior history; the subsequent runs indicate lower creep rates of nearly a factor of ten. A similar effect is shown in Figure 7 for tests from 200MPa and 1350C. Tests 6-1, 4-2, and 8-1 on separate specimens are quite repeatable, whereas 6-2 and 6-3 indicate a substantial reduction in creep rate. In this sequence 4-2 is the second run in a sequence but is from a higher stress than 4-1 and apparently is not influenced by the first run in that case.

Figure 8 indicates there is no effect of test sequence for relaxation from 1200C and 350MPa and 1350C and 200MPa. This result

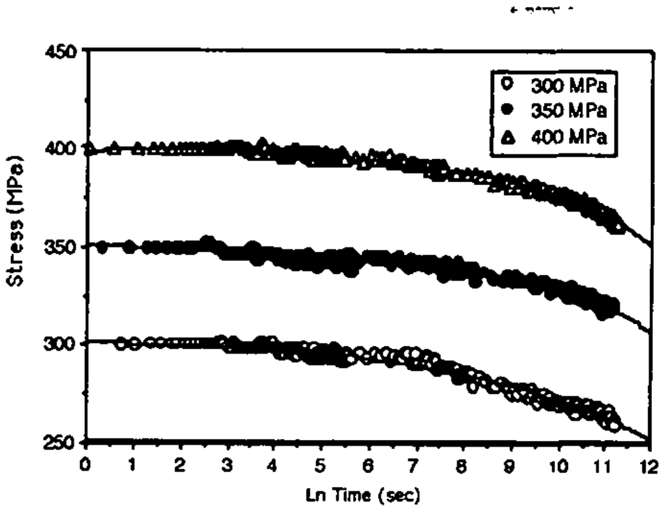

Figure 3 Stress relaxation in specimen #1 at 1200C

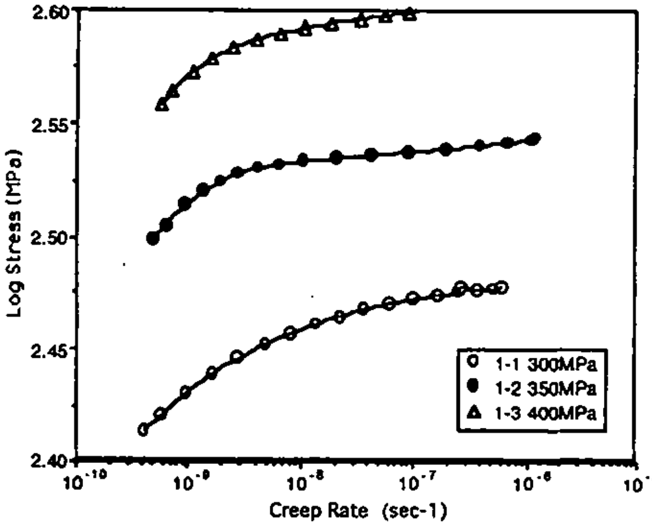

Figure 4 Stress vs. creep rate at 1200C for specimen #1

appears to indicate that for large changes of stress and temperature prior history effects are wiped out. For thermal exposure alone, however, a small systematic strengthening effect was observed with increasing temperature of prior exposure for testing at 1200C and 300MPa (Figure 9). This systematic behavior was not observed for the tests from 1350C and may be within the level of repeatability. In no case in any series of these experiments was there evidence that prior thermomechanical exposure could lead to a reduced creep strength.

## DISCUSSION

Five areas are covered in this discussion: comparison with

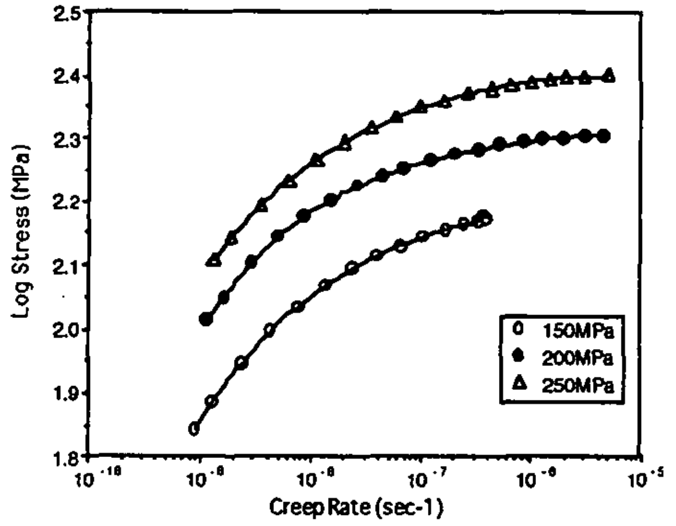

Figure 5 Stress vs. creep rate at 1350C for specimen #4

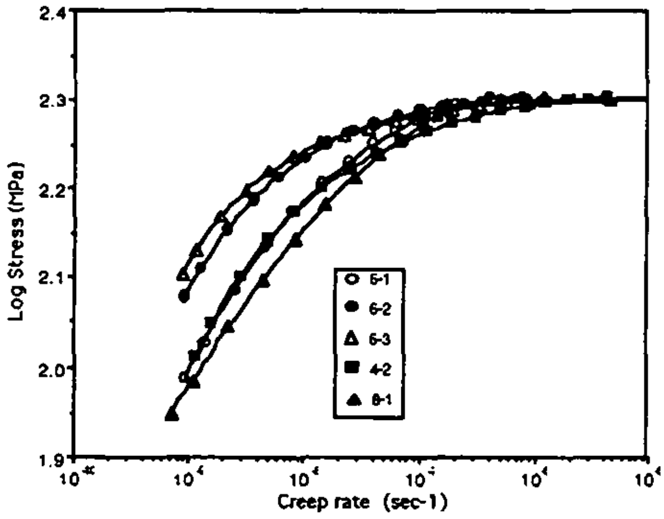

Figure 7 Specimens 6 ,4 and 8 relaxed from 200MPa and 1350C

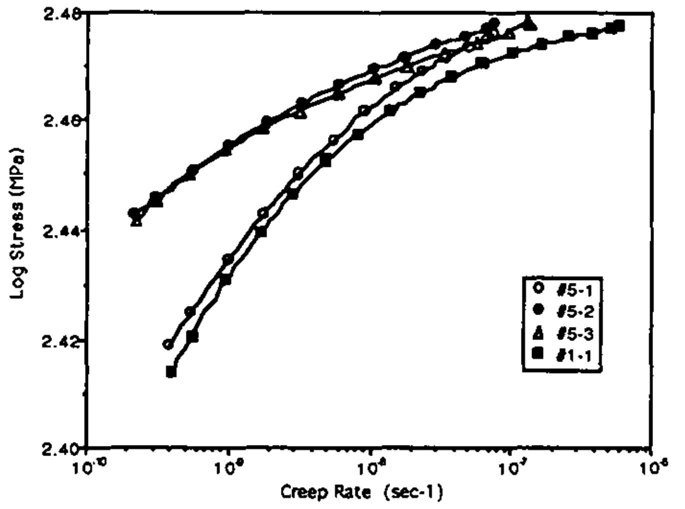

Figure 6 Specimens 5 and 1 relaxed from 300MPa and 1200

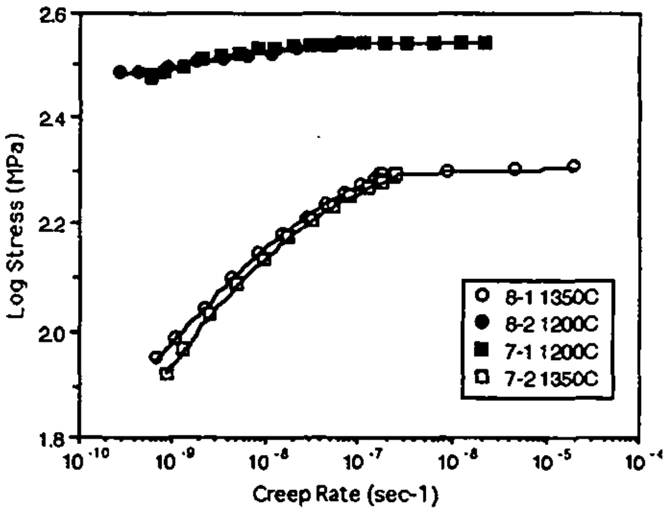

Figure 8 Effect of test sequence in specimens 7 and 8

previous SRT results, comparison with Allied Signal creep data, viscoelastic/plastic behavior, effect of prior thermal/mechanical history, and creep design for silicon nitride.

## Comparison with other SRT data

Comparisons were made at three test temperatures with previously generated data for silicon nitride on the GE/EPRICeramics for Gas Turbines Program. Examples at 1200C and 1300C are shown in Figures 10 and 11. The filled circles are the new data. In these figures AS800 is an earlier vintage of the Allied Signal material and SN88 is from NGK Insulators. The current AS800 was clearly superior in creep strength to both of the other tested materials at all three temperatures. At the lowest stresses the creep rates were more than an order of magnitude lower than those for the other materials.

## Comparison with Allied Signal Creep Data

Creep data were generated both on NIST dogbone specimens and ORNL buttonhead specimens. The data were supplied by John Pollinger of Allied Signal and the creep rates were used in comparison with the SRT data. No distinction was made between the two specimen types in the comparison. The Allied Signal minimum creep rate data at 1300C are compared with two SRT tests from 300MPa in Figure 12. Test #12 was a three day test and extended almost to $10^{-10}$ $sec^{-1}$. The creep data split the trends for the two SRT tests and gave a stress exponent of 6.25. Test 3-3 has

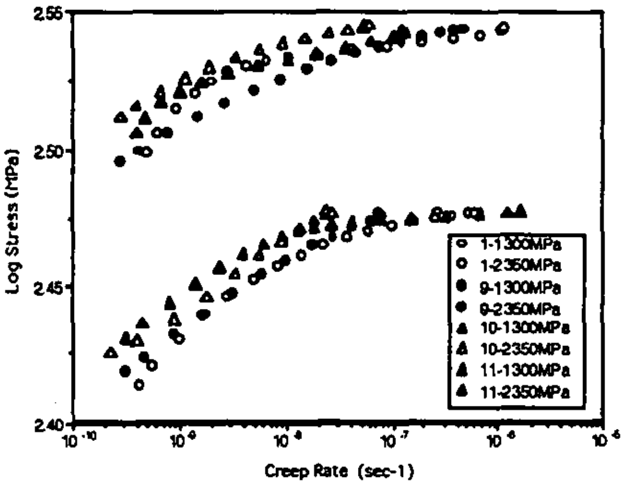

Figure 9 Effect of thermal exposure on relaxation from 300MPa and 350MPa in specimens 1,9,10,11

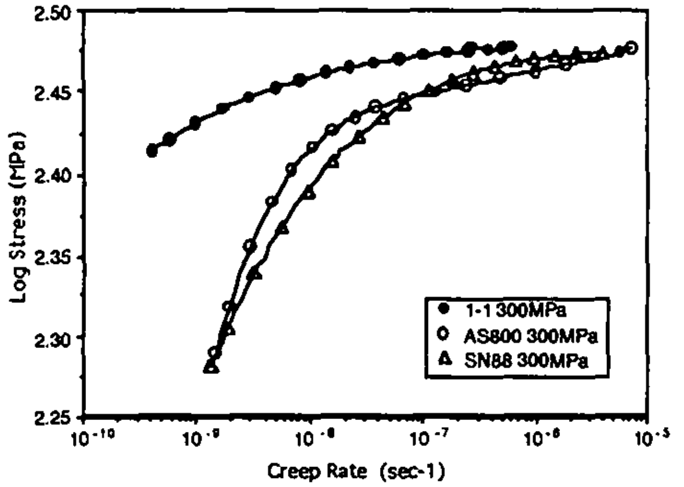

Figure 10 Comparison data at 1200C from 300MPa

lower creep rates after prior SRT tests at 200MPa and 250MPa than does test 12. This result is consistent with the results shown in Figures 6 and 7 in that prior SRT runs lead to increased creep resistance. Less extensive data at 1250C and 1350C gave stress exponents of 6.5 and 7.8 respectively.

## Viscous Elastic/Plastic Deformation

After unloading at the end of the test the strain was monitored for two hours in each run to estimate the anelastic recovery rate. Between one third and two thirds of the accumulated inelastic strain recovered during this period. The fraction recovered did not clearly depend on test temperature. However, there was some indication that a higher fraction recovered in later tests of a series at increasing stress or constant stress. These observations demonstrate that an appreciable, and perhaps a major portion of the creep strain, is recoverable. It was found that the creep recovery could be empirically fit to a time to the one third law quite well. This is illustrated in Figure 13 for specimen #12 at 1300C.

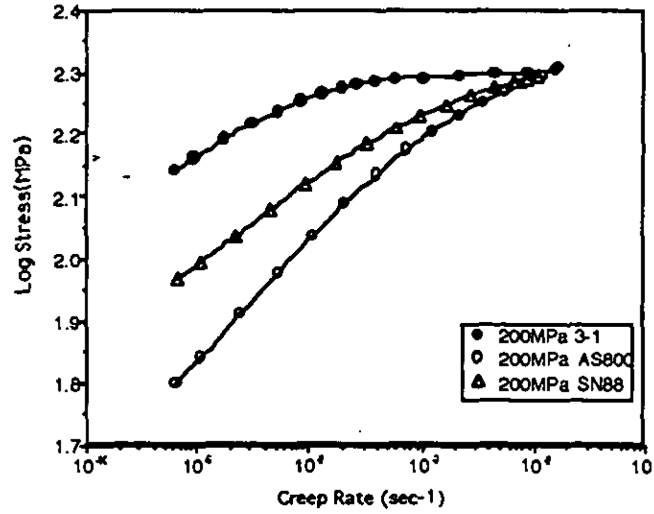

Figure 11 Comparison Data at 1300C from 200MPa

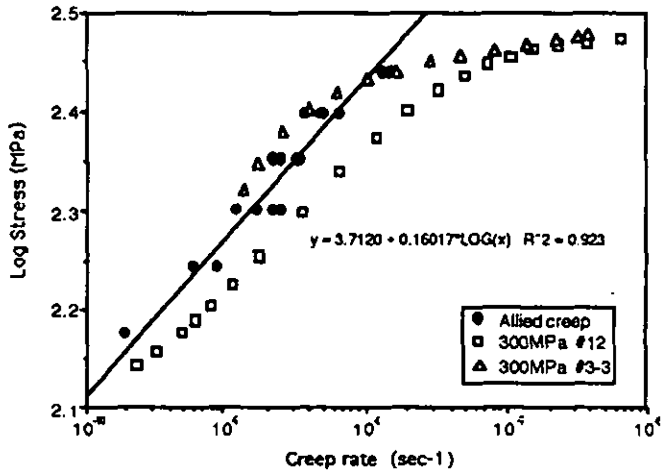

Figure 12 Comparison with Creep data at 1300C

In previous studies on similar material the first significant stress relaxation and subsequent creep recovery was observed at 800C and progressively increased to 1300C (Woodford, 1996, 1998). It appears that linear elastic behavior may be assumed up to about 800C and that viscoelastic/plastic behavior becomes increasingly important at higher temperatures.

For conventional creep testing the anelastic component would affect the magnitude of the transient strain depending on the loading rate as well as the temperature and stress. In addition, if much of the creep strain is recoverable, then its magnitude is expected to be history dependent. Even in metals, the relative contribution of anelastic strain to total creep strain increases as the

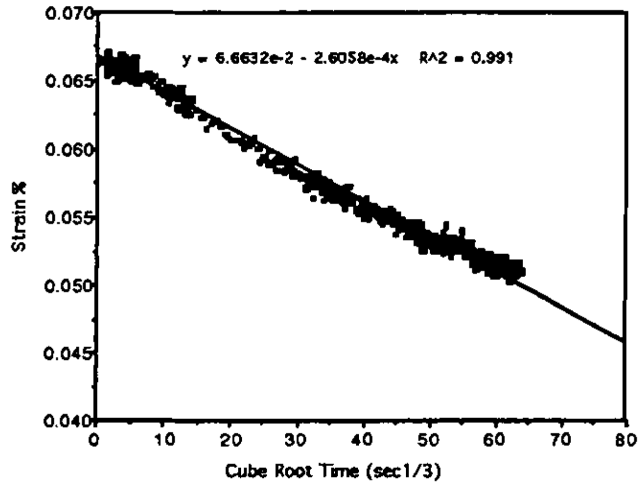

*Figure 13 Anelastic Contraction at 1300C*

stress decreases and may become significant under equipment operating conditions. Therefore, unless a concerted effort is made to separate the components of creep strain, the conventional creep analysis could not provide a useful basis for analysis of non-steady test conditions.

For viscoelastic flow, all or part of the inelastic creep strain is recoverable. Whereas plastic shortening never occurs, anelastic strain may lead to an extension on loading and a shortening or contraction on unloading. Clearly, the extent of creep recovery is going to depend on the stress, temperature and previous deformation history.

The fact that repeat runs at the same stresses (#5 at 1200C and #6 at 1350C in Figures 6 and 7) creep much more slowly than the first run may be because of continued anelastic contraction stemming from the previous test in each case. Since full recovery was not allowed after the first run, some anelastic contraction is expected during the second run. This in turn would lead to the lower than expected creep rates.

Whereas the importance of creep recovery is well recognized in polymer design, it is usually ignored in metal design. On the basis of the current experiments, it appears that the phenomenon may be very important for ceramics design, at least for the silicon nitride studied in this program.

## Effect of Prior Thermal/Mechanical History

The first run tests in Figures 6 and 7 ( e.g. #5-1 and #1-1, and #6-1 and #8-1) indicate good repeatability in separate specimens. However, subsequent tests on the same specimen at the same stress for both 1200C and 1350C show a reduced creep rate (e.g. #5-2,3 and #6-2,3). As indicated above this most likely stems from the complexity of anelastic deformation. However, for large changes in test conditions there is no effect of test sequence history. This is clearly shown in Figure 8 for tests between 1200C and 1350C. The fact that testing at 1200C has no significant effect on the response at 1350C may be easier to accept than the reverse sequence, but the results are clear. Figure 9 shows, especially for the first run from 300MPa, that unstressed thermal exposure may lead to reduced creep rates at 1200C. It is interesting to note that in no case did prior thermal or thermal/mechanical treatment lead to a significant reduction in creep strength. Figure 12 shows another example where the third run in a test sequence, #3-3, has a higher creep strength than the first run from the same stress, #12.

## Creep Design for Silicon Nitride

Creep testing and analysis of ceramics have tended to follow methods and procedures developed for metallic materials. Thus parametric correlations of minimum creep rates and rupture times are normally used as bases for comparison and optimization. These have required the use of expensive and time consuming tests. Moreover, because of the major contribution of anelastic strain to creep deformation the methodology may not be appropriate. The phenomenology and the mechanism of deformation in polymers are recognized to be quite different from those in metals. The dominant role of viscoelasticity in polymers, with complete recovery of strain after unloading possible in many situations, has led to different design approaches (Crawford, 1987)). Based on the current and previous tests (Woodford, 1996,1998)) it is believed to be more appropriate to draw from these approaches rather than the traditional approaches to metallic alloy mechanical design. Thus, the ceramic is viewed as a viscoelastic/plastic material.

Polymer components are often designed using a Pseudo Elastic Method. The classical equations for the design of springs, beams, plates, cylinders etc. have all been derived under the assumptions that:

(i) the strains are small
(ii) the modulus is constant
(iii) the strains are independent of loading rate or history and are immediately reversible
(iv) the material is isotropic
(v) the material behaves the same way in tension and compression

In the Pseudo Elastic Method appropriate time-dependent values of properties such as modulus are selected and substituted into the classical equations. It is common to generate stress-strain curves as a function of rate (or time) either directly or indirectly. The latter may be done by crossplotting using constant time sections through conventional creep curves. These produce isochronous curves. Alternatively, pseudo stress-strain curves as a function of strain rate or stress rate may be generated from stress relaxation tests in polymers(Grwzwinski and Woodford,1995) or ceramics (Woodford, 1996). The appropriate time-dependent modulus is then the stress divided by the strain for a particular strain. This is referred to as the secant modulus.

In the previous studies (Woodford, 1996, 1998) it was shown that stress relaxation was insignificant up to about 800C and that a linear elastic design analysis based on a tangent (Young's) modulus was appropriate. At higher temperatures it is proposed that an

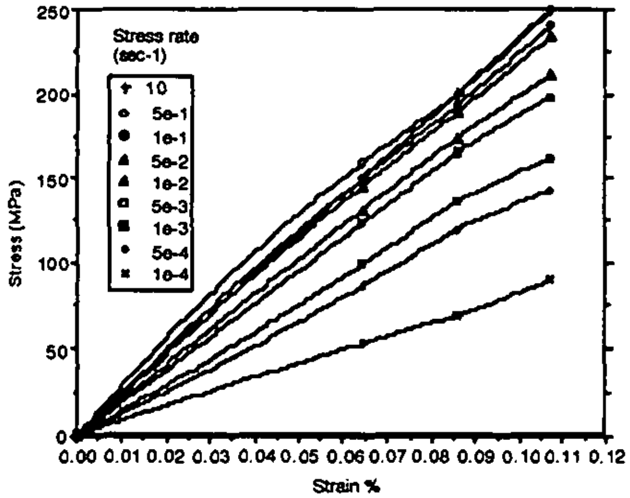

Figure 14 Pseudo stress strain curves for specimen #4 at 1350C

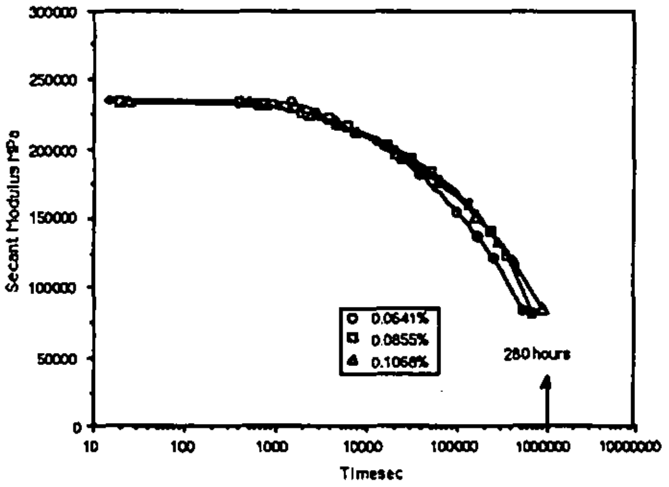

Figure 15 Secant modulus for #4 at 1350C

elastic analysis using a time dependent secant modulus may be most appropriate.

To generate the secant modulus curves it was first necessary to plot pseudo stress strain curves. These could be done either as a function of inelastic (creep) strain rate or stress rate. The latter was chosen since it is much easier, if necessary, to run actual tests under stress rate control. Also, since the initial loading was under stress rate control this allowed an additional high rate point to be included. The procedure involved curve fitting of derived stress vs. stress rate curves. These were then used to generate pseudo stress-strain curves as a function of stress rate as shown in the example at 1350C in Figure 14. The secant modulus values for each of the three tests at each temperature were then plotted against pseudo time (stress divided by stress rate). It was found that there appeared to be no clear effect of strain (see Figure 15 at 1350C). Accordingly, all

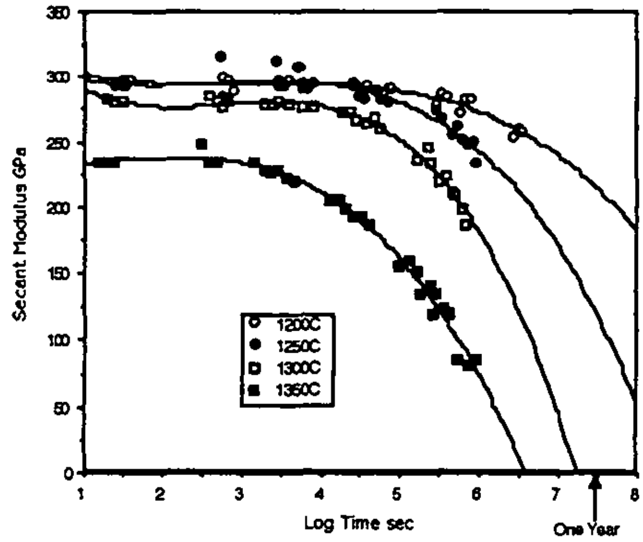

Figure 16 Secant modulus curves for all temperatures

the results of secant moduli could be plotted on unique temperature dependent but strain independent curves vs. log time. The resulting plot is shown in Figure 16.

Time-dependent design in this approach would simply use pseudo elastic analysis with the appropriate modulus being selected from figure 16. For example, the value at one year indicates that the allowable tensile loading at 1250C for a strain of 0.1% would be 115MPa. (i.e. 115GPa x 0.001).

## CONCLUSIONS

1 Stress relaxation tests with a strain control to $\pm 1.5x10^{-5}$ were successfully conducted to produce stress vs. creep rate curves covering between three and four decades in rate.

2 After unloading, all tests showed between 40% and 60% recovery of the inelastic strain in two hours, demonstrating the importance of viscoelasticity in the overall deformation.

3 Prior SRT testing, depending on the particular test sequence, either had no effect or resulted in increased creep strength (reduced creep rate).

4 Prior thermal exposure also resulted in increased creep strength.

5 None of the prior exposures led to a reduction in creep strength. This offers a new perspective on remaining life assessment of components.

6 An SRT test conducted at 1300C for three days clearly showed a sigmoidal shape.

7 Comparison with previous data on silicon nitride indicated that the present material is the strongest tested so far.

8 Long time creep data on similar pedigree material showed comparable creep rates.

9 Creep recovery at a fixed stress obeyed a time to the one third law quite well.

10 Pseudo stress strain curves were constructed as a function of stress rate. These were used to measure secant modulus values which were found to be essentially independent of strain for a fixed time.

11 It was proposed that a master plot of secant modulus vs. log time could be used in a pseudo elastic design framework for silicon nitride at temperatures above about 800C.

## ACKNOWLEDGEMENT

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