95-GT-235

Dynamic Fatigue Testing of Candidate Ceramic Materials for Turbine Engines to Determine Slow-Crack-Growth Parameters

Nora R. Osborne
George A. Graves
Metals and Ceramics Division
University of Dayton Research Institute
Dayton, Ohio

Matt K. Ferber
High Temperature Materials Laboratory
Oak Ridge National Laboratory
Oak Ridge, Tennessee

ABSTRACT

The purpose of this study was to evaluate the mechanical strength and slow-crack-growth parameter values for two commercially available silicon nitrides, SN-88 and NT164, at three high temperature conditions. Weibull analysis and dynamic fatigue slow-crack-growth parameters were used to characterize the material strength and resistance to slow crack growth at high temperatures for use in life prediction models. Although both materials are commercially available Si_3N_4, their high temperature behavior was found to be significantly different.

INTRODUCTION

Over the past few decades silicon-based ceramic materials, such as silicon nitride and silicon carbide, have shown promise as potential candidates for advanced heat engine application. These materials have high strength, and good oxidation and thermal shock resistance. The use of these materials offers a number of advantages including higher temperature operation, decreased weight, greater efficiency, lower life-cycle cost, and reduced dependency on the use of strategic materials.

Many companies have been involved in the development of improved Si_3N_4 materials. Because of limited characterization information, these materials have found limited application as structural components in the hot section of prototype and demonstration engines. For silicon nitride to become a reality for its intended applications a number of requirements must be met; namely, the development of reliable overall life-prediction methodology, appropriate test methods, and demonstration of feasibility for ceramic component design.

BACKGROUND

Under applied stress brittle materials exhibit a wide variability in strength. When macroscopic applied stresses reach critical values at crack tips, rapid and catastrophic failure will occur. The strength of brittle components can thereby be related to the flaw populations that are distributed on tensile surfaces, within the bulk, or along the edges of ceramic parts.

The Weibull two-parameter distribution is most often used to characterize the strength distribution of brittle materials [1]. The two physical constants determined by using the Weibull model are the scale parameter, σ_o, and the Weibull modulus, m. The scale parameter is used to perform a normalizing function, and the Weibull modulus reflects the degree of variability in strength -- the higher the m the less variability in the strength. Values of 5 to 20 for m are typical for ceramic materials [2].

The two-parameter Weibull model is determined using the maximum likelihood method. The maximum likelihood approach provides a means of determining the statistical error values for the test parameters and is somewhat less conservative than a linear regression technique [3].

Many brittle materials are susceptible to slow crack growth, or static fatigue, while under tensile load in a corrosive environment. The crack growth rate can be expressed as a power law relationship with the stress intensity level, K_I, described by equation 1

$$v = v_0\left(\frac{K_I}{K_{Ic}}\right)^n = AK_I^n \tag{1}$$

where v_o and A are material constants, and $K_{Ic} = Y\,\sigma_a\,c^{1/2}$. For semicircular flaws located at the surface of a brittle component, $Y = 2.24/\pi^{1/2}$. The constant n is a measure of the stress

Presented at the International Gas Turbine and Aeroengine Congress & Exposition
Houston, Texas - June 5-8, 1995
This paper has been accepted for publication in the Transactions of the ASME
Discussion of it will be accepted at ASME Headquarters until September 30, 1995

corrosion susceptibility of the material and is determined from the slope of the ln stressing rate versus ln median strength plot (see equation 2)

$$n = \left(\frac{1}{slope}\right) - 1 \tag{2}$$

and B, the alternative second crack growth parameter, is given by equation 3 as

$$B = \frac{2}{(n-2)\,A\,Y^2\,K_{Ic}^{(n-2)}} . \tag{3}$$

To determine the dynamic fatigue, strength measurements at two or more constant stress rates combined with a knowledge of the intrinsic strength are needed for the evaluation of n and B. The variability in n depends on the fatigue resistance of the material, and large n values generally represent materials with greater fatigue resistance [4].

EXPERIMENTAL PROCEDURE

Two sets of 150 test-ready flexure bars of each material were supplied to the University of Dayton Research Institute (UDRI) by Oak Ridge National Laboratory for dynamic fatigue testing. The materials, SN-88 and NT164, were produced by NGK, Nagoya, Japan, and Norton Advanced Ceramics, St. Gobain/Norton Industrial Ceramics, East Granby, CT, respectively. The test specimens were used to characterize the flexural strength of the materials based on the results of experimental evaluation of four groups of ten specimens at each of four stressing rates for three different test temperatures.

The flexural strength was measured using SiC four-point bend fixtures in a universal testing machine[1] following ASTM C1161-90 (MIL-STD-1942 (MR)). Measurements at 1038°C, 1150°C, and 1350°C were conducted in a high-temperature furnace[2] adapted for use in the testing machine. The test specimens were 3 mm x 4 mm x 50 mm with the tensile surfaces ground and the long edges of the tensile surfaces rounded to minimize failures initiated by edge chips. Specimens were loaded at machine crosshead speeds of 2×10^{-1}, 2×10^{-2}, 2×10^{-3}, and 2×10^{-4} in/min (8.4×10^{-5}, 8.4×10^{-6}, 8.4×10^{-7}, and 8.4×10^{-8} m/s).

The Weibull parameter, m, and median strength, σ_m, were determined for each test condition, and dynamic fatigue was characterized by ln-ln plots of fracture strength σ_f versus loading rate ($\dot{\sigma}$) which is expressed by the relationship

$$\sigma_f = A'\dot{\sigma}^{\left(\frac{1}{n+1}\right)} \tag{4}$$

where A' and n are material constants.

RESULTS AND DISCUSSION

The values determined for the Weibull parameter m and scale parameter, σ_o, using maximum likelihood regression for SN-88 are found in Table 1. The Weibull plots for the four stressing rates at each of the three temperatures used in this study are shown in Figures 1 through 3. The dynamic fatigue plots for SN-88 generated from the Weibull results are shown in Figure 4. Similar information for NT164 is found in Table 2 and Figures 5 through 8. A comparison of the median strengths of the materials for all four machine crosshead speeds at the three test temperatures is shown in Figure 9.

The Weibull modulus, m, for both materials was higher than expected. This may be attributed to the small number of specimens (10) tested for each temperature/loading rate condition. It is generally accepted that at least 30 specimens be tested at each test condition before the results are considered statistically reliable.

The fast fracture strength of SN-88 from 20 to 1350°C decreased as the temperature increased. At 1038 and 1150°C SN-88 was not subject to slow crack growth. At 1350°C strength actually increased as the stressing rate decreased. Additions to this material produces an intergranular glassy phase which may act to redistribute applied stress as it softens at the higher temperatures. This would account for both the negative n values and the relatively high median strengths found at the lower stressing rates.

Ritter, et al. [5] suggests that variability in the values of n and B depends on the total number of samples tested, the stressing rate range, the Weibull parameter, and the fatigue resistance of the material. The statistical uncertainty in n can be very large for samples sizes much smaller than 100. It is recommended that before meaningful conclusions can be drawn regarding the effect of a test variable on n as measured by the dynamic fatigue data, the statistical reproducibility of n should be evaluated.

A significant drop in strength with increasing temperature was observed in NT164, and no effects of dynamic fatigue were seen at 1038 and 1150°C. At 1350°C a dramatic decrease in strength with decreased loading rate was observed. This was in sharp contrast with the SN-88 whose strength increased with decreasing loading rate.

Ongoing studies at UDRI indicate that the SN-88 material has much higher creep rates under identical conditions than the NT164. A fractographic evaluation of the materials at 1350°C at the lowest loading rate might provide the necessary information concerning the behavior of the two materials.

CONCLUSIONS

SN-88 was not subject to slow crack growth at 1038 and 1150°C. At 1350°C and above the material behavior indicated some type of stress relief. Stress redistribution due to softening of a glassy grain boundary phase might be occurring.

At 1150 and 1350°C NT164 was subject to slow crack growth behavior, while fast fracture strength from room temperature to 1350°C decreased as temperature increased.

---

[1] Instron model 1123A with 1000 pound load cell and 500 pound range, Instron Corp., Canton, MA.

[2] ATS model 3320, Applied Test Systems Inc., Butler, PA.

Although both materials are commercially available Si_3N_4, their high temperature behavior is significantly different.

REFERENCES

1. Weibull, W. "A Statistical Distribution Function of Broad Applicability." *J. Appl. Mech.* 18(3):293-297, 1951.
2. Davidge, R.W. *Mechanical Behavior of Ceramic:Cambridge Solid State Science Series.* Cambridge University Press, 1979.
3. Berens, A. 1994: personal communication.
4. Evans, A.G. "Fracture Mechanics Determinations," *Fracture Mechanics of Ceramics*, edited by R.C. Grandt , vol. 4. Plenum Press, NY, 1978.
5. Ritter, J.E., Bandyopoadhyay, N., and Jakus, K. "Statistical Reproducibility of the Crack Propagation Parameter N in Dynamic Fatigue Test," *J. Amer. Ceram. Soc.* 62(9-10): 542-543.

TABLE 1. FLEXURAL STRENGTH MEASURED FOR NGK SN-88

Test Temperature °C	Crosshead Speed in/min	Atmosphere	Number of Tests	Weibull Modulus *m*	Weibull Scale Parameter σ_o	Median Strength σ_m	Dynamic Fatigue Shape Parameter n	Dynamic Fatigue Scale Parameter B ($secMPa^2$)
20	0.2	Air	30	23	838	922		
1038	0.2	Air	10	25	703	693	-202.0	-7×10^{30}
	0.02	Air	10	26	682	672		
	0.002	Air	10	38*	699	692		
	0.0002	Air	10	42	721	715		
1150	0.2	Air	10	21	697	685	392.3	9.8×10^{-50}
	0.02	Air	10	16	671	656		
	0.002	Air	10	23	679	669		
	0.0002	Air	10	25	679	669		
1350	0.2	Air	10	42	641	635	-51.8	-1×10^{13}
	0.02	Air	10	23	667	656		
	0.002	Air	10	18	741	726		
	0.0002	Air	10	28	735	725		

* Used estimated stressing rate for this series to calculate n.

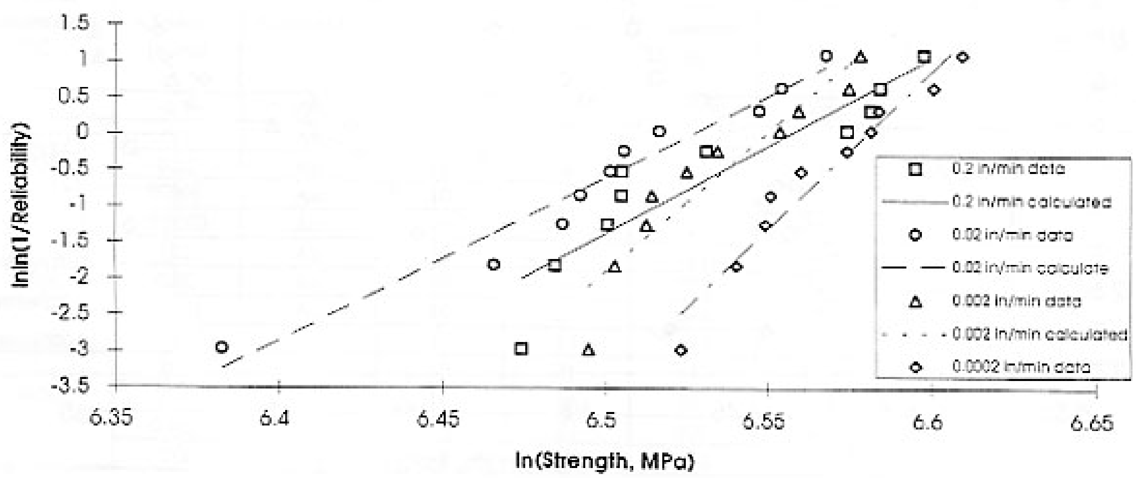

FIGURE 1. SN-88 MAXIMUM LIKELIHOOD PROBABILITY PLOTS FOR STRENGTHS MEASURED AT ALL FOUR DISPLACEMENT RATES AND 1038°C.

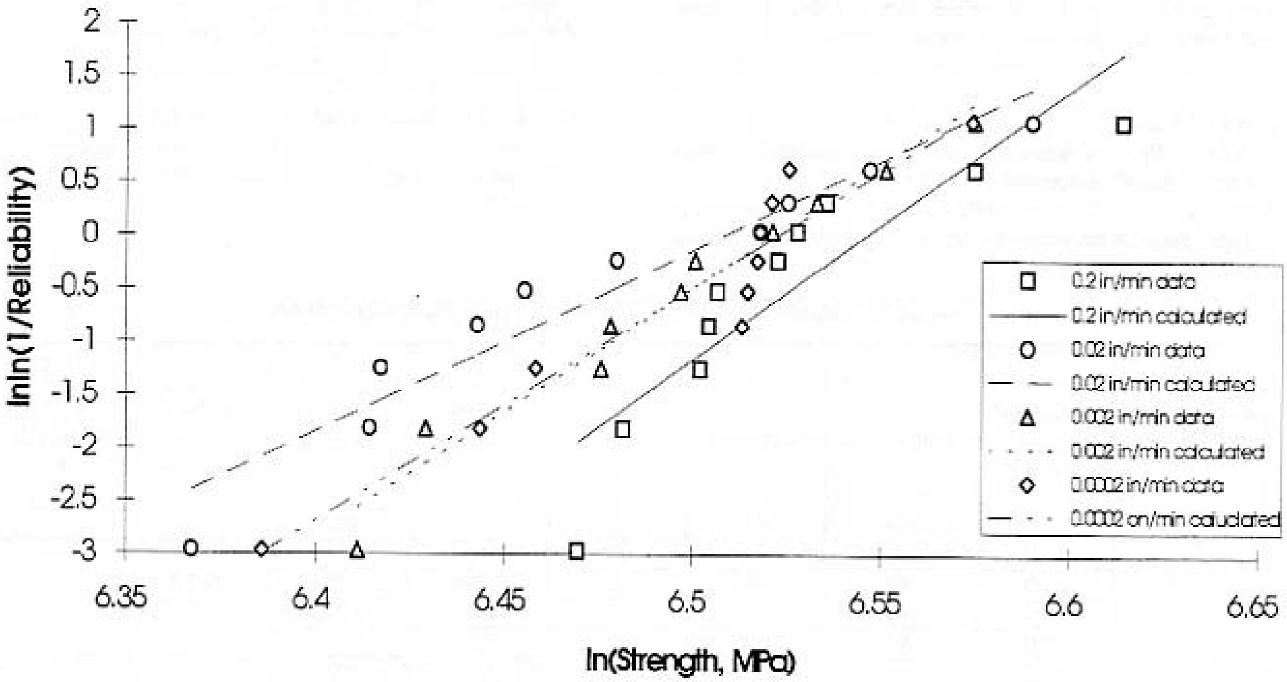

FIGURE 2. SN-88 MAXIMUM LIKELIHOOD PROBABILITY PLOTS FOR STRENGTHS MEASURED AT ALL FOUR DISPLACEMENT RATES AND 1150°C.

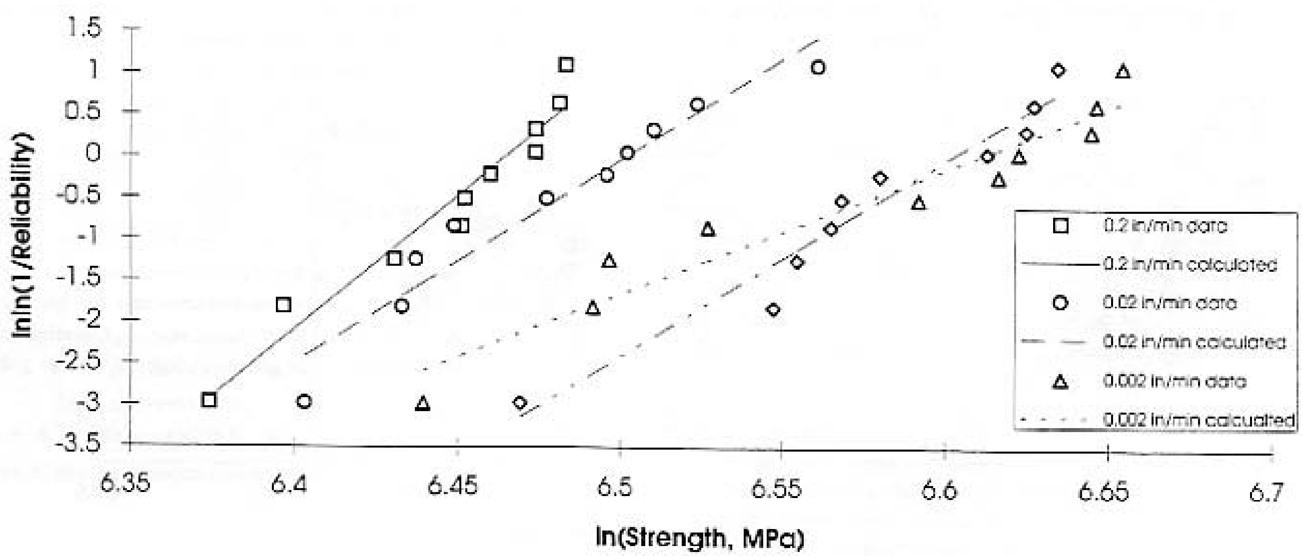

FIGURE 3. SN-88 MAXIMUM LIKELIHOOD PROBABILITY PLOTS FOR STRENGTHS MEASURED AT ALL FOUR DISPLACEMENT RATES AND 1350°C.

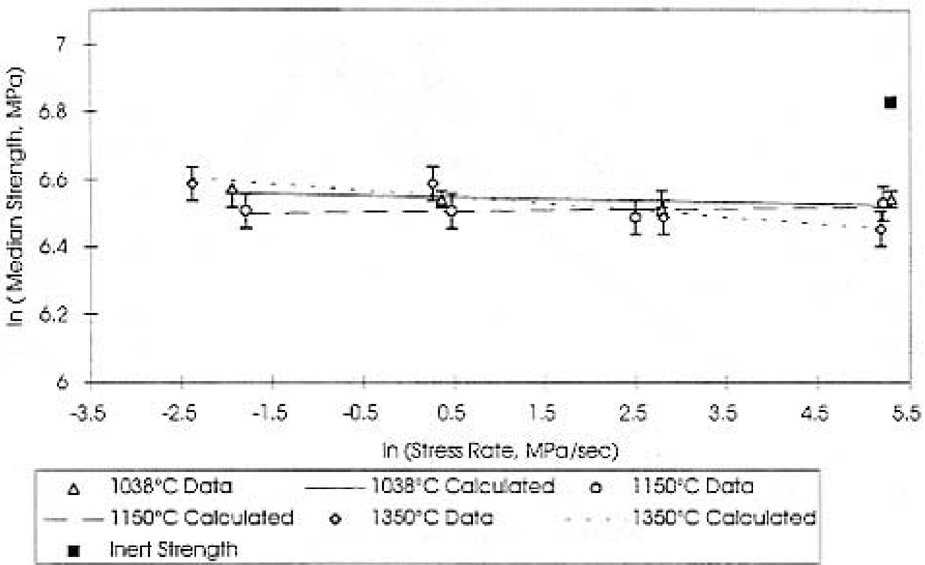

FIGURE 4. SN-88 DYNAMIC FATIGUE PLOTS AT ALL THREE TEST TEMPERATURES.

TABLE 2. FLEXURAL STRENGTH MEASURED FOR NORTON NT164

Test Temperature °C	Crosshead Speed in/min	Atmosphere	Number of Tests	Weibull Modulus m	Weibull Scale Parameter σ_0	Median Strength σ_m	Dynamic Fatigue Shape Parameter n	Dynamic Fatigue Scale Parameter B (secMPa²)
20	0.1	Air	10	14	1134	1106		
1038	0.2	Air	10	15	768	749	405.5	1.5×10^{-70}
	0.02	Air	10	8	710	680		
	0.002	Air	10	17	760	744		
	0.0002	Air	10	25	724	713		
1150	0.2	Air	10	23	781	769	34.5	0.371
	0.02	Air	10	21	733	721		
	0.002	Air	10	28	688	659		
	0.0002	Air	10	17	645	631		
1350	0.2	Air	10	21	647	635	17.8	9.61
	0.02	Air	10	16	559	546		
	0.002	Air	10	12	500	486		
	0.0002	Air	10	20	432	424		

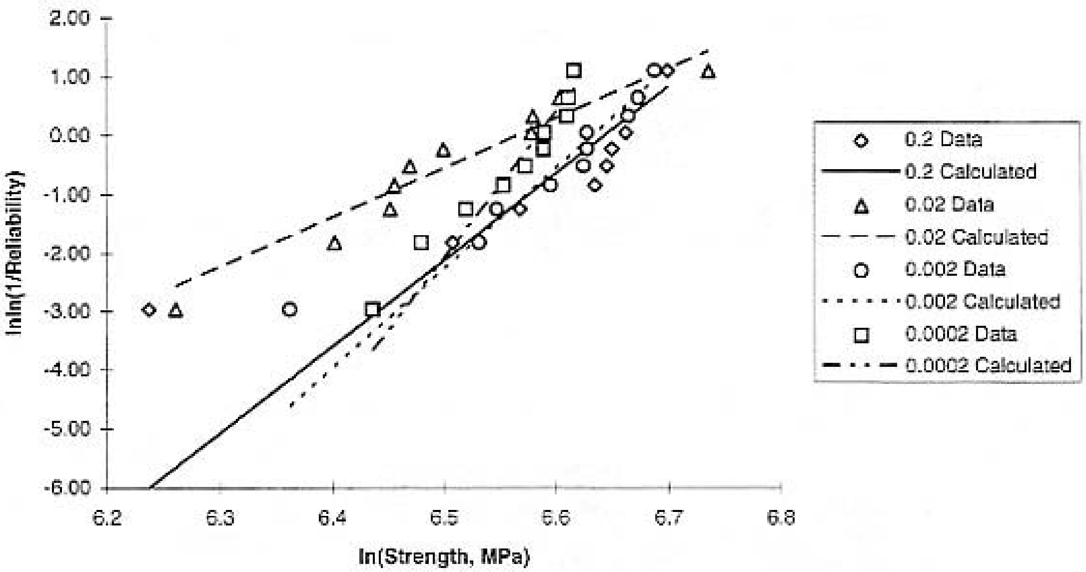

FIGURE 5. NT164 MAXIMUM LIKELIHOOD PROBABILITY PLOTS FOR STRENGTHS MEASURED AT ALL FOUR DISPLACEMENT RATES AND 1038°C.

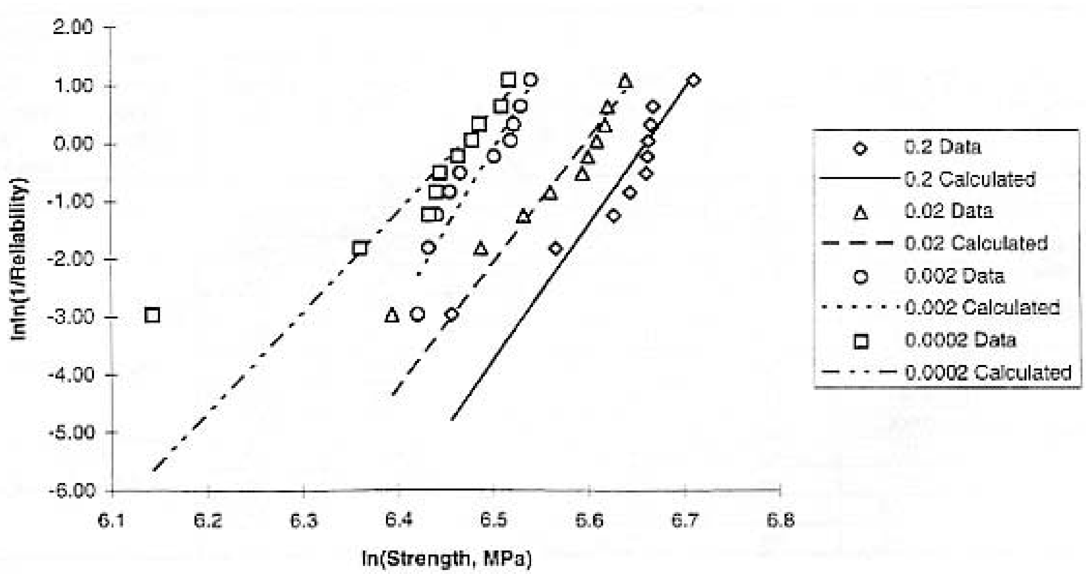

FIGURE 6. NT164 MAXIMUM LIKELIHOOD PROBABILITY PLOTS FOR STRENGTHS MEASURED AT ALL FOUR DISPLACEMENT RATES AND 1150°C.

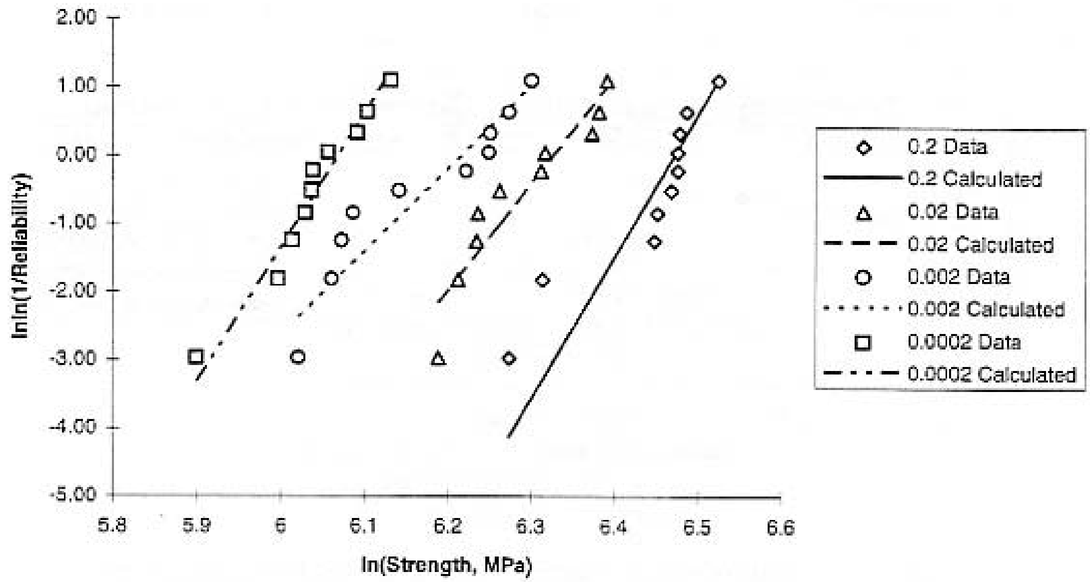

FIGURE 7. NT164 MAXIMUM LIKELIHOOD PROBABILITY PLOTS FOR STRENGTHS MEASURED AT ALL FOUR DISPLACEMENT RATES AND 1350°C.

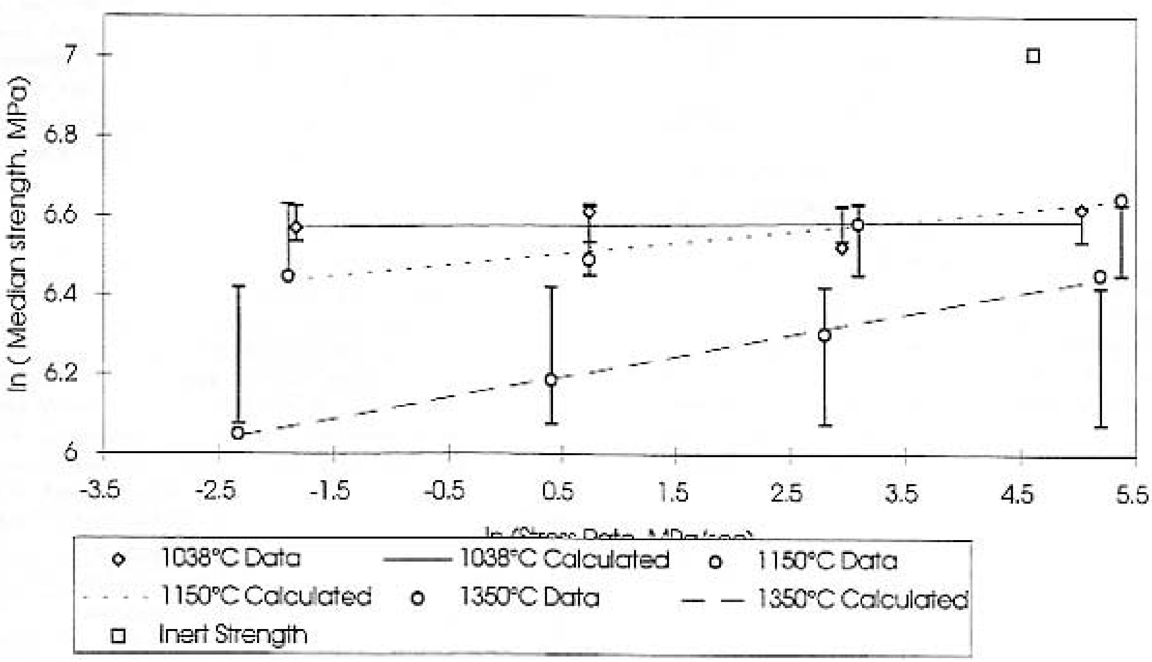

FIGURE 8. NT164 DYNAMIC FATIGUE PLOTS AT ALL THREE TEST TEMPERATURES.

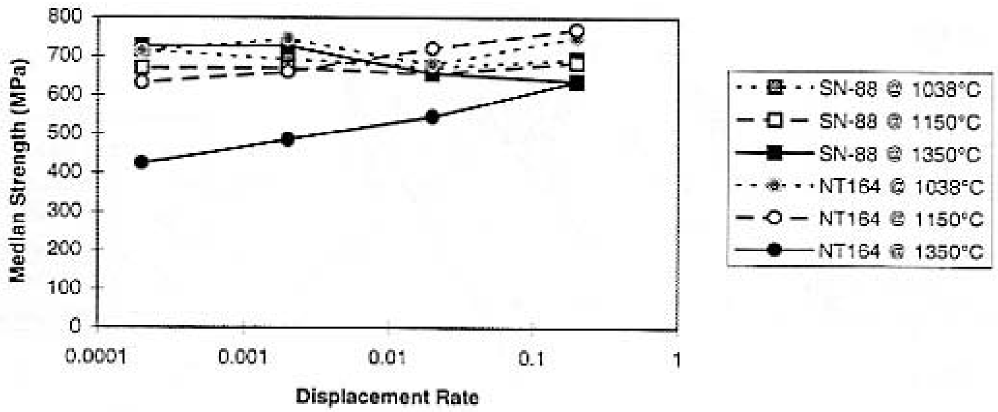

FIGURE 9. COMPARISON OF MEDIAN STRENGTH FOR NT164 AND SN-88.