ACCELERATED TESTING METHODOLOGY FOR THE DETERMINATION OF SLOW CRACK GROWTH OF ADVANCED CERAMICS

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Introduction

Constant stress-rate ("dynamic fatigue") testing has been used for several decades to characterize slow crack growth behavior of glass and ceramics at both ambient and elevated temperatures (refs. 1,2). The advantage of constant stress-rate testing over other methods lies in its simplicity: Strengths are measured in a routine manner at four or more stress rates by applying a constant crosshead speed or constant loading rate (Figs. 1 and 2). The slow crack growth parameters (n and A) required for design can be estimated from a relationship between strength and stress rate (refs. 1,2).

With the proper use of preloading in constant stress-rate testing, an appreciable saving of test time can be achieved (Fig. 2). If a preload corresponding to 50 % of the strength is applied to the specimen prior to testing, 50 % of the test time can be saved as long as the strength remains unchanged regardless of the applied preload. In fact, it has been a common, empirical practice in strength testing of ceramics or optical fibers to apply some preloading (<40%). The purpose of this work is to study the effect of preloading on the strength to lay a theoretical foundation on such an empirical practice. For this purpose, analytical and numerical solutions of strength as a function of preloading were developed. To verify the solution, constant stress-rate testing using glass and alumina at room temperature and alumina, silicon nitride, and silicon carbide at elevated temperatures was conducted in a range of preloadings from 0 to 90 %.

Solution

The analytical and numerical solutions of strength as a function of preloading has been obtained previously (refs. 3,4). For the natural flaw system with no residual stress field, the normalized (or 'reduced') strength as a function of preloading can be expressed as follows:

$$\bar{\sigma}_f = (1+\alpha_p^{n+1})^{1/(n+1)} \tag{1}$$

where $\bar{\sigma}_f$ is the normalized strength, in which the strength with preloading is normalized with respect to the strength with zero preloading, α_p is the preloading factor $(1 \le \alpha_p < 1)$, where the preloading stress is normalized with respect to the strength with zero preloading, and n is the slow

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crack growth (SCG) parameter. In this analysis, slow crack growth was described by the following well-known power-law equation

$$v = da/dt = A[K_I / K_{IC}]^n \tag{2}$$

where v is the crack growth rate, a the crack size, t time, A the SCG parameter, K_I the mode I SIF, and K_{IC} fracture toughness of a material. The resulting plot of Eq .(1) for various values of n is shown in Fig. 4(a). For the indentation crack system where an additional term appears in the net SIF, due to the residual contact stress produced by elastic/plastic indentation deformation, analytical solution was not feasible. The solution was thus made via numerical methods (ref. 4). The result of the numerical solution of strength as a function of preloading is presented in Fig. 4(b).

Experimental

The materials used were soda-lime glass, 96 wt % alumina (ALSIMAG 614, G.E. Ceramics), NC132 silicon nitride (Norton Co.), and NC203 silicon carbide (Norton Co.). Constant stress-rate testing was conducted in four-point flexure using soda-lime glass plates and as-machined MOR alumina bars at room- temperature distilled water, and using as-machined alumina bars at 1000°C air, as-machined NC132 silicon nitride bars at 1100°C and as-machined NC203 silicon carbide bars at 1300°C. Indentation-induced surface flaws both with and without residual contact stress field were used for glass specimens. A typical range of applied stress rates was 0.03 to 333 MPa/s. After the regular constant stress-rate testing (i.e., without preloading), additional testing was performed to determine the influence of preloading on strength and to verify the solutions. Typically, five preloads ranging from 50 to 90 % were used at each chosen stress rate.

Results and Discussion

1) Constant Stress-Rate Testing

The results of the constant stress-rate testing for each material are shown in Fig. 6. The SCG parameter n was found to be: $n = 17.1 \pm 0.5$ for indented-and-annealed glass, $n' = 17.4 \pm 0.4$ for as-indented glass, $n = 41.7 \pm 2.3$ for as-machined alumina at RT, $n' = 40.2 \pm 4.9$ for as-indented alumina at RT, $n = 7.6 \pm 0.3$ for alumina at 1000°C, $n = 18.6 \pm 1.7$ for NC132 at 1100°C, and $n = 29.7 \pm 1.5$ for NC203 at 1300°C.

2) Strength as a Function of Preloading

The results of the preloading experiments for each material are shown in Fig. 7, where strength was plotted as a function of preloading from 0 to 90 %. The line in the figures represents the strength obtained with zero preloading at each test rate. It is evident from these figures that the strength is almost insensitive to preloading for most of the materials tested either at room temperature or at elevated temperatures.

3) Comparison with Theoretical Solutions

A comparison of the solutions with the experimental data can be made if the strength with preloading are normalized with respect to the strength without preloading at each stress rate, obtaining $\bar{\sigma}_f$ in accordance with Eq. (1) and the numerical solution. The resulting plots are shown in Fig. 8. The theoretical line by Eq. (1) or by the numerical solution calculated with the estimated SCG parameter *n* for each case was also included. Except for NC203, the theory is in good agreement with the experimental data, thereby indicating that the solutions are valid not only at room temperature but at elevated temperatures. Note that the variation in strength (about 5%) is attributed to the inherent scatter of strength exhibited by the materials. NC203 SiC exhibited the highest variation because of its low Weibull modulus ($m \approx 8$). The effect of strength scatter (Weibull modulus) on preloading is illustrated in Fig. 9. Excellent agreement is found in the indented glass specimens and the alumina specimens, since the specimens exhibited a high Weibull modulus of greater than 20.

4) Crack Growth Behavior

The reason why the preloading technique is workable is due to the fact that most of the crack growth occurs close to and/or at failure time at which fracture strength is defined. The nature of this long "incubation" time of an initial crack is a key aspect that makes the preloading technique feasible in constant stress-rate testing (see Fig. 10).

5) Implications

The most direct and powerful effect of preloading technique is the saving of test time, which gives a great impact on testing efficiency (Fig. 11). For example, if it takes about 9 h to test one ceramic specimen in constant stress rate testing and if a minimum of 20 specimens are required to obtain reliable statistical data, then total testing time at that stress-rate would be 180 h. But if a preloading of 80% is applied, the total testing time would be reduced to 36 h so that 80 % of the total test time can be saved. And 70 % saving for a preload of 70 %, and so on. This great advantage of the preloading technique has been adopted to a recently established ASTM standard on slow crack growth testing for advanced ceramics. Also, the preloading technique can be used as a tool identifying a mechanism associated with failure at elevated temperatures (ref. 3), as shown in Fig. 12.

References

1. Evans, A. G., Slow Crack Growth in Brittle Materials under Dynamic Loading Conditions, Int. J. Fract., vol. , no. 2, 1974, pp. 252-259.
2. Ritter, J. E., Engineering Design and Fatigue Failure of Brittle Materials," pp. 661-686 in Fracture Mechanics of Ceramics, vol. 4, edited by R. C. Bradt, D. P. H. Hasselman, and F. F. Lange, Plenum, New York, 1978.
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4. (a) Choi, S. R., Salem, J. A., and Gyekenyesi, J. P., Fatigue Strength as a Function of Preloading in Dynamic Fatigue Testing of Glass and Ceramics, ASME Paper # 96-GT-342, accept for publication in J. Eng. for Gas Turbines and Power, Trans. of ASME; (b) Choi, S. R., and Salem, J. A., Preloading Technique in Dynamic Fatigue Testing of Glass and Ceamics with an Indentation Flaw System, J. Am. Ceram. Soc., vol. 79, no. 3, 1996, pp. 1228-1232; (c) Choi, S. R., and Salem, J. A., Preloading Technique in Dynamic Fatigue Testing of Ceramics: Effect of Preloading on Strength Variation, J. Mater. Sci. Lett., vol. 15, 1996, pp. 1963-1965.

DETERMINATION OF SLOW CRACK GROWTH OF ADVANCED CERAMICS

$$v = A\,[K_I/K_{IC}]^n$$

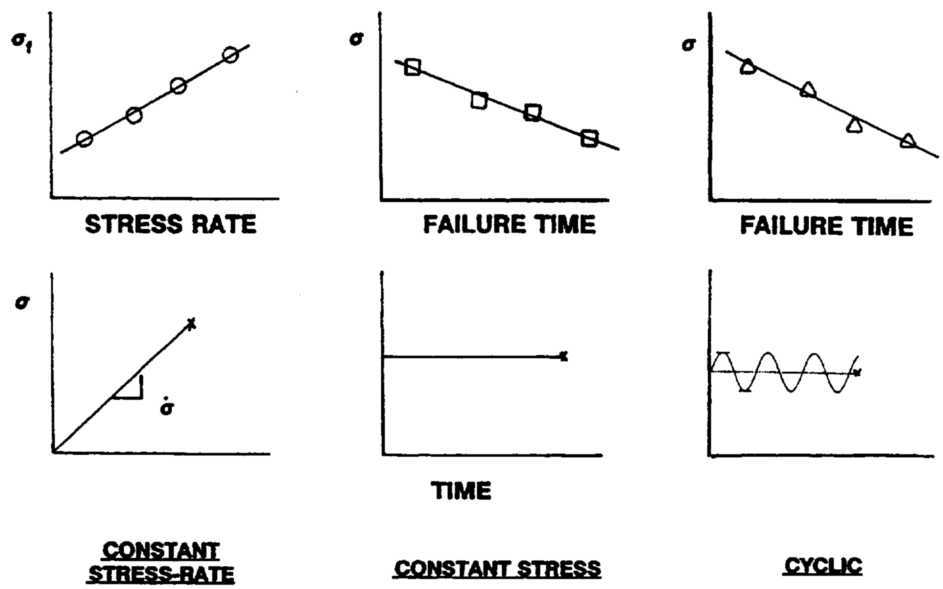
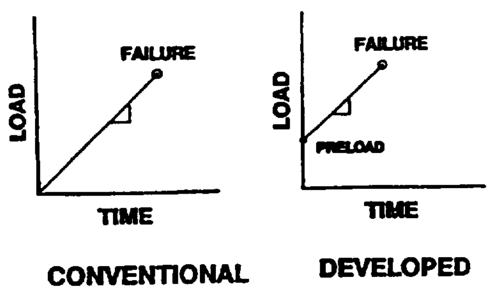


Fig. 1

CONSTANT STRESS-RATE ("DYNAMIC FATIGUE") TESTING

- ¤ QUICK AND SIMPLE
- ¤ STILL TIME CONSUMING
- ¤ EMPIRICAL USE OF PRELOADING



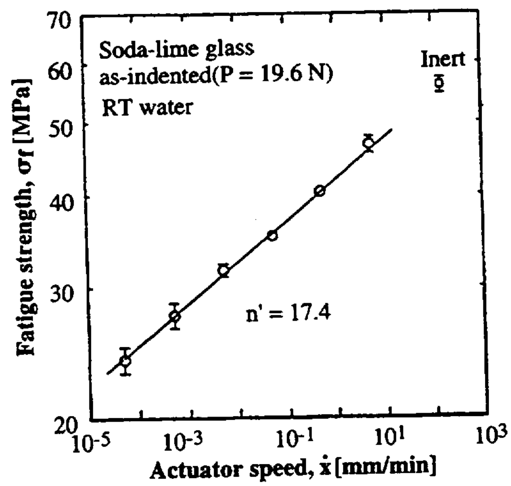


Fig. 2

PURPOSE OF STUDY

- ¤ TO DETERMINE EFFECT OF PRELOADING ON STRENGTH
- ¤ TO LAY A THEORETICAL FOUNDATION
- ¤ TO VERIFY WITH EXPERIMENT
- ¤ TO ESTABLISH AS A TESTING METHODLOGY

Fig. 3

ANALYTICAL AND NUMERICAL SOLUTIONS

1. NATURAL FLAWS

$$v = A\left[\frac{K_I}{K_{Ic}}\right]^n$$

$$K_I = Y \sigma_a \sqrt{a}$$

$$\sigma_a = \sigma_o + \dot{\sigma} t$$

$$\boxed{\bar{\sigma}_f = [1 + \alpha_p^{n+1}]^{\frac{1}{n+1}}}$$

where

$$\bar{\sigma}_f = \frac{\sigma_{fp}}{\sigma_{fn}} = \frac{\textit{Fatigue Strength w/ Preload}}{\textit{Fatigue Strength w/o Preload}}$$

$$\alpha_p = \frac{\sigma_o}{\sigma_{fn}} = \frac{\textit{Preload Stress}}{\textit{Fatigue Strength w/o Preload}}$$

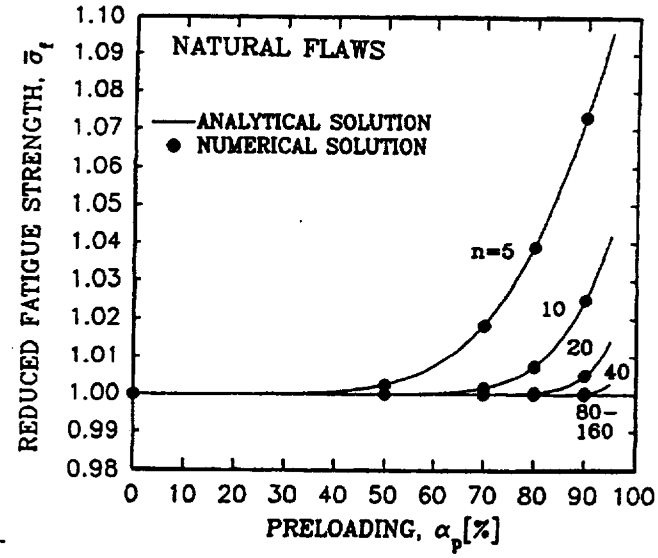


Fig. 4(a)

2. INDENTATION-INDUCED FLAWS (NUMERICAL SOLUTION)

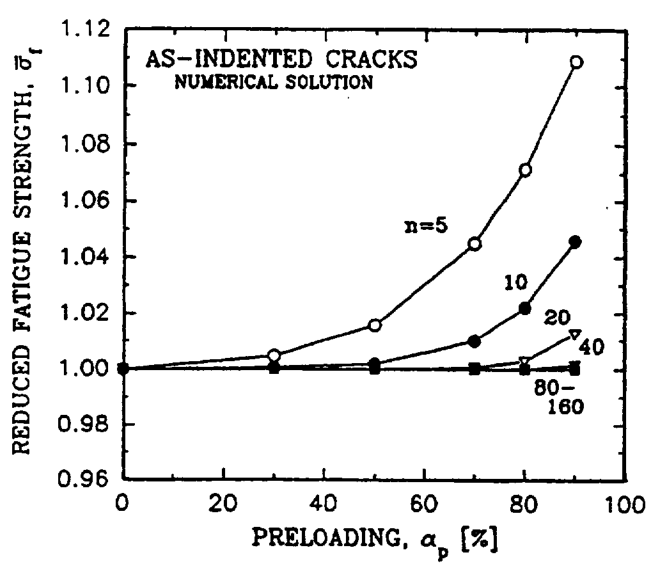


Fig. 4(b)

EXPERIMENTAL

¤ MATERIALS:
- ROOM TEMPERATURE:
  - GLASS (AS-INDENTED & ANNEALED) (2)
  - ALUMINA (AS-INDENTED & AS-MACHINED) (2)
- ELEVATED TEMPERATURES:
  - ALUMINA (1000°C)
  - NC132 SILICON NITRIDE (1100°C)
  - NC203 SILICON CARBIDE (1300°C)

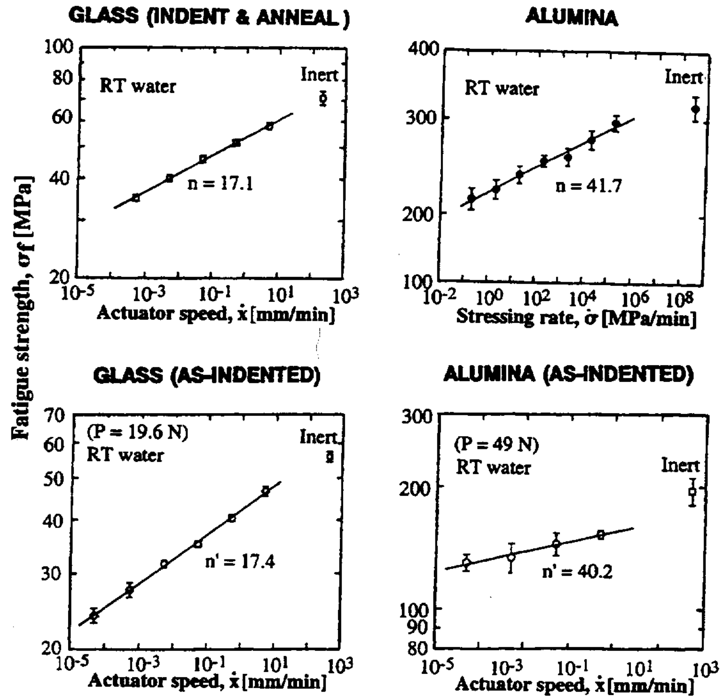
¤ CONSTANT STRESS-RATE TESTING:
- FOUR-POINT FLEXURE
- 0.03 TO 333 MPa/s (A RANGE OF STRESS RATES)

¤ PRELOADING EXPERIMENT:
- PRELOAD RANGING FROM 50 TO 90 %

Fig. 5

RESULTS OF CONSTANT STRESS-RATE TESTING

(ROOM TEMPERATURE)



(HIGH TEMPERATURES)

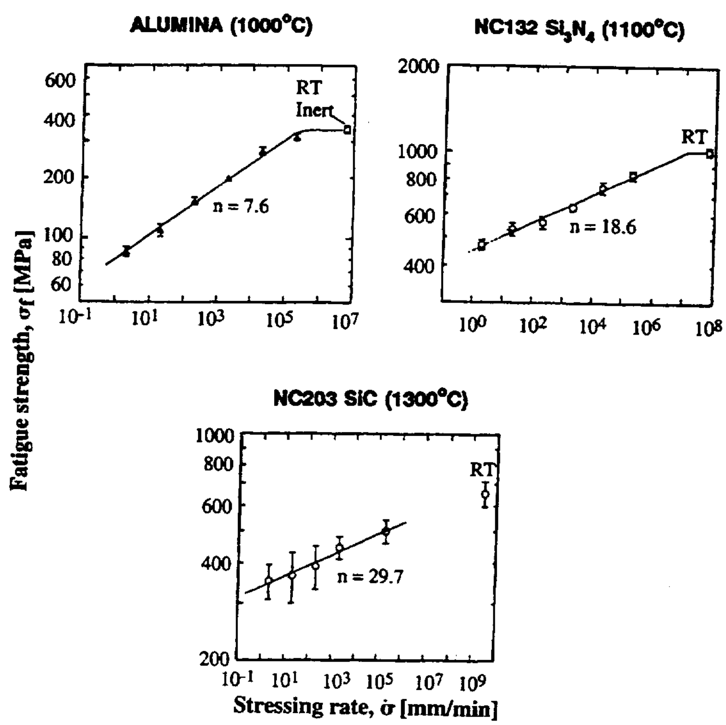
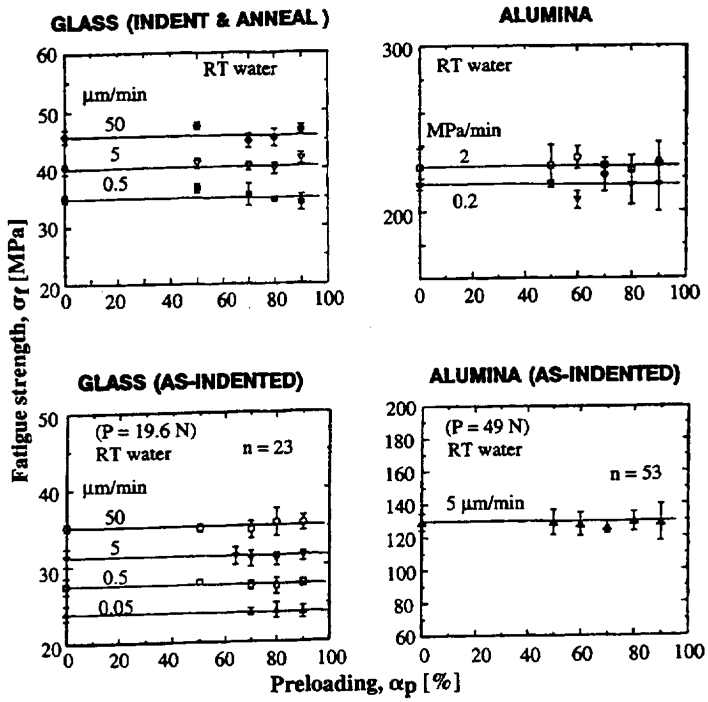


Fig 6

RESULTS OF PRELOADING EXPERIMENTS

(ROOM TEMPERATURE)



(HIGH TEMPERATURES)

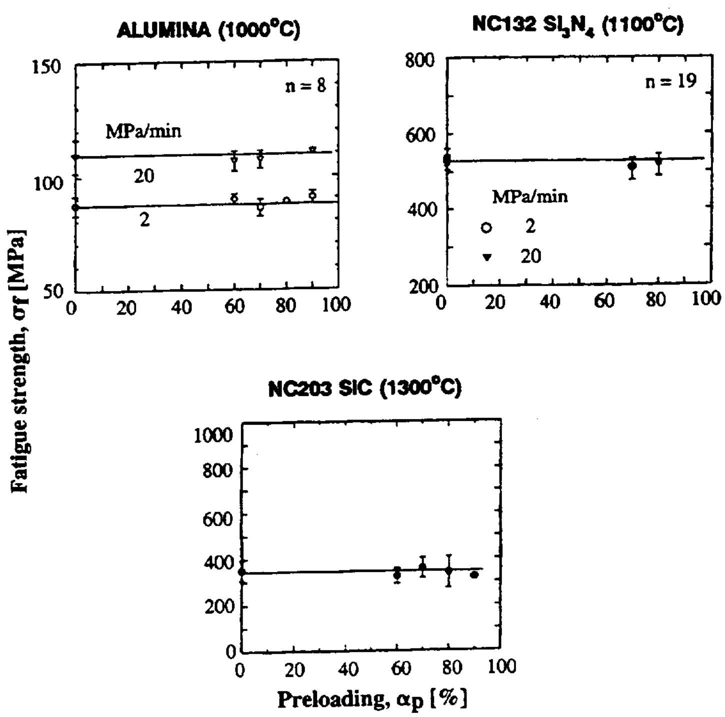
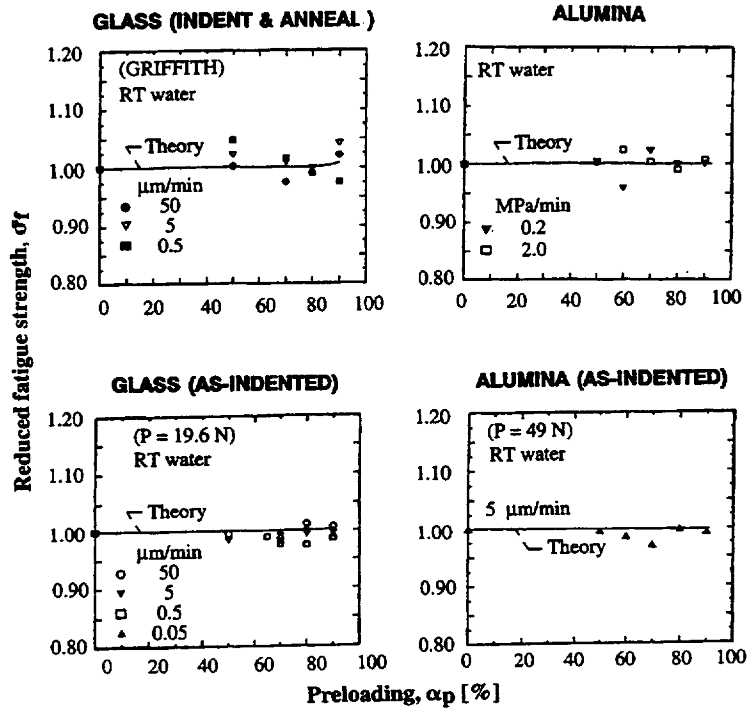


Fig. 7

COMPARISON WITH THEORY

(ROOM TEMPERATURE)



(HIGH TEMPERATURES)

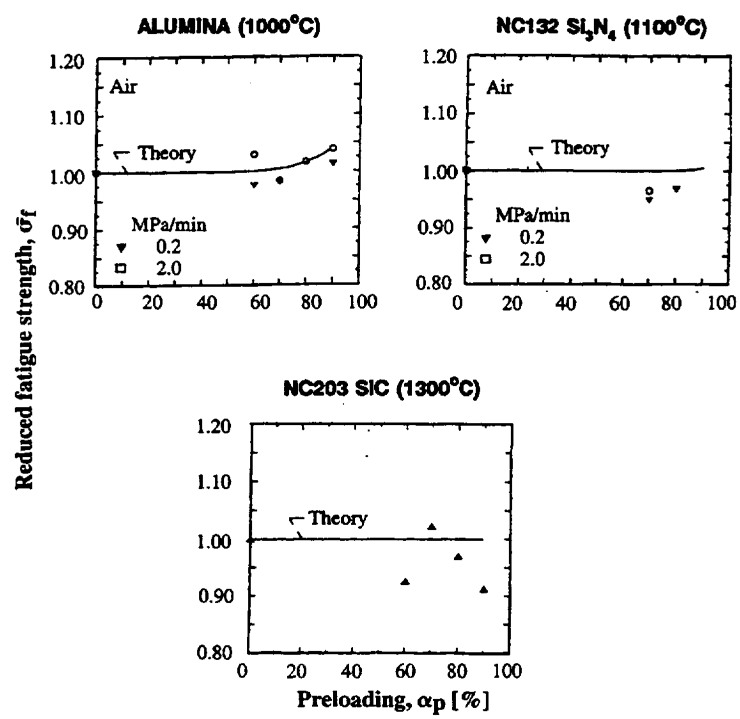


Fig. 8

EFFECT OF STRENGTH SCATTER ON PRELOADING

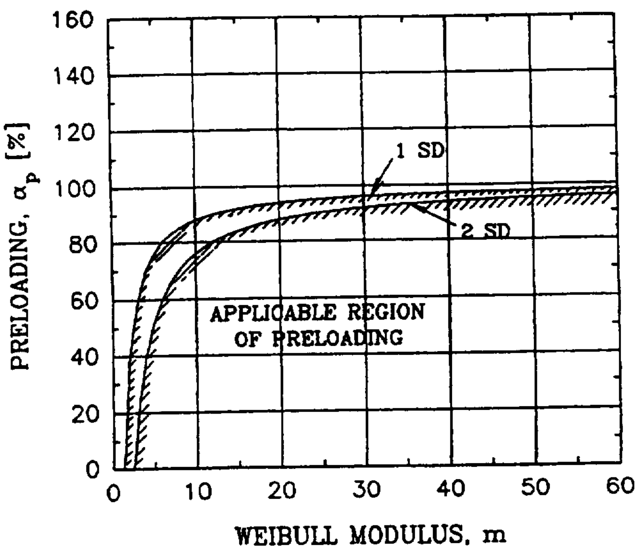
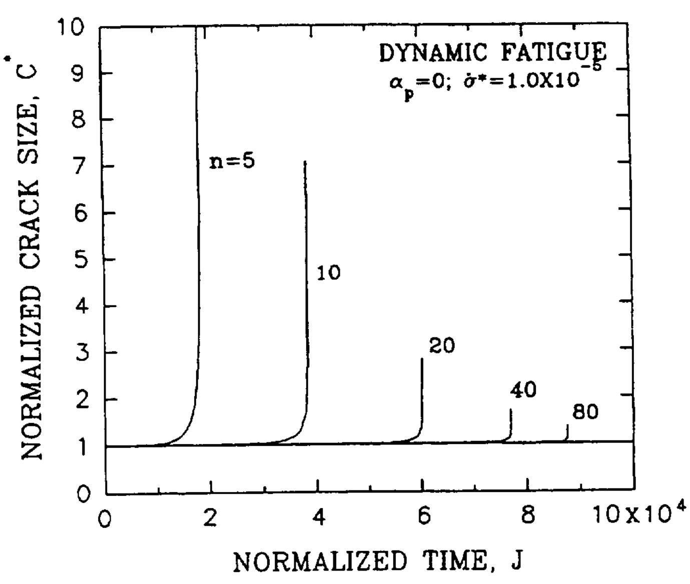


Fig. 9

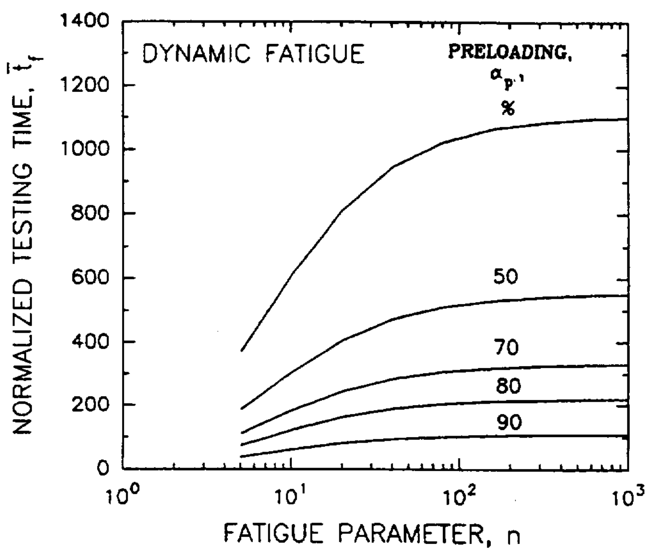
REASON FOR WORKABILITY OF PRELOADING



A CRACK STARTS TO GROW AFTER A LONG “INCUBATION” TIME !

Fig. 10

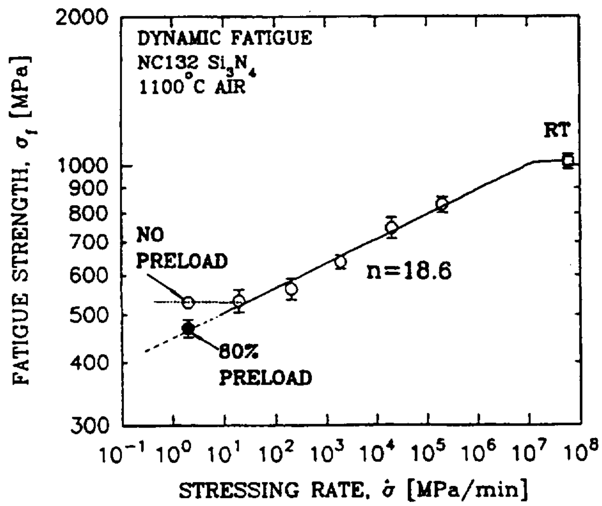
DRAMATIC TEST-TIME SAVING



90 % PRELOAD ---- 90 % SAVING IN TEST TIME
80 % PRELOAD ---- 80 % SAVING
AND SO ON !
A GREATER IMPACT ON TEST EFFICIENCY!

Fig. 11

A TOOL IDENTIFYING A FAILURE MECHANISM



- **PROLONGED TIME — CREEP DEFORMATION**
  CRACK BLUNTING
  STRENGTH INCREASE

- **80% PRELOADING — DECREASED EXPOSURE TIME**
  CREEP MINIMIZED/OR VANISHED
  MAXIMIZES SLOW CRACK GROWTH

Fig. 12

CONCLUSIONS

¤ THE SOLUTION VERIFIED BY EXPERIMENTS

¤ PRELOAD UP TO 80 % POSSIBLE FOR MOST CERAMICS (n>20)

¤ THE TECHNIQUE --- DRAMATIC TEST-TIME SAVING
A GREAT IMPACT ON TEST EFFICIENCY

¤ THE TECHNIQUE --- ADOPTED IN AN 'ASTM' STANDARD ON
SLOW CRACK GROWTH TESTING OF CERAMICS

Fig. 13