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Proof testing of ceramics

Part 1 *Experiment*

J. E. RITTER Jr, P. B. OATES
Mechanical Engineering Department, University of Massachusetts, Amherst, Massachusetts 01003, U.S.A.

E. R. FULLER Jr, S. M. WIEDERHORN
National Bureau of Standards, Washington, D.C. 20234, U.S.A.

The effectiveness of proof testing as a method of improving component reliability was studied by comparing strength distributions of soda-lime–silica glass before and after proof testing. The effects of unloading rate from the proof stress, hold time at the proof stress, and proof test environment were examined. The results indicate that the proof test must be conducted with rapid unloading rates and good environmental control to be effective. The theoretical implications of these results are discussed.

1. Introduction

Proof testing is one means used to assure the mechanical reliability of structural ceramics. In proof testing, ceramic components are subjected to stresses that are greater than those expected in service in order to break the weak components and thus truncate the low end of the strength distribution. In this manner, weak components are eliminated before they can be placed in service. Proof testing has been applied to spacecraft windows [1, 2], electrical porcelain insulators [3], and vitrified grinding wheels [4].

Wiederhorn, Evans, and Fuller [5–7] have provided a mathematical foundation for the selection of the proof test stress and the establishment of proof test conditions. Their analysis is based on the assumption that failure of ceramics occurs from the growth of pre-existing flaws. By characterizing this crack growth and coupling crack growth parameters with proof testing, they derived the strength after proof testing assuming flaw growth during the proof stress, load–unload cycle. The resulting theory indicates that crack growth must be minimized to have effective proof testing. This can be achieved by having rapid unloading from the proof stress and good environmental control during the proof test.

In support of the proof test theory, Ritter [8] has shown recently for soda-lime–silica glass after proof testing that the inert strength distribution and failure time under static stress agree with that predicted from theory. Aside from this study, there has been no extensive experimental confirmation of proof test theory. The purpose of the present study, therefore, was to conduct a detailed study of the proof test technique and to assess the validity of the theory in predicting the strength after proof testing. In particular, the effectiveness of proof testing and the applicability of proof test theory was determined by comparing the inert strength distributions of soda-lime–silica glass before and after proof testing over a range of proof test conditions. The major proof test variables were unloading rate fromthe proof stress and the proof test environment. Soda-lime–silica glass was chosen as the model material for this study because it is readily available and because its subcritical crack behaviour is well characterized.

2. Experimental procedure

All specimens used in this study were soda-lime–silica glass, microscope slides* ($7.62 \times 2.54 \times 0.10\,cm^3$). The slides were annealed at 500° C for 1 h, furnace cooled, and then abraded in the centre with a standard blast of No. 240 SiC grit. After abrasion, the samples were aged for 24 h in

*Corning No. 2947.

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distilled water to normalize their strength and then stored in a desiccator prior to testing. Over 2000 samples were prepared in this manner and they were randomly selected for testing.

The inert strength measurments before and after proof testing were made in liquid nitrogen using four point bending. The bending apparatus had inner and outer supports of 2.54 and 5.08 cm, respectively. All strength testing was done on an universal testing machine† using a constant cross-head speed of 0.2 cm min^{-1}, corresponding to a stressing rate of 5.29 MPa sec^{-1}. The initial inert strength distribution was determined from 79 samples while the after-proof strength distributions were determined from about 30 samples.

The bend apparatus was also used for proof testing. The samples were loaded up to the proof stress at a stressing rate of 5.29 MPa sec^{-1} and then unloaded at various rates (132.33, 5.29, and 1.32 MPa sec^{-1}). The time at the proof stress varied from momentary (less than 0.5 sec) to 60 sec. The proof test environments were liquid nitrogen, dry nitrogen gas (about 5% relative humidity), ambient air (55 to 65% relative humidity) and water. The last three test environments were at room temperature, about 23° C. Groups of 50 samples were proof tested for each proof test condition and proof test stresses were chosen to break approximately 40% of a set of test specimens, leaving about 30 samples for the determination of the inert strength distribution after proof testing.

3. Results and discussion

The strength after proof testing has been derived for 3 conditions [5–7]: no flaw growth during proof testing, flaw growth up to unloading, and flaw growth during the entire proof stress cycle. If no flaw growth occurs during the proof test, then the inert strengths for a given specimen before and after proof testing are equal; however because the weak samples have been eliminated from the initial distribution, the cumulative failure probability after proof testing (F_a) will have changed [5]

$$F_a = \frac{F - F_p}{1 - F_p} \tag{1}$$

where F is the cumulative failure probability before proof testing and F_p is the cumulative failure probability of the proof test.*

Assuming that a single power law relationship exists between subcritical crack velocity and the stress intensity factor

$$V = AK_I^n \tag{2}$$

where A and n are constants for a given material and test environment, the inert strength after proof testing (S_f) can be derived accounting for crack growth during the proof stress cycle. Considering crack growth up to unloading but not during unloading, the S_f distribution is given by [5, 6]

$$\left(\frac{S_f}{S_o}\right)^{n-2} = \left(\ln\frac{1}{1-F_a} + \ln\frac{1}{1-F_p}\right)^{\frac{n-2}{m}} - \left(\ln\frac{1}{1-F_p}\right)^{\frac{n-2}{m}} + \left(\frac{\sigma_p}{S_o}\right)^{n-2} \tag{3}$$

where n is the crack propagation parameter appropriate for the proof test environment, σ_p is the proof stress, and m and S_o are the Weibull shape and scale parameters, respectively, of the initial inert strength distribtuion. If flaw growth occurs during the entire proof stress cycle, the S_f distribution is now [6]

$$\left(\frac{S_f}{S_o}\right)^{n-2} = \left(\ln\frac{1}{1-F_a} + \ln\frac{1}{1-F_p}\right)^{\frac{n-2}{m}} - \left(\ln\frac{1}{1-F_p}\right)^{\frac{n-2}{m}} \tag{4}$$

For purposes of identification in the following discussion, the various predicted S_f distributions willl be labelled: Type I, based on Equation 1 for no flaw growth, Type II, based on Equation 3 for no flaw growth on unloading, Type III, based on Equation 4 for flaw growth during entire proof stress cycle. For all three types of distributions it can be shown that S_f is greater than the initial strength at all levels of failure probability, provided $m < n - 2$. For Type I and II distributions, the S_f distribution is truncated at σ_p; hence, σ_p represents the minimum inert strength after proof testing.

†Instron Corp., Canton, MA.

*F is obtained by ordering a set of strength data. F is given by $r/(N+1)$ where N is the total number of datum points and r is the position of each point in the ordered set. $r = 1$ for the lowest strength, $r = 2$ for the second lowest and so forth.

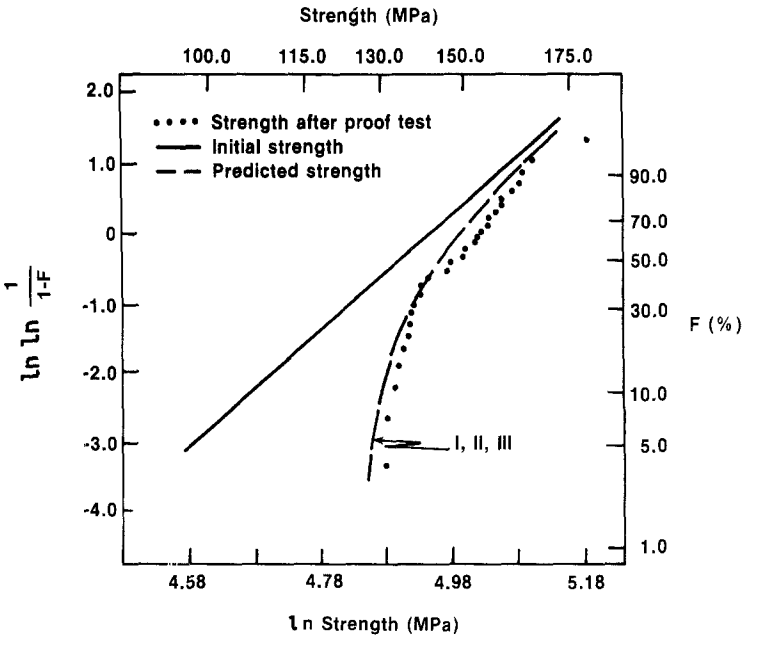

Figure 1 Inert strength distributions of soda-lime–silica glass before and after proof testing compared to the theoretical, after proof distributions. Proof testing was in liquid nitrogen at an unload rate of 5.29 MPA sec^{-1} and $\sigma_p = 127.5$ MPa, $F_p = 0.36$, $n = 120$, $m = 8.19$, $S_o = 137.4$ MPa.

When flaw growth occurs on unloading (Type III distribution), the strength distribution is not truncated and no assurances of a minimum strength can be given.

The experimental inert strength distributions after proof testing could be divided generally into one of four distributions depending on the proof test conditions. Firstly, under inert proof test conditions (liquid nitrogen) the strength distributions after proof testing agreed with that predicted from either a Type I, II, or III distribution (Fig. 1). In this case the Type II and III distributions were determined by assuming an $n = 120$ [6]. As can be seen in Fig. 1 there was no significant difference between the three predicted distributions within the range of the after proof data. This observation just reflects the fact that for an inert environment such as liquid nitrogen little or no crack growth can occur because of the high n value appropriate for this environment.

Secondly, with rapid unloading rates in the dry nitrogen and air environments, the strength distributions after proof testing agreed with that predicted from either a Type II or III distribution

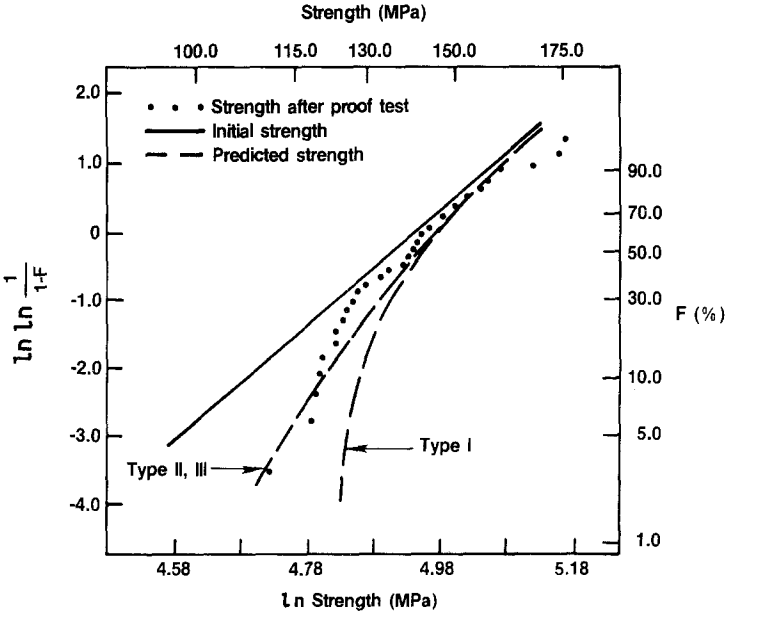

Figure 2 Inert strength distributions of soda-lime–silica glass before and after proof testing compared to the theoretical, after proof distributions. Proof testing was in air at unload rate of 132.3 MPA sec^{-1} and $\sigma_p = 79.3$ MPa, $F_p = 0.33$, $n = 18.4$, $m = 8.19$, $S_o = 137.4$ MPa.

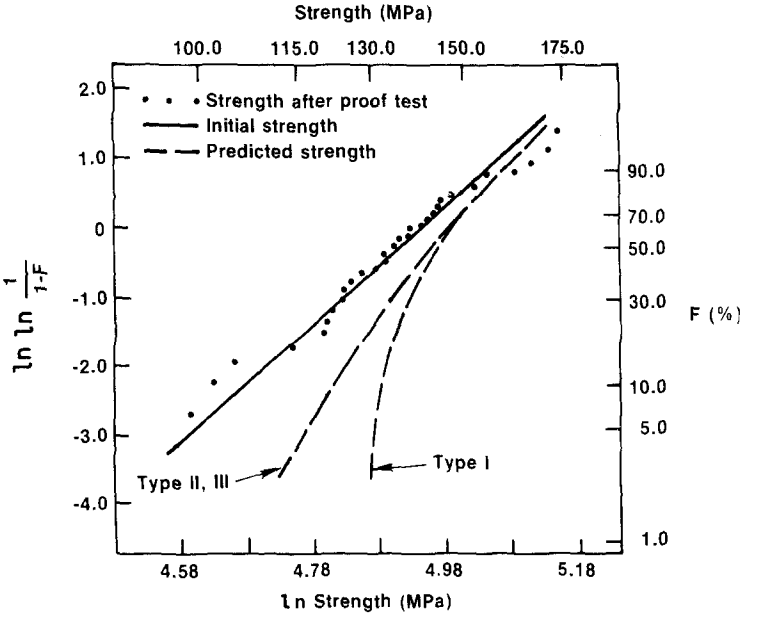

Figure 3 Inert strength distributions of soda-lime–silica glass before and after proof testing compared to the theoretical, after proof distributions. Proof testing was in water at unload rate of 132.3 MPA sec^{-1} and $\sigma_p = 68.6$ MPa, $F_p = 0.37$, $n = 18.4$, $m = 8.19$, $S_o = 137.4$ MPa.

where $n = 18.4$ [8,9] (Fig. 2). Since the strength distributions after proof testing are shifted significantly to the left of the Type I distribution, it is evident that crack growth occurs during proof testing in these "moist" environments; however, it could not be determined if the after proof strength distributions were truncated at σ_p. To conclusively show truncation, much larger sample size after proof testing would have to be used (about 10 000 samples).

Finally, when good proof test controls were not used, i.e. relatively slow unloading rates and/or moist proof test environments, the strength distributions after proof testing either were not significantly different from the initial distribution (Fig. 3) or were weaker than the initial distribution (Fig. 4). In these cases none of the theoretical distributions could explain the observed distributions after proof testing since the strengths were much weaker than predicted from theory.

Table I summarized all the proof test results. It is evident that, for effective proof testing, crack

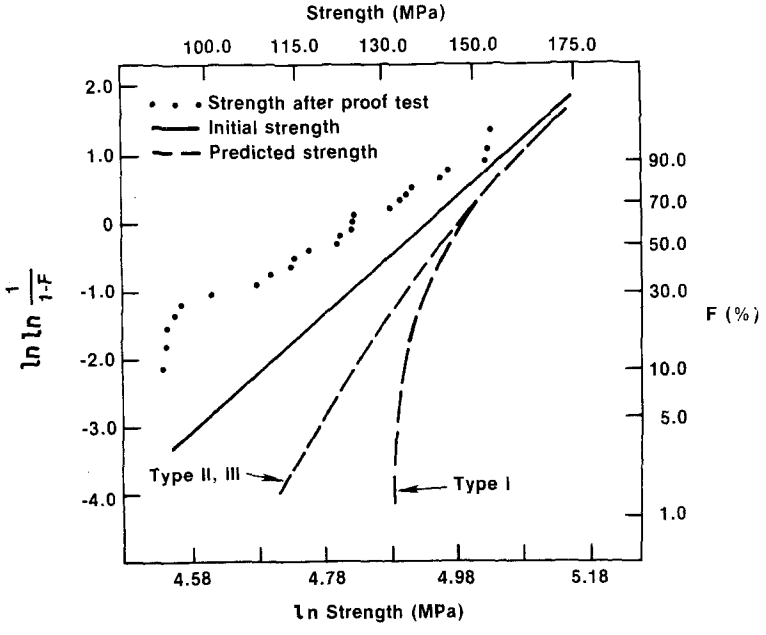

Figure 4 Inert strength distributions of soda-lime–silica glass before and after proof testing compared to the theoretical, after proof distributions. Proof testing was in air at unload rate of 5.29 MPA sec^{-1} and $\sigma_p = 79.3$ MPa, $F_p = 0.47$, $n = 18.4$, $m = 8.19$, $S_o = 137.4$ MPa.

TABLE I Summary of the proof test data for soda-lime–silica glass

Proof test environment	Proof stress unload rate (MPa sec^{-1})	Hold time at proof stress (sec^{-1})	After proof inert strength distribution
Liquid nitrogen	132.22	< 0.5	I, II, III*
Liquid nitrogen	5.29	< 0.5	I, II, III
Dry nitrogen gas	132.33	< 0.5	II, III
Dry nitrogen gas	5.29	< 0.5	Same as initial
Dry nitrogen gas	1.32	< 0.5	Same as initial
Dry nitrogen gas	5.29	60	Same as initial
Air	132.33	< 0.5	II, III
Air	5.29	< 0.5	Weaker than initial
Air	1.32	< 0.5	Weaker than initial
Air	5.29	5	Same as initial
Water	132.33	< 0.5	Same as initial
Water	5.29	< 0.5	II, III
Water	5.29	5	Same as initial

*Type theoretical distributions that agreed with after proof data.

growth must be minimized through use of good proof test conditions, namely, rapid unloading rates and good environmental control. In addition, under experimental conditions where proof testing was not effective*, none of the theoretical, after proof distributions agreed with the data. There was only one exception to these trends and that was for the proof test conducted in water at an unloading rate of 5.29 MPa sec^{-1}. It is not known why this one set of results did not fit into the general pattern.

To provide additional support to the general conclusions made above regarding the effectiveness of proof testing, a large group of as-received microscope slides were proof tested in very dry nitrogen (less than 0.1% r.h.) and in moist nitrogen (50% r.h.) using an unloading rate of about 13.2 MPa sec^{-1}. Because the dry nitrogen

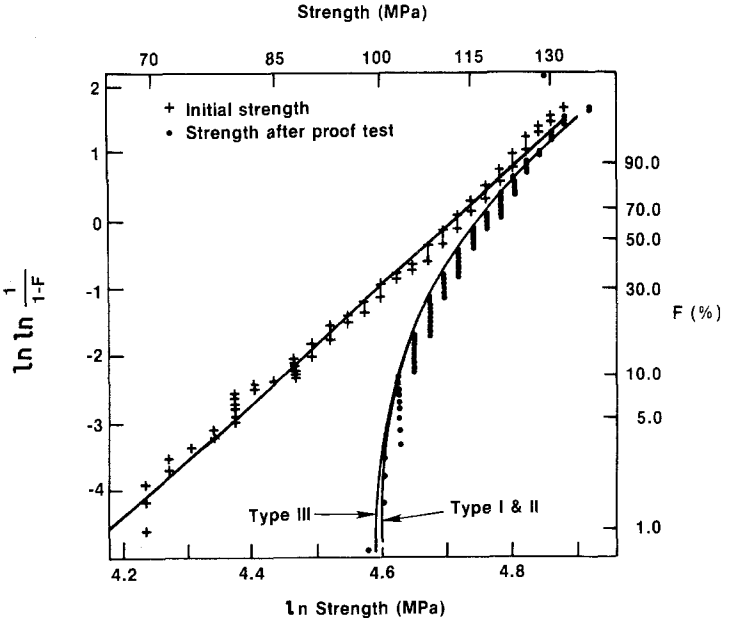

Figure 5 Strength distributions for soda-lime–silica glass in very dry nitrogen gas before and after proof testing compared to the theoretical, after proof distributions. Proof testing was in very dry nitrogen gas at unload rate of 13.3 MPa sec^{-1} and σ_p = 103.9 MPa, $F_p = 0.30$, and n = 120.

*The reader is cautioned not to jump to the conclusion that when good proof test controls are not used, proof testing will not be beneficial. It must be remembered that our conclusion is based on a group of uniformly abraded samples that could statistically be characterized by a single strength, i.e. flaw distribution. If in a set of samples to be proof tested some samples contain gross flaws, perhaps due to incorrect manufacturing, then proof testing even without ideal conditions, would undoubtedly eliminate these weak samples from the population, thereby, improving the reliability of the remaining samples.

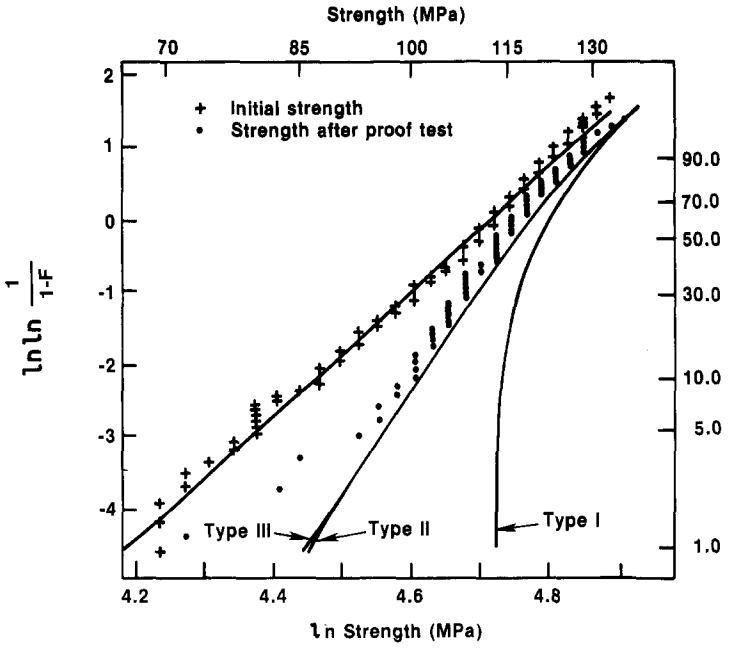

Figure 6 Strength distributions for soda-lime–silica glass in very dry nitrogen gas before and after proof testing compared to the theoretical, after proof distributions. Proof testing was in 50% r.h. nitrogen gas at unload rate of 13.2 MPa sec^{-1} and σ_p = 103.9 MPa, $F_p = 0.58$, and n = 18.4.

gas was a relatively inert environment, it was used as the test environment for the strength measurements before and after proof testing. Figs. 5 and 6 show that these results are in agreement with the trends shown in Table I. When good proof test controls are used, proof testing is effective and the strength distribution after proof testing can be characterized theoretically (Fig. 5). On the other hand, when a relatively slow unloading rate is used in a moist environment, proof

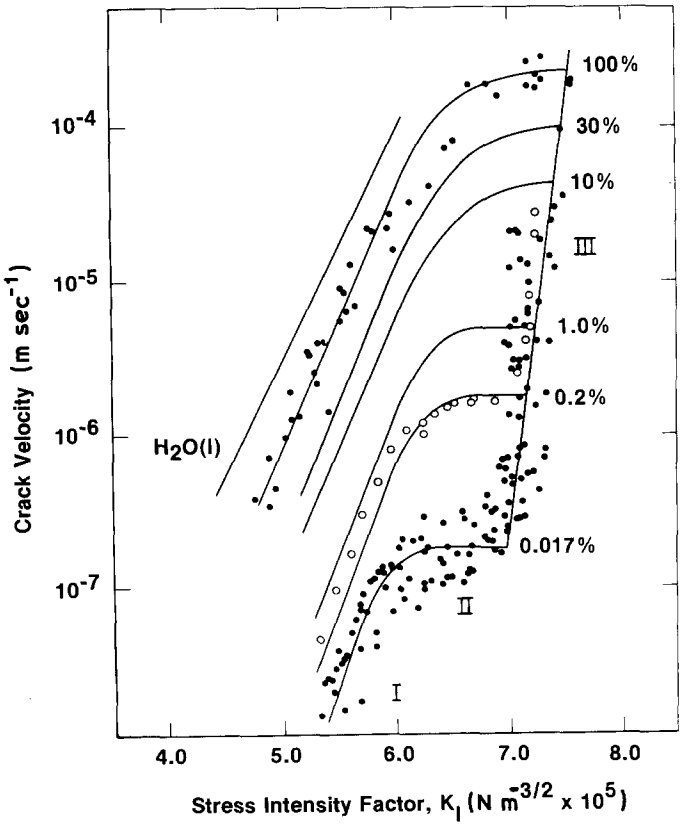

Figure 7 Crack propagation behaviour of soda-lime–silica glass in moist nitrogen gas environments. Per cent relative humidity is given on the right-hand side of diagram (after Wiederhorn [101]).

testing is not effective in truncating the distribution and the after proof strength distribution does not agree well with the theoretical distributions (Fig. 6).

Based on the above proof test results, the question of why strengths after proof testing under non-ideal conditions are significantly less that those predicted must be addressed. The use of Equation 2 in deriving the strength after proof testing (Equations 3 and 4) may be questioned, since experiments have been conducted that characterize the dependence of crack velocity on stress intensity factor by three principal regions of behaviour (Fig. 7) [10]. Regions I and II result from a stress corrosion reaction between the glass and water in the environment. The rate of the reaction between water and the glass controls crack motion in Region I, whereas diffusion of water to the crack tip controls crack motion in Region II, where the crack velocity is essentially constant. In Region III the stress intensity factor is close to K_{IC} and crack velocity does not depend on water in the environment. Since Equation 2 represents only Region I crack growth, the equations derived for the strength after proof testing are based on the assumption that the strength is controlled exclusively by subcritical crack growth in Region I. If the behaviour shown in Fig. 7 is important, then Equation 2 would overestimate the crack velocity when the stress intensity factor of the crack was in the range that characterizes Region II, and the theory would predict failure when, in fact, the samples may just pass the proof test. The samples that just survive the proof test would then be weak, and the distribution after the proof test could not be predicted from Equations 3 or 4. Therefore, an analysis that takes into account Region II crack growth is necessary to account for the observed strength distributions after proof testing under non-ideal conditions. An analysis of this sort is presented in Part II of this paper.

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