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DYNAMIC FATIGUE TESTING OF ZERODUR GLASS-CERAMIC

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ABSTRACT

The inherent brittleness of glass invariably leads to a large variability in strength data and a time dependence in strength. Loading rate plays a large role in strength values. Glass is found to be weaker when supporting loads over long periods of time as compared to glass which undergoes rapid loading. These properties complicate the structural design allowables for the utilization of glass components in an application such as the AXAF.

This report describes the test methodology to obtain parameters which can be used to predict the reliability and lifetimes of Zerodur glass-ceramic which is to be used for the mirrors in the AXAF.

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List of Figures

INTRODUCTION

The aim of this study is to provide the necessary data to predict the reliability and lifetime of Zerodur Glass-Ceramic which will be utilized as the material for the grazing incidence mirrors in the Advanced X-ray Astrophysics Facility (AXAF).

Zerodur is a alumino-silica glass-ceramic containing 70 to 78% by weight of high-temperature quartz as the crystalline material. The mean crystal size of the crystals is in the region of 50 to 55 NM. The crystalline phase has a negative expansion coefficient, while the vitreous phase has a positive expansion coefficient. This pratically balances out linear thermal expansion leading to long term stability even with temperature fluctuations. Zerodur, is therefore an ideal candidate for AXAFS and other applications such as mirror substrates mounts where changes in the material due to temperature variations could impair the quality of observations. The composition and mechanical, thermal and optical properties have been reported by Schott Glass Technologies, producers of Zerodur. However, the question of subcritical crack growth in this material has not been explored in detail.

The design of the stresses in the AXAF mirrors is dictated by the ultimate strength of the material and a nominal value of 1450 psi (MOR) is recommended by the manufacturer. This is a minimum value or worst case value. In glassy materials, the modulus of rupture depends upon primarily the surface condition of the material. In other words, a surface with a large number of flaws will have a lower strength than one with few on no flaws. The reduction in strength is caused by surface flaws which concentrate applied stresses leading to failure at much lower than theoretical applied loads. These flaws can be introduced by machining or normal handling. Thus one method to increase strength is to polish the surface thereby removing surface flaws. Schott Glass reports an MOR of approximately 11,000 psi with a failure probability of 0.5% when the surface is polished with 600 grit polishing paper. Even higher strengths could be expected with a finer polishing medium. Even if one were to accept a value in the range of 11,000 psi one must consider the effects of delayed failure under constant applied loads. Uner these loads, even if small, failure can occur after a given period of time has passed. This has been attributed to the mechanism of stress corrosion whereby water molecules from a humid environment enhance crack growth. Cracks continue to grow over a period of time until catastrophic failure occurs.

Therefore one needs a reliable method of predicting life times under applied loads. The fatigue parameters for making lifetime predictions must be obtained under the service environment, if at all possible and can be obtained from one of three types of experiments: crack velocity, static fatigue and dynamic fatigue.

Crack velocity can be measured directly as a function of the stress intensity factor on specimens specifically designed for fracture mechanics experiments.[1] These speicmens contain a macroscopic crack which allows accurate measurement of crack velocity and stress intensity factor. For purposes of failure prediciton crack velocity data is not as reliable as that from static or dynamic fatigue strength experiments because data from large, preformed cracks is not necessarily relevent to the propagation of microscopic cracks present in brittle materials.[2]

Static fatiue data is generally obtained by measuring the time to failure of a large number of samples at several constant applied stresses.[3]

Dynamic fatigue is generated by measuring fracture strength as a function of stress rate.[3] For this study dynamic fatigue was chosen to develop the time-to-failure equation necessary to a design diagram. This concept will be developed fully in the Technical Discussion section.

Due to problems in obtaining samples in time to perform the actual experiments no data will be presented in the report. Instead attention will be paid to theoretical development of the design diagram and the proposed experimental procedure to obtain the necessary data for the diagram.

Technical Discussion

Initial Strength

Modulus of Rupture (MOR) will be determined using the double ring bending strength test method.[4] Figure 1 is a graphical representation of this technique. In this case a disc shaped specimen is loaded between two concentric rings. For limited forces, a tensile stress field will be set up in the central region of the convexly bent speicmen surface. Outside the load ring the radial and tangential stresses in the specimen decrease toward the edge, so that the possibility of fracture there is small. By increasing the force the tensile stress in the specimen center is increased at a constant rate until fracture occurs, with the expectation that the fracture is initiated in the region of the surface subjected to the maximum stress, underneath the load ring.

The major advantage of this technique, as compared to techniques such as 3 and 4 point bending, is the elimination of edge effects.

To calculate the rate of increase of the bending stress in circular specimens the following is applicable:

$$\frac{\Delta \sigma_b}{\Delta t} = \frac{1.08}{S^2} \cdot \frac{\Delta F}{\Delta t} \qquad (1)$$

Where:

Δ F = Increase of test force, measured in the time interval near the fracture initiation

Δ S = Specimen thickness

1.08 = Numerical constant related to the load ring and specimen support ring diameters and Poisson's Ratio

The bending strength (σ_{bB}) belonging to the maximum force (Fmax) from equation (1) is:

$$\sigma_{bB} = 1.08 \frac{Fmax}{S^2} \qquad (2)$$

Where:

σ_{bB} = bending strength

Fmax = greatest force

S = specimen thickness

Weibull Statistics

In order to determine the distributions of strength Weibull plots will be utilized. Fracture of brittle materials can be regarded as a statistical process, since the material has a distribution of flaw sizes. Weibull[5] described the mathematics for this process in terms of probability functions. His equations can take forms:

$$F = 1 - \exp\left(-\frac{\sigma}{\sigma_o}\right)^m \tag{3}$$

or

$$F = 1 - \exp\left(-\frac{\sigma - \sigma_u}{\sigma_o}\right)^m \tag{4}$$

where:

F = probability that the material will fail at stress

σ_u, σ_o = constants which describe the distribution

m = constant (Weibull modulus)

Equation (3) is the two-parameter model and represents an unbounded distribution. Equation (4) is the three-parameter model and represents a lower bounded distribution.

A number of dynamic fatigue studies of glass and glass-ceramics have shown the two-parameters model to be acceptable in describing the flaw population and it's relation to material failure.[6,7,8] Therefore, in this study equation (3) will be utilized.

One can rearrange equation (3) to obtain a more workable equation. Starting with equation (3)

$$F = 1 - \exp\left(-\frac{\sigma}{\sigma_o}\right)^m \tag{3}$$

$$-1 + F = -\exp\left(-\frac{\sigma}{\sigma_o}\right)^m$$

$$1 - F = \exp\left(-\frac{\sigma}{\sigma_o}\right)^m$$

$$(1 - F)^{-1} \quad \exp\left(\frac{\sigma}{\sigma_o}\right)^m$$

or

$$\frac{1}{1 - F} = \exp\left(\frac{\sigma}{\sigma_o}\right)^m \tag{5}$$

Taking ln of both sides of (5)

$$\ln\left(\frac{1}{1-F}\right) = \left(\frac{\sigma}{\sigma_0}\right)^m \tag{6}$$

and taking ln again;

$$\ln\left(\ln\frac{1}{1-F}\right) = m \ln \sigma - m \ln \sigma_0 \tag{7}$$

Where:

σ = Failure stress (MOR)

And:

$$F = \frac{m - 0.5}{N}$$

Where:

m = Rank of sample in terms of strength

N = Number of samples tested

Thus a plot of $\ln\left(\ln\frac{1}{1-F}\right)$ versus $\ln \sigma$ will yield a slope of m. The slope indicates the distribution of strengths. As he slope (m) increases the distribution narrows, i.e. there is less scatter in the results.

Lifetime Prediciton Equation

Weibull statistics can be combined with data on static or dynamic fatigue to yield predictions of the lifetime of brittle materials under various environmental conditions.[3] The derivation of this equation combines elements of fracture mechanics theory and Weibull statistics.

The rate of crack growth in a material can be expressed as a power law funciton:[9]

$$V = \frac{da}{dt} = AK_I \tag{8}$$

Where:

a = crack length

K_I = stress intensity factor

A, N = constant for a given material and environmental

The stress intensity factor can be expressed as:

$$K_I = \sigma Y \sqrt{a} \qquad (9)$$

Where:

a = crack length

σ = applied stress

Y = crack geometry parameter which is constant for a given material

Combining equations (8) and (9) to eliminate K_I yields:

$$\frac{da}{dt} = A \sigma^N Y^N a^{N/2} \qquad (10)$$

For the case of constant applied stress (σ_a):

$$\frac{da}{dt} = A \sigma_a^N Y^N a^{N/2}$$

or

$$a^{-N/2} da = A \sigma_a^N Y^N dt \qquad (11)$$

To obtain the material lifetime, this expression is integrated over the initial-to-final crack length and from the time the stress was applied ($t = 0$) to the time of fracture ($t = t_f$).

$$\int_{a_0}^{a_f} a^{-N/2} da = \int_0^{t_f} A \sigma_a^N Y^N dt \qquad (12)$$

$$\frac{a^{-N/2+1}}{-N/2+1}\Big]_{a_o}^{a_f} = A\sigma_a^N Y^N t\Big]_0^{t_f}$$

<div style="text-align:center">or</div>

$$\frac{2a^{-(N-2/2)}}{N-2}\Big]_{a_o}^{a_f} = A\sigma_a^N Y^N t\Big]_0^{t_f}$$

which yields:

$$\frac{2}{N-2}\left(a_f^{-(N-2/2)} - a_o^{-(N-2/2)}\right) = A\sigma_a^N Y^N t_f \tag{13}$$

Rearranging equation (9):

$$a = \left(\frac{K_I}{\sigma Y}\right)^2$$

and substituting into equation (13):

$$\frac{2}{N-2}\left[\left(\frac{K_{If}}{\sigma_f Y}\right)^{-(N-2)} - \left(\frac{K_{Io}}{\sigma_o Y}\right)^{-(N-2)}\right] = A\sigma_a^N Y^N t_f \tag{14}$$

The stress intensity factor when fracture occurs, K_{IC}, and the fracture stress σ_f is the stress at which fracture would occur in an inert enviornment unaffected by subcritical crack growth (σ_i)

Substituting into equation (14):

$$\frac{2}{N-2}\left[\left(\frac{K_{IC}}{\sigma_i Y}\right)^{-(N-2)} - \left(\frac{K_{Io}}{\sigma_o Y}\right)^{-(N-2)}\right] = A\,\sigma_a^N Y^N t_f \qquad (15)$$

Generally $K_{IC}^{-(N-2)} << K_{Io}^{-(N-2)}$

Therefore one can write:

$$\frac{2}{N-2}\left(\frac{\sigma_i Y}{K_{Ic}}\right)^{N-2} = A\,\sigma_a^N Y^N t_f \qquad (16)$$

Solving for t_f:

$$t_f = \frac{2}{N-2}\frac{\sigma_i^{N-2} Y^{N-2}}{A\,\sigma_a^N K_{Ic}^{N-2} Y^N} = \frac{2}{N-2}\frac{\sigma_i^{N-2}}{A\,\sigma_a^N K_{Ic}^{N-2} Y^2} = B\,\sigma_i^{N-2}\sigma_a^{-N} \qquad (17)$$

Where:

$$B = \frac{2}{AY^2 K_{IC}^{N-2}(N-2)}$$

Taking ln of of both sides of equation (17)

$$ln\,t_f = ln\,B + (N-2)ln\,\sigma_i - N\,ln\,\sigma_a \qquad (18)$$

Rearrangement of Equation (7) yields:

$$ln\left(ln\frac{1}{1-F}\right) + m\,ln\,\sigma_o = m\,ln\,\sigma$$

or

$$ln\,\sigma = \frac{1}{m}\left[ln\left(ln\frac{1}{1-F}\right) + m\,ln\,\sigma_o\right] \qquad (19)$$

In an enviornment where no subcritical flaw grow occurs prior to fracture one can write equation (19) in terms of inert strength (σ_i) i.e.

$$\ln \sigma_i = \frac{1}{m_i}\left[\ln\left(\ln\frac{1}{1-F}\right) + m_i \ln \sigma_i\right] \quad (20)$$

Substitution of equation (20) into equation (18) for $\ln \sigma_i$ yields:

$$\ln t_f = \ln B + \frac{N-2}{m_i}\left[\ln\left(\ln\frac{1}{1-F}\right) + m_i \ln \sigma_i\right] - N \ln \sigma_a \quad (21)$$

Where:

t_f represents the time required for a flaw to grow from an initial subcritical size to dimensions critical for catastrophic crack propagation leading to material failure.

And:

B and N are constants which characterize the subcritical flaw growth.

Determination of B and N

Evans 10 derived an expression relating the strength of a material (σ) to the stressing rate ($\dot{\sigma}$) at constant temperature for the case of subcritical flaw growth as follows:

$$\frac{d\sigma}{dt} = \dot{\sigma} \tag{22}$$

or,

$$d\sigma = \dot{\sigma}\, dt \tag{23}$$

Dividing by da ;

$$\frac{d\sigma}{da} = \dot{\sigma}\, \frac{dt}{da} \tag{24}$$

Since $\dfrac{da}{dt}$ = V (crack velocity):

$$\frac{d\sigma}{da} = \frac{\dot{\sigma}}{V} \tag{25}$$

Now, we know that,

$$V = AK_I$$

Therefore we can write:

$$d\sigma = \frac{\dot{\sigma}\, da}{AK_I^N} \tag{26}$$

We also know:

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

or

$$K_I^N = Y^N \sigma^N a^{N/2}$$

Substituting this expression into equaiton (26) gives:

$$d\sigma = \dot{\sigma}\, da / A\sigma^N a^{N/2} Y^N$$

or

$$\sigma^N d\sigma = \dot{\sigma}\, da / A Y^N a^{N/2} \tag{27}$$

Integrating over the extent of crack growth from an intial stress of zero to a final failure stress or obtains:

$$\int_0^{\sigma_f} \sigma^N d\sigma = \frac{\dot{\sigma}}{A Y^N} \int_{a_i}^{a_c} a^{-N/2} da \tag{28}$$

which yields:

$$\frac{\sigma_f^{N+1}}{N+1} = \frac{2\dot{\sigma}}{A Y^N (2-N)} \left[a_c^{(2-N)/2} - a_i^{(2-N)/2} \right] \tag{29}$$

Since, $a_c^{(2-N)/2} >> a_i^{(2-N)/2}$

the former can be dropped leading to:

$$\frac{\sigma_f^{N+1}}{N+1} = \frac{2\dot{\sigma}\left(-a_i^{(2-N)/2}\right)}{A Y^N (2-N)} \tag{30}$$

The intial flaw size can be taken as:

$$a_i^{\frac{1}{2}} = K_{IC}/Y \sigma_{ic}$$

or

$$a_i = \frac{K_{IC}^2}{Y^2 \sigma_{ic}^2} \tag{31}$$

Substituting equation (31) into (30) for a_i yields:

$$\frac{\sigma_f^{N+1}}{\theta} = \frac{2(N+1)\dot{\sigma} \sigma_{ic}^{N-2}}{A Y^2 (N-2) K_{IC}^{N-2}} \tag{32}$$

Can rewrite equation (32) as:

$$\nabla_f^{N+1} = B(N+1)\nabla_{ic}\dot{\nabla}$$ (33)

where:

$$B = \frac{2}{AY^2(N-2)K_{Ic}^{N-2}}$$

Taking ln of both sides of equation (33):

$$(N+1)\ln\nabla_f = \ln\left[B(N+1) + (N-2)\nabla_{ic}\right] + \ln\dot{\nabla}$$

or

$$\ln\nabla_f = \frac{\ln\left[B(N+1) + (N-2)\nabla_{ic}\right] + \ln\dot{\nabla}}{N+1}$$ (34)

Rewriting Equation (34):

$$\ln\nabla_f = \left(\frac{\ln\left[B(N+1)\right] + (N-2)\ln\nabla_{ic}}{N+1}\right) + \frac{\ln\dot{\nabla}}{N+1}$$ (35)

or

$$\ln\nabla_f = a_0 + a_1\ln\dot{\nabla}$$ (36)

where:

$$a_0 = \frac{\ln\left[B(N+1)\right] + (N-2)\ln\nabla_{ic}}{N+1}$$

$$a_1 = \frac{1}{N+1}$$

Thus, to obtain values for B and N, one fits fracture stress and stress rate data for samples tested in humid enviornment to a straight line g iven by equation (36), that is a plot $\ln \dot{\sigma}_f$ versus $\ln \dot{\sigma}$. To ensure that the values of B and N are reliable one needs to test over a wide range of stress rates (i.e. over several orders of magnitude). The inert strength (σ_i) should be obtained at high enough stress rates to insure that crack growth is independent of environmental (i.e. no stress corrosion at the crack tip is occurring).

Design Diagrams

Once the values of the constants B and N are determined, the time-to-failure for various values of failure probability (F) and applied stress (σ_a) can be computed. Figure 2 shows an exmaple of a design diagram for polished ULE glass at F = 0.001, 0.01 and 0.20. Comparing this to a diagram for unpolished ULE glass (Figure 3) one can note decrease in strength at a given time period (Ex. 10 yrs.) as compared to polished glass. This infers that at a given probability of failure the polished glass should withstand an applied stress for a longer period of time. For examples at F = 0.01 and an applied stress of 24 x 10^6 MPa the polished material will fail in one hour whereas the unpolished fails in one second.

Experimental Approach

The investigation will be carried out in three phases.

Phase I- Residual Stress Investigation

In order to determine the effects of residual stresses on the modulus of rupture of Zerodur, 10 samples with ground surfae finish will be annealed and compared with 10 duplicate unannealed samples using the double ring bend test method. All tests will be performed at a stress rate of 290 psi/sec, which is in accordance with test standard DIN 52292.

Phase II - Generation of Design Diagram

Weibull plots [$\ln (\ln \frac{1}{1-F})$ vs. $\ln \dot{\sigma}_f$] will be made in order to determine the strength distributions for three types of sample preparation (AS cut, ground, and ground with acid etch). The etching technique is to be supplied by Schott Glass Co. MOR data will be obtained at four different stress rates, 3.5 x 10^{-3} MPa/s, 2.7 x 10^{-2} MPa/s, 2 MPa/s and 200 MPa/s. Thirty five samples will be tested. The final stress at each stress rate will be used to determine the inert strength. In this instance samples will be heated at 150°C for 24 hours in a dry N$_2$ environment and put in a vacuum dessicator before testing.

Each sample will be exposed to flowing dry initrogen for fifteen minutes before testing, in a plexiglass chamber fitted to the testing machine. To insure that this stress rate leads to crack growth independent of environment, ten samples will be placed in distilled water for 24 hours and then tested at 200 MPa/s. The sample to be tested at the three lower stress rates will be stored at ambient conditions (T = 25°C, R.H. = 50%) for at least one week before testing.

Using the time-to-failure equation developed in this paper, a time-to-failure diagram will be developed for each sample preparation. That is a separate diagram for the as cut, ground, and ground and acid etched samples.

Phase III- Verficiation of Diagrams

In order to verify the three time-to-failure diagrams, applied stress values which intersect the F = 0.005 probability curve corresponding to time-to-failures of 1 minute, 1 hour, and 1 day will be selected and used to test 10 specimens each using the double ring test method.

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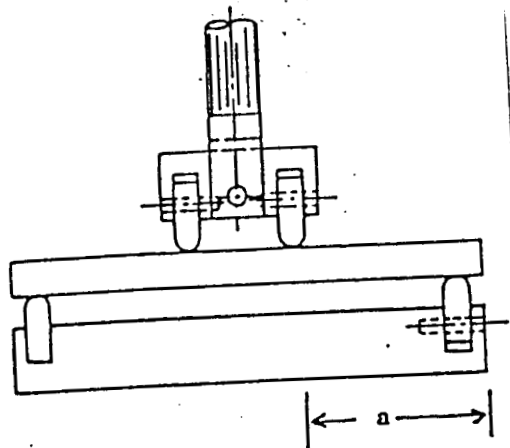10. Evans, A. G., Int. J. Fract. 10 251-259 (1974).

Figure 1

Figure 2

Figure 3