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EFFECTS OF WATER ON THE STRENGTH OF ZERODUR

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16. Abstract

An experimental design matrix was constructed to determine the effects of time and temperature water soak on the strength of Zerodur glass-ceramic. It was found that strength does increase in a nonlinear manner which is consistent with existing theories of crack tip blunting and residual stress reduction.

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INTRODUCTION

Glasses and glass-ceramics used in optical systems undergo controlled grinding and polishing to achieve the required optical properties. During early stages of grinding, rough surfaces are produced due to the large grit sizes of the grinding medium. One result of the rough grind is introduction of surface and subsurface damage which leads to strength reduction of the glass article. As the polishing process continues, this damage is removed and the strength increases. It has been observed that for a freshly ground glass the strength will increase with time [1,2]. Studies have indicated that the strength can also be increased by annealing or exposing the surface to high humidity [3,4]. Supposedly, the grinding introduces residual stress in the glass which effectively lowers the glass strength by increasing the local stress intensity factor at the crack tip. The increased stress intensity factor is due to the elastic/plastic zone at the crack tip [5]. This residual stress can be relieved by thermal annealing or exposing the crack tips to water. The exact mechanism is not understood at present.

This present study does not have as its goal determination of this mechanism, but rather determining the effect of water as a function of temperature and time on the strength of Zerodur glass ceramic. This material is being used as mirror element material for the Advance X-Ray Physics Facility which will be launched at the end of this decade. Zerodur, being a glass-ceramic, cannot be annealed, since this would lead to crystal growth and property alteration. Unpublished data by this author showed that allowing rough ground (230 to 270 grit) Zerodur samples to soak in distilled water for 48 h at room temperature led to a 14-percent increase in strength. This data led to the more extensive study described in the next section.

EXPERIMENTAL METHODS

In order to study the effects of water as a function of temperature and soak time on glass strength, an experimental design matrix was developed using Q-Edge statistical software. This design is a full factorial with two factors (water temperature and soak time) at two levels with 10 replicates. This design matrix is shown in table 1. After each set of samples was run according to the design matrix, it was dried at 105 °C for 24 h. Samples were then mechanicallly tested using a concentric ring bend test [6]. Failure analysis was performed on each sample using a stereomicroscope to determine the origin of failure. In order to test the prediction of the design software, two more sets of samples were soaked at 25 °C and 80 °C for 335 h.

RESULTS AND DISCUSSION

The results of the strength testing are given in table 2. Also given in table 2 are the standard deviation and Weibull moduli. It can be seen that as the time increases at room temperature, the strength increases to 8,176 psi at 72 h and then deceases to 7,793 at 335 h. At 80 °C the strength is maximum at a 2-h soak and then decreases as the time increases. The strength changes are not really that significant when one takes into account the standard deviations. However, what is important is that the strength does increase dramatically when one compares the strength of samples which are not exposed after rough grinding to other than a laboratory environment. For 230 to 270 grit ground Zerodur with no water soak, the average breaking strength was 7,000 psi. By soaking for 2 h at 80 °C, the strength was increased by almost 20 percent. This has practical implications for large mirror elements. A time-temperature soak of the elements could be used to increase the glass strength before lifting and handling operations associated with further polishing is begun. Also increasing strength allowables would be possible. The changes in strength are most likely due to a multiple mechanism involving diffusion of water to the crack tips and chemical reaction at the crack tip which blunts the flaw and reduces residual stress intensity. This residual stress is due to the rough grinding process which produces subsurface damage. At high temperatures preferential etching may take place which could cause the strength to decrease. At longer time periods there may be water, not removed by the subsequent drying, which remains in the subsurface flaws. It is noted that the Weibull modulus increases with time for the room temperature samples. This would indicate that the size distibution of blunted flaw tips is becoming more narrow over time. Thus, the probability of failure is more equal from sample to sample. The reverse is observed for the high temperature samples. The Weibull modulus decreases with time. It may be that certain flaws are preferentially etched and at a faster rate due to the increase in kinetics. This would be likely if one assumes an exponential effect with temperature (i.e., Arrhenius).

One notes in table 2 that one of the predicted strength values does not agree with the tested value. The Q-edge software, based on information from the basic design matrix, predicted a strength of 9,446 psi for the 335-h soak at room temperature. The actual tested value was only 7,793 psi. This could be due to the fact that the prediction equation is linear, whereas the mechanisms which affect the glass strength are nonlinear. For example, diffusion of water to the crack tip is an exponential process as is the chemical reaction at the crack tip. Another possible explanation for the discrepency is that the time values used in the prediction were well outside the limits of the original matrix (i.e., 335 h versus 72 h).

CONCLUSIONS

Although the designed experiment did not yield a valid prediction equation, valuable information was obtained concerning the effects of time and temperature water soaking on the strength of Zerodur glass-ceramic. It was found that soaking the glass-ceramic specimens for 2 h at 80 °C led to the largest increase in strength. This is consistent with theoretical considerations. It may be possible to institute a more powerful design which takes into consideration exponential effects. Also the effect of water pH should be considered in a new experimental design since the proton concentration in the crack tip would affect chemical reaction rates.

Table 1. Experimental design matrix.

Run	Temperature	Time
1	20	2
2	20	72
3	80	2
4	80	72

Table 2. Strength, standard deviation and Weibull modulus of Zerodur as a function of time and temperature water soak.

Time (h)	Temperature (°C)	Strength (psi)	Std. Dev. (psi)	Weibull Modulus
2	25	7,839	1,073	8.60
72	25	8,176	809	11.97
335	25	7,793 (9,446)*	564	15.40
2	80	8.648	513	19.60
72	80	8,417	918	10.70
335	80	7,694 (7,548)*	1,059	7.73

*Predicted values from Q-edge software.

REFERENCES

1. Holland, A.S., and Turner, W.E.S.: Journal of the Society of Glass Technology, vol. 21, 1937, p. 383.

2. Mould, R.E.: Journal of the American Ceramic Society, vol. 43, 1960, p. 160.

3. Roach, D.H., and Cooper, A.R.: Journal of the American Ceramic Society, vol. 68, 1985, p. 632.

4. Chantikul, P., Lawn, B.R., and Marshall, D.B.: Journal of the American Ceramic Society, vol. 64, 1981, p. 322.

5. Marshall, D.B.: "Fracture from Surface Flaws." Fracture in Ceramic Materials. Noyes Publications, A.G. Evans, ed., 1984.

6. "Testing of Glass and Glass-Ceramics," German Standard DIN 52 292, part 1, 1984.

APPROVAL

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The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

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