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STRESS MEASUREMENT WITH NANO-INDENTATION

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The feasibility of using nano-indentation to measure residual stress in glasses was studied. Indents were placed on the side of flexure specimens at four different distances from the neutral axis while the specimens were under load in four-point-bending. Three different glasses (soda-lime, boro-silicate, and fused silica) were indented with a cube-corner indenter using 2 to 30 mN indentation loads. A high resolution scanning electron microscope was used to measure the length of the cracks emanating from the corners of the indents while the specimen remained under load. The measured crack lengths were correlated to the local stress using indentation theory. For the correlation, elastic beam theory was used to calculate the magnitude of the local stress at the indentation sites. Results derived from crack lengths were in good agreement with local stress within experimental scatter. However, this scatter was found to be rather large as a result of the stochastic nature of crack formation. It can be concluded from this study that nano-indentation can be used to measure residual surface stresses with high spatial resolution provided that a sufficient number of indents are used to assure good statistical accuracy.

1. INTRODUCTION

Indentation techniques to measure mechanical properties of brittle materials has gained wide spread acceptance in the ceramics community due to their relative simplicity and reasonable accuracy. These measurement techniques evolved from the pioneering work of such investigators as B. R. Lawn, M. V. Swain, E. R. Fuller, A. G. Evans, and others whose contribution to the development of the indentation

crack length method to measure fracture toughness has been critically reviewed recently by C. B. Ponton and R. D. Rawlings.[1] Of particular significance is Lawn, Evans, and Marshall's elastic-plastic model of indentation[2] which was evaluated for its utility for toughness testing by G. R. Anstis, P. Chantikul, B. R. Lawn, and D. B. Marshall.[3]

The elastic-plastic model can be used also to assess the effect of pre-existing residual stresses. Accordingly, the critical stress intensity factor (fracture toughness) K_c, can be related to the indentation load, P, the post-indentation crack length, c, and the pre-existing residual stress, σ_{res}, as:

$$K_c = \frac{\chi P}{c^{3/2}} + Y\sigma_{res}\sqrt{c} \tag{1}$$

The first term on the right hand side of this equation represents the effect of the indentation which is characterized by the parameter, χ. The second term represents the effect of the pre-existing local, or residual stress. Implicit in the form of the second term is the assumption that the indent crack is a half penny surface crack placed in a uniform residual stress field whose depth is greater than the depth of the indentation crack. Y is the shape parameter for a half-penny surface crack.

The applicability of the indentation theory has been restricted, by-and-large, to Vickers indents with fully developed half-penny cracks whose diameter is at least 2.5 times the impression diagonal.[1] This restriction led to the use of relatively large indentation loads by Wu et al.[4] in their study to measure stresses in uniaxially loaded Si_3N_4 bars with Vickers indentation. Consequently, the length of the cracks that were analyzed to predict the stress in the test bars were of the order of hundreds of micro-meters. The purpose of the study presented in this paper was to assess the applicability of Eq. (1) to measure residual stress on a much smaller scale using a cube-corner nano-indenter to produce cracks with lengths of several micro-meters. Such small cracks would enable the use of indentation for the measurement of stress with high spatial resolution.

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2. EXPERIMENTAL PROCEDURE

Specimens made of three different glasses (soda-lime, boro-silicate, and fused silica) were indented in a nano-indenter (Nano II, Nano Instruments, Oak Ridge, TN) with a cube-corner indenter using 2 to 30 mN indentation loads. Indents were placed on the side of flexure specimens at four different distances from the neutral axis while the specimens were under load in four-point-bending. Figure 1 is a schematic showing the location and arrangement of the indents on the four-point-bend specimen.

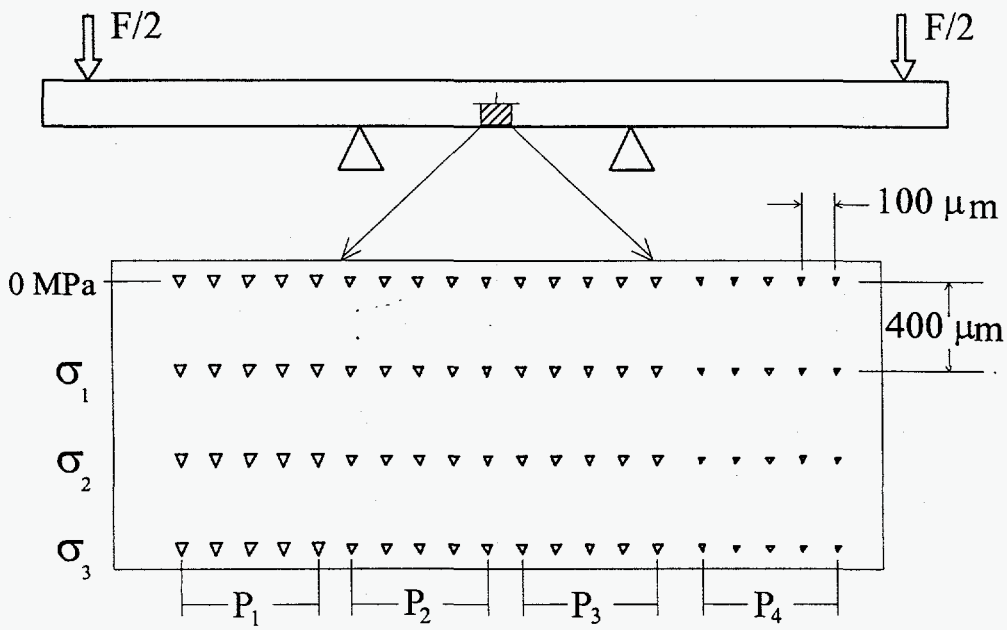

FIGURE 1
Schematic of the location and arrangement of the indents on the bend-specimens

A high resolution scanning electron microscope (Philips XL30) was used to measure the length of the cracks emanating from the corners of the indents while the specimen remained under load. Only the cracks perpendicular to the bending stress were measured.

For correlating observed crack lengths with the local stress Eq. (1) was rearranged to express P as a function of c, namely:

$$P = \frac{K_c}{\chi} c^{3/2} - \frac{Y\sigma_{res}}{\chi} c^2 \qquad (2)$$

Eq. (2) was first fitted using non-linear regression to the measured lengths of cracks at the neutral axis where σ_{res} is zero. This regression gave a value for χ, assuming $K_c = 0.75$ MPa(m)$^{1/2}$ for each of the three glasses. The crack lengths of indents placed in the compression side of the specimen at three different distances from the neutral axis were regressed successively using the above obtained χ value and Y = 1.29.[5] These regressions gave the estimate for the local stress, σ_{res}.

3. RESULTS AND DISCUSSION

Figure 2 shows the average crack length data for soda-lime glass plotted as indentation load versus crack length. Error bars are also shown for the cracks measured at the neutral axis (solid circles). Error bars of similar magnitude for the other locations are omitted for sake of clarity. In addition, the figure shows the regression lines (Eq. (2)) for the four different local stress locations. It can be seen that the crack length derived stresses are within 30% of the actual bending stresses in the beam. This is reasonable agreement considering that the crack lengths were obtained from only five cracks at each indentation load / local stress combination.

Similar results were obtained for the fused silica and boro-silicate glasses with the only exception of one stress level in fused silica glass where the estimate was 50% higher than the local stress. All other estimates for the three glasses were better than 30%. These results are summarized on Fig. 3 which shows the estimated stress versus the applied (local) stress. The reasonable correlation between the estimated and applied stress is evident in the figure.

The most important result of this study is that the elastic-plastic indentation theory relating crack length, load, and local stress is valid for very small cube-corner indentations. However it should be noted that the indentation constant χ is different for these small indents than for the larger Vickers indents. For example, in soda-lime glass χ is 0.22 for the nano-indents used in study, whereas it is 0.055

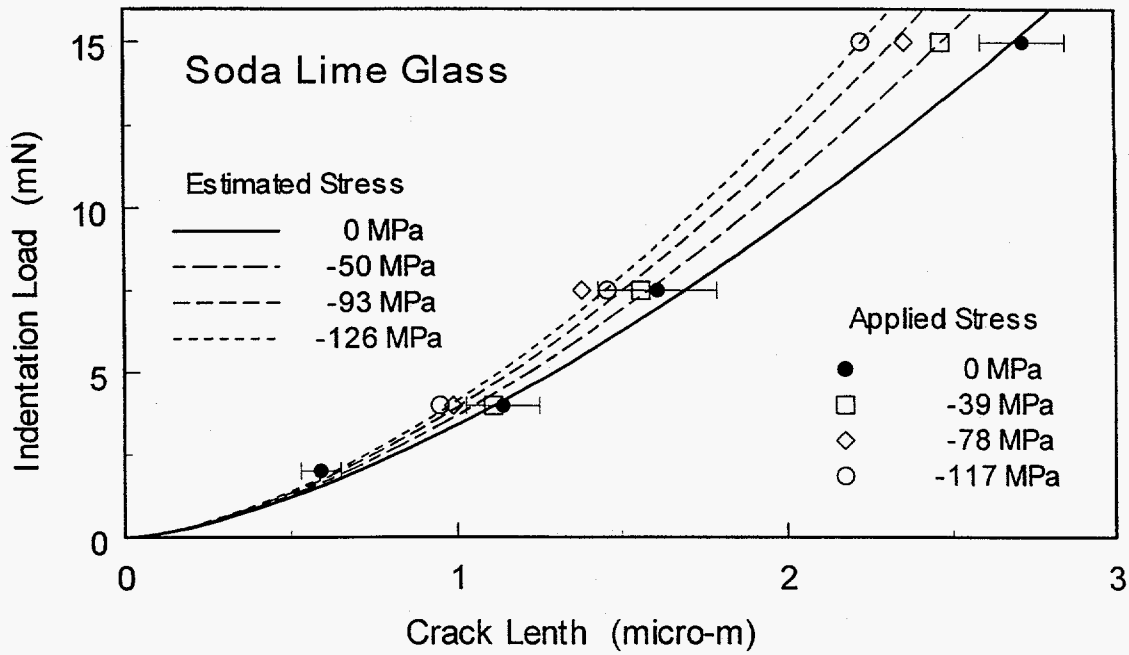

FIGURE 2
Effect of local stress on nano-indentation crack length. Lines are best fit Eq. (2)

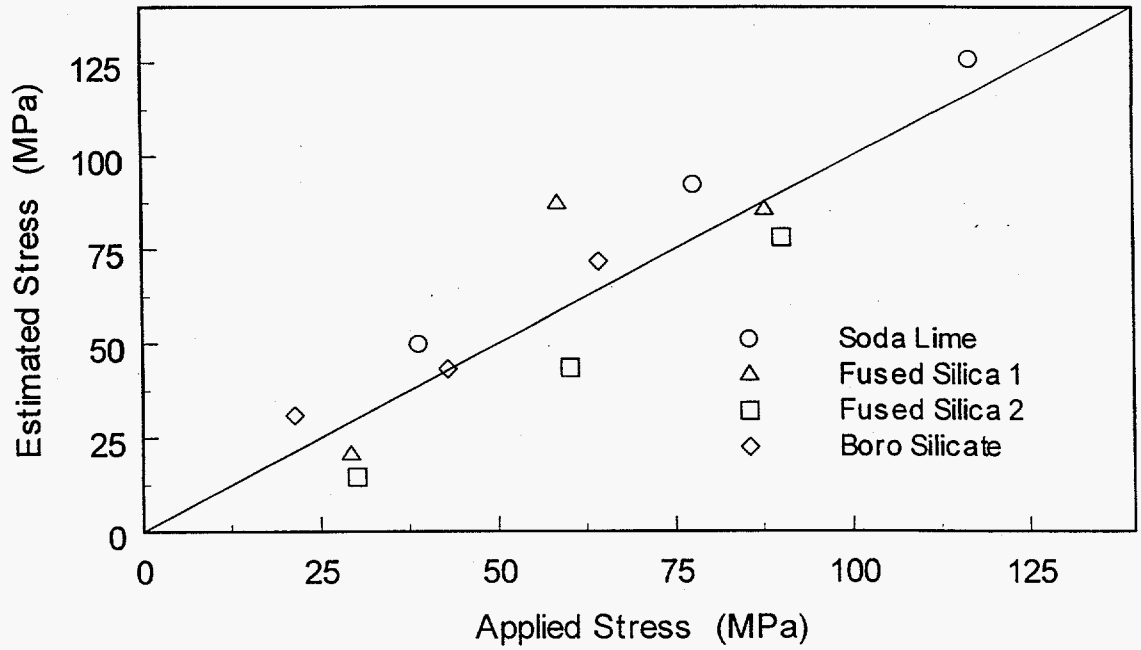

FIGURE 3
Comparison of the crack length estimated stress to the applied stress for soda-lime, fused silica, and boro-silicate glasses.

for Vickers indents.[3] This difference is most likely caused by the radical difference in indenter geometry.

4. CONCLUSIONS

The length of cracks emanating from the corners of nano-indentation impressions was analyzed on the basis of elastic-plastic indentation theory that was developed for Vickers indentation. The analysis resulted in reasonable estimates of the known local stresses in test specimens of soda-lime, fused silica, and borosilicate glasses. This finding is of a significant practical importance because nano-indent cracks were only a few micro-meter in length in comparison to hundreds of micro-meter Vickers cracks. These small scale cracks, thus, enable the use of nano-indentation in mapping the residual stress fields with high spatial resolution.

ACKNOWLEGEMENT

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