

# SURFACE FLAWS IN GLASS

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The nature of surface flaws in glass is examined in the light of indentation fracture theory. First, the mechanical response of well-developed indentation cracks to applied stresses is described. A characteristic feature of the predicted response is a stage of precursor growth to a critical failure configuration, due to the stabilizing influence of residual elastic-plastic stresses. This characteristic is confirmed by direct observations of the crack evolution. Then it is demonstrated from acoustic scattering experiments that machining damage flaws have essentially the same characteristic response, implying that they are true microcracks. Finally, aging experiments on indented, machined and abraded surfaces, in aqueous environments, are described. Strength recovery with aging time is observed in all cases, provided residual stresses remain active during exposure to the environment, and is directly attributable to secondary crack growth effects.

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## I. INTRODUCTION

In an earlier paper in this volume<sup>1</sup> the use of indentation cracks as a means of studying fracture behavior and flaw characteristics was foreshadowed. The chief merit of this approach lies in the unique degree of control that is achievable; it is possible to generate a strength-controlling flaw of predetermined location and size, and hence to observe directly the subsequent response of the cracks to applied loading and/or environmental influences. Such direct observations provide valuable insight into the mechanisms of failure.

The ideal indentation fracture system closely resembles the "natural" contact damage that glass surfaces experience during surface finishing and service. Several studies have already demonstrated similarities in the general damage configurations associated with indentation, machining, abrasion, scratching, and particle impact,<sup>2-5</sup> all of which are caused by penetration of the surface by hard, sharp particles. However, before indentation fracture mechanics can be extended to natural flaws, correlations in the responses to applied stresses need to be established.

This paper has two purposes. One is to survey a growing body of experimental evidence in support of the contention that indentation flaws provide a representative model for naturally occurring flaws. Some of that evidence comes from direct observations of indentation cracks and machining cracks under sustained loads.<sup>6</sup> Other, less direct, evidence comes from comparative measurements of strength variations in surface-damaged specimens after aging in reactive environments.<sup>7</sup> The second purpose is to examine the implications of these observations concerning the nature of flaws and mechanisms of failure. Of particular concern will be the question raised in the earlier paper<sup>1</sup> as to whether flaws in glass behave as atomically sharp cracks or as blunt "notches."<sup>8-10</sup>

Our attention here will be confined to "post threshold" indentations with well-developed cracks. Severe surface damage processes, such as machining and abrasion, fall well within this domain. The flaws in high strength glass surfaces (such as optical fibers) relate more closely to "sub-threshold" indentations,<sup>1</sup> and will be dealt with in detail elsewhere.<sup>11</sup>

## II. RESPONSE OF INDENTATION FLAWS TO APPLIED STRESSES

The residual stresses that are responsible for creating radial indentation cracks<sup>1</sup> exert a major influence on the subsequent response of these same cracks to applied stresses and to environmental interactions. In this section we briefly outline how these residual stresses may be incorporated into a fracture mechanics formulation of strength, emphasizing the differences in

behavior in relation to flaws which experience no such influence. Experiments which identify these difference will be described.

**A. FRACTURE MECHANICS ANALYSIS**

The residual indentation stress field acts in concert with any subsequently applied tensile loading in driving the radial cracks to failure.<sup>12</sup> Accordingly, determination of the stress intensity factor associated with the residual field is central to any fracture mechanics analysis involving indentation cracks. In the spirit of the derivation of Eq. (5) in Ref. 1, this stress intensity factor is given by<sup>13</sup>

$$K_r = \chi P/c_r^{3/2} \tag{1}$$

where  $c_r$  is the radial crack dimension,  $P$  is the indenter load and  $\chi \propto (E/H)^{1/2}$  ( $E$  is Young's modulus,  $H$  is hardness). The stress intensity factor due to the applied stress  $\sigma_a$  alone is given by

$$K_a = \psi \sigma_a c_r^{1/2} \tag{2}$$

where  $\psi$  is a crack geometry parameter. Thus the net stress intensity factor is

$$K = \chi P/c_r^{3/2} + \psi \sigma_a c_r^{1/2} . \tag{3}$$

For cracks which grow under equilibrium conditions, e.g. as in truly inert

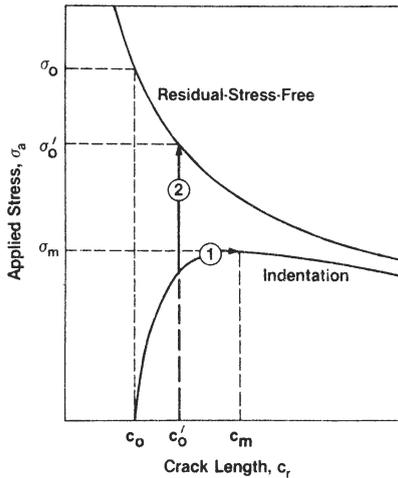


Fig. 1. Plot of applied stress as a function of equilibrium crack size for indentation cracks ( $\chi_r \neq 0$ ) and residual stress-free cracks ( $\chi_r = 0$ ). The corresponding crack responses during breaking tests are indicated by paths 1 and 2.

environments, we may put  $K = K_c$  in Eq. (3),  $K_c$  defining the toughness, to obtain

$$\sigma_a = \left( K_c / \psi c_r^{1/2} \right) \left[ 1 - \chi P / K_c c_r^{3/2} \right]. \quad (4)$$

This equation serves as a master relation for determining strength characteristics.

To illustrate, Eq. (4) is plotted in Fig. 1 for two cases, one with the residual stress term intact ( $\chi \neq 0$ ) and one with the same term removed ( $\chi = 0$ ). In this figure,  $c_0$  is the equilibrium crack size immediately after indentation, obtained by putting  $\sigma_a = 0$  in Eq. (1). In practice, exposure of the newly formed cracks to non-inert environments causes some subcritical crack growth to occur, from  $c_0$  to  $c'_0$ , say.<sup>1</sup> The routes to failure for the two cases shown are via paths 1 and 2. For the latter, the crack fails spontaneously from its initial configuration at  $c'_0$  at a stress level  $\sigma'_0$ . This is the well known response for ideal "Griffith" flaws. For the flaws with residual stress, the crack undergoes stable extension from  $c'_0$  to  $c_m$ , before failing at the stress  $\sigma_m$ . This instability is defined by the maximum in the  $\sigma_a(c)$  function,

$$\sigma_m = 3K_c / 4\psi c_m^{1/2} \quad (5a)$$

$$c_m = \left( 4\chi P / K_c \right)^{2/3}. \quad (5b)$$

We note that these failure conditions are independent of  $c'_0$ , provided  $c'_0 \leq c_m$ .

Thus the critical distinguishing feature for equilibrium flaws with and without residual stress is the presence (or otherwise) of a precursor stage of stable growth enroute to failure.

## B. EVIDENCE FOR RESIDUAL STRESS INFLUENCE

The predicted stable precursor growth of indentation cracks during failure testing has been observed directly in many brittle materials.<sup>14-18</sup> As an example, a Vickers indentation in a soda-lime glass flexure specimen, as viewed through a microscope attachment, is shown in Fig. 2 at three stages of loading. The stable growth halted whenever the loading was stopped to take the photographs, indicating that the test environment was sufficiently inert to prevent any spurious influence of subcritical extension in the observations.

Comparative experiments were run on indentation cracks that had been annealed prior to strength testing.<sup>18</sup> The annealing relieved the residual stresses (as confirmed by stress birefringence measurements), thereby

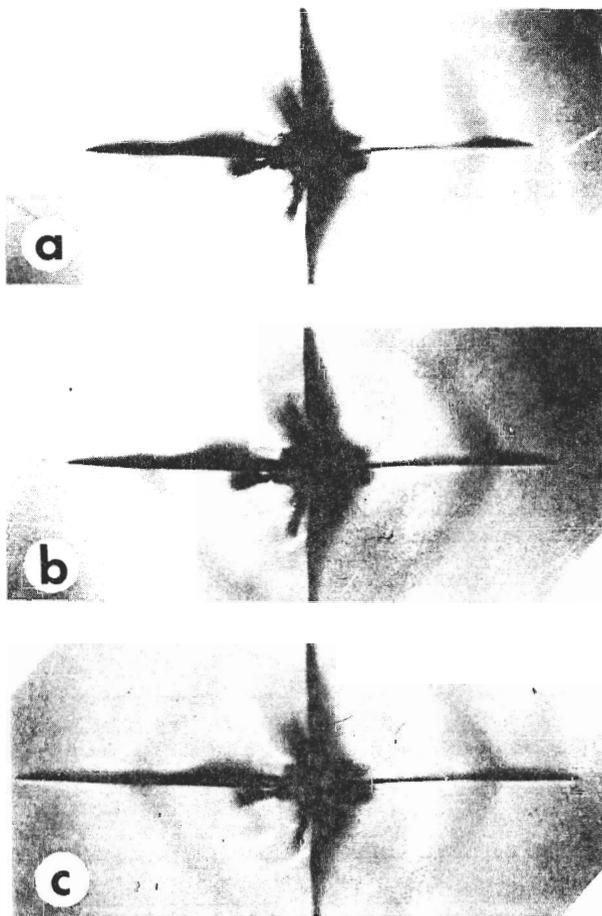


Fig. 2. Response of Vickers indentation ( $P = 50$  N) in soda-lime glass during failure test in dry nitrogen environment. Applied stress,  $\sigma_a =$  (a) 0, (b) 28.3, (c) 31.5 MPa. Specimen failed at 32.0 MPa. Width of field 1 mm. After Ref. 12.

effectively converting the configuration from the lower to the upper curve in Fig. 1. Thus for soda-lime glass indented at  $P = 50$  N the strength increased from  $32 \pm 3$  MPa to  $45 \pm 5$  MPa, corresponding to the relative levels  $\sigma_m$  and  $\sigma'_0$  in Fig. 1. Moreover, no stable precursor crack growth was observed in the annealed specimens.

It will be seen from the above description of the indentation mechanics that there is one major advantage of working with flaws that are under the influence of local residual stresses. The critical conditions for instability are determined unequivocally by the observable growth of a sharp crack, whereas for analogous flaws free of such stresses there is no precursor

development to provide a clue to the underlying failure processes. We shall make use of this distinction in the sections that follow.

### III. ACOUSTIC ANALYSIS OF FLAW TYPES

Unfortunately, optical monitoring of natural flaws during failure testing is not generally feasible because the location of the failure origin cannot be determined a priori. Moreover, failure can occur from subsurface flaws, and these may be obscured by extraneous surface damage. Such difficulties can be circumvented by using a technique based on the scattering of acoustic waves from crack interfaces. With this technique two transducers are used, one to excite surface (Rayleigh) waves at normal incidence to the cracks, and a second to detect backscattered waves. The relative amplitude of the detected signal is related, via scattering analysis, to the crack dimensions, whereas the time delay between generation and detection defines the crack location. The most definitive tests to date have been carried out on silicon nitride,<sup>6</sup> but the principles apply to any brittle material. Once the signals have been "calibrated" using indentation cracks, the method may be applied to characterize the response of suitable natural flaws.

#### A. INDENTATION CRACKS

Acoustic scattering from surface cracks depends on the effective, free crack area. This dependence has been demonstrated by monitoring the scattering

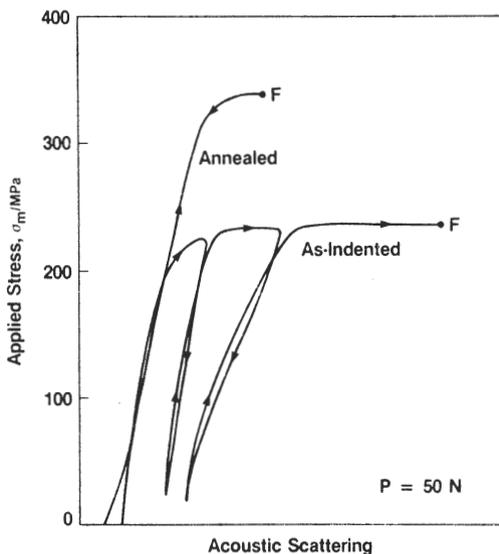


Fig. 3. Variation of acoustic scattering from Knoop indentation cracks in  $\text{Si}_3\text{N}_4$  during tensile loading to failure (F). Note reversibility in acoustic scattering with applied tension for annealed cracks, irreversibility for as-indented cracks.

from Knoop indentation cracks in silicon nitride, Fig. 3. The signals on as-indented surfaces differ significantly from those on corresponding indented surfaces with the residual stresses removed. At zero applied load the cracks for both surface types are partially open; even in the absence of residual contact stresses the crack walls are prevented from closing intimately by interfacial asperities.<sup>6</sup> On applying the loading the cracks open further, thereby increasing the signal intensity in both crack configurations.

An important difference in the signal characteristics is observed on unloading the cracks prior to failure. The curve for the surfaces without residual stresses retraces its original path, whereas that for as-indented surfaces shows hysteresis. In the latter instance the signal for the completely unloaded cracks is larger than the initial intensity, implying that some increase in the crack area must have occurred. Thus, irreversibility in the scattering response may be taken as a definitive indicator of the residual stress influence on crack growth.

Simultaneous optical observations of the crack behavior during the tests described above did in fact confirm the correspondence between signal irreversibility and crack expansion enroute to failure.<sup>6</sup>

## B. MACHINING FLAWS

Acoustic scattering tests were accordingly run on silicon nitride surfaces which had been machined.<sup>6</sup> The characteristic signal response is shown in Fig. 4. It is immediately clear that the response shows the same irreversibilities as the curve for as-indented surfaces in Fig. 3. It may therefore be concluded that the machining flaws undergo stable crack growth prior to failure. This is not altogether surprising, since machining damage

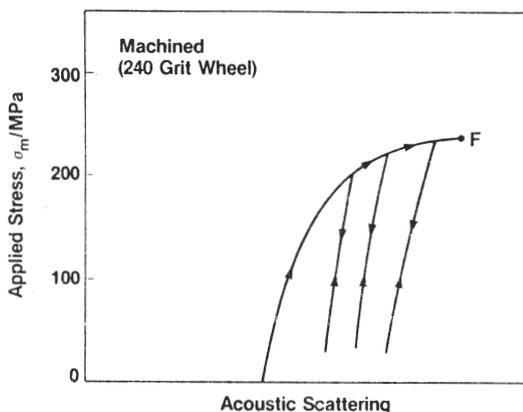


Fig. 4. Acoustic scattering for flaws in machined  $\text{Si}_3\text{N}_4$  surfaces. After Ref. 6.

can be regarded as the accumulated effect of a large number of individual contact events. Certain differences in quantitative crack relations may be expected, due for instance to more complex contact geometry (linear instead of axisymmetric) and interactions with neighbors. Nevertheless, the essential distinctive element of the ideal indentation flaw is preserved, namely that strength is controlled by a well-defined crack extension process under the driving action of a residual stress field.

These results serve to provide a solid justification for adopting indentation fracture mechanics as the basis for modelling a wide range of strength-controlling flaws in glasses and ceramics.<sup>2</sup>

#### IV. AGING EFFECTS

The strength of freshly formed flaws in glass tends to increase on aging in aqueous environments. This strengthening has been reported for many damage configurations, e.g. scratching,<sup>19</sup> abrasion,<sup>20</sup> and cutting.<sup>21,22</sup> In these examples the flaws are clearly rendered less effective by chemical interactions with water. In this section we examine the aging evidence, some old and some new, in terms of indentation fracture mechanisms.

##### A. SODA-LIME GLASS IN WATER AT ROOM TEMPERATURE

In a classic early aging study on soda-lime glass in water Mould<sup>20</sup> showed that:

- (i) The strength of specimens with grit blast abrasion and sharp particle scratch damage increased, by 30% and 60% respectively, after immersion for one day at room temperature;
- (ii) Annealing of freshly damaged specimens increased the strength by approximately the same amount as the aging treatment;
- (iii) The strength of specimens with annealed damage did not increase further with more aging;
- (iv) Annealed damage was less susceptible than fresh damage to "fatigue" (i.e. time-dependent reductions in strength) in water.

The same characteristics have since been observed in Vickers indentation flaws.<sup>18,23,24</sup> Figure 5 shows the variation of strength with aging time for indented soda-lime glass specimens. The aging environment in this case was silicone oil, but trace quantities of water were inevitably present. A strength increase of  $\approx 25\%$  is realized after several hours, similar in trend to the result for abrasion damage mentioned in (i) above. As to the effects referred to in

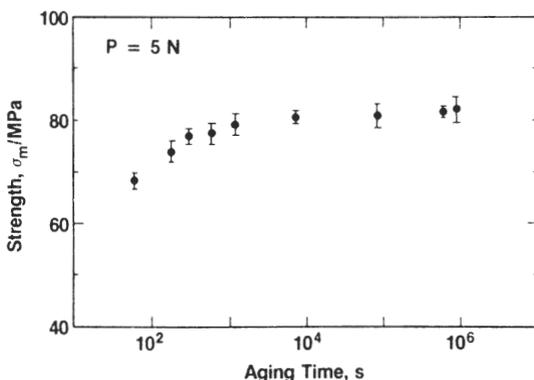


Fig. 5. Strength of Vickers-indented soda-lime glass as a function of aging time in oil between indentation and strength tests.

(ii) and (iii), we have already noted a comparable degree of strengthening on annealing indentation cracks (Sect. 2.2), and the strength thereafter is found to be insensitive to prolonged exposure to laboratory environments. Finally, the fatigue behavior mentioned in (iv) has been reproduced with indented specimens;<sup>24</sup> an extended discussion of this last point will be given in another paper in this volume.<sup>11</sup> Thus the aging responses of the present indentation cracks and Mould's artificial damage in soda-lime glass are in all respects similar.

This correlation between indentation and natural flaw types provides us with a powerful means for identifying the actual mechanism of aging. Indentation flaws, it will be recalled, can be observed directly during their entire evolutionary process, a luxury not available in the case of natural flaws. Thus, as we saw previously in Fig. 7 of Ref. 1, indentation cracks are observed to extend appreciably in their post-indentation lifetime. Figure 6, which shows the same crack system at two stages in its aging history in a moist environment, illustrates this extension clearly. Thus any hypothetical aging mechanisms which specifically exclude crack lengthening, notably those based on crack sharpening concepts,<sup>8-10</sup> can be immediately ruled out for the damage conditions considered here.

At first sight it may seem improbable that an increase in crack size could lead to a strength increase during aging. However, we recall from Sect. 2.1 that the critical conditions for failure given in Eq. (1) are independent of the initial *radial* crack size. In the absence of any other changes, the subcritical growth of the radial crack from  $c_0$  to  $c'_0$  simply reduces the extent of precursor extension that ensues in the subsequent strength test (Fig. 1); the ultimate strength  $\sigma_m$  is unaffected. In reality, other changes do occur, notably

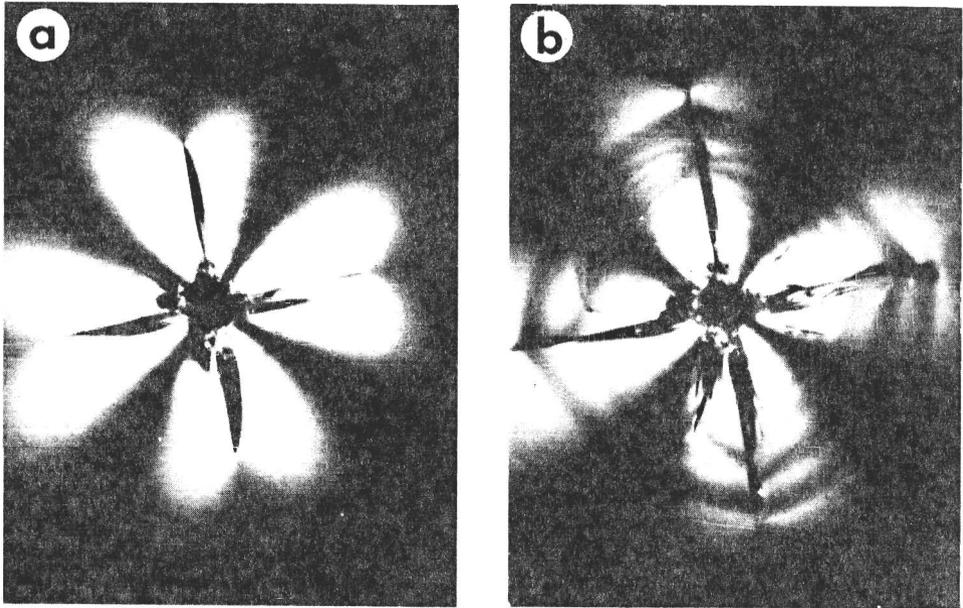


Fig. 6. Vickers indentation ( $P = 50$  N) in soda-lime glass, (a) immediately after indentation, (b) after aging in moist environment for 1 hr.

in the *lateral* crack configuration (faintly visible in Fig. 6). In fact, the lateral cracks tend to grow more rapidly than their radial counterparts during aging. It has been demonstrated that these two countervailing crack components can interact in such a way as to reduce  $\chi$  or  $\psi$  in Eq. 5, and thereby increase the stress level  $\sigma_m$  needed to produce failure.<sup>22,23</sup> In this sense, aging is analogous to annealing in its effect on strength, consistent with Mould's second observation listed earlier in this section.

## B. SILICA GLASS IN SILICIC ACID AT ELEVATED TEMPERATURE

In Ref. 1 we alluded to the distinction that can be made between "normal" and "anomalous" glasses: in the first glass type, typified by soda-lime glass, contact deformation occurs via a volume-conserving shear process, and the intensity of residual stress is high; in the second, typified by high-silica glass, the deformation involves densification of the network structure, with correspondingly reduced residual stress intensity. This has led some workers to suggest that the model of residual-stress relief proposed above for soda-lime glasses may be inappropriate for explaining aging effects observed in silica glass.

This contention is examined here. We refer specifically to aging experiments on abraded silica glass (Corning Vycor) in silicic acid solutions at

TABLE I. Aging of Silica Glass in Aqueous  $\text{Si}(\text{OH})_4$  solution at  $90^\circ\text{C}$ 

Flaw Type	Strength (MPa)	
	t = 0	t = 10 days
Abrasion	$63.3 \pm 2.4$	$83.2 \pm 13.0$
Machining	$50.2 \pm 1.9$	$67.2 \pm 10.0$
Indentation Radial Crack	$47.0 \pm 3.7$	$58.5 \pm 7.8$
Indentation Cone Crack	$36.0 \pm 2.2$	$39.6 \pm 3.0$

elevated temperatures ( $90^\circ\text{C}$ ) conducted by Ito and Tomozawa.<sup>10</sup> We have repeated the conditions of their experiments, but have extended the study to other flaw types, including indentations. In our tests comparative strength tests were run at zero and extended (10 day) aging times. The results are shown in Table 1. For the surfaces with the abrasion and machining damage and with the indentation radial cracks the strength increases are in the range 25-35%, i.e. comparable with the results for soda-lime glass. For the surfaces with the indentation cone cracks, however, the increases are negligibly small. The feature which distinguishes the cone cracks in this group of flaw types is the absence of any associated residual stress field; the contact (sphere on flat) is purely elastic to the point of fracture.<sup>2</sup> Hence we must again conclude that the existence of residual stresses is a critical element of the aging process, and that strength increases simply reflect some time-dependent relaxation of these stresses.

## V. DISCUSSION

We have demonstrated that certain important flaw types in glass, notably those associated with machining and abrasion damage, are governed in their strength behavior by the laws of crack propagation. This we have been able to do by drawing analogies between the responses of these flaws and of indentation cracks, as in the acoustic scattering and the aging experiments. Central to our argument has been the stabilizing role of residual contact stresses in the crack growth characteristics. Precursor extension prior to failure, directly observable in the Vickers indentation configurations, is cited as the most definitive manifestation of this residual stress influence.

It is interesting to consider these observations in the context of current, opposing views of failure in glass. In one view failure originates from cracks with atomically sharp tips.<sup>25</sup> The behavior of sharp cracks can be described in terms of a fracture mechanics formulation in which the crack length is the sole strength-controlling dimension. In the other view strength-controlling flaws are considered to be intrinsically "blunt." The strength is then related to the radius of curvature of the tip as well as to the length of the crack.<sup>8-10</sup>

Thus in the interpretation of fatigue strengths, for example, the conflict becomes one of flaw *lengthening* (subcritical crack growth) vs flaw *sharpening* (crack initiation from a notch-like defect). It is, of course, conceivable that these two steps could occur sequentially in any failure process, in which case the slower would control the kinetics. Such transitions in character have in fact been claimed to occur in large-scale (double-cantilever beam) cracks in glass.<sup>26-27</sup> Once the stress intensity factor is lowered below  $\approx 0.25 \text{ MPa m}^{1/2}$ <sup>27</sup> some hysteresis becomes apparent in the loading needed to restart the propagation. For the conditions of our experiments on contact-related flaws the identification of a well-defined crack propagation stage would appear to be beyond dispute; the question remains as to whether the stress intensity factor for these flaw types could ever fall below the "blunting" limit just indicated.

Accordingly, in the aging tests described in Sect. 4 it is possible that  $K_r$  in Eq. (1) could decrease below the limit at sufficiently long times. For this to happen the crack size would have to extend beyond  $\approx 2 c_0$  (recall  $K_r = K_c$  at  $c_r = c_0$ ), an amount in excess of any post-indentation growth observed in our data range (e.g. Fig. 5; see also Refs. 1, 28, 29). Of course, any processes which act to relax the  $\chi$  term in Eq. (1) (e.g. lateral crack growth, annealing) would contribute to a reduction in  $K_r$ . Controlled indentation tests in this region of behavior could prove a ripe area for further research.

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## DISCUSSION

**Q: J. J. Mecholsky.** Although I agree that residual stress is important in indentation and machining cracks, I think there are differences that have to be taken into account. The grinding groove can be deep and many of the chips removed can relieve the residual stress. I think that the asymmetry of cracks from machined surfaces, i.e. elongated crack along the direction of grinding and the more uniform flaw perpendicular to grinding, can influence the amount of residual stress and amount of relief due to lateral growth.

**A: B. R. Lawn.** There is indeed strong evidence that residual stress relief can occur at contact sites due to interactions with neighboring events, and we present evidence for this in a later paper in this volume. Nevertheless, the intensity of residual stress around machining flaws can remain sufficiently large as to exert a dominant influence on the characteristic flaw response, as the data in Fig. 4 unequivocally demonstrate.

**Q: M. M. Chaudhri.** (i) What was the sensitivity of the acoustic technique?

(ii) Does this technique have any advantages over the optical methods?

**A: B. R. Lawn.** (i) The sensitivity of the acoustic method used was such that cracks down to a size of about 20  $\mu\text{m}$  could be detected. This is about the limit currently available.

(ii) Yes. Opaque materials can be studied, and subsurface flaws can be monitored. Also, the location of the failure-controlling flaw does not have to be predetermined.

**Q: M. Tomozawa.** Relating to the aging of machined high silica glass, we arrived at a model of crack blunting by dissolution and precipitation (*J. Amer. Ceram. Soc.* 65, 368, 1982), since the mechanical strength increases faster in saturated silicic acid solution than in distilled water, while the

dissolution rate of the glass is faster in distilled water. I do not think the residual stress model can explain this phenomenon.

**A: B. R. Lawn.** In our tests we duplicated the conditions of the experiments just described, but using indentation as well as machining-damage flaws (see Sect. IV.B). Under these conditions the indentation cracks grew steadily, under the action of the residual driving force, throughout the aging process. Hence the blunting/dissolution argument cannot apply to indentation cracks, at least over the aging times covered by Ito and Tomozawa. Now in our tests we could not directly observe the response of the machining flaws in the same way as the indentation cracks, but we were able to demonstrate that the same degree of strength increase with aging did occur in the two cases, *provided residual stresses were present*. This result, plus the observation in the acoustic scattering tests that machining flaws show the same characteristic irreversible crack growth behavior under stress (Sect. III), is strong evidence for the proposed model. The suggestion that blunting mechanisms are responsible for the aging effects observed by Ito and Tomozawa is based on circumstantial argument, although this is not to rule out the possibility that such mechanisms might not operate under different conditions (see Sect. V).