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Residual stress in glass: indentation crack and fractography approaches

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Abstract

Objective—To test the hypothesis that the indentation crack technique can determine surface residual stresses that are not statistically significantly different from those determined from the analytical procedure using surface cracks, the four-point flexure test, and fracture surface analysis.

Methods—Soda-lime-silica glass bar specimens (4 mm × 2.3 mm × 28 mm) were prepared and annealed at 650 °C for 30 min before testing. The fracture toughness values of the glass bars were determined from 12 specimens based on induced surface cracks, four-point flexure, and fractographic analysis. To determine the residual stress from the indentation technique, 18 specimens were indented under 19.6 N load using a Vickers microhardness indenter. Crack lengths were measured within 1 min and 24 h after indentation, and the measured crack lengths were compared with the mean crack lengths of annealed specimens. Residual stress was calculated from an equation developed for the indentation technique. All specimens were fractured in a four-point flexure fixture and the residual stress was calculated from the strength and measured crack sizes on the fracture surfaces.

Results—The results show that there was no significant difference between the residual stresses calculated from the two techniques. However, the differences in mean residual stresses calculated within 1 min compared with those calculated after 24 h were statistically significant (p=0.003).

Significance—This study compared the indentation technique with the fractographic analysis method for determining the residual stress in the surface of soda-lime silica glass. The indentation method may be useful for estimating residual stress in glass.

Keywords

Indentation; residual stress; fractography; fracture toughness

Introduction

Residual stresses are often present in ceramic materials either by design or as a consequence of processing [1]. Any manufacturing process that changes the shape of a glass-phase ceramic or results in very large temperature gradients causes residual stress to develop [2]. In some cases, compressive residual stresses are intentionally introduced to improve the mechanical

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property performance by virtue of the inhibiting effect of compressive stress crack propagation [3–6]. Regardless of the source of residual stress, it plays an important role in serviceability of a component. A variety of techniques have been used to determine the magnitude of residual stresses, and each method has some limitations. For example, X-ray or neutron diffraction techniques can be applied effectively only to crystalline materials. Birefringent methods or the photoelastic technique are limited to transparent materials, and electrical-strain gauge applications are limited in their ability to distinguish residual stresses from the total stress [2, 7].

The crack indentation technique based on a pyramidal diamond indenter has been widely used to measure mechanical properties of brittle materials such as hardness, brittleness, and fracture toughness [8]. This technique has the potential for rapid evaluation of material properties of small samples and may be useful for materials development or quality control [9]. Recently, the Vickers indentation method was criticized as an unreliable test for fracture toughness determination or for any other fracture resistance parameter [10,11]. Additionally, calibration constants have been empirically determined and adjusted according to the material under investigation to achieve reasonable results [10]. However, because of its expediency, convenience, and small sample requirement, this technique still maintains its popularity as a qualitative tool to determine mechanical parameters of ceramic materials.

Indentation methods have been proposed since the 1930s as means of measuring or detecting surface residual stresses [2]. The effective compressive or tensile residual stresses can be estimated by comparing the fracture stress of stressed samples to that in unstressed samples [12]. The indentation technique has also been employed to calculate global residual stresses in bilayer dental ceramics [13]. These global stresses are calculated using a fracture mechanics equation combined with local residual stresses from contact damage. A new indentation method was proposed to measure the residual stresses around a Vickers indentation crack [14], and this method can be extended to other cases. In order to determine the residual stress around an indentation, first, an indentation must be made in unstressed material, and the stress intensity factor for the indentation can be obtained from the equation [15]:

$$K_c = \alpha \left[\frac{E}{H} \right]^{1/2} \left[\frac{P}{(c_0)^{3/2}} \right] \quad (1)$$

where K_c is the fracture toughness, P is the indentation load, c_0 is the crack length at that load, E/H is the ratio of elastic modulus to hardness, and α is a dimensionless constant. Under the influence of residual stress, the crack grows to a new equilibrium length (c) at the same indentation load (P) as in the stress-free case. At equilibrium, the crack will experience a composite stress intensity, and K_c is given by the equation:

$$K_c = \alpha \left[\frac{E}{H} \right]^{1/2} \left[\frac{P}{c^{3/2}} \right] \pm \psi \sigma_a c^{1/2} \quad (2)$$

Combining equation (1) and (2), residual stress (σ_R) can be calculated using equation 3:

$$\sigma_R = K_c \left[\frac{1 - \left(\frac{c_0}{c} \right)^{3/2}}{\psi c^{1/2}} \right] \quad (3)$$

where ψ is a crack geometry factor. For the radial-median crack, the annealed surface crack is assumed to be semicircular in shape, and has a value of 1.24. The crack length in the unstressed material is c_0 , and c is the crack length under the influence of the residual stress.

A similar method that involves the superposition of stress intensity factors was used to measure near-surface residual stresses in tempered glass based on another fracture mechanics approach [12], and was also used to calculate the local residual stress in bilayer dental ceramics [13]. This method employs a beam having a surface residual stress that is subjected to flexural loading. For a semi-elliptical crack of depth a and half width b , c is the equivalent semi-circular crack size where $c = (ab)^{1/2}$. The stress intensity factor at the border of the semicircular crack is calculated from the following equation:

$$K_I = \left(\frac{2}{\pi^{1/2}} \right) \sigma_A c^{1/2} Y_F(\theta) - \left(\frac{2}{\pi^{1/2}} \right) \sigma_R c^{1/2} Y_R(\theta) \quad (4)$$

where $Y_F(\theta)$ is the crack border correlation factor associated with flexural loading, and $Y_R(\theta)$ is the crack-border correction factor associated with the residual stress field [12]. In the case of flexural loading, $Y_F(\theta)$ is readily available, and $Y_R(\theta)$ in a residual stress field is assumed to be unity. Residual stress can be calculated as follows:

$$\sigma_R = \frac{\left(\frac{2}{\pi^{1/2}} \right) \sigma_A c^{1/2} Y_F(\theta) - K_I}{Y_R(\theta) \left(\frac{2}{\pi^{1/2}} \right) c^{1/2}} \quad (5)$$

We will test the hypothesis that the indentation crack technique can determine surface residual stresses that are not statistically significantly different from those determined from the analytical procedure using surface cracks, the four-point flexure test, and fracture surface analysis.

Materials and Methods

Specimen preparation

Glass bars (2.3 mm × 4 mm × 28 mm) were prepared from a soda-lime silicate glass plate. All sharp edges were beveled by grinding with a 30- μ m grit metallographic polishing disk to remove high stress concentration areas. The glass transition temperature (T_g) of soda-lime silicate glass was determined using DSC (Differential scanning calorimetry) at a heating rate of 3 °C/min. All specimens were annealed at 650 °C (75 °C over T_g) for 30 min to eliminate all residual stresses before testing. The specimens were cooled down slowly by programming the furnace to be opened gradually for 30 min from the annealing temperature at 650 °C to room temperature. **Fracture toughness test:** Twelve beam specimens were indented using a Vickers hardness indenter at a load of 19.6 N while viewing under an optical microscope at 400× magnification (Fig. 1). Crack measurements were made within 1 min and 24 h after indentation using a calibrated imaging system (OmniMet Modular Imaging System, Buehler, Lake Bluff, IL) at 100× magnification. The mean crack lengths measured within 1 min and 24 h after the indentation were chosen as the crack size of the unstressed materials (c_0) to calculate the residual stress from equation 3. Specimens were divided into two groups.

For Group 1, the annealed and indented specimens were subjected to four-point flexure to failure with a 20.0 mm lower span and 6.7 mm upper span at a crosshead speed of 0.5 mm/min. For Group 2, the specimens were annealed at 650 °C for 30 min after indentation to remove the residual stress from the indent prior to the four-point flexure. The failure strengths were

calculated, the critical crack sizes were measured, and the fracture toughness (K_C) was then determined from the equation:

$$K_C = Y \sigma_f c^{1/2} \quad (6)$$

where Y is a numerical constant that accounts for location, loading condition and crack geometry [1.65 for indentation cracks (Group 1) and 1.24 for indentation cracks with local residual stresses from the indentation being removed (Group 2)]. c is the equivalent semi-circular critical flaw size calculated from $(ab)^{1/2}$, where a is the semiminor axis, and b is the semimajor axis of a semi-elliptical crack [16]. Assuming a small crack relative to the thickness, the geometrical factor (Y) was calculated based on Randall's interpretation [17] of Irwin's work [18,19]:

$$Y = [0.515 Q^{1/2}]^{-1} \quad (7)$$

Q ranges in value from 1.00 for long, shallow cracks ($Y=1.94$) to $(\pi/2)^2$ for semicircular cracks ($Y=1.24$). If we calculate the area of an elliptical crack of depth, a , and half-width, b , and equate that to the area of a semi-circular crack, i.e., $(a b)^{1/2} = c$, then we can use the Y factor for a semi-circular crack with radius c , which is 1.24. For the effect of local residual stress from indentations, the value of Y is changed to $4/3$ of the original value, hence, 1.65 [20].

Crack indentation approach: A total of 18 specimens were heated at 650 °C for 30 min and compressive residual stress was intentionally introduced within the surface by applying compressed air at a pressure of 0.05 MPa at a controlled distance of 2.5 cm from the center of the 18 bars for 1 min after removing the specimens from the furnace. Indentation cracks were produced at the middle of each bar under a 19.6 N load using a Vickers hardness indenter. Crack measurements were made on each bar within 1 min (Group A) and 24 h (Group B) after indentation. The crack length, which is perpendicular to the long axis of the bar, was measured because this crack was subjected to tensile loading (Fig. 1). The crack lengths (c) from this group were compared with the mean crack length values (c_0) of the annealed group within 1 min and 24 h after indentation. Residual stresses were calculated using equation 3.

Fractography approach: A total of 18 specimens from above were tested under four-point flexure (Group C). Fracture strength was calculated, fracture surfaces were analyzed, and residual stresses of each bar were calculated using equation 5.

Statistical analysis

Residual stresses, which were calculated using equation 3 within 1 min (Group A) and 24 h (Group B) after indentation and from equation 5 for each specimen in Group C, were compared using a randomized block design. Because all measurements were made on the same specimens and were compared among each other using different approaches, the variability within each specimen (within each block) was less than that between specimens (between blocks). A statistical analysis was performed at a significance level of 0.05 (SAS 9.1.3 Service pack 4).

Results

Fracture toughness and strength values of the soda-lime glass are shown in Table 1. Fracture surfaces were analyzed to determine critical crack dimensions (Fig. 2). Although strengths from Group 1 and Group 2 were different, the fracture toughness values are statistically the same regardless of the method used in this study [$p=0.62$]. The fracture toughness value in Table 1 was used in the determination of surface residual stresses in the soda-lime-silica glass.

Indentation cracks of unstressed and stressed specimens are shown in Fig. 3. The average crack size on the surface of glass bars without residual stress was larger than that with residual compressive stress for the same indentation load.

Compressive residual stresses, which were calculated using the crack indentation technique for Groups A and B, were not significantly different from those determined using the quantitative fractographic approach (Group C) [$p=0.19$ and 0.13 , respectively]. However, the mean residual stress for Group A was significantly different from that for Group B [$p=0.003$]. The calculated residual stress values are shown in Table 2. We could not measure the crack length of four specimens from Group A because no cracks initiated during that period of time. However, the crack lengths of the same specimens were observed at 24 h (Group B).

Discussion

Table 2 shows the residual stress of Group A and Group B calculated using equation 3 compared well with that calculated using equation 5 (Group C). There was no significant difference between both techniques; the results show good agreement between the indentation technique at 24 h (Group B) and the fractographic approach (Group C). Because of the greater effect of slow crack growth during the early period after indentation, the crack measurements of Group A were more difficult to obtain than those of Group B as rapid crack propagation occurred during the early period. Thus, a computer controlled, rapid imaging system was connected to the indenter and optical microscope. Furthermore, cracks were not well-developed in some specimens of Group A, and they could not be observed (four specimens), but they could be detected at 24 h.

The techniques presented in this study were based on fracture mechanics principles for determining the compressive residual stress on glass surfaces. Both techniques are similar in that they involve the superposition of stress intensity factors. For indentation cracks, the Vickers indenter was used with a moderate load and a permanent indentation was formed with cracks of approximately equal radial crack lengths from each corner of the impression. Residual stress was determined by comparing crack lengths of indentation with a residual stress field, with those crack lengths from indentations in the unstressed material using the same indentation load [21]. Therefore, cracks from the materials with a tensile stress field surrounding the crack are longer than those in the unstressed materials, whereas cracks under a compressive stress field will be shorter. For four-point flexure with a surface-indented crack, the applied loading results in a flexural stress distribution with a maximum tensile stress (σ_A) at the surface of the bars (Fig. 4) due to the superposition of stresses, and the critical stress intensity at the crack tip in this case will be greater than the critical stress intensity factor of the unstressed material [22]. This method is useful for determining surface residual stresses. For the indentation technique, it is essential to understand the residual stress distribution within materials. This approach was used to investigate the residual stress distribution around a primary indented crack [14,21,23]. However, the indentation technique requires a high quality polished specimen and a completely flat surface; otherwise, the crack length cannot be correctly measured.

In conclusion, there was good agreement between residual stresses calculated using the indentation technique and the fractographic approach based on induced surface cracks, four-point flexure, and fracture surface analysis. Therefore, the indentation technique may be employed under carefully controlled conditions as a simplified method to determine the effective residual stress value within brittle materials such as glasses, and glass ceramics.

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Figure 1. Schematic of specimen dimensions showing indentation crack used for residual stress calculation.

Figure 2.

Optical photographs of the fracture surface showing a critical crack of a fracture toughness test specimen (a) with local residual stress from an indent (Group 1) and (b) without local residual stress from an indent (group 2) [annealed after indentation]. Arrows denote the critical crack boundary.

Figure 3.

Optical photograph of an indentation crack induced at a load of 19.6 N in (a) an annealed specimen and (b) a specimen with compressive residual stress. The crack length of specimens with compressive residual stress is shorter.

Figure 4.

A schematic of a semicircular crack with indentation crack depth $[a]$ within the global compressive residual stress region on the side of indent $[\sigma_R]$ under uniform flexural stress from loading $[\sigma_A]$ [22].

Table 1

Fracture strength (MPa) and fracture toughness (MPa m^{1/2}) value of soda-lime glass used in this study

Condition	Strength (MPa)	Fracture Toughness (MPa m ^{1/2})
Anneal + Indent (Group 1)	47.6 (2.7)	0.82 (0.07)
Anneal + Indent + Anneal (Group 2)	64.7 (2.1)	0.81 (0.04)

Table 2

Residual stress value (MPa) of each specimen from indentation technique and the superposition of the stress intensity factor method

Specimen #	Residual stresses calculated from indentation technique(MPa)		Residual stress calculated from fractography (MPa)
	Group A (1 min)	Group B (24 h)	Group C
1	-6.65	-5.84	-7.27
2	-11.53	-15.12	-13.16
3	-15.25	-22.69	-23.35
4	N/A *	-29.84	-16.88
5	N/A	-24.92	-25.55
6	N/A	-17.92	-13.31
7	-12.32	-20.61	-2.08
8	-23.30	-22.27	-13.51
9	-12.72	-23.13	-23.46
10	-18.52	-20.21	-14.24
11	-23.30	-27.29	-28.33
12	N/A	-31.46	-31.70
13	-13.54	-20.61	-19.38
14	-14.38	-21.85	-24.00
15	-13.96	-15.79	-16.54
16	-13.96	-20.21	-19.34
17	-6.97	-10.29	-12.36
18	-7.95	-12.29	-11.73

* no crack observed