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Strength and fracture toughness of indented glass–nickel compacts

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Strength and fracture toughness of glass–nickel compacts have been measured in a four-point bend test. Knoop microhardness indentation technique was used to introduce controlled surface flaws in hot-pressed sodium borosilicate glass containing up to 10 vol% spherical nickel powders for the bend test and also to assess the strength of the compacts. It was found that as the number of voids increased, the load to induce indentation flaws, the subsequent fracture strength and toughness all increased.

1. Introduction

When a crack front interacts with an array of second-phase inclusions, normally it bows out between them in a way similar to a dislocation bowing between obstacles. Lange [1] suggested that the increase in strength and fracture surface energy for a composite are primarily due to a line tension effect at the crack front. Various brittle matrix composites have indicated that the line tension is the major contribution for increase in strength [2]. The interaction of a crack front with the dispersed phase and toughening of brittle matrix composites were studied by other investigators [3–5] by varying second-phase particles of different thermal expansion coefficient and elastic modulus.

In the present investigation, a model system was used to measure the strength and fracture toughness of glass–nickel compacts containing controlled surface flaws which were introduced by using a Knoop microhardness indenter [6–8]. The samples were tested in four-point bending to measure the strength. Fracture toughness was then calculated from the strength and flaw depth which was measured, by using an optical microscope, from the fracture surface.

2. Experimental procedure

Sodium borosilicate glass of composition 16 wt% Na_2O, 14 wt% B_2O_3 and 70 wt% SiO_2 was used as a matrix [9, 10] and nickel microspheres of 20 μm average diameter were used as a dispersed phase. Thermal expansion of nickel ($13 \times 10^{-6}\,{}^\circ C^{-1}$) was much higher than that of glass ($7.8 \times 10^{-6}\,{}^\circ C^{-1}$). Different volume fractions of glass and nickel powders were vacuum hotpressed at 725° C using 13.80 MN m^{-2} pressure for 10 min. On cooling, nickel microspheres shrink faster than glass, leaving voids with nickel inside them. Glass-matrix compacts were cut into strength specimens with a diamond saw. Rectangular bar specimen ($25 \times 4 \times 1.2\,mm^3$) were indented by Knoop indenter which produced a semicircular flaw. The indentation load was varied from 200 g to 2000 g. The samples were tested at room temperature in a four-point bend jig with an outer and inner span of 19 and 6 mm, respectively. A cross-head speed of 0.0254 cm min^{-1} was used. The flaw depth was measured from the fractured specimen using an optical microscope.

3. Results

Fig. 1 shows the fracture surfaces of glass and glass–nickel compacts with the semicircular indentation-induced flaws. Fig. 2 shows an enlarged fracture surface of glass–5 vol% Ni. The semicircular crack front is impeded by a random array of void/nickel obstacles during propagation and it bows out between them. Fig. 3 shows the plot of fracture strength versus the indentation load. In ordinary sodium borosilicate glass, the strength decreases rapidly with the indentation load and levels off at around 1000 g indenter load. In the case of glass–nickel compacts, the strength initially

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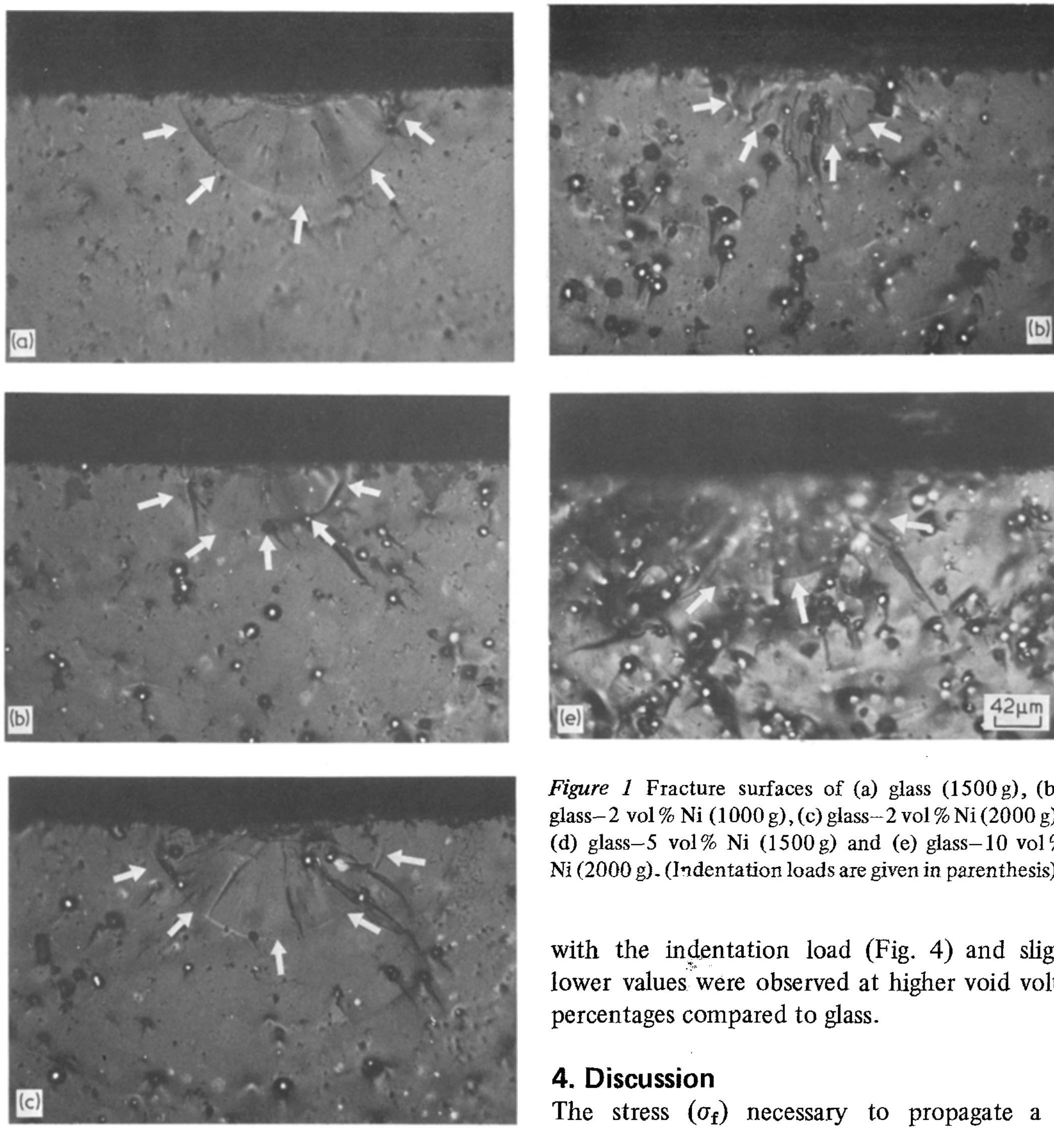

Figure 1 Fracture surfaces of (a) glass (1500 g), (b) glass–2 vol % Ni (1000 g), (c) glass–2 vol % Ni (2000 g), (d) glass–5 vol% Ni (1500 g) and (e) glass–10 vol% Ni (2000 g). (Indentation loads are given in parenthesis).

remains unchanged with the indentation load and then drops (particularly 2 and 5 vol% compacts) and levels off above 1000 g load. Up to 1000 g load, the inherent flaws (surface machining flaws and voids) are probably more critical than the indentation-induced flaws. For 10 vol% compacts, the strength is totally independent of indentation load. Fig. 3 also indicates that the strength of glass–nickel compacts increases with the void concentration after 1500 g load. The drop in strength of the compacts decreases gradually compared to that of non-indented samples. This is probably due to the resistance of crack propagation in the presence of void/particle obstacles (as indicated in Fig. 2) and the number of voids at higher volume fraction. The flaw depth increases with the indentation load (Fig. 4) and slightly lower values were observed at higher void volume percentages compared to glass.

4. Discussion

The stress (σ_f) necessary to propagate a pre-existing flaw through a series of obstacles was given by Lange [1] as

$$\sigma_f = \left[\frac{2E}{\pi a}\left(\gamma + \frac{T}{d}\right)\right]^{1/2} \qquad (1)$$

using Griffiths equation

$$\sigma_f = \left(\frac{2E\gamma}{\pi a}\right)^{1/2}, \qquad (2)$$

where E is the elastic modulus, γ is the fracture surface energy of the matrix, a is the flaw depth, T is the line tension (energy) of the crack front, and d is the mean free path [11] (taken as the interparticle spacing). The second term in Equation 1 depends on the value of the line energy of the crack front (T) and the distance between the dispersed particles (d). The line energy just prior

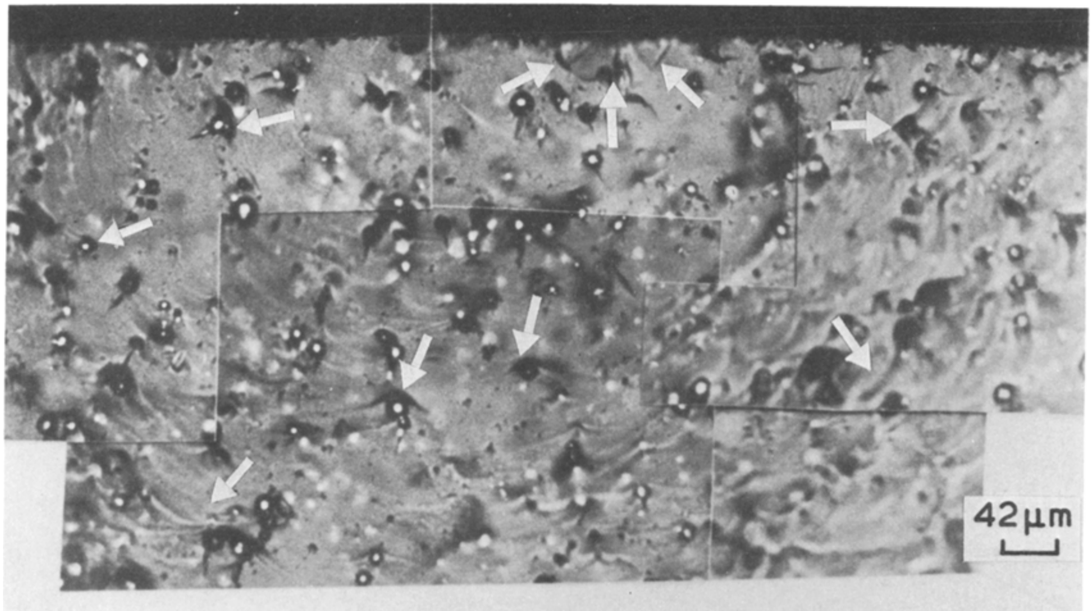

Figure 2 Enlarged fracture surface of glass–5 vol% showing the interaction of the crack front with the void/particle obstacles.

to crack propagation can be derived [1] as

$$T = \tfrac{2}{3}\gamma a. \tag{3}$$

The expression for the mean free path (d) between particles of uniform diameter (D) distributed randomly into the matrix was derived by Fullman [11] as

$$d = \frac{2D(1-\phi)}{3\phi} \tag{4}$$

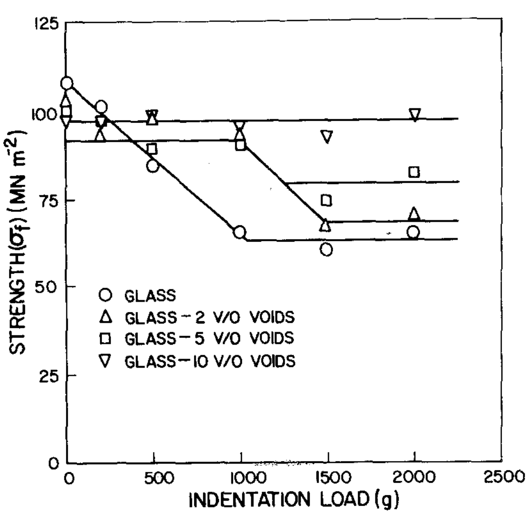

Figure 3 Strength is plotted against the indentation load for glass–Ni compacts.

where ϕ is the volume fraction of the dispersed phase. Substituting T and d into Equation 1.

$$\sigma_f = \left[\frac{2E\gamma}{\pi}\left(\frac{1}{a} + \frac{\phi}{D(1-\phi)}\right)\right]^{1/2}. \tag{5}$$

Substituting K_{IC} for $(2E\gamma)^{1/2}$ (fracture toughness), Equation 5 reduced to

$$\sigma_f = \left[\frac{K_{IC}}{(\pi)^{1/2}}\left(\frac{1}{a} + \frac{\phi}{D(1-\phi)}\right)\right]^{1/2}. \tag{6}$$

The semicircular flaw which is produced by indentation, can be better approximated by the thumbnail crack. In thin plates, the leading edge of such a thumb nail crack should see, essentially, plane strain constraint [12] so the corresponding fracture toughness may be low. As the load is in-

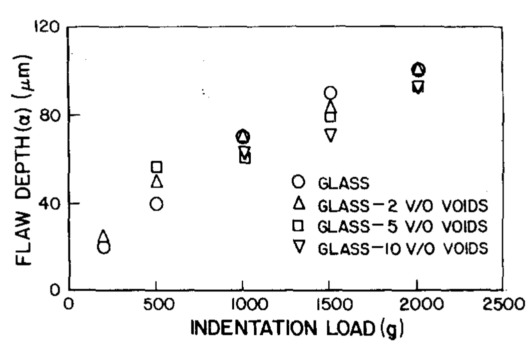

Figure 4 Flaw depth versus indentation load.

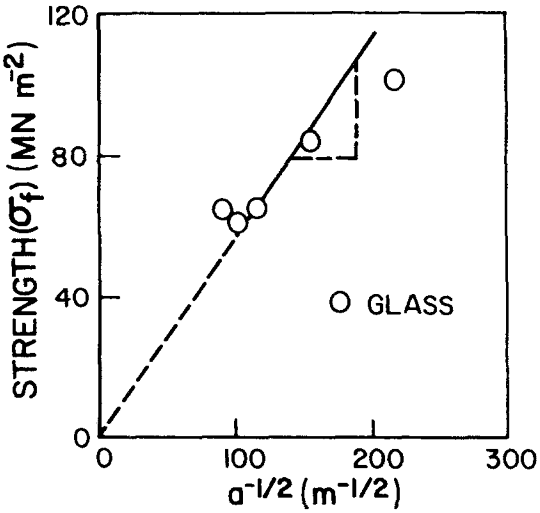

Figure 5 Strength of glass is plotted against (flaw depth)$^{-1/2}$.

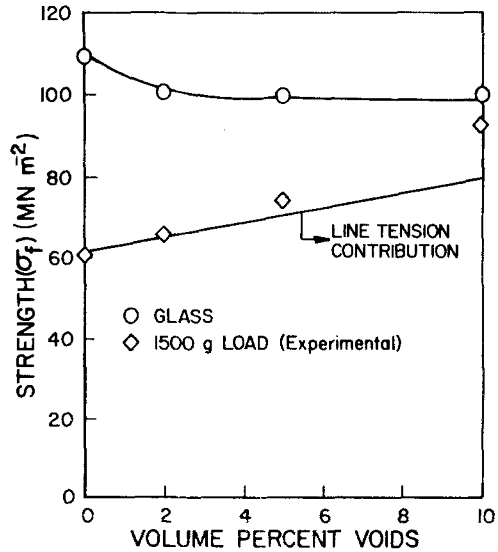

Figure 6 Strength of glass with respect to vol% voids.

creased the thumb-nail will "click" through and become a through crack. The stress intensity factor for a surface semicircular crack was calculated [13] from the equation given by Irwin [14] as

$$K_{IC} = 1.162\sigma_f(a)^{1/2}. \quad (7)$$

From Equation 2, when σ_f is plotted against $a^{-1/2}$, a straight line relationship for glass is obtained (Fig. 5). The slope gives the fracture toughness of glass as $1.0\,\text{MN}\,\text{m}^{-3/2}$. This value is consistent with previous results [10]. Substituting K_{IC} values in Equation 6, the strength values for a composite using a line tension contribution are

$$\sigma_f \simeq 0.57\left[\frac{1}{a}+\frac{\phi}{D(1-\phi)}\right]^{1/2}. \quad (8)$$

Thus as the volume fraction of the dispersed phase increases and the flaw depth decreases, the strength should increase. Taking the average diameter of the particle as 20 μm and flaw depths from Fig. 4, σ_f is calculated and plotted in Fig. 6. In actual measurements, the strength at higher volume fraction of voids was even higher than the values predicted by using line tension contribution. Fig. 6 also shows the strength reduction of approximately 11% with the 10 vol % voids. The strength reduction is slightly less than that observed by Bertolotti and Fulrath [15] (18% in their experiment).

The calculated K_{IC} values of glass using this technique show a lower value than is obtained from Equation 2. This is probably due to the residual stress [6] resulting from the microhardness indentation [16, 17]. The residual stresses can be eliminated by annealing [6, 13] the indented sample. K_{IC} is independent [13] of the indentation load as shown in Fig. 7. For glass–nickel compacts, K_{IC} values show a definite increase of more than 20%, with increase in voids as shown in Fig. 8. This indicates that glass–nickel compacts containing higher volume percent of voids are quite resistant to crack propagation. When the crack starts propagating it loses its energy temporarily at the void–matrix interface and it is impeded by the void/particle obstacle, and needs an additional energy to propagate the crack, leading to failure. Therefore, in glass–nickel compacts, the void/nickel particles dispersed phase is quite resistant to crack propagation. As the volume percent of dispersed phase is increased, the load necessary to propagate the indentation flaw is increased and subsequently

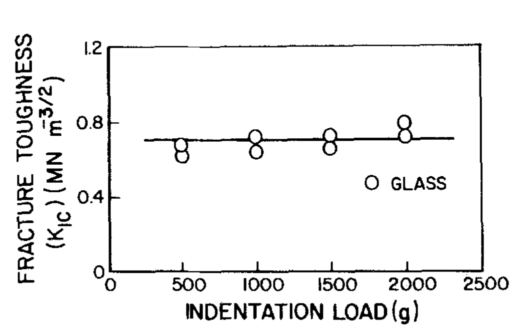

Figure 7 Fracture toughness is independent of indentation load.

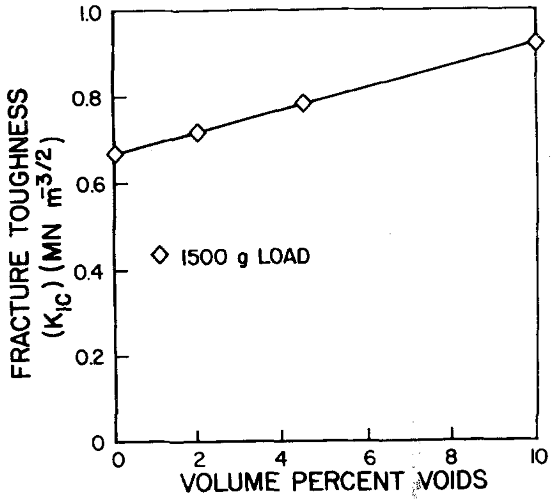

Figure 8 Fracture toughness is showing a definite increase with increase in vol% voids.

the strength and fracture toughness are increased. Thus, in application of glass specimens where the resistance of crack propagation is of particular interest (e.g. in thermal shock resistance), some void/particle inclusions are quite effective from its catastrophic failure. In the case of polycrystalline ceramics containing voids or pores, a similar concept can be applied to improve the crack propagation resistance but the effect might be less dramatic compared to the ceramics containing inclusions (high strength and elastic modulus), of similar thermal expansion coefficient with good bonding with the matrix.

5. Summary and conclusions

In glass, the strength decreases rapidly with increase in indentation load up to 1000 g and then levels off. But in glass–Ni compacts, as the volume percent of void/particle dispersed phase increases, the strength and fracture toughness are increased compared to glass containing similar strength controlling indented flaws. The fracture toughness of glass is independent of identation load and it increases with increase in void/particle inclusions which are resistant to crack propagation. Therefore, for crack propagation resistance in glass, 10 vol% nickel inclusion is found to be very effective and it can be used to improve the thermal shock resistance of sodium borosilicate glass.

Acknowledgement

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