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MICROMECHANICAL STRESS CONCENTRATION EFFECTS ON STRENGTH OF POROUS BRITTLE CERAMICS

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Raymond L. Bertolotti and Richard M. Fulrath

Inorganic Materials Research Division, Lawrence Radiation Laboratory, and Department of Mineral Technology, College of Engineering, University of California, Berkeley, California

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ABSTRACT

The effect of micromechanical stress concentrations around elastic discontinuities in a glass was investigated. Specimens of glass containing porosity of controlled size and volume fraction were fabricated by vacuum hot pressing and strength tested under uniaxial and biaxial stress conditions. Results were interpreted as a function of the relative sizes of Griffith flaws and the volume of material over which stress concentrations act.

For the glass investigated, assuming an adequate flow density, the strength was found to be dependent on pore size and volume fraction as well as the loading conditions. Differences in uniaxial and biaxial

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At the time this work was done the writers were, respectively, graduate student research assistant and associate professor of ceramic engineering, Department of Mineral Technology, University of California. R. L. Bertolotti is now associated with the Advanced Products Operation, General Electric Company, Sunnyvale, California.

strength are suggested to be due to differences in stress concentration geometry.

I. INTRODUCTION

It has been known for many years that the fracture strength of most brittle materials is less than the theoretical strength by two or three orders of magnitude. Griffith[1] attributed this discrepancy to microscopic flaws or cracks that exist throughout the bulk of brittle materials. Later, as the realization grew that fractures in glass invariably originate at the surface, Griffith's postulated cracks were restricted to the surfaces of the material. Fracture could originate at these cracks at an average stress level well below the theoretical strength of the material due to micromechanical stress concentrations. The existence of these flaws has recently been verified by several investigators.[2-4]

It is found that the strength of homogeneous brittle materials is highly dependent on the surface condition due to the effects of Griffith flaws. Many of the brittle materials of current technological importance are not truly homogeneous, being composed of mixtures of materials differing in mechanical, thermal, and other properties. Under applied load, the differences in elastic properties of the components lead to the existence of stress inhomogeneities. Although this can be used to advantage in many cases, such as fiber reinforcement of a ductile matrix, stress inhomogeneities can lead to a large decrease in the strength of composite materials. Such is the case when pores, which can be regarded as a second phase, are present in a brittle matrix.

Much experimental work has been done to determine the effect of porosity on the strength of brittle materials. Several investigators

have used a "cross-sectional area" approach and predicted the strength of matricies containing various forms of inclusions.[5,6] This approach predicts a smooth monotonic decrease in strength with porosity. Various other investigators used a "stress concentration" approach.[7-9] They calculated the stress concentrations around cavities of various kinds. This approach predicts an instantaneous decrease in strength with the introduction of the first pore. Based on observations of many dispersed phase-glass matrix composites, Hasselman and Fulrath[10] postulated that the effect of stress concentrations on strength is governed by the relative size of the Griffith flaw and the volume of material over which the stress concentrations act. On this basis, the effect of porosity on strength can be divided into three distinct regions.

When the pore size is much larger than the flaw size, Hasselman and Fulrath's Case I, under loading conditions flaws located near pores will be entirely within a stress concentration field. These flaws will nucleate fracture at average stresses equal to the zero-porosity strength divided by the maximum value of the stress concentration factor. A precipitous decrease in strength would be expected with the introduction of the first pore into the loaded area.

As the size of the pore approaches the flaw size, Hasselman and Fulrath's Case II, the flaws will not be entirely located in areas of high stress concentration. A smaller decrease in strength would be expected than for Case I, but there would still be a precipitous decrease in strength with the first pore although not as pronounced.

When the pore size is much smaller than the flaw size, Hasselman and Fulrath's Case III, the stress concentration field will no longer

be large enough to appreciably effect the strength of the material. Only a monotonic decrease in strength with porosity should be observed with no precipitous drop. The amount of material available to carry the load will determine the strength in this case.

The purpose of this investigation was to experimentally determine the effect of stress concentrations on the strength of a brittle matrix system. Different stress concentrations and stress concentration distributions were investigated by determining the strength of a glass with very closely controlled porosity. Both uniaxial and biaxial stress conditions were used and results were interpreted as a function of the relative size of Griffith flaws and the volume of material over which the stress concentrations act.

II. EXPERIMENTAL PROCEDURE

A. Materials

A sodium borosilicate glass composed of 16% Na_2O, 14% B_2O_3, and 70% SiO_2 was chosen for use in this study. The glass was manufactured by mixing suitable proportions of sodium carbonate, boric acid, and silica and melting the mixture in a platinum crucible at 1350°C overnight. The glass was then milled to a particle size less than about 2 microns.

In order to introduce controlled porosity, nickel microspheres were vacuum hot-pressed into the glass matrix. Due to the much larger coefficient of thermal expansion of the nickel than that of the glass and due to a lack of bonding, the nickel spheres on cooling developed pores which would leave the spheres free even under the strain conditions

employed in the bending tests. However, some difficulty was encountered with interfacial bonding in the minus 10 micron range (see Results and Discussion).

The nickel spheres* were purchased in bulk form and were separated by sieves and by air elutriation. No oxide was visible on the nickel surface and none could be detected by x-ray analysis.

B. Specimen Fabrication

The porous glass specimens were prepared by mixing appropriate amounts of nickel spheres and glass and vacuum hot-pressing at 680°C and 2000 psi for 10 minutes in suitable graphite dies. The pressing temperature was about 60°C above the softening point of the glass. The hot pressing arrangement used is similar to that used by Rossi and Fulrath.[11] Figure 1 shows the microstructure of a typical specimen. Some of the spheres have pulled out during polishing.

The usual procedure for mixing the nickel spheres and powdered glass was to weigh out the appropriate proportions and then mix by tumbling in a container. However, with some specimens (see Results and Discussion) it was necessary to treat the mixed powders in isopropyl alcohol. This was done by weighing as usual and then mixing the powders in isopropyl alcohol and vacuum drying overnight. These treated powders were placed in hot dies (about 150°C) and the time of exposure to the atmosphere was minimized.

Uniaxial test specimens were made by pressing two-inch diameter by 0.075 inch thick disks. These disks were wet polished on 240, 400,

* Purchased from the Federal-Mogul Division, Federal-Mogul-Bower Bearings, Inc.

and 600 grit SiC paper to insure an adequate density of Griffith flaws and to produce a uniform surface. The disks were then cut with a high precision diamond saw into bars about 0.25 inch wide by 0.075 inch thick. The polished surface was used for the tensile surface in bend testing.

The biaxial specimens were one inch diameter disks 0.075 inch thick. The disks were polished in the same manner as the uniaxial specimens. It was found that test disks could not readily be sliced from a thick disk without excess surface damage, especially with the high nickel specimens. This difficulty seemed to be due to the ductility of the nickel.

C. Testing Procedure

Uniaxial strengths were determined by loading the 0.25 by 0.075 inch bars to failure on a four-point hydraulic loading device with an overall span of 0.75 inch. The specimens were loaded such that the ground surface was stressed. Time to failure was approximately 15 to 30 seconds. Approximately sixteen data points were obtained for each composition of pore size and volume fraction.

The modulus of rupture for the uniaxial specimens was calculated from the expression

$$\sigma = \frac{Mc}{I} \qquad (1)$$

where σ is the modulus of rupture (calculated outer fiber stress), M is the maximum bending moment, c is the distance from the surface to the neutral axis, and I is the moment of inertia of the cross section about the neutral axis. The dimensions of each specimen were measured individually at the point of failure.

Biaxial strengths were determined by loading one-inch diameter by 0.075 inch thick disks to failure. The disks were placed on a stiff rubber O-ring and then a hydraulic plunger with a 3/16 in. diameter ball tip was loaded against the center of the disk. Figure 2 shows a diagram of the breaking device. The time to failure was about 15 to 30 seconds.

The maximum tensile stress, hereafter referred to as the biaxial strength, was calculated from the expression[12]

$$\sigma = \frac{P}{h^2}\left[(1+\nu)(0.485 \log \frac{a}{h} + 0.52) + 0.48\right] \qquad (2)$$

where P is the load on the ram, h is the thickness, ν is Poissons ratio (0.20), and a is the radius of the supporting O-ring.

To observe the density of Griffith flaws on the surface of the strength specimens, the lithium ion-exchange process described by Ernsberger[4] was used. Several specimens were polished on successive grades of abrasive down to Linde A alumina and then were placed in contact with a eutectic mixture of $LiNO_3$-KNO_3 for 1-1/2 hours at 200°C. The specimens were washed in water, dried, and gold plated to increase the reflectivity of the surface.

The lithium ions in the melt exchange with sodium ions in the glass. This causes a tensile stress in the glass due to the smaller size of the lithium ions and subsequent decrease in thermal expansion effective on cooling from 200°C. Moistening the glass catalyzes the propagation of cracks, and these cracks are thought to originate at Griffith cracks or be representative of them.

III. RESULTS AND DISCUSSION

Figure 3 shows the experimental data for the uniaxial strength of glass specimens containing spherical porosity. Table I lists the experimental data along with the statistical data. The strength of the glass was found to be 11,930 psi. In general, the results agree very well with those predicted on the basis of Hasselman and Fulrath's hypothesis on the strength of brittle-matrix composites.[10]

The strengthening of the untreated glass by 5-10 micron nickel spheres was unexpected. It was found that treating the mixture in isopropyl alcohol prior to hot-pressing gave strengths that would be predicted on the basis of the data for other porosity sizes at given volume fractions of porosity. This same treatment was tried on zero porosity glass and on a 30 volume percent of 36-44 micron porosity sample. It was found that the strength of these samples was not significantly effected. In view of the work of Rossi and Fulrath[11] with a similar treatment on alumina, the effect of the isopropyl alcohol is thought to reduce the amount of absorbed water on the glass powder surface. It is known that water vapor will hasten the oxidation of nickel and that nickel oxide will form a good bond to the glass. It is suggested here that with the large surface area and the relatively small thermal contraction of the 5-10 micron spheres as compared to the larger sizes, the effect is for the spheres to bond to the glass and not separate on cooling. The result would be localized areas of glass in tangential compression and radial tension around the spheres. In this case the effect of the nickel microspheres is not to create porosity but to

dispersion strengthen the glass as was observed by Hasselman and Fulrath[13] with glass-alumina systems.

Figure 4 shows the experimental data for the biaxial strength of glass specimens containing spherical porosity. Table II lists the experimental data along with the statistical data. The biaxial strength of the glass was found to be 11,510 psi, in good agreement with the uniaxial value.

In general, the standard deviations of the biaxial data were higher than those for the corresponding uniaxial data. This is probably due to a larger surface being exposed to the maximum stress in the uniaxial bend test than in the biaxial test used. In the uniaxial case the maximum stress is present over a 1/4 by 1/4 inch area while in the biaxial case the maximum stress is present only at a point.

When stress concentrations are present under load, the relative sizes of the Griffith flaws and the volume of material over which stress concentrations act determine the strength of the material. The stress concentrations for conditions of uniaxial loading are:*[10]

$$\sigma_r = 2T\left[-\frac{a^2}{4r^2} + \left(\frac{3}{4}\frac{a^4}{r^4} - \frac{a^2}{r^2}\right)\cos 2\theta\right] + T\cos\theta \qquad (3)$$

$$\sigma_\theta = 2T\left[\frac{a^2}{4r^2} - \frac{3}{4}\frac{a^4}{r^4}\cos 2\theta\right] + T\sin\theta \qquad (4)$$

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* These equations are for stress concentrations around a circular hole in a flat plate and are used because of the relatively high stress gradients present in the test procedure.

Under biaxial loading, the stress concentrations are:

$$\sigma_r = 4T\left[-\frac{a^2}{4r^2}\right] + T \tag{5}$$

$$\sigma_\theta = 4T\left[\frac{a^2}{4r^2}\right] + T \tag{6}$$

where σ_r and σ_θ are the radial and tangential stress components, respectively, T is the applied stress, a is the pore radius, r is the distance from the center of the pore, and θ is the angle between the tensile direction and the line joining the center of the pore and the point in question.

Under conditions of tensile load (T positive), tensile stresses greater than the applied stresses occur in the tangential components as can be seen from equations 4 and 6. Therefore, the tangential components should be responsible for failure of the glass.

The maximum value of stress concentrations that occur in uniaxial and biaxial loading differ. The maximum value under uniaxial conditions is 3T and under biaxial conditions is 2T. The maximum occurs at two points only in the uniaxial case and only along the edge of the pore in the biaxial case. Figure 5 is a map of stress concentration contours. The area of concentration greater than 1.5 (a typical value of calculated stress concentration observed for failure) for the two loading conditions is not significantly different. However, there is an area of higher concentration than 2T in the uniaxial case. This can account for the lower observed strengths in the uniaxial tests compared to those in the biaxial tests. When the pore size is very much larger than the flaw

size, the uniaxial strength should be considerably lower than the biaxial strength due to the probability of flaws being present in areas of stress concentration greater than 2. Since the uniaxial strength zero porosity intercept for the largest pores gives a stress concentration factor less than 2, the data for larger pore sizes seems to correspond to Hasselman's Case II. That is, the flaws are not located entirely in regions of high stress concentation. The same situation applies to the largest pore size biaxial data.

Case III seems to be approached more rapidly in the biaxial case than the uniaxial case. The biaxial data for 20-36μ porosity seems to fall in this region although the same is not true for the uniaxial data. Case III seems to be approached with the smallest pores in the uniaxial samples.

The strengthening of the alcohol treated glass by 5-10 micron spheres was unexpected in the biaxial case in view of the uniaxial results with the same treatment. One possible explanation of this is that only a fraction of the spheres in this size range actually separate from the treated glass on cooling. Assuming that a large fraction do not, the bonded fraction would lead to dispersion strengthening as explained in the uniaxial case. Since there is a very large stress gradient from the center to edge of the disk in the biaxial test used, the probability of a pore-flaw combination being present at the point of maximum stress is low and failure will be nucleated from an area of lower than maximum stress. In the uniaxial case there is a sufficiently large area (1/4 by 1/4 inch) under maximum stress to nucleate a failure within that area.

The result is that the biaxial specimens break at a higher apparent stress than the uniaxial specimens. Another biaxial test that loads a larger area would be desirable and could be used to investigate this hypothesis.

Figure 6 shows the results of the lithium ion-exchange technique. There appears to be an adequate flaw density around pores as was assumed in this investigation. Some of the nickel spheres have pulled out of the glass during polishing as would be expected.

IV. SUMMARY

The effect of stress concentration on the strength of a glass was investigated. It was found that the strength was determined by the porosity size and volume fraction as well as the loading conditions.

The results were interpreted as a function of the relative sizes of Griffith flaws and the volume of material over which the stress concentrations act. The experimental results agree very well with Hasselman's hypothesis on the effect of porosity on strength of a brittle matrix.

It was found that with the pore size much greater than the flaw size the strength would be determined by stress concentrations around the pores. This is due to the probability of a flaw being present in an area of high stress concentration. As the pore size becomes much smaller than the flaw size, the effects of stress concentration are not significant and a smooth, monotonic decrease in strength with porosity occurs.

It is suggested that for very large pores, the uniaxial strength should be less than the biaxial strength due to higher stress concentrations in the uniaxial case.

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Table I. Uniaxial strength of sodium borosilicate glass containing dispersed spherical pores, in psi x 10^{-3}

[Glass strength = 11930 pxi (12.7,46)]*

Pore diameter range (microns)	Volume percent porosity					
	2%	5%	10%	20%	30%	40%
5-10	11.75 (13.5,17)	12.81 (13.4,13)	13.93 (12.2,18)	15.68 (8.9,13)	--	--
5-10**	10.98 (14.0,17)	11.11 (10.9,17)	11.14 (11.1,15)	11.49 (8.3,16)	--	--
10-20	11.25 (11.9,18)	10.39 (6.7,16)	9.58 (11.2,15)	9.75 (7.9,15)	8.71 (6.7,16)	--
20-36	10.47 (10.2,17)	9.83 (9.4,30)	8.52 (8.7,17)	8.14 (7.8,16)	7.29 (3.2,15)	6.97 (4.2,15)
36-44	8.51 (13.0,17)	7.27 (8.0,29)	6.23 (13.1,14)	6.02 (6.5,14)	5.03 (7.9,14)	4.80 (5.4,15)
74-105	7.50 (10.6,18)	6.74 (7.1,16)	6.01 (5.9,16)	4.96 (10.0,15)	4.35 (7.9,15)	4.21 (8.3,16)
105-186	7.37 (14.6,15)	6.18 (9.0,17)	5.62 (7.1,14)	4.63 (8.4,16)	4.06 (3.9,16)	3.68 (9.9,13)

* Numbers in parentheses represent standard deviation in percent of average and number of specimens, respectively.

** Alcohol treated.

Table II. Biaxial strength of sodium borosilicate glass containing dispersed spherical pores, in psi x 10^{-3}

[Glass strength = 11510 psi (18.4,11)]*

Pore diameter range (microns)	Volume percent porosity					
	2%	5%	10%	20%	30%	40%
5-10**	14.13 (8.0,4)	14.53 (13.8,3)	14.60 (13.0,3)	16.32 (19.8,4)	--	--
20-36	11.38 (14.4,4)	10.72 (20.8,4)	9.03 (10.2,4)	8.20 (10.9,4)	7.67 (3.2,4)	8.11 (7.4,4)
74-105	7.91 (9.2,4)	7.38 (8.9,4)	6.74 (11.3,4)	5.80 (9.8,3)	5.68 (12.8,4)	5.32 (5.6,4)

* Numbers in parentheses represent standard deviation in percent of average and number of specimens, respectively.

** Alcohol treated.

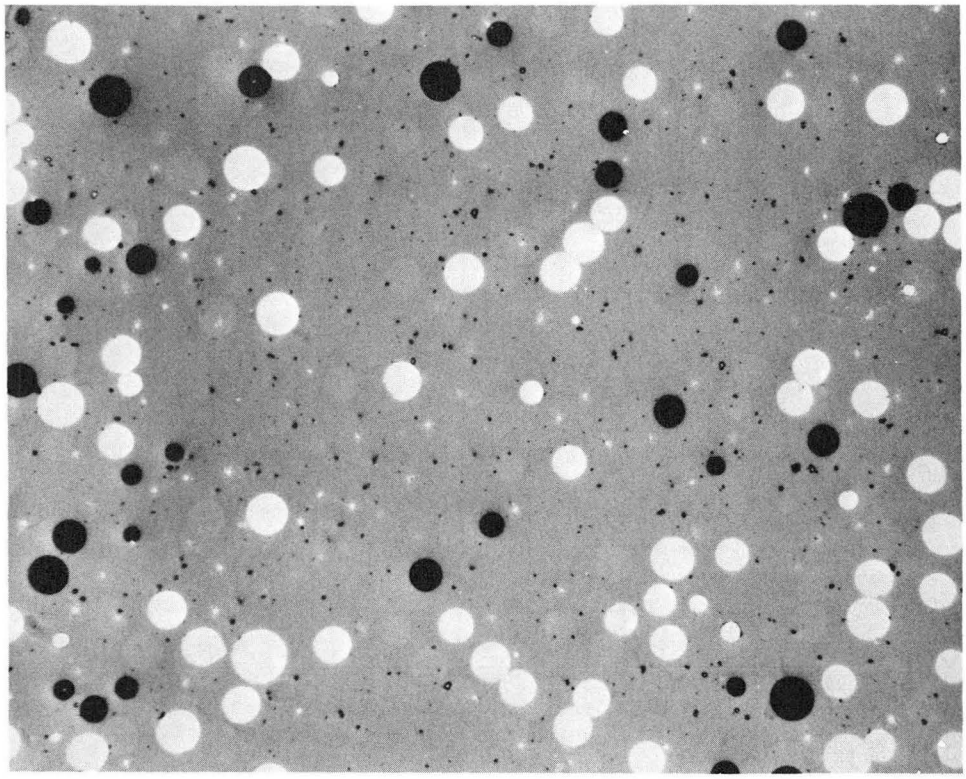

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Fig. 1 Microstructure of sodium borosilicate glass containing 10 volume percent spherical nickel particles, 36 to 44 microns in diameter.

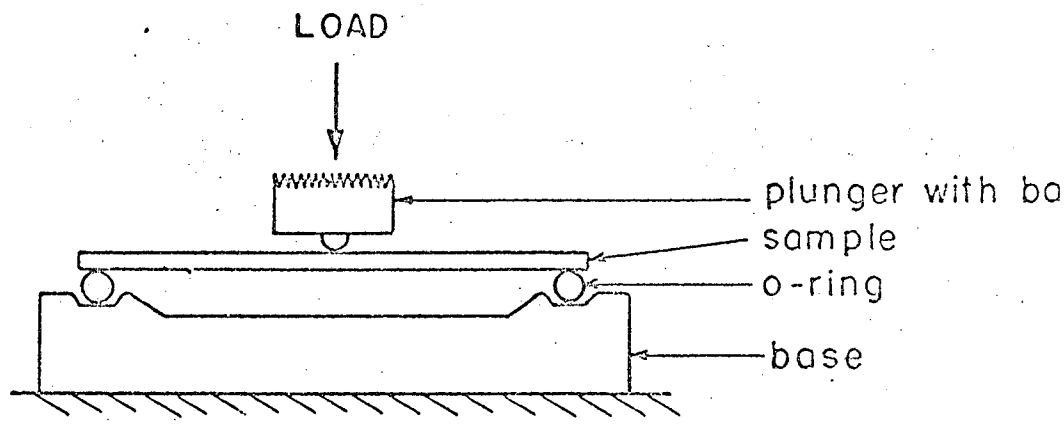

Fig. 2 Cross sectional view of biaxial stressing device.

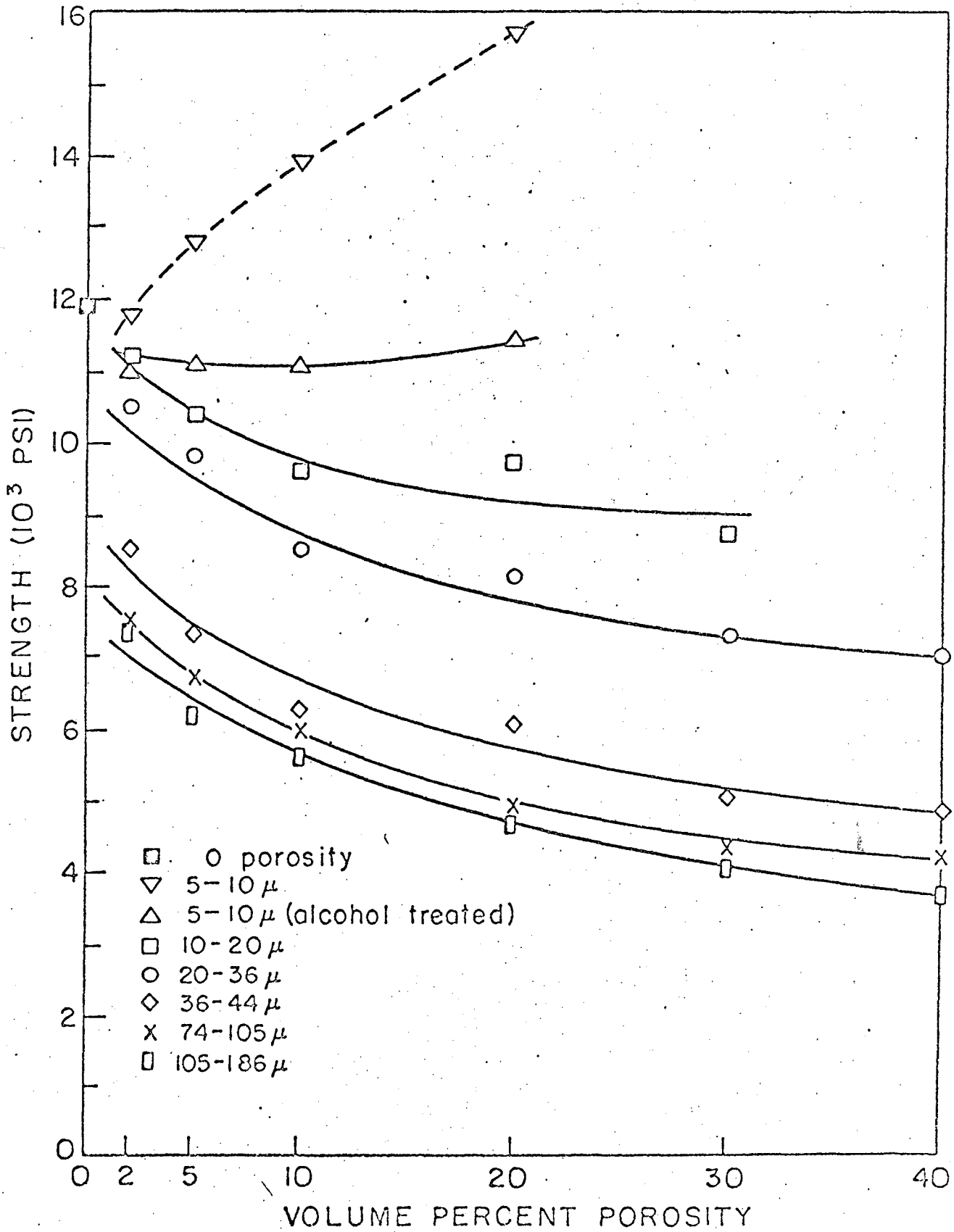

Fig. 3 Uniaxial tensile strength of sodium borosilicate glass containing spherical pores.

R- 78%
5'4"

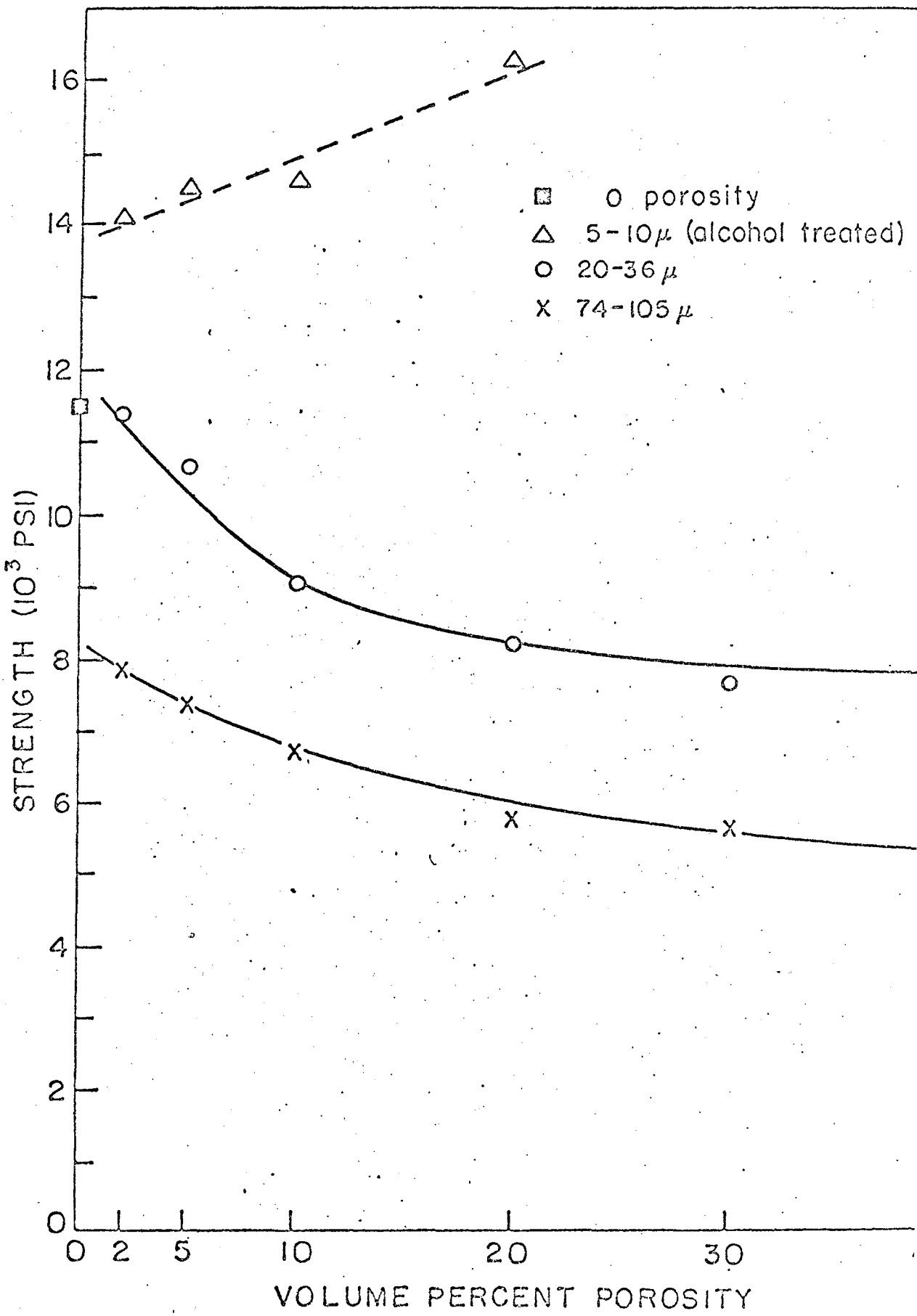

Fig. 4 Biaxial tensile strength of sodium borosilicate glass containing spherical pores.

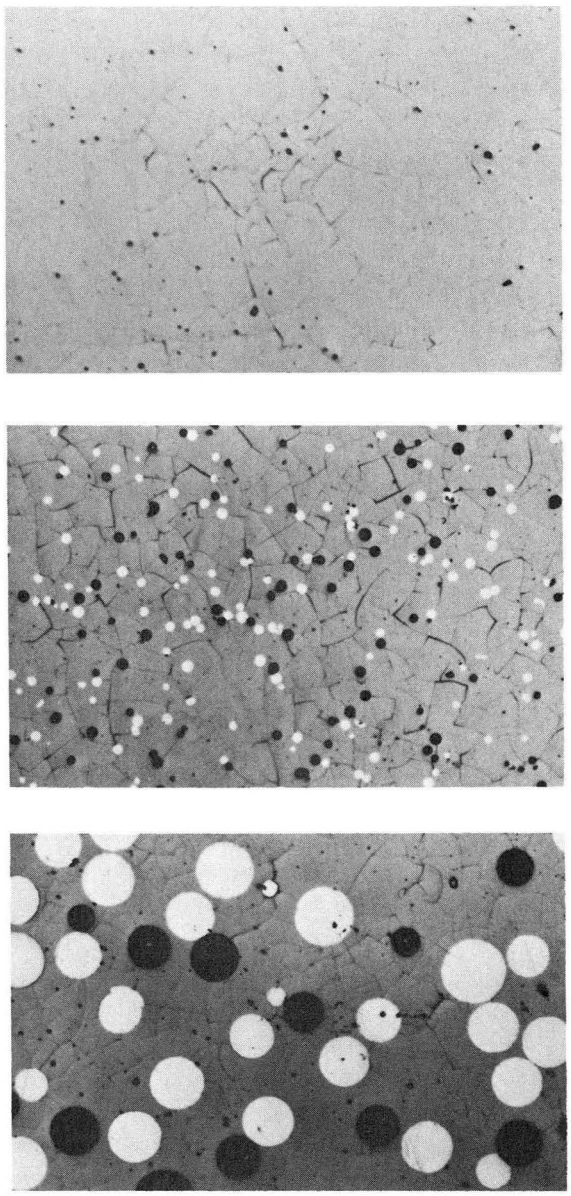

Fig. 6 Griffith flaws on surface of glass matrix containing (a) 0, (b) 5 vol. % 5-10 micron porosity and (c) 30 vol. % 36-44 micron porosity.

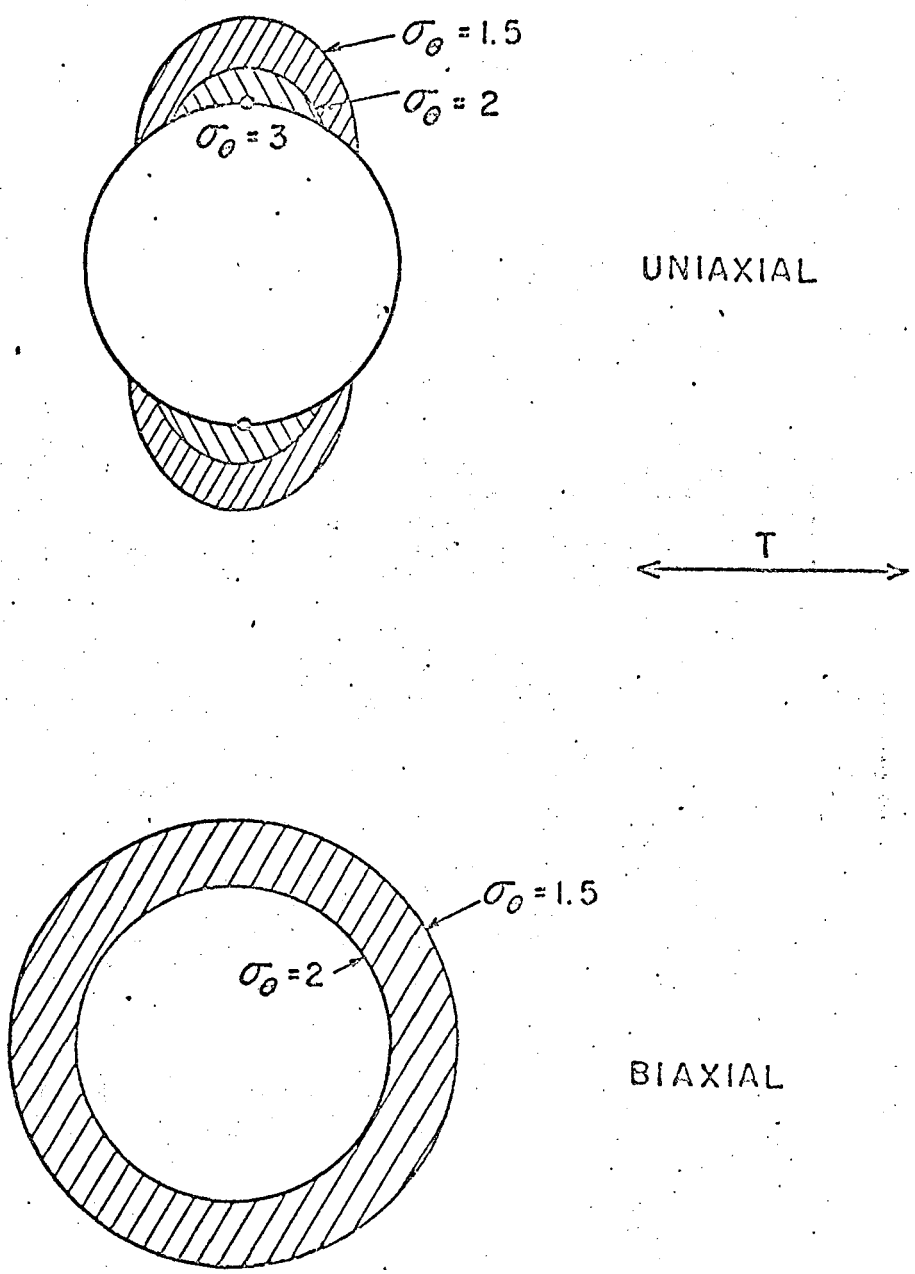

Fig. 5 Approximate tangential tensile stress concentration around a flat cylindrical pore under uniaxial biaxial loading.

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