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# INFLUENCE OF SURFACE DAMAGE ON THE STRENGTH OF A BRITTLE-MATRIX COMPOSITE

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Subhash Risbud (M.S. Thesis)

September 1971



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#### INFLUENCE OF SURFACE DAMAGE ON THE STRENGTH OF A BRITTLE-MATRIX COMPOSITE

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#### INFLUENCE OF SURFACE DAMAGE ON THE STRENGTH OF A BRITTLE-MATRIX COMPOSITE

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#### ABSTRACT

The strength of a model two-phase glass-alumina system was investigated relative to the extent of surface damage. The composite system was fabricated by the vacuum hot pressing of finely powdered mixtures of glass and alumina. Strength specimens cut from hot-pressed compacts were subjected to varying degrees of surface damage by controlled and reproducible surface treatments.

Uniaxial strengths were found to be independent of the grit size of diamond saw blades, decreased significantly with surface abrasion and were found to improve at a critical annealing temperature after abrasion. The effects of surface condition on strength are discussed in relation to the microcracks and edge chips generated during sawing or abrasion.

#### I. INTRODUCTION

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The interest in studying properties of two-phase systems has been inspired by the promising improvements in thermal and mechanical behavior of practical composite materials. A variety of composite systems have been developed on the basis of experimental work. Fiber-reinforced metals,<sup>1,2</sup> dispersion strengthened metals and alloys,<sup>3</sup> resin-matrix composites. 4 and plastics. 5,6 are classical examples. Plastics reinforced with glass filaments are reported to be three times stronger than steel on a comparable density basis. From the ceramic point of view. composite systems of interest are the cermets, glass-ceramics, and dispersion strengthened glasses.<sup>10,11</sup> Ceramics possess good oxidation resistance, high modulus of elasticity and strength up to fairly high temperatures, low density, and chemical stability. As such, they would comply with the major requirements for a composite, but for their brittleness. An ideal composite system would be one which retains the desirable properties of the ceramic and at the same time provides a means of arresting crack propagation through the brittle matrix.

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Extensive work in this laboratory has resulted in the identification of several parameters that affect the mechanical properties of a model brittle matrix composite system. Among the variables recognized so far are: porosity, internal stresses, interfacial bonding, and differences in the elastic properties of the constituents. It is known that the damage introduced by abrasive operations can affect the properties of a ceramic material.<sup>12,13</sup> The extent and nature of damage introduced varies from material to material. Abrasion of completely brittle materials like germanium, silicon, and sapphire generally leads to generation of greater non-crystallographic cracks than sub-surface defects.<sup>14,15</sup> Semi-brittle materials, on the other hand, show a great deal of sub-surface damage, like broad slip bands, in addition to some cracking.<sup>16</sup>

This study describes the results of an investigation of surface damage resulting from various carefully controlled surface treatments on a glass-alumina composite. Sawing and surface abrasion were chosen as treatments constituting the major source of flaws.

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II. THEORETICAL BACKGROUND AND LITERATURE REVIEW

The useful life of brittle materials is often limited by their susceptibility to catastrophic failure under tensile loads. One of the major consequences of usually unavoidable operations like grinding, cutting or mechanical handling, is the generation of microscopic flaws in the material. The classical Griffith crack analysis<sup>17</sup> has become a central concept in rationalizing the low tensile strength of brittle materials and in developing statistical theories of strength. The Griffith crack concept is, broadly speaking, still basic to the theory of brittle fracture. Recent research<sup>18</sup> on the strength of glass, however, has attempted to characterize the Griffith crack. Such work has been supplemented by the growing body of information on pristine glass surfaces.

The properties of brittle-matrix composites have received much less attention than ductile matrix systems. The available results, however, appear to present a reasonable and consistent pattern. Since this work is concerned with a brittle-matrix system, no attempt has been made to refer to the voluminous literature on the subject of ductile-matrix composites. The reader is referred to the reviews by Kelley and Davies,<sup>19</sup> Cratchely<sup>20</sup> and more recently by Broutman and Krock.<sup>21</sup>

Analysis of composite systems based on models, although successful for thermal expansion and conductivity studies, has been less successful in the prediction of mechanical strength.<sup>22</sup> The fundamental requirement for two dissimilar materials to act in concert is the development and maintenance of a strong interfacial bond. In glass-metal systems thermodynamic equilibrium at the interface is an essential condition for the formation of a strong bond.<sup>23</sup> In analyzing brittle matrix-composities, both internal stresses and localized stress concentrations must be considered. Fulrath<sup>24</sup> demonstrated the existence of internal stresses in a model glass-alumina system, and concluded that strength decreased with increasing internal stresses for a constant particle size of the dispersed phase. Grossman<sup>25</sup> made a similar study on glass-thoria composites. A recent study<sup>26</sup> has, however, concluded that internal stresses do not significantly affect the strength of well bonded brittlematrix composites.

Extensive studies have been made at this laboratory correlating strength in a model composite system to several parameters which include interfacial bonding,<sup>27</sup> micromechanical stress concentration,<sup>28</sup> porosity<sup>29</sup> and water concentration in the glass.<sup>30</sup> In general, it has been observed that a good bond between phases and small size of dispersed phase particles contribute to strength. Hasselman and Fulrath<sup>31</sup> developed a hypothesis for strengthening a glass-alumina composite, based on the rationale that introduction of a hard crystalline dispersed phase in the glass matrix limits the size of Griffith flaws which can be produced in the surface. Lange,<sup>32,33</sup> has suggested that the interaction of a crack front with the dispersed phase in a brittlematrix composite leads to increased fracture energy and may be expected to lead to higher strength.

To analyze the Hasselman-Fulrath postulate quantitatively, the mean free path between dispersed phase particles can be expressed  $^{34}$  as

 $d = \frac{\mu}{3} R \left( \frac{1}{V_{f}} - 1 \right)$  (1)

where R = particle radius

 $V_{p}$  = volume fraction of dispersion

d = mean free path.

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Since the hypothesis assumes the most severe flaw in the glass matrix to be proportional to the mean free path, the classical Griffith<sup>17</sup> equation for fracture strength

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$$=\frac{K}{(\pi a)^{1/2}}$$

can be written as

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$$= K' (R)^{-1/2} \left(\frac{1}{V_{f}} - 1\right)^{-1/2}$$
(3)

(2)

where a = flaw size and K = constant depending upon the elastic modulus E and the surface energy  $\gamma$  and K' = constant.

For comparison, Broutman and Krock<sup>21</sup> evaluated the constants in Eq. (3) above by fitting the strength versus volume fraction data reported by Nason and Fulrath<sup>35</sup> for a glass-tungsten composite. The trend of the curve given by Eq. (3) was reasonable. However, it is obvious that Eq. (3) implies cracks of infinite length when  $V_f = 0$ . Hence good fit to data is not obtained as  $V_f$  tends to zero. Two empirical approaches have been suggested to correct Eq. (3). First is the introduction of a finite crack size for  $V_f = 0$  by adding a correction factor  $V_o$  to  $V_f$  and secondly introducing a lower limiting strength  $\sigma_o$  into Eq. (3). The latter correction overestimates strength data. It appears that these corrections give a reasonable fit to the data. The hypothesis that spacing between particles limits the most severe flaw therefore appears quite sound and further data fitting serves little purpose. Several experimental data on brittle matrix composites have been published from time to time. Binns<sup>36</sup> studied the effect of alumina and

zirconia dispersions on the elastic moduli of various glasses and

reported that the results were independent of particle size. Davidge and Green<sup>37</sup> reported the strength of various glasses containing thoria spheres of different sizes. They reported that both elastic constants and thermal expansion differences led to lowered strengths for thoria spheres greater in size than a critical diameter. Hamijian and Lidman<sup>38</sup> reported rupture strength of up to 60,000 psi for a zirconium carbidematrix composite with niobium metal dispersions. Walton and Poulos<sup>39</sup> studied reinforcement of silica and alumina matrices by wires of a Ni-Cr steel alloy. Tummala and Friedberg<sup>40</sup> studied glass-zirconia compacts and have reported increased mechanical strength for acid treated zirconia dispersions in a glass-matrix. Rossi<sup>41</sup> has reported thermal expansion data on BeO-SiC composites at 1000°C. Based on thermal expansion measurements, the SiC matrix appears to be crack free.

It is apparent from the above review that efforts in brittlematrix systems have been directed mainly at obtaining fundamental information rather than immediate application. Applications have, however, been suggested for ceramic fiber composites in rocket nozzle inserts, aircraft and missile radomes, printed circuits and antenna windows.<sup>42</sup> While it is doubtful that any one or even a few ceramic material composites will provide the solution to the thermal problems of extreme environments, it may still be possible to utilize the best properties of ceramics, without being limited by their inherent weakness.

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#### A. <u>Preparation of Powders</u>

The glass and alumina powders used in this study were chosen because of their nearly identical thermal expansion coefficients. This resulted in composites with minimum internal stresses.

The glass used was prepared by air-melting the component oxides in a platinum crucible at approximately 1350°C. One 1500 gram glass batch required 375 g of boric acid, 411 g of sodium carbonate and 1050 gm of silica flour. Melted and refined batches were cast into graphite molds, and reduced to -325 mesh by crushing and dry milling in an alumina lined ball mill with flint pebbles.

The alumina used in this study was similarly reduced to -325 mesh by dry ball milling agglomerated A-14 alumina. The size of agglomerates was about 30 microns. Particle size measurements "made on the D-glass and alumina powders prepared by the above procedure, yielded values of an average particle size of 3-3.5 microns for alumina and 3-4 microns for the glass. The alumina particles had a platy morphology. Composite powders were prepared by mixing the proper volume fractions of glass and alumina powders in a rotating plastic bottle containing several alumina balls for 24 hours or more.

#### B. Fabrication of Compacts

Specimen compositions were determined by assuming theoretical density in a compact 2 in. in dia.  $x \ 1/4$  in. thick. The appropriate weight of powders was loaded into graphite dies, and cold compacted to 1000 psi before loading into the hot press. Hot pressing was carried out under a vacuum of approximately  $10^{-4}$  torr. Controlled and uniform

#### \*Alcoa product.

\*\*Fisher sub-sieve sizer, Fisher Scientific Co., St. Louis, Mo.

fabrication schedules were used for all compacts. The materials were heated at a rate of approximately 5°C/min to 500°C and a 1000 psi load was applied at this temperature. This load was sustained until the temperature reached 700°C. Compacts were soaked at this temperature for 10 minutes and allowed to cool under vacuum for several hours. No postfabrication annealing treatment was considered necessary in view of the very slow rate of cooling.

C. <u>Density Measurements and Microstructural Evaluation</u> Bulk densities of compacts were measured by taking the saturated weight, suspended weight and dry weight of samples. The suspended weight was taken by immersing the sample in water. Densities of approximately 98% ± 0.5 were achieved in most compacts.

Microstructures of the load-bearing surfaces were prepared for examination by progressive polishing on diamond wheels of 220 mesh, 30 microns and 15 microns. Final finishing was achieved by polishing the samples on No. 3 and No. 1 diamond on Syntron polishing machines for 30 hours or more. The sample surfaces were gold-plated to aid examination. Mean free paths were calculated from the microstructures examined by techniques similar to those outlined by Underwood et. al.<sup>43</sup> and used by Nivas and Fulrath.<sup>44</sup>

#### D. Sawing and Surface Treatments

The compacted slugs were cemented to high aluminum porcelain ceramic plates and cut into strength specimens approximately 0.06 to 0.08 in. in thickness, with a high precision diamond saw using kerosene as a coolant. The diamond blade was 0.05 in. thick and 6 in. in outside diameter with a speed of approximately 3500 surface ft/min.

The saw was advanced at a rate of 0.25 in./min. through the compact and

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through a small part of the underlying ceramic in order to avoid chipping of specimen edges. Specimens were removed from the plate by dissolving away the adhesive cement with methyl alcohol and were then cleaned in an ultrasonic cleaner.

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The specimens were divided into three batches for testing under the following surface conditions:

(1) Specimens sawn with a 80 grit diamond saw blade at a constant feed rate and saw speed.

(2) As-sawn specimens uniformly abraded by hand grinding in an identical manner using 240 grit SiC and kerosene lubricant on a glass plate.

(3) As-sawn and abraded specimens, subjected to annealing at different temperatures and times.

#### E. Specimen Testing

Specimens were tested on a 4-point loading device with an overall span of 3/4 in. The abraded surfaces were subjected to tensile stresses during the test. Time to failure was approximately 80 secs. for composites and 40 secs. for glass. From the breaking load the modulus of rupture was calculated by the flexure formula.

 $\sigma = \frac{MC}{I}$  where  $\sigma = modulus$  of rupture.

M = maximum bending moment

C = distance from surface of sample to the neutral axis
I = moment of inertia of the cross section about the neutral axis.

For the rectangular beam used in the above tests, this formula

reduced to

 $\sigma = \frac{PL}{bd^2}$  where b = width at the point of failure

d = thickness at point of failure

P = load to failure

L = span.

The average of several specimens was reported in each test. The as-sawn, abraded, annealed and fractured surfaces were examined by optical and scanning electron microscopy. A thin layer ( $\sim 150 \text{\AA}$ ) of Pt-Pd was deposited on surfaces before examination.

#### IV. RESULTS AND DISCUSSION

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The properties of materials used in this investigation are summarized in Table I. In the design of materials of the type used in this study, it has been suggested<sup>37</sup> that particle and matrix expansion coefficients be matched and that particle size be as small as possible. In keeping with these observations, the glass and alumina used in this study had nearly identical coefficients of thermal expansion. The average particle size of the platy alumina dispersions was approximately 3.5 microns.

About 98% ± 0.5% of the theoretical density was achieved in hotpressed compacts. A representative microstructure of the composite showing platy alumina dispersions in a continuous glass matrix is illustrated in Fig. 1. From repeated microstructural examination of such areas, it was concluded that the mixing and pressing techniques used were adequate to yield a composite system of randomly dispersed alumina particles in the glass matrix. Calculations of the mean free path between alumina dispersions gave values of approximately 3 to 3.5 microns in all composites. These mean free path values clearly lie in the region where Hasselman and Fulrath<sup>31</sup> postulated strengthening effects in glassalumina systems. The 50 volume fraction alumina-glass composite corresponds to a minimum flaw size of approximately 15 microns, based on calculations using Griffith's equation and observed strengths of composites. This difference is not surprising in view of the non-uniform distribution of flaw sizes obtained by severe abrasion of the specimen surfaces, and further illustrates the flaw-limitation postulate.

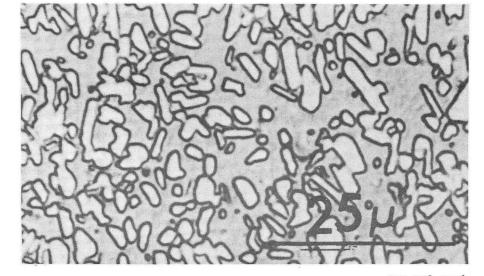
In order to invoke statistical reliability, strength data were obtained for a series of glasses and composites fabricated in an identical manner, but subjected to different degrees of surface damage.

#### Table I. Properties of materials

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Material	Composition	Thermal Expansion Coefficient (Room temp. to 450°C)	Density g/cc	Elastic Modulus psi	· · · · · · · · · · · · · · · · · · ·
Glass	70 wt% SiO2	7.8 x 10 <sup>-6</sup>	2.48	10 x 10 <sup>6</sup>	₹.
	14 wt% B2O3	in/in per <sup>o</sup> C			
	16 wt% Na <sub>2</sub> 0				
Alumina	A-14 <sup>*</sup> ;	8 x 10 <sup>-6</sup>	3.98	60 x 10 <sup>6</sup>	
	mainly $\alpha$ -Al <sub>2</sub> O <sub>3</sub>	in/in per <sup>o</sup> C	· · · · ·		•
Glass- Alumina Composite	50 vol% Al2O3 50 vol% glass	-	3.19 ± 1%	24.4 x 10 <sup>6</sup> **	
* Product	of ALCOA.				
		45	and the second		

\*\*Data reported by Hasselman and Fulrath. 42

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Fig. 1 Representative microstructure of composite showing platy alumina particles in the continuous glass martix.

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It was anticipated at the beginning of this study that diamond sawing with different grit sizes would show different strengths for glass but no differences in the strength of composities; the rationale being that the different grits would introduce different sizes of microcracks in the glass, while the microcracks in the composite would always be limited by the mean free path between the alumina particles.

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Previous work<sup>30</sup> in this laboratory has shown that the standardized strengths of severely abraded composites decrease even through the maximum flaw size, dictated by interparticle spacing, had been achieved. This suggests another strength controlling variable besides interparticle spacing. It was speculated that while flaws were limited by the alumina particles at the surface, the depth of the flaws may vary depending on the extent of surface damage. Thus, a deeper flaw may bypass alumina dispersions beneath the surface, while being limited to the interparticle spacing at the surface. To test this hypothesis, composite slugs were sawn with diamond blades of different grit sizes ranging from a coarse 80 grit to a fine 270 grit. Strength specimens were cut from one half of a compact using the 80 grit diamond saw blade. Similar specimens were then cut from the other half of the same compact using the 270 grit diamond saw blade.

The cross bending strengths and associated statistical data for these experiments are summarized in Table II. As is clear, there is no correlation between the strength and grit size of the diamond saw blade for either the glass or composites. A possible explanation of these results is that regardless of the grit size of the diamond blade, a fairly uniform sawn surface is obtained due to the fairly high surface feet per minute of the blade and the comparatively low feed rate used in

-13-

Compact Grit No. 1 80 2 80 2 80 3 120	Bo Bo Bo Bo Bo PTO PTO PTO	Com <sup>d</sup> <sub>B</sub> v, Psi 19,200 20,000 20,000 23,853 23,853 22,575 22,575 21,604	Composites Std. Dev. (% of 0 <sub>av</sub> ) 6 8.2 11.3 10.5	No. of Samples 8 12 12 12 9	Compact No.	<sup>α</sup> <sub>av</sub> ,psi 10,200 9,090 9,434 8,973	Glass Std. Dev. (% of σ <sub>av</sub> ) 11 14.5 7.8 9.8	No. of Samples 7 11 10 10
170	0	20,563	15	01		ı		I.

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this study.

Following the above findings, attempts were made to introduce deliberate surface abrasion by unformly abrading as-swan surfaces with SiC grit. Trial tests made on a small sample basis indicated that 120 grit, 240 grit, and 600 grit SiC resulted in reducing the strengths of composites by approximately the same amount. Abrasion by 240 grit SiC was chosen as the standard treatment given to all as-sawn surfaces. The results show that there is a significant decrease in strength in both glass and composites upon abrasion. The abraded samples were found to improve in strength upon annealing at a critical temperature. Composite samples showed a maximum recovery in strength from the abraded value, when annealed at 700°C for 1 hour. Glass samples recovered best when annealed at 680°C for 1 hour. The cross-bending strengths and associated statistical data for the glass and composities as a function of surface conditions is summarized in Table III and IV.

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-16-

Each of the as-sawn, as-sawn and abraded, and as-sawn, abraded and annealed surfaces were examined by optical and scanning electron microscopy techniques. Severe edge chipping was observed in the as-sawn and abraded samples of both the glass and composites. Figures 2 and 3 illustrate the effect for the glass and the composite. In each case microphotographs at a relatively low magnification were taken by placing the edges of the as-sawn and as-sawn and abraded specimens adjacent to each other. Edge chipping is clearly seen in the as-sawn and abraded specimen while the edge of the as-sawn specimen is chip-free. The lowered strengths observed in as-sawn and abraded samples is postulated to be due to these gross edge cracks. From these results, the depth of flaws beneath the surface does not appear to control strength. Edge chipping is thus the likely rationale for lowered strengths in severely

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Compact No.	Surface Condition	Average Strength, σ <sub>av</sub> , psi	Std. Deviation $(in \% of \sigma)$	No. of Samples	Measured Mean Free Path
	10	20,950	7.8	7	
1.	AS	14,035	15.5	8	3.50
	ASA ASAA	17,160	4.8	8 8	microns
2.	AS	20,481	7	9	
	ASA	14,227	8.3	12	3.04
	ASAA	17,964	11.5	11	microns
3.	AS	28,610	8.7	9	
	ASA	21,435	9.9	10	3.20
	ASAA	26,932	7.4	9	microns
4.	AS	18,022	12.3	9	
	ASA	13,800	4.5	10	3.00
	ASAA	17,698	7.5	9	microns
	ASA = As se	awn (80 grit diamond saw R awn and Abraded (240 grit awn, Abraded, and Annealed	SiC)		

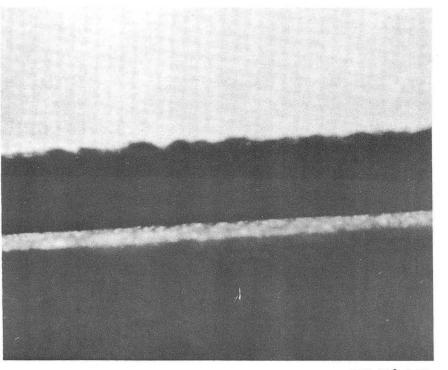
Table III. Crossbending strengths and statistical data for composites

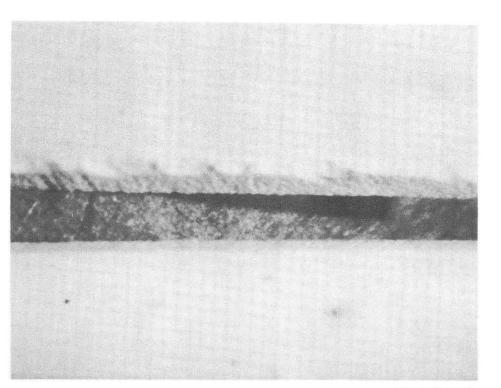
Table IV.	Crossbending	strengths	and	statistical	data	for	glass	
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			M	a state Charles and
Compact No.	Surface Condition	Average Strength, σ <sub>av</sub> , psi	Std. Deviation (in % of σ <sub>av</sub> )	No. of Samples
1.	AS	8,423	13.5	11
	ASA	6,470	16.2	10
	ASAA	8,838	12.3	8
2.	AS	8,910	11.5	9
an e e	ASA	7,465	18	10
	ASAA	8,526	16.5	10
3.	AS	10,814	8.8	8
1. S.	ASA	8,297	13	7
	ASAA	9,903	8	6
+.	AS	8,039	8.2	8
	ASA	5,113	15	8
	ASAA	10,537	9.7	8
	ASA = As saw	n (80 grit diamond saw blade) n and abraded (240 grit SiC) n, abraded, and annealed (680		

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-17-





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Fig. 2 Photomicrograph showing edge chipping in an as-sawn and abraded sample (above) compared to chip free as sawn sample (below) Magnification: 20x XBB 718-3778

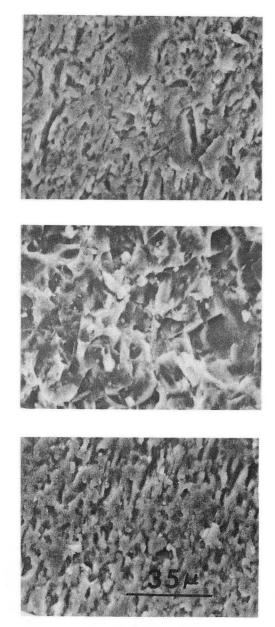
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Fig. 3 Photomicrograph showing edge chipping in as sawn and abraded sample (above) compared to chip free as sawn sample (below) Magnification: 20X

abraded composites, where maximum flaw sizes, dictated by interparticle spacing, have been achieved.

Annealing treatments on the as-sawn and abraded surfaces gave further validity to the edge chipping hypothesis. Strengths of as-sawn and abraded specimens recovered upon annealing at a critical temperature but were not higher than the as-sawn strengths. Thus, annealing treatments appear to round off the gross edge cracks generated during abrasion and seem to have little healing effect on the microcracks generated during sawing.

Scanning electron micrographs of the different surfaces obtained by different surface treatments are shown in Figs. 4 and 5. As-sawn and abraded surfaces clearly appear rough compared to as-sawn surfaces. Annealing of the as-sawn and abraded surface seems to result in a smoother surface. Typical fracture surfaces of the glass and the composites are illustrated in Figs. 6 and 7. The fracture path in composites can be seen to travel around the dispersed phase particles.

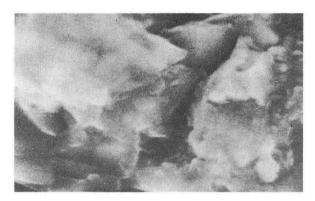


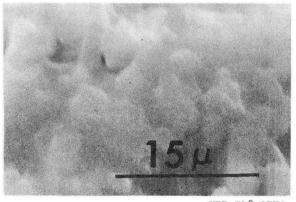
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Fig. 4 Scanning electron micrographs of glass surfaces subject to various degrees of Surface damage: a) As-sawn surface b) As-sawn and abraded surface c) Assawn, abraded and annealed surface.

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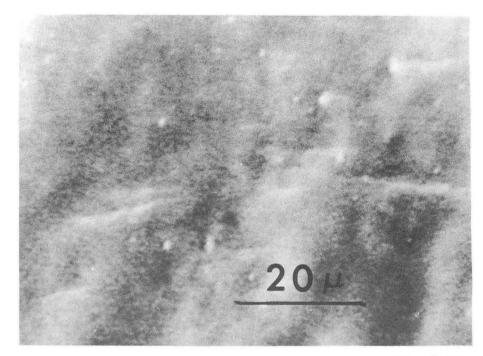






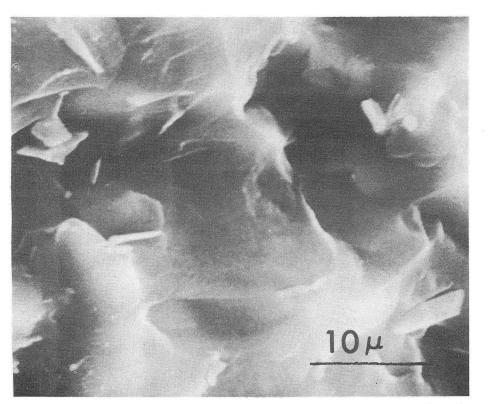
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Fig. 5 Scanning electron micrographs of composite surfaces subjected to various degrees of surface damage: a) As-sawn surface b) Assawn abraded surface c) As-sawn, abraded and annealed surface.



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Fig. 6 Scanning electron micrograph of typical fracture surface of glass.



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Fig. 7 Scanning electron micrograph of typical fracture surface of a composite.

#### V. CONCLUSIONS

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The uniaxial strength of a glass-alumina composite system has been shown to be independent of the grit size of the diamond saw blade used in sawing. Strengths of as-sawn specimens of glass and composites decrease significantly upon surface abrasion, and recover considerably upon annealing the abraded samples at a critical temperature. Lowered strengths of as-sawn and abraded specimens have been postulated to be due to severe edge chipping and annealing is suggested as a means of rounding off these gross edge cracks. Annealing treatments do not result in raising the strength of as-sawn and abraded samples to the assawn level.

#### ACKNOWLEDGEMENTS

The patient counsel, encouragement, and cooperation extended by Professor R. M. Fulrath is respectfully acknowledged. I wish to thank Neal Houlding, Don Whitaker, and George Georgakopoulos for technical assistance.

Gratitude and appreciation is expressed to my parents, Mr. and Mrs. H. N. Risbud of Poona, India, for making my graduate study possible.

This work was performed under the auspices of the United States Atomic Energy Commission.

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