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IN BRITTLE MATRIX COMPOSITE

Mark A. Stett and R. M. Fulrath

July 1968

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Bertolotti and Fulrath[1] experimentally studied the effect of stress concentrations on the strength of a porous glass by fabricating composites of nickel microspheres in D glass (16% Na_2O, 1 % B_2O_3, 70% SiO_2). The thermal expansion coefficient of the glass was less than that of the nickel. Upon cooling the nickel shrank away from the glass forming spherical pores which weakened the glass. Samples that were fabricated using small (5-10 μm diameter) spheres, however, showed an anomalous strengthening of the glass. It was proposed that adsorbed water on the surface of the glass powder used in fabricating the composite caused oxidation of the nickel surface and resulted in a bond between the oxidized nickel microspheres and the glass.

According to Pask and Fulrath,[2] a chemical bond can occur between a metal and glass when a balance of bond energies is achieved across the transition zone at the interface between the metal and the glass. This balance occurs when thermodynamic equilibrium is obtained at the interface. By thermodynamic equilibrium it is meant that each of the phases is saturated with the other and that there is no possibility of further reaction to form a new phase. In order to determine if an oxide is needed to develop the optimum chemical bond, composites of 20 vol. % oxidized nickel microspheres and 80 vol. % D glass were vacuum hot

pressed into samples at 700°C and 2000 psi for 10 minutes, free cooled to 600°C and then cooled for the remainder of the time under one atmosphere of argon to prevent the 0.050 inch thick samples from cracking. Nickel microspheres 44-63 μm in diameter were oxidized for various lengths of time in air prior to sample fabrication. Lengths of time required for various percentages of weight gain (and therefore various initial oxide thicknesses) were determined from weight gain measurements at 750°C and 800°C.

The hot-pressed sample discs were ground, introducing a large concentration of surface flaws, and cut into bars 0.050" x 0.180" in cross-section. These bars were then broken in bending on a four-point loading device with a 3/4" span. Strengths that were measured are given in the table. The variation of strength with percentage of weight gain during oxidation is shown in Fig.1. It can be clearly seen that an optimum chemical bond is developed and that the magnitude of strengthening (about a 50% increase over D glass alone) is similar to that observed by Bertolotti and Fulrath. Further investigation of the effect of such a bond and the mechanism of strengthening that is involved is being undertaken.

The path of fracture in a brittle material is altered by the inclusion of particles of a second phase or pores. It is further altered when a chemical bond exists between the brittle material and the inclusion. If the inclusion is a pore, the fracture will propagate to the pore and around its diameter, leaving a hemispherical cavity in the fracture surface. When there is a bond between a metallic inclusion and the glass the fracture will propagate through the glass around the sphere, but

Crossbending strength and statistical data for oxidized nickel-D glass composites

Oxidation Temp.(°C)	Oxidation Time (hrs)	Weight gain (%)	Average strength (psi)	Number of samples	Standard deviation (% of average)
800	1/4	0.3	10727	17	8.4
"	1/2	0.7	11958	17	6.2
"	1	1.1	12018	18	9.5
"	2	1.7	10872	15	12.6
"	3	2.2	10910	9	11.7
"	4	2.8	10033	17	13.4
"	6	3.3	10348	19	10.6
"	12-1/4	5.8	10260	12	12.2
750	1/6	0.18	9557	18	13.7
"	1/2	0.3	10190	19	12.1
"	1-1/2	0.9	11743	21	7.0
"	2	1.1	11445	22	7.3
"	4	1.7	10117	21	7.3
"	10	2.8	11185	23	7.7
"	15	3.6	10392	18	5.4
D glass alone			7742	36	13.5
D glass & unoxidized nickel			6435	32	8.1

still within the glass. Fracture surfaces of composites of nickel-D glass (non bonded) and oxidized nickel-D glass (bonded) were examined using a scanning electron microscope and confirmation of this fracture behavior can be seen in Figs. 2 and 3.

In the case of the non-bonded nickel (pore)-D glass system the fracture will propagate directly to and around the sphere due to stress concentrations around a spherical cavity. During the hot pressing of the oxidized nickel-D glass composite, an interfacial bond is formed by the migration of nickel oxide into the glass until the glass is saturated with nickel oxide near the sphere. Because of the thermal expansion difference between the nickel and the matrix glass, a radial tensile stress is developed. In order to attempt to relieve this tension, a fracture will propagate around the sphere at a finite distance in the glass phase. A more quantitative investigation of this mechanism is being undertaken, but the proposed hypothesis of Bertolotti and Fulrath is confirmed by the behavior observed in Figs. 2 and 3.

REFERENCES

1. Bertolotti, Raymond L. and Fulrath, Richard M., Effect of micromechanical stress concentrations on strength of porous glass, J. Am. Ceram. Soc., 50(11), 558-562 (1967).

2. Pask, Joseph A. and Fulrath, Richard M., Fundamentals of Glass-to-Metal bonding: VIII Nature of Wetting and Adherence, J. Am. Ceram. Soc., 45(12) 592-596 (1962).

ACKNOWLEDGMENT

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FIGURE CAPTIONS

Fig. 1. Composite strength as a function of weight gain during oxidation.

Fig. 2. Scanning electron microscope photograph of fracture surface of nickel-D glass (non-bonded) composite.

Fig. 3. Scanning electron microscope photograph of fracture surface of oxidized nickel-D glass (bonded) composite.

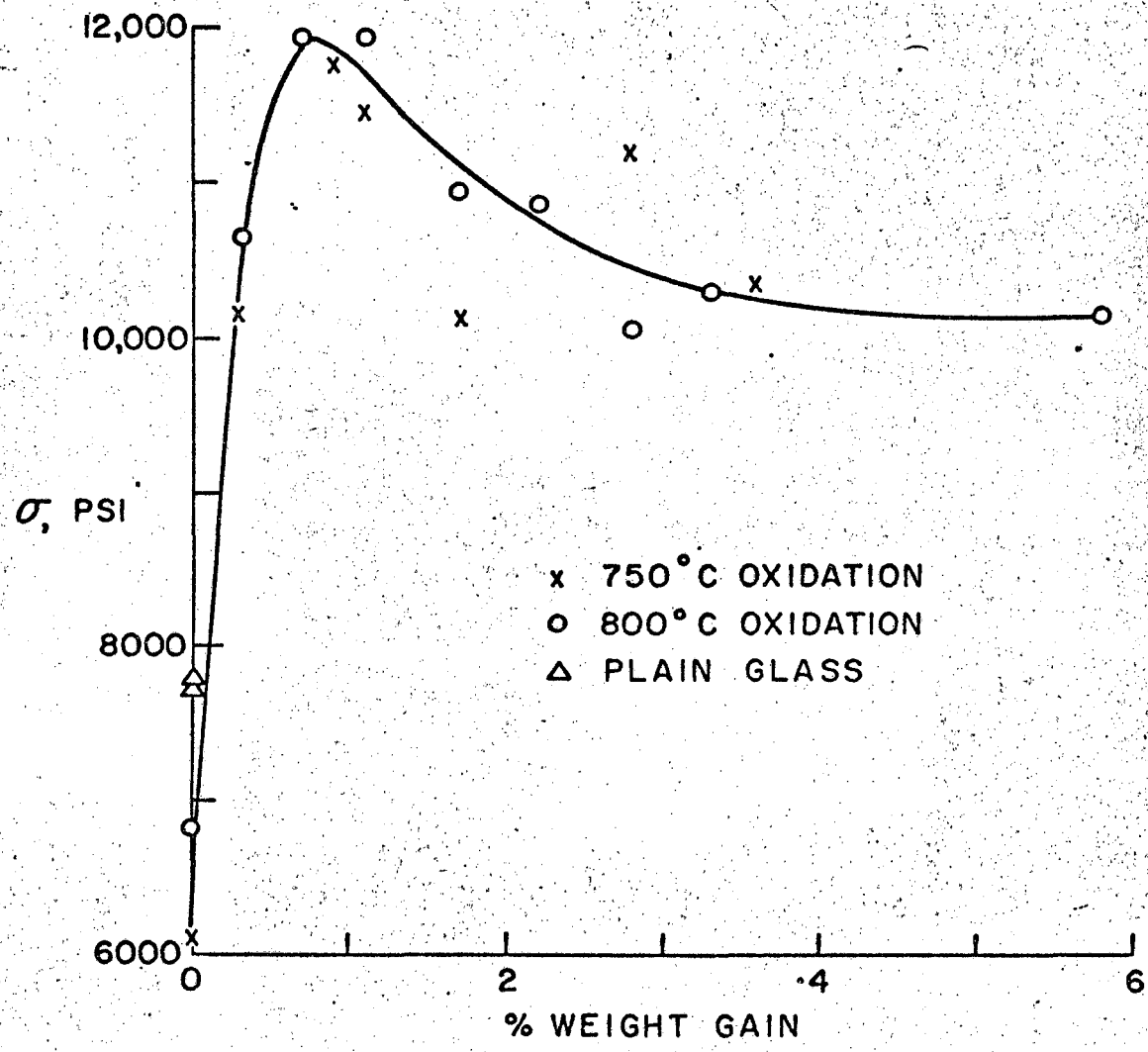

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Fig. 1

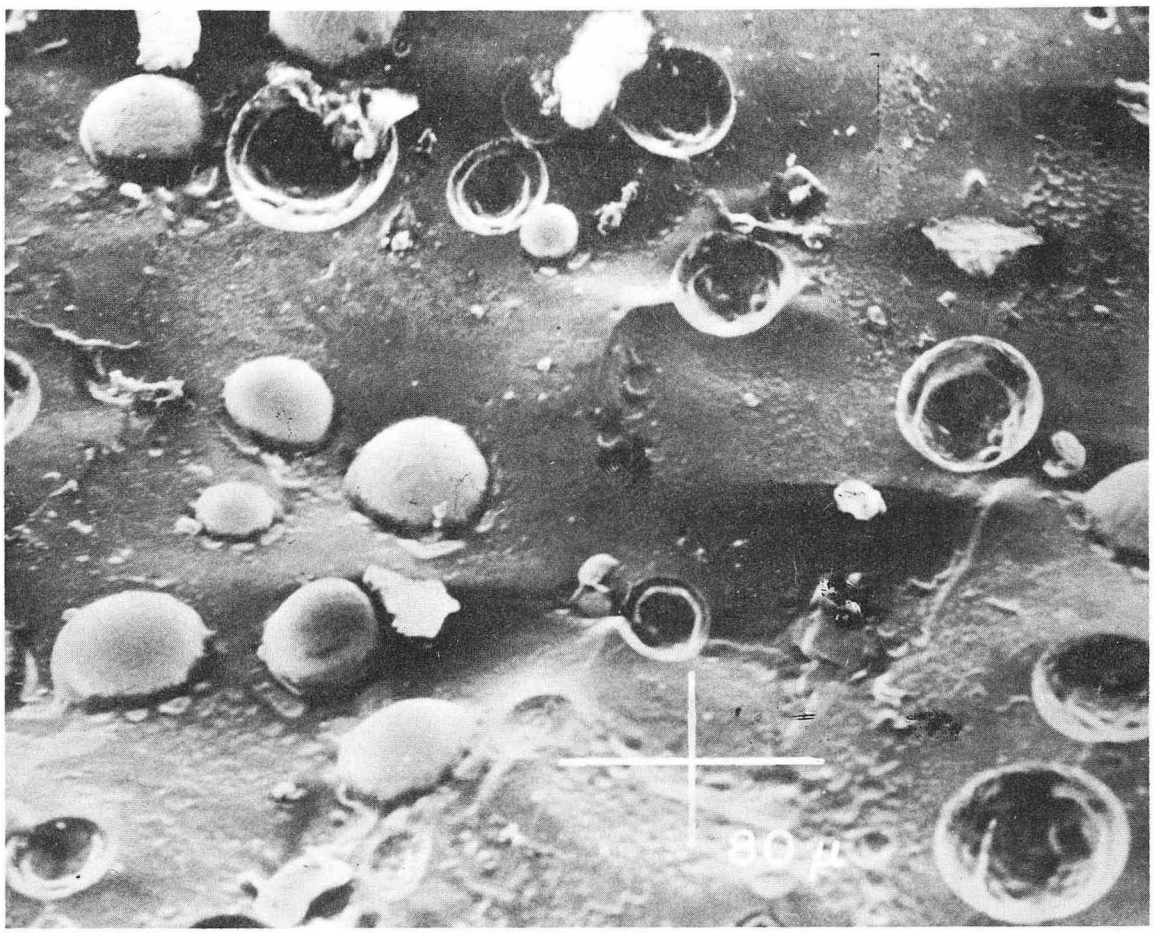

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Fig. 2

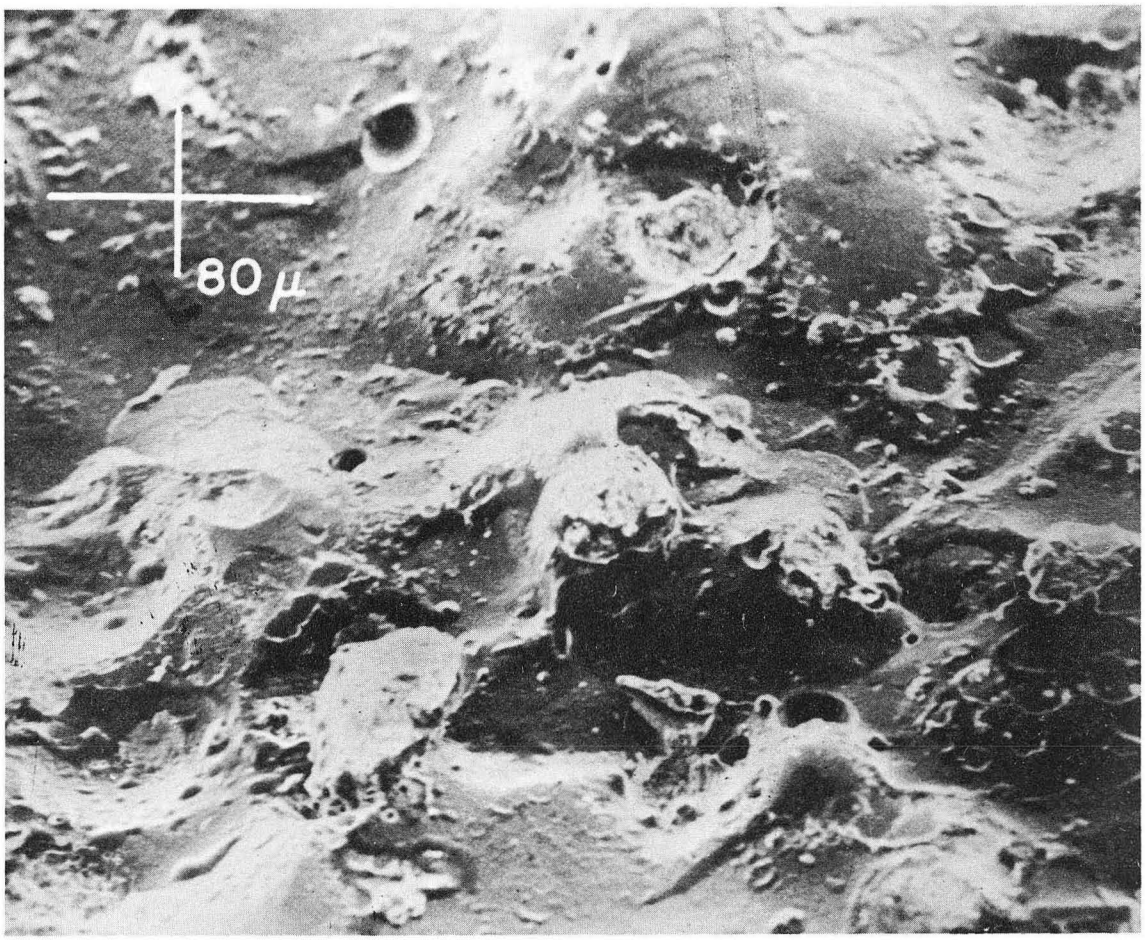

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Fig. 3

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