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## ADHESION AT CERAMIC INTERFACES

by

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Many of the properties associated with ceramic materials such as high hardness, high dielectric constant, refractoriness, and good optical properties will play a critical role in the development of devices for new and emerging technologies. In many cases, the combination of properties that is required demands that a composite material be designed to fulfill these complex materials needs. The increasing emphasis upon composite materials design and performance necessarily focuses greater attention upon the structure and properties of interfaces in ceramic materials. One of the most important aspects of interfacial behavior is the adhesive stability. As an example, high hardness ceramic coatings for tribological applications require a high degree of interfacial adhesion with the underlying substate material. Alternatively it has been shown [1] that fiber reinforced ceramic composites that are designed for high fracture toughness must contain weak interfaces that allow for fiber pull-out to toughen the intrinsically brittle ceramic matrix. Our ability to design ceramic interfaces for specific interfacial adhesive behavior dictates that we develop a full understanding of the factors that control the adhesive bond in these systems.

We report on the use of continuum fracture mechanics techniques to identify the molecular source of adhesion between oxide surfaces and introduce a new approach to measuring interfacial adhesive forces using an Interfacial Force Microscope.

When two brittle materials are fractured along the bi-material interface the fracture energy is a direct measure of the interfacial bond energies. Since fracture mechanics methods can be used to determine the interfacial fracture energy, this continuum approach can be used to explore molecular models for interfacial bonding.

We have used fracture mechanics test techniques [2,3] to measure the adhesive bond energy formed between hydrated silica glass surfaces and silica species deposited from solution. In the case of silicate surfaces hydrated in room temperature water vapor, intermolecular bonding between hydrated surfaces can be attributed to either hydrogen bonding interactions between adsorbed water molecules (0.15  $J/m^2$ ) or electrostatic bonds formed between adsorbed cations and anionic-nonbridging surface oxygen groups  $(2.0 \text{ J/m}^2)$ . The bonding interaction observed at room temperature depends upon the glass surface composition and the degree of surface hydration. When hydrated silicate solution species are added to the interface and heat treated, adhesion energies as large as the cohesive energy of vilica glass can be obtained with heat treatments as low as 200 C. The adhesion of the silicate interfacial film produced by the addition of solution species is greatest for silicate precursors showing a low degree of molecular crosslinking. In addition, the presence of alkali in the silicate solution greatly enhances interface adhesion for heat treatments below 200 C.



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Although the fracture mechanics technique that we have described in the previous section can be used to sort out possible mechanisms of interfacial bonding, a detailed description of the interfacial force versus interface separation is required to critically test theoretical models for adhesive bonding. It is also necessary that these measurements be made for well characterized surfaces so that the effects of interfacial impurities can be determined. In order to address this problem, we have combined aspects of atomic force and scanning tunneling microscopy to develop a new method for measuring interfacial forces on an atomic scale. This new Interfacial Force Microscope [4] is capable of measuring the force versus separation between the atomically flat surface of a field-emitter tip and a single-crystal surface using a unique force-feedback sensor design. A major limitation in previous attempts to measuring the interfacial force versus displacement relation is the instability that occurs when the interfacial force is greater that the compliance of the force sensor. In this new force feedback sensor design, we dynamically control the modulus of the force sensor through the electronic feedback loop so that the entire range of interfacial forces can be probed in one experiment.

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