



A Simplified Diagnostic Method for Elastomer Bond Durability

Less time and equipment are needed.

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A simplified method has been developed for determining bond durability under exposure to water or high humidity conditions. It uses a small number of test specimens with relatively short times of water exposure at elevated temperature. The method is also gravimetric; the only equipment being required is an oven, specimen jars, and a conventional laboratory balance.

Elastomers (rubbers) are frequently bonded to other surfaces, including glasses, metals, and occasionally other polymers. Due to mismatches in chemistry, thermal expansion and stiffness, the bonded interfaces usually require the use of a primer. Initial bond strengths can be determined by tensile testing, but the long-term durability of these bonds is uncertain, as is the effectiveness of the primer. This is especially the case when these bonds are exposed to humidity, thermal cycling, and possibly ultraviolet light.

Decline in bond strength is usually due to the intrusion of water at the bond interface. Water may chemically attack the primer chemistry, draw ions out from the glass or metal surface, and add stress from expansion. The long-term stability of bonded elastomers in humid environments is difficult to determine without very large numbers of test specimens and very long periods of water exposure.

A new method for determining rubber/glass bond durability was devised in which finely divided substrate (glass beads) was dispersed in the elastomer and the amount of absorbed water measured by weight gain. The difference between primed and unprimed glass beads was immediately apparent, and the stability of the bond was determined by measuring the time to a sudden increase in weight gain.

The materials used in this study were: (a) EVA; an elastomer of ethylene-vinyl acetate (33 percent) copolymer, (b) spheri-

cal glass micro-beads as the substrate, and (c) a primer consisting of methacryloxy propyl trimethoxy silane. The glass micro-beads had an average diameter of 30 microns, and were primed with 5 percent by weight of the primer deposited from an alcohol solution. The test specimens were made by blending 30 percent by volume of the glass beads (primed and unprimed) into the EVA polymer with the use of a two-roll differential mill, while adding 1.5 percent of a curing (vulcanizing) agent. A heat-activated peroxide agent was used [t-Butyl-O-(2-ethylhexyl)monoperoxy carbonate]. Compounded specimens were compression molded into sheets of 0.060-in. (1.52-mm) thickness, and cured at 150 °C for 30 minutes. The curing process vulcanizes the EVA rubber and also activates the primer to create a strong bond to the glass surface.

Table 1 provides strength and weight gain data for primed and unprimed

Temperature°C		40	40	60	60	60	80	80	80
UNPRIMED (Hrs)	Control	500	2,000	250	500	2,000	250	500	1,000
Strength,psi	1,380	1,230	1,210	960	530	50	285	310	0
Elongation,%	600	570	510	515	300	60	120	100	0
Weight gain, %	---	19	51	29	410	2,015	568	503	0
PRIMED (Hrs)	Control	500	2,000	250	500	2,000	250	500	1,000
Strength,psi	905	900	1,150	930	990	830	1,010	910	725
Elongation, %	350	190	390	285	315	120	245	220	140
Weight gain, %	---	2	4	4	6	35	13	17	62

Table 1. Water Gain and Strength are shown for EVA rubber containing primed and unprimed glass beads

Temperature°C		40	40	60	60	60	80	80	80
UNPRIMED (Hrs)	Control	500	2,000	250	500	2,000	250	500	1,000
Strength,psi	1,380	1,280	1,210	1,340	960	480	560	0	0
Elongation,%	600	565	570	575	500	280	290	0	0
Absorbed water,%	0	0	0	0	0	0	0	0	0
PRIMED (Hrs)	Control	500	2,000	250	500	2,000	250	500	1,000
Strength,psi	905	990	860	890	980	850	920	920	880
Elongation, %	350	315	260	240	240	240	310	310	260
Absorbed water, %	0	0	0	0	0	0	0	0	0

Table 2. Recovery of Water-Exposed Specimens is shown for primed and unprimed cases.

specimens after water immersion at 40, 60, and 80 °C. Cured EVA rubber containing no glass beads gained only 1 weight percent water. This shows that the immense weight gain of other specimens is due to water absorption at the glass/elastomer interface. This was also verified by FTIR (Fourier Transform Infrared) spectroscopy.

The amount of water absorption in specimens containing glass beads is dramatically changed by the presence of the primer, particularly in the specimens immersed in water at the 80 °C temperature. Unprimed specimens aged in water at 60 °C for 2,000 hours showed a 2015-percent increase in weight, whereas primed glass bead specimens gained only 35 percent. Reduction in tensile strength also followed primed and unprimed spec-

imens, and increasing exposure times and temperatures.

Importantly, this method can provide semi-quantitative measurement of the bond resistance to water by measuring the time to the sudden increase in weight. This effect is due to degradation of the primer chemistry at the interface. The longer the time to weight increase, the more effective is the primer and the more water resistant is its chemical bond.

Duplicate specimens of those reported in the first table were dried to constant weight and retested for tensile strength. This was done to determine the recoverability of hydrothermal attack after water removal. Specimens containing primed glass microbeads recovered their initial properties almost entirely, whereas the unprimed glass beads, especially in the 80 °C

water exposure, deteriorated too badly to be tested.

Table 2 presents the results as percentage retention of control values: This method offers a simplified way of (a) determining the effectiveness of primers used for bonding elastomers to glass, and perhaps other substrates, in environments with humidity or water exposure; (b) a semi-quantitative technique for determining bond stability; and (c) a method for determining the long term durability and onset of hydrolytic attack at the interface. This method has the advantages of requiring very few specimens and only a laboratory balance to perform measurements.

This work was done by Paul White of Caltech for NASA's Jet Propulsion Laboratory. Further information is contained in a TSP (see page 1). NPO-43912

Complex Multifunctional Polymer/Carbon-Nanotube Composites

CNTs are treated and incorporated into composites to obtain enhanced properties.

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A methodology for developing complex multifunctional materials that consist of or contain polymer/carbon-nanotube composites has been conceived. As used here, "multifunctional" signifies having additional and/or enhanced physical properties that polymers or polymer-matrix composites would not ordinarily be expected to have. Such properties include useful amounts of electrical conductivity, increased thermal conductivity, and/or increased strength. In the present methodology,

these properties are imparted to a given composite through the choice and processing of its polymeric and CNT constituents.

The methodology involves utilization of CNTs in any or all of several ways:

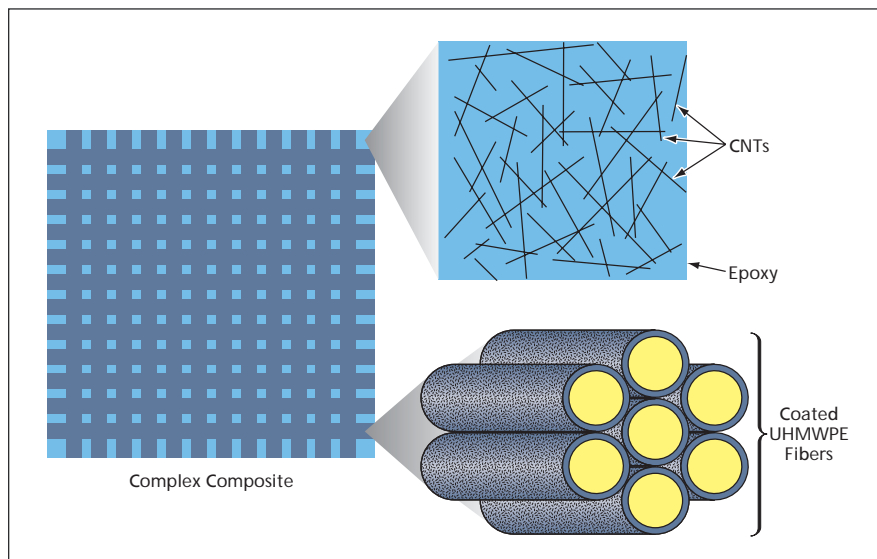
- Coating the CNTs to impart desired properties — for example, coating them with electrically and/or thermally conductive polymers, which could be dissolved in solvents;
- Incorporating uncoated or coated CNTs into a polymeric matrix, possibly in such

a manner as to improve the properties of the CNTs, the matrix, and/or the resulting composite; and/or

- Using a polymer/CNT composite as the matrix ingredient of a complex composite that includes any of a variety of other fibrous reinforcing materials.

The figure is a simplified illustration of an example of such a complex composite. In this case, a fabric made of coated ultra-high-molecular-weight polyethylene (UHMWPE) fibers is embedded in a matrix that is, itself, a composite of CNTs in an epoxy matrix. Typically, heretofore, such a composite would be designed and fabricated to obtain high strength, would not contain CNTs, and would be electrically insulating and, to some extent, thermally insulating. By incorporating a suitable quantity of CNTs, one can obtain enough electrical conductivity to drain off excess static electricity to prevent static discharge or to render the composite effective as a barrier against electromagnetic interference, and to obtain usefully large degrees of thermal conductivity and thermal stability, all without sacrificing mechanical strength.

This work was done by Pritesh Patel, Gobinath Balasubramaniam, and Jian Chen of Zyvex Corp. for Marshall Space Flight Center. For further information, contact Sammy Nabors, MSFC Commercialization Assistance Lead, at sammy.a.nabors@nasa.gov. Refer to MFS-32355-1.



This Complex Composite consists of a fabric of coated UHMWPE fibers in a matrix material consisting of an epoxy/CNT composite.