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James F. Carley

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Lawrence
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PRETREATMENT OF KAPTON*-COATED CABLE FOR EPOXY ADHESION

James F. Carley
Lawrence Livermore National Laboratory
Livermore, California

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ABSTRACT

Preliminary testing of a new system for protecting bonded strain gages that will be attached to the MFTF magnets indicated falling electrical resistance to ground, attributed to the infiltration of moisture. The most likely infiltration route seemed to be along the Kapton* lead cable, which has an outer surface of FEP fluorocarbon resin. Samples of the cable were pretreated with a fluorocarbon etchant, Tetra-Etch, for periods of 10, 25, and 40 s at room temperature, followed by rinsing with demineralized water. The treated ends were embedded in the proposed epoxy sealant, Hysol EA 934, a compound containing 70 wt % of asbestos. The tensile-shear stresses required to pull the wires out of these embedments were measured. Results show that the three levels of treatment are equally effective in raising the bond strength from 377 psi for the untreated cable to about twice that, 763 psi. The 40-s exposure to Tetra-Etch appears to have penetrated the 0.5-mil fluorocarbon coating and attacked the Kapton film and the conductor coatings inside it.

*Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Department of Energy to the exclusion of others that may be suitable. Kapton is a registered trademark of E. I. duPont de Nemours and Company.

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INTRODUCTION

Seven hundred strain gages are to be attached to various surfaces of the large magnets of the magnetic fusion apparatus now under construction at LLNL. In the preceding experiment, some of the epoxy-bonded, but uncoated sensors debonded over a year's time during which they were repeatedly taken down to 4 K and returned to ambient temperatures. It appeared that infiltrating moisture was the cause of the problem. In preparation for the next, larger experiment, improved systems of bonding and protecting strain gages were explored with the aid of an accelerated test in which the test specimens were exposed to 100% RH at 120°F for varying times. One of the most promising systems, sketched in Figure 1, is a multilayer construction. It consists of bonding the gage to a stainless-steel substrate (with suitable surface preparation), attaching the 4-wire Kapton-coated cable, applying a layer of asbestos-filled epoxy adhesive, covering the assembly with a layer of aluminum foil sealed along its edges with a nitrile-rubber sealant, then overlaying this with a second layer of the epoxy adhesive, Hysol EA 934. The Kapton wire was chosen over other candidates because of its high resistance to damage from neutron radiation. EA 934 was chosen for its excellent bonding properties, resistance to moisture and low thermal expansion (due to its high asbestos content).

When test gages prepared according to this recipe, but lacking the second EA 934 overcoat, were exposed to 100% RH at 120°F for some days, some showed a reduced electrical resistance to ground. We noticed, too, that the bond of the EA 934 along the surface of the leadwire cable seemed to pull apart easily, making that interface a prime suspect as a penetration route for moisture. I was asked to investigate this possibility and to suggest ways of improving the bond. In the short time available before the work of attaching gages to the MFTF magnets had to begin, it was felt that wholly new systems of gage bonding and sealing could not be initiated.

APPROACH TAKEN AND EXPERIMENTS MADE

In conversation with BarCal Wire and Cable, suppliers of the leadwire, I learned that the Kapton film used in the final wrap of the cable was coated with FEP fluorocarbon resin on both sides. This coating is heat-fusible, which the Kapton polyimide material itself is not; by passing it through an oven after wrapping, a continuous, moisture-resistant overcoat is generated. However, fluorocarbon resins ("Teflon") are noted for their very poor adhesion properties, and for the strenuous pretreatments that must be applied to them in order to make them adherable with common adhesives. It seemed likely that by removing the FEP film on the outside of the cable, or by pretreating it, we might achieve better bonds to the EA 934.

I first tried soaking the ends of some cable samples in boiling hexafluorobenzene (HFB), a chemical listed in the Polymer Handbook as a solvent for poly(hexafluoropropylene), a polymer closely related to FEP copolymer. Boiling times of 2, 4, and 24 h were used. There was no change in the appearance or texture of these cable ends, but the samples were given to James W. Fischer, who examined their surfaces by x-ray photoelectron spectroscopy, a technique of surface analysis which was capable of finding any fluorine present. Dr. Fischer reported strong F and C-F signals from the untreated and treated samples, indicating to me that the boiling treatments had not removed the FEP half-mil coating.

A commercially available etchant for fluorocarbons is Tetra-Etch, made by W. L. Gore and Associates. Bill Barnes of BarCal said they routinely treat cable with Tetra-Etch and recommended 20 to 30 s immersion at room temperature, followed by rinsing in deionized water. To test this reagent's effectiveness in our system, I planned the following experiment. Fifteen wire samples were cut and straightened as well as I could manage with my fingers. Six were left untreated; the ends of the others were etched. Three each were immersed in Tetra-etch for 10 s, 25 s and 40 s, and immediately rinsed thoroughly with deionized water* and dried with a clean wipe. The wires were arranged vertically in a rack with the treated ends down. A small amount of EA 934, thoroughly mixed, was placed in each of 15 5-cc polyethylene cups with

*One should not confuse this with unionized water, in which all the molecules are members of the Amalgamated Brotherhood of HOH Workers.

an inside bottom circumference of 3/4 inch. The cups were centered beneath the wire ends which were then pressed down into the soft (but stiff) epoxy mix and allowed to stand overnight. The cure was completed by placing the embedded wires in an air oven at 200°F for an hour. The finished embedment looked as sketched in Figure 2A.

A tensile-adhesion-test set up was devised for an Instron Model TDM testing machine located in 231 Building. As Figure 2A shows, the upper surfaces of the epoxy plugs were uneven, since this material is too stiff to flatten out by gravity and is difficult to flatten with a spatula. To accommodate this unevenness, I made a laminated washer consisting of ordinary flat steel washers cemented together, one 7/8-in OD, 3/8-in ID, the other 3/4-in OD, 5/16-in ID, to which was cemented a felt washer of the same ID, 0.7-in OD, and 0.2-in thickness (Figure 2B). When the specimen was pulled, the felt surface was pressing against the upper surface of the epoxy plug and the steel surface was retained by the grip of the testing machine. A plotter recorded the force-cell signal and the crosshead displacement. From those plots were obtained the breaking loads; these were used to calculate the shear stresses at failure.

Each of the epoxy plugs pulled away in one piece. Because of the unevenness of the upper surface, the depth of the shear surface in each plug varied considerably around the circumference. To obtain a representative mean depth, each plug was cut along a diameter by a sharp blow with a surgical scalpel, and the depth was measured along four elements around the circumference and the four values averaged. In six of the plugs there were bubbles impinging the shear surface; the unfilled part of the hole height was deducted prior to averaging. The wide variation in these depths was the largest source of variation in the breaking strengths.

Specimens 13, 14 and 15, exposed for 40 s in Tetra-Etch, showed fragments of the cable covering adhering to the epoxy plug with corresponding tears and gaps in the cable ends. This seemed to indicate that the etchant had entirely removed the FEP coating and, perhaps, had weakened the underlying polyimide. Cable specimen No. 14 was taken to Jim Fisher for surface analysis. Jim found that there was still detectable F present, but that the wrap was flimsy and the etchant seemed to have penetrated the wrap and dulled the insulation on the wires inside.

RESULTS AND RECOMMENDATIONS

The data and calculated shear stresses at failure are listed in Table 1.

For the 6 specimens that were cleaned but untreated, the mean stress at failure was 377 psi (2.60 MPa), the standard deviation was 98 psi (0.68 MPa), and the standard error of the mean was 40 psi (0.28 MPa).

For specimens 7-9, etched for 10 s, the corresponding figures are 773, 25 and 14.5 psi (5.33, 0.17, 0.100 MPa).

For specimens 10-12, etched 25 s, the corresponding figures are 762, 154, and 89 psi (5.25, 1.06, 0.61 MPa). The much larger standard deviation here is due to the very low failure stress of No. 11.

For specimens 13-15, etched 40 s, the figures are 757, 54 and 31 psi (5.22, 0.38, 0.22 MPa).

Clearly, the three levels of treatment provided nearly equal mean shear strengths. The strength was more consistent at 10 s than at 0, 25 or 40 s, however, this difference in variance is not statistically significant at the 95% level (risk of Type I error = 0.05). Pooling the sums of squares and degrees of freedom (11) for all 15 specimens provides a pooled standard deviation of 96.8 psi. When this value is used to test the significance of the difference between the treated and untreated means, 387 psi, the t-value is +7.59, far beyond any reasonable critical value. Etching makes a big, real difference!

Considering the apparently severe attack of the Kapton overwrap at 40 s, *and the evidence of etchant penetration into the cable end, I recommend that* the cable be treated for 10 s in Tetra-Etch at room temperature, as shown in Figure 3, and immediately be thoroughly rinsed with demineralized water, dried, and cut off. After the electrical connections have been made, the length of cable that will be embedded in EA 934 should be wiped with alcohol and allowed to dry prior to applying the adhesive. Care should be taken in applying the final overcoat to make sure that plenty of EA 934 gets beneath the cable and completely wets and surrounds the entire circumference.

Table 1. Bond Strength Measurements

Number	Time of Exposure to Tetra-Etch, s	Bond Area, 10^{-3} in^2	Load at Failure lb_f	Shear Stress at Failure, psi	Mpa
1	0	70.5	24.4	346	2.39
2	0	56.9	23.8	418	2.88
3	0	49.5	26.4	533	3.68
4	0	72.1	18.0	250	1.72
5	0	61.4	19.0	309	2.13
6	0	50.3	20.3	404	2.78
7	10	25.4	19.7	776	5.35
8	10	34.3	27.3	796	5.49
9	10	27.6	20.6	746	5.15
10	25	24.0	21.0	875	6.03
11	25	45.9	26.9	586	4.04
12	25	37.6	31.0	824	5.68
13	40	29.9	24.0	803	5.53
14	40	37.6	29.0	771	5.32
15	40	42.6	29.7	697	4.81

Section A-A

Fig. 1: Proposed multilayer system for bonding and protecting MFTF strain gages.

Fig. 2: A is a profile view of test specimen of Kapton cable embedded in EA 934 epoxy plug. B shows cushioned washer whose face was set against upper face of plug during tensile-shear test.

Fig. 3: This shows suggested way to etch end of Kapton lead cable that will be embedded in EA 934 adhesive overcoat, without allowing Tetra-Etch to infiltrate cable end.