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Lap Shear Strength of Selected Adhesives (Epoxy, Varnish, B-Stage Glass Cloth) in Liquid Nitrogen and at Room Temperature

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ABSTRACT

The lap shear strengths of several adhesives were measured in liquid nitrogen and at room temperature. The adhesives included several epoxy resins, a varnish, and a B-stage glass cloth (a partially cured resin in a fiberglass cloth matrix). Several parameters critical to bond strength were varied: adhesive and adherend differences, surface preparation, coupling agents, glass cloth, epoxy thickness, fillers, and bonding pressure and temperature. The highest lap shear strengths were obtained with the B-stage glass cloth at both liquid nitrogen and room temperatures with values of ∿20 MPa (3000 psi) and ∿25.5 MPa (3700 psi) respectively.

INTRODUCTION

Adhesives will be used in future superconducting machinery for electrical insulation and/or structural strength. The application of the adhesive, whether for structural support, spacing, thermal insulation, or electrical insulation, will determine just how strong the adhesive must be. Our particular application of an adhesive is to provide good mechanical strength and electrical insulation between turns or layers of the superconducting coils to be fabricated for tokamak type fusion machines. For that purpose, a program was initiated by the Materials Evaluation Group of the Superconducting Magnet Development Program to evaluate the lap shear strengths of selected adhesives at room and at liquid nitrogen temperatures. Liquid nitrogen was chosen over liquid helium because of the difficulties and expense of liquid helium testing and also because the mechanical and physical properties of the adherends and adhesives change little from 77K to 4.2K.

The purpose of the lap shear tests was to quantify and compare the effects on shear strength induced by several parameters: adhesive and adherend differences, surface preparations, coupling agents, glass cloth, adhesive thickness, fillers, and curing pressure and temperature. The

comparison yielded the best adhesive, of the ones tested, for use at room temperature and/or liquid nitrogen. The best adhesive was determined in relation to winding a large coil: i.e., ease in adhesive application, surface preparation of conductor, working setup time of the adhesive, curing temperature and cycle, and overall mechanical strength.

The adhesives were subdivided into two groups, epoxies and B-stage glass cloth, because of the two distinct methods of sample fabrication with respect to surface preparation, temperature and pressure cure cycles, adhesive application, and adherend type and size.

EPOXIES

The several epoxies used in the shear strength evaluation are listed in Table 1 (which indicates the epoxy trade name, manufacturer, mixture ratios, typical setup time, cure cycle, and relative viscosity). The Shell Epon, Emerson and Cummings Stycast, and General Mills Versamid epoxies were less than one year old by the time the samples were tested, whereas the Hexcel Uralite, Epotek, and Crest epoxies were mixed and tested slightly after the expiration date of the shelf life. The effect of aging on shear strength should be minimal, particularly since the epoxies had been stored in a refrigerator.

A large percentage of the epoxies tested was a mixture of 70% Epon 871, 30% Epon 828, and 13 pph Epon Z curing agent because this particular mixture had been used in the study of the effect of surface preparation on shear strength. This particular mixture was optimized for cryogenic use by the addition of the flexibilizer Epon 871 (an aliphatic epoxy resin), to the Epon 828 (an unmodified bisphenol — an epoxy resin). The proportion of 70% Epon 871, 30% 828, and 13 pph Epon Z curing agent had the highest overall strength in our cryogenic tensile and impact tests of the 871-828 group and was therefore used for the shear tests. This Epon 871-828 mixture was easily mixed, outgassed, and poured, and it lends itself to vacuum coil encapsulation applications. Setup time is long — over 5 hours @ 75°F — and the cure cycle is mild — about 150°F for 24 hours.

Table 1.

Epoxy Trade Name	Manufacturer	Mixing Ratio	Typical Setup Time	Cure Cycle	Viscosity
Epon 871 Epon 828 Z-Hardener	Shell Shell Shell	871 - 70% 828 - 30% Z - 13pph	∿5 hrs@75°F ∿2 hrs@150°F	24 hrs@150°F	Thin
Epon 871 Epon 828 Z-Hardener Silane 6020	Shell Shell Shell Dow Corning	871 - 70% 828 - 30% Z - 13pph .5%/wt	∿5 hrs@75°F ∿2 hrs@150°F	24 hrs@150°F	Thin
Stycast 2850 FT (Blue) 24 LV Hardener	Emerson & Cummings	Stycast 2850 FT 100% 24 LV - 7pph	1 hr@75°F	24 hrs@75°F	Med. Thick ∿15,000 CPS @70°F
Stycast 2850 FT (Black) #9 Hardener	Emerson & Cummings	Stycast 2850 FT 100%; #9 Hardener 3.5pph	1 hr@75°F	24 hrs@75°F	Thick 90,000 CPS @70°F
General Elec. No. 7031	General Electric	100% G.E. Adhesive & insulating varnish	∿6 hrs@75°F	6 hrs@75°F plus 12 hrs @150°F	Thin
Epon 871 Epon 828 Z-Hardener Silane 6040	Shell Shell Shell Dow Corning	70% - 871 30% - 828 13pph-Z .5% Silane	∿5 hrs@75°F 2 hrs@150°F	24 hrs@150°F	Thin
Epon 815 Versamid 140	Shell General Mills	100 parts 40 parts	4-6 hrs@75°F	24 hrs@75°F	Thin
Hexcel Uralite	Hexcel	100 parts "A" 40 parts "B"	∿ 10 min.	24 hrs@150°F	Thick

Table 1. (cont.)

Epoxy Trade Name	Manufacturer	Mixing Ratio	Typical Setup Time	Cure Cycle	Viscosity
Crest	Crest	100 parts "A" 26 parts "B" (by wt.)	∿ 10 min.	2 hrs@250°F	Thick
Epo-Tek 920 FL	Epoxy Technology Inc.	100 parts "A" 3 parts "B" (by wt.)	In Excess of 8 hrs	5 min.@120°F	Medium Thick 14,000 CPS
Epon 815 Versamid 140 Silane 6020	Shell General Mills Dow Corning	100 parts 40pph 1%	5 hrs@75°F 2 hrs@150°F	24 hrs@150°F	Thin
Epon 871 Epon 828 Z-Hardener Silane 6020	Shell Shell Shell Dow Corning	70% - 871 30% - 828 13pph - Z 1% Silane	∿5 hrs@75°F	24 hrs@150°F	Thin
Epon 815 Versamid 140 Silane 6020	Shell General Mills Dow Corning	100 parts 40pph 2%	4-6 hrs@75°F	24 hrs@75°F	Thin

The Epon 815-Versamid 140 mixture has about the same flexibility and texture after curing as the Epon 871-828 mixture but seems more fluid during the working time before gelation, which makes the Epon 815-Versamid 140 combination very appropriate for vacuum coil encapsulation. The mixture used for testing was 100 parts Epon 815 and 40 parts Versamid 140. Typical setup time is 4-6 hours @ 75°F with a total cure after 24 hours.

Two Stycast epoxies were mixed and tested for shear strength. Stycast 2850 FT (Blue) with 7 pph 24 LV hardener is a medium-thick epoxy with a room temperature cure cycle, and Stycast 2850 FT (Black) with 3.5 pph # 9 hardener is a thick epoxy which has a room temperature cure.

The remaining epoxies were off-the-shelf, two-part adhesives which were generally easy to mix, difficult if not impossible to outgas, and hard to pour because of their thick textures. The time from mixing to gelation was short — approximately 10-15 minutes for the Hexcel Uralite and Crest epoxies and about 8 hours for the Epotek epoxy.

The only varnish tested was an all-purpose insulating varnish, General Electric 7031, which has a room temperature cure time of about 6 hours @ 75°F plus 12 hours @ 150°F. GE 7031 is a one-part liquid varnish which becomes tacky in about 15 minutes when used in thin applications.

B-STAGE GLASS CLOTH

A large sample of B-stage glass cloth was obtained from Synthane Taylor of LaVerne, California for testing and evaluation. This particular sample, EF 527-5M, required a medium cure cycle, i.e., 350°F at 300-400 psi for ∿1 hour. It should be pointed out that the medium cure cycle required for the glass cloth is obtained in industry by integrating a hydraulic press with a furnace such that the pressure and temperature can be raised simultaneously to the desired levels. Lacking a large press and furnace, we used an alternate method of applying pressure and temperature. C-clamps outfitted with strain gauges and calibrated using a load cell were used in a small oven to apply the desired pressure to

the shear samples. Although exact control over temperature and pressure was not possible, excellent and consistent shear results were obtained. In fact, the first samples had a 360°F @ 50 psi cure cycle and the shear strength was still higher than any of the epoxies. The refrigerated B-stage glass cloth was very easy to use and withstood severe abuse (handling, cutting, bending) with no noticeable decrease in shear strength.

OTHER PARAMETERS

Three distinct fabrication techniques were used to assemble the shear samples because the epoxies were of different viscosities, the glass cloth was added to some, and the bond thickness and curing pressures were varied from sample to sample. The first technique was to use a hypodermic needle to inject the low viscosity epoxies into a mold in which the prepared samples had been placed. This method was simple and allowed the bond thickness to vary depending on the mold and adherend size. Samples 1Cu-6Cu, 1SS, 2SS, and 1Al-7Al were prepared in this way.

For the thicker epoxies, a method was devised to enable the application of an epoxy, the alignment of the adherends, and the curing of the epoxy with a constant bond pressure. A Teflon sample holder was used to position the adherends and epoxy while a large spring clamp was applied to the outside adherends. The clamp allowed for thinner bond thicknesses and an approximate 210 KPa (30 psi) cure pressure. Samples 7Cu-14Cu, 3SS-6SS, and 8Al-13Al were prepared in this manner.

The third and simplest of the techniques was required for the B-stage glass cloth sample. The fabrication consisted of the adherend surface preparation as outlined on Table 3, the cutting of the B-stage glass cloth to fit the adherend size with a pair of scissors, and the clamping of the sample with the calibrated C-clamp. The samples were then placed in the oven with the curing pressures being continuously monitored with a strain indicator. The fabrication technique for the B-stage glass cloth samples was the easiest of the techniques since no mixing, outgassing, or pouring was required and also since there was no stringent setup time.

Several surface preparations were tried, ranging from sophisticated etching and cleaning solutions to simple abrasion and degreasing. The wide variations of surface preparations are listed in Table 2 for the varnish and epoxies samples and in Table 4 for the B-stage glass cloth samples.

A coupling agent, Dow Corning Z 6020, was added to some of the Epon 871-828 and Epon 815-Versamid 140 epoxies to evaluate its effect on shear strength. Z 6020 silane is an amino-functional material widely used as a coupling agent in the fabrication of resin-glass laminates. The key to the coupling ability of this product is the dual reactivity of the silane molecule, which enables it to serve as an effective chemical bridge between the organic and inorganic. The liquid Z 6020 silane was added directly to the epoxy prior to mixing and outgassing but was applied directly to the copper adherends in a couple of the B-stage glass cloth samples, sample set number 13.

Glass cloth was added to a few samples to act as a spacer between adherends and to possibly increase the shear strength by decreasing the thermal contraction gradient between adherend and epoxy. The glass cloth was .18mm (.007") thick, untreated, and finely woven. The cloth was simply cut to size and inserted between adherends. By nature, the B-stage glass cloth is 53.6%-55% glass cloth by weight.

A filler, 300-mesh fused quartz, was added to some of the Epon 871-828 and Epon 815-Versamid 140 epoxy samples to see the effect it might have on shear strength. It was hoped that the quartz filler would reduce the large thermal contraction gradients between the adherends and the epoxies. A 40% (by weight) addition of the fused quartz to the epoxies still enabled simple mixing, outgassing, and pouring into the mold. Three-hundred mesh fused quartz was used since it readily dispersed throughout the epoxy and would not settle to the bottom of the mixing container. Larger sized fillers tend to stratify throughout the epoxy unless constantly stirred. The addition of the quartz filler is indicated in Tables 3 and 4.

A double lap joint (see Fig. 1) was chosen for the test configuration for ease in assembly and alignment. With this arrangement, bending

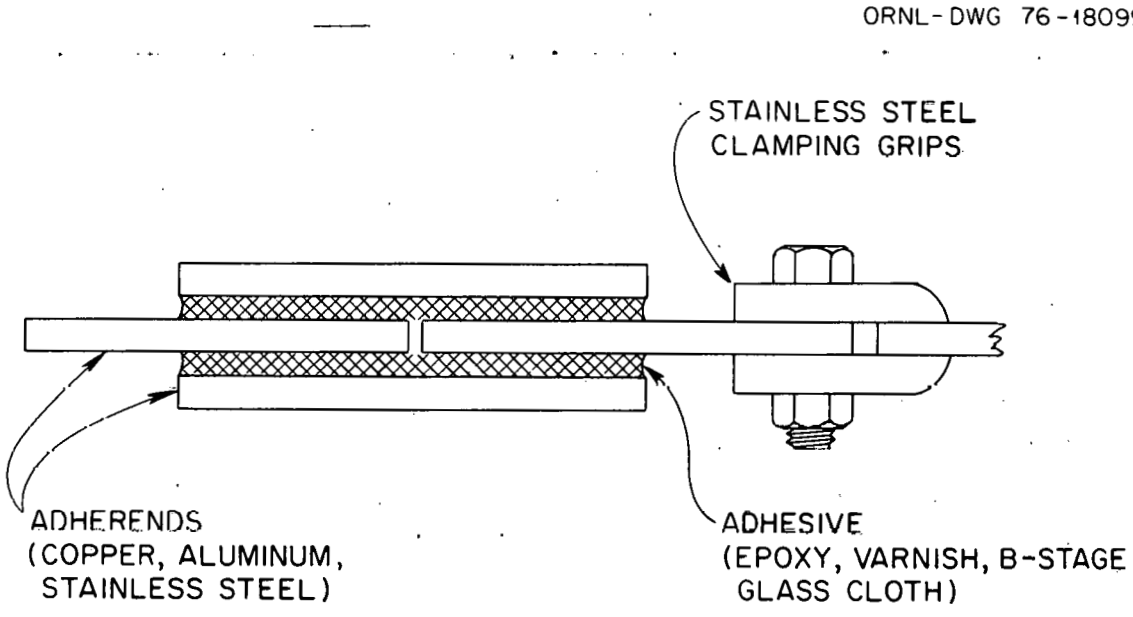

Fig. 1. Typical double lap shear sample with grips.

Table 2. Surface Preparations of Shear Specimens

Material: Cu, ASTM B 152, Light Cold-Rolled
1.59mm × 15.88mm × 50.80mm
(1/16" × 5/8" × 2")

Sample Number	
1Cu	1 - Degreased with trichloroethylene. 2 - Rinsed in distilled H_2O for five minutes. 3 - Etched in a solution of: A - Concentrated phosphoric acid - 75% B - Concentrated nitric acid - 10% C - Demineralized H_2O - 15% 4 - Rinsed in distilled water and dried at room temperature.
2Cu	1 - Degreased in trichloroethylene. 2 - Rinsed in distilled H_2O for five minutes. 3 - Etched in the following solution for 1/2 minute: A - Phosphoric acid - 75% B - Sulfuric acid - 10% C - Distilled H_2O - 15% 4 - Rinsed in distilled H_2O for five minutes. 5 - Stored in desiccator in $CaSO_4$ and pumped to 500μ.
3Cu	1 - Used 240 lining cloth, then number 400 lining cloth until oxidized surface was removed. 2 - Stored overnight in alcohol bath. 3 - Degreased in trichloroethylene. 4 - Rinsed in alcohol. 5 - Air dried - rubbed with facial tissue.
4Cu	1 - Brushed with steel brush until shiny. 2 - Abraded with # 100 (coarse) emery cloth. 3 - Immersed in a beaker of trichloroethylene overnight. 4 - Poured off in the morning - immersed in fresh trichloroethylene - samples then removed and blotted dry.
5Cu	1 - Mechanically abraded using coarse emery cloth. 2 - Degreased in trichloroethylene. 3 - Air dried at room temperature. Note: 1/8" samples instead of 1/16" for 5Cu only.
6Cu	1 - Degreased in trichloroethylene. 2 - Air dried. 3 - Abraded with medium emery cloth, until all oxidation removed. 4 - Degreased in trichloroethylene. 5 - Air dried.
7Cu	1 - Mechanically abraded using coarse emery cloth. 2 - Degreased in trichloroethylene. 3 - Air dried at room temperature.
8Cu	1 - Mechanically abraded using coarse emery cloth. 2 - Degreased in trichloroethylene. 3 - Air dried at room temperature.
9Cu	1 - Mechanically abraded using coarse emery cloth. 2 - Degreased in trichloroethylene. 3 - Air dried at room temperature.
10Cu, 11Cu 12Cu & 13Cu	1 - Mechanically abraded using coarse emery cloth. 2 - Degreased in trichloroethylene. 3 - Air dried at room temperature.

Material: Stainless Steel 304L ASTM A 240 Bright Cold-Rolled
1.59mm × 15.88mm × 50.80mm
(1/16" × 5/8" × 2")

1SS	1 - Degreased with trichloroethylene. 2 - Rinsed in demineralized water for five minutes. 3 - Immersed 10 minutes in the following solution: A - 50-50 solution of HCl and H_2O 4 - Rinsed in deionized H_2O at 200°F. 5 - Dried at 200°F.

Table 2. Surface Preparations of Shear Specimens (Cont.)

Sample Number	
2SS	1 - Degreased in trichloroethylene. 2 - Rinsed in demineralized H_2O for five minutes. 3 - Rinsed in the following solution for five minutes: A - Oxalic acid - 9 parts by wt. B - Sulfuric acid - 1 part by wt. C - Demineralized H_2O - 90 parts by wt. 4 - Immersed in above solution 8-10 minutes at 180°F - 190°F. 5 - Black smut removed by brushing with a steel brush under running water. 6 - Rinsed thoroughly in demineralized water. 7 - Rinsed in alcohol and dried at room temperature.
3SS, 4SS, 5SS & 6SS	1 - Mechanically abraded using coarse emery cloth. 2 - Degreased in trichloroethylene. 3 - Air dried at room temperature. Material: Al ASTM B 209 AA 1100 H14 1.50mm × 15.88mm × 50.80mm (1/16" × 5/8" × 2")
1Al	1 - Etched in following solution: 200 ml H_2O (distilled) 50 ml 96% H_2SO_4 2 gr. sodium dichromate 2 - Degreased in trichloroethylene, rinsed in distilled water, then etched in above solution at 60°C for 12 minutes.
2Al	1 - Degreased in trichloroethylene. 2 - Rinsed in demineralized water for five minutes. 3 - Etched in a solution of: A - Demineralized H_2O - 30 parts B - Sulfuric acid (96.3%) - 10 parts C - Sodium dichromate - 1 part Immersed in above solution at 160°F for 10 minutes. 4 - Rinsed in demineralized H_2O for ten minutes. 5 - Stored until tested in desiccator with $CaSO_4$ & pumped to 500μ until tested.
3Al	1 - Degreased in trichloroethylene. 2 - Rinsed in demineralized H_2O for 5 minutes. 3 - Etched in a solution of: A - Chromic acid - 81 grams B - Sulfuric acid (96.3%) - 236 grams C - Enough demineralized H_2O to make 1 gallon Immersed in above solution at 160°F for 10 minutes. 4 - Rinsed in demineralized H_2O for 5 minutes. 5 - Stored in desiccator with $CaSO_4$ & pumped to 500μ until tested.
4Al	1 - Degreased in trichloroethylene. 2 - Rinsed in demineralized H_2O for five minutes. 3 - Etched in following solution: A - Demineralized H_2O - 200 ml B - NaOH (50% sol) - 50 ml Immersed in above solution for 10 minutes. 4 - Rinsed in demineralized H_2O for 5 minutes. 5 - Stored in desiccator with $CaSO_4$ & pumped to 500μ until tested.
5Al	1 - Degreased in trichloroethylene. 2 - Rinsed in demineralized H_2O for 5 minutes. 3 - Cleaned surface with #240 & #600 emery cloth. 4 - Degreased in trichloroethylene. 5 - Rinsed in demineralized H_2O for five minutes. 6 - Stored in desiccator.
6Al	1 - Degreased in trichloroethylene. 2 - Rinsed in demineralized H_2O for five minutes. 3 - Etched in the following solution: A - Demineralized H_2O - 30 parts B - Sulfuric acid - 10 parts C - Sodium dichromate - 1 part

Table 2. Surface Preparations of Shear Specimens (cont.)

Sample Number	
	Immersed in above solution 160°F for 10 minutes - then stored in desiccator with $CaSO_4$ & pumped to 500μ until tested.
7Al	1 - Degreased with trichloroethylene. 2 - Rinsed in demineralized H_2O for five minutes. 3 - Rinsed in methyl alcohol. 4 - Dried at room temperature. 5 - Etched in the following solution: A - Demineralized H_2O - 30 parts B - Sulfuric acid (96.3%) - 10 parts C - Sodium dichromate - 1 part 6 - Rinsed in demineralized H_2O for 5 minutes. 7 - Rinsed in methyl alcohol and dried.
	Size: 3.5mm × 15.88mm × 50.80mm (1/8" × 5/8" × 2")
8Al, 9Al 10Al & 11Al	1 - Mechanically abraded using coarse emery cloth. 2 - Degreased in trichloroethylene. 3 - Air dried at room temperature

Table 3. Shear Test Results of Epoxies

Copper: Type: ASTM B 152, Light Cold-Rolled
Size: 1.59mm × 15.88mm × 50.80mm
(1/16" × 5/8" × 2")

Sample No.	Epoxy Formula	Lap Shear Strength				Average Bond Thickness	
		77K		293K			
		MPa	(psi)	MPa	(psi)	mm	(in.)
1Cu	70% Epon 871			.19	(27)	1.52	(.060)
	30% Epon 828			.24	(35)		
	13 pph Z curing agent			.27	(39)		
2Cu	70% Epon 871	.48	(69)	6.76	(980)	1.52	(.060)
	30% Epon 828	.08	(12)	5.93	(860)		
	13 pph Z curing agent	.16	(23)	9.45	(1374)		
3Cu	70% Epon 871	2.13	(308)	8.06	(1169)	1.52	(.060)
	30% Epon 828	.92	(134)	8.49	(1231)		
	13 pph Z curing agent	.99	(144)	8.49	(1231)		
4Cu	70% Epon 871	8.49	(1231)	7.93	(1149)	1.52	(.060)
	30% Epon 828	9.48	(1374)	8.77	(1272)		
	13 pph Z curing agent	8.06	(1169)	8.49	(1231)		
5Cu	70% Epon 871	11.58	(1678)	6.22	(901)	.50	(.020)
	30% Epon 828	10.59	(1535)	9.04	(1310)		
	13 pph Z curing agent	10.45	(1515)				
	+.5% Z 6020 Silane						
6Cu	70% Epon 871	6.19	(898)	7.92	(1147)	1.5	(.060)
	30% Epon 828	6.72	(974)	7.78	(1128)		
	13 pph Z curing agent	7.38	(1070)	8.04	(1166)		
	+.007" glass cloth						
	+.5% Z 6020 Silane						
7Cu	Stycast 2850 FT	4.39	(636)	5.93	(860)	.08	(.003)
	Blue + 7 pph 24 LV	5.93	(860)	5.62	(815)		
	curing agent	4.57	(663)	6.00	(869)		
		5.25	(761)	4.76	(690)		
8Cu	Stycast 2850 FT	7.19	(1044)	7.16	(1039)	.25	(.010)
	Blue + 7 pph 24 LV	8.02	(1164)	8.12	(1179)		
	curing agent	8.34	(1210)	6.62	(961)		
	+.007" glass cloth						
9Cu	Stycast 2850 FT	8.28	(1201)	7.16	(1039)	.25	(.010)
	Blue + 7 pph 24 LV	8.28	(1201)	7.41	(1075)		
	curing agent						
	+.007" glass cloth						
10Cu	Stycast 2850 FT	4.55	(661)	2.96	(430)	.10	(.004)
	Black + 7 pph 24 LV	3.95	(573)	3.10	(450)		
	curing agent	4.32	(627)	3.25	(471)		
11Cu	Stycast 2850 FT	5.68	(824)	2.88	(418)	.30	(.012)
	Black + 7 pph 24 LV	3.82	(554)	3.30	(479)		
	curing agent	6.18	(896)	4.41	(640)		
	+.007" glass cloth						
12Cu	100 parts Epon 815	4.61	(669)			.13	(.005)
	40 parts Versamid 140	4.61	(669)				
	+ 40% wt. 300 mesh fused quartz						
	+ 2% Silane 6020						
	+.007" glass cloth	7.51	(1089)			.43	(.017)
13Cu	Hexcel Uralite	9.07	(1315)			.13	(.005)
14Cu	Epo-tek	7.19	(1042)			.13	(.005)
		6.59	(956)			.13	(.005)
	+.007" glass cloth	7.69	(1115)			.43	(.017)

Table 3. Shear Test Results of Epoxies (cont.)

Stainless Steel: Type: 304L ASTM A 240 Bright Cold-Rolled
Size: 1.59mm × 15.88mm × 50.88mm (1/16" × 5/8" × 2")

Sample No.	Epoxy Formula	Lap Shear Strength 77K MPa	77K (psi)	293K MPa	293K (psi)	Average Bond Thickness mm	(in.)
1SS	70% Epon 871 30% Epon 828 13 pph Z curing agent	6.77 3.97 6.03	(981) (576) (875)	5.88 10.01 10.01 10.29	(853) (1451) (1451) (1493)	1.52	(.060)
2SS	70% Epon 871 30% Epon 828 13 pph Z curing agent	7.52 5.01	(1090) (726)	6.21 8.11 9.57	(900) (1176) (1387)	1.52	(.060)
3SS	70% Epon 871 30% Epon 828 13 pph Z curing agent + 40% wt. 300 mesh fused quartz + 1% Silane 6020	10.81 10.81 12.14	(1568) (1568) (1760)			.13 .13 .13	(.005) (.005) (.005)
	+.007" glass cloth	17.14 17.40	(2485) (2523)			.43 .43	(.017) (.017)
4SS	100 parts Epon 815 40 parts Versamid 140 + 1% Silane 6020	11.99 11.99 9.41	(1739) (1739) (1365)			.13 .13 .13	(.005) (.005) (.005)
	+.007" glass cloth	16.94 15.17	(2457) (2200)			.43 .43	(.017) (.017)
5SS	100 parts Epon 815 40 parts Versamid 140 + 40% wt. 300 mesh fused quartz + 1% Silane 6020	6.57 6.57	(953) (953)			.13 .13	(.005) (.005)
	+.007" glass cloth	11.59 12.57	(1680) (1822)			.43 .43	(.017) (.017)
6SS	100 parts Epon 815 40 parts Versamid 140 + 40% wt. 300 mesh fused quartz + 2% Silane 6020	7.91 7.91	(1147) (1147)			.13 .13	(.005) (.005)
	+.007" glass cloth	10.94 11.12	(1587) (1612)			.43 .43	(.017) (.017)
	Aluminum: Type: ASTM B 209 AA 1100 - H14 Size: 1.59mm × 15.88mm × 50.80mm (1/16" × 5/8" × 2")						
1A1	70% Epon 871 30% Epon 828 13 pph Z curing agent	4.67 5.52 6.23	(677) (800) (903)	5.09 5.52 >5.58	(738) (800) (809)	1.52	(.060)
2A1	70% Epon 871 30% Epon 828 13 pph Z curing agent	4.82 3.58	(700) (520)			1.52	(.060)
3A1	70% Epon 871 30% Epon 828 13 pph Z curing agent			>5.65 >5.44	(820) (789)	1.52	(.060)
4A1	70% Epon 871 30% Epon 828 13 pph Z curing agent			>5.23 >5.09	(759) (738)	1.52	(.060)

Table 3. Shear Test Results of Epoxies (cont.)

Aluminum: Type: ASTM B 209 AA 1100 - H14
Size: 1.59mm × 15.88mm × 50.80mm (1/16" × 5/8" × 2")

Sample No.	Epoxy Formula	Lap Shear Strength 77K MPa	77K (psi)	293K MPa	293K (psi)	Average Bond Thickness	
5A1	70% Epon 871 30% Epon 828 13 pph Z curing agent			>5.65	(820)	1.52	(.060)
6A1	70% Epon 871 30% Epon 828			6.62 5.76 6.33 2.19 >5.49 >5.31	(961) (836) (918) (318) (796) (770)	1.52	(.060)
7A1	70% Epon 871 30% Epon 828 13 pph Z curing agent +.5% Z 6020 Silane +.007" glass cloth	9.87 9.72 9.72 9.87	(1433) (1411) (1411) (1433)	>5.93 >5.77 >5.86	(860) (838) (850)	.76	(.030)
8A1	GE 7031 varnish +.007" glass cloth	2.16 2.04 2.96 2.47	(314) (296) (430) (358)	3.33 2.71	(483) (394)	.23	(.009)
	Size: 3.18mm × 15.88mm × 50.80mm (1/8" × 5/8" × 2")						
9A1	100 parts Epon 815 40 parts Versamid 140 + 40% wt. 300 mesh fused quartz	9.43 9.43	(1367) (1367)			.13	(.005)
	+.007" glass cloth	12.22	(1772)			.43	(.017)
10A1	Hexcel Uralite	12.26	(1778)			.13	(.005)
	+.007" glass cloth	11.22 12.78	(1627) (1853)			.43 .43	(.017) (.017)
11A1	Crest 7410	11.62	(1685)			.13	(.005)
	+.007" glass cloth	10.70	(1552)			.43	(.017)
12A1	Crest 7425	8.36 12.63	(1212) (1832)			.13 .13	(.005) (.005)
13A1	Epo-tek	13.55 13.84	(1965) (2007)			.13 .13	(.005) (.005)
	+.007" glass cloth	14.32	(2077)			.43	(.017)

Table 4. Shear Test Results of B-Stage Glass Cloth

	Composite Adhesive	Average Lap Shear 77K MPa	Average Lap Shear 77K (psi)	Average Lap Shear 293K MPa	Average Lap Shear 293K (psi)	Surface Preparation
1.	B-stage glass cloth[1]	20.79	(3014)	25.65	(3720)	Copper[2] - abraded with 180 grit sandpaper, alcohol cleaned
2.	B-stage glass cloth	16.21	(2350)	12.92	(1874)	Copper - sandblasted (25 grit @ 65 psi), alcohol cleaned
	G-10 (.060")					G-10 - sandblasted (25 grit @ 65 psi), alcohol cleaned
3.	B-stage glass cloth	20.01	(2901)	15.06	(2183)	Copper - sandblasted (25 grit @ 65 psi), alcohol cleaned
	Kapton H (.005)					Kapton - sandblasted (25 grit @ 65 psi), alcohol cleaned
4.	B-stage glass cloth Micarta (.031")	19.89	(2884)	23.84	(3457)	Copper - abraded with 180 grit sandpaper, alcohol cleaned
	Micarta (.058")	20.17	(2925)	25.50	(3697)	Micarta - abraded with 400 grit sandpaper, alcohol cleaned
5.	B-stage glass cloth			12.39	(1797)	Copper - abraded with 180 grit sandpaper, alcohol cleaned
	Nomex paper (.020")					Nomex - abraded with 400 grit sandpaper, alcohol cleaned
6.	B-stage glass cloth			25.59	(3750)	Copper - abraded with 180 grit sandpaper, alcohol cleaned
	G-10 (.060")					G-10 - abraded with 180 grit sandpaper and water, oven dried
7.	B-stage glass cloth			12.88	(1868)	Copper - abraded with 180 grit sandpaper, alcohol cleaned
	G-10 (.060")					G-10 - abraded with cleanser & water, oven dried
8.	B-stage glass cloth			9.66	(1400)	Copper - abraded with 180 grit sandpaper, alcohol cleaned
	Kapton H (.005")					Kapton - abraded with 400 grit sandpaper, acetone cleaned
9.	B-stage glass cloth			16.19	(2347)	Copper - abraded with 180 grit sandpaper, alcohol cleaned
	Kapton H (.005")					Kapton - abraded with 400 grit sandpaper, alcohol cleaned
10.	B-stage glass cloth			7.75	(1124)	Copper - abraded with 180 grit sandpaper, alcohol cleaned
	Kapton H (.005")					Kapton - alcohol cleaned
11.	B-stage glass cloth			12.46	(1807)	Copper - abraded with 180 grit sandpaper, alcohol cleaned
	Kapton H (.001")					Kapton - alcohol cleaned
12.	B-stage glass cloth			4.26	(618)	Copper - abraded with 180 grit sandpaper, alcohol cleaned
	G-10 (.010")					G-10 - abraded with 400 grit sandpaper, alcohol cleaned; acetone samples fell apart after curing
13.	B-stage glass cloth			3.60	(522)	Copper - abraded with 180 grit sandpaper, alcohol cleaned
	G-10 (.010)			2.71	(393)	G-10 - abraded with 400 grit sandpaper, alcohol cleaned; G-10 was then coated with coupling agents A(522)[3], B(373)[4]

1. Synthane Taylor EF 527-5m, glass B-stage cloth; samples were cut to size and used as received; medium cure cycle was used as advised by Synthane Taylor (350°F, 300-400 psi pressure for 1 hour).
2. OFHC Copper, 5/16" x 1/4" x 2".
3. Silane Z 6040 Coupling agent, Dow Corning.
4. Silane Z 6020 Coupling agent, Dow Corning.

moments on the sample are kept to a minimum. Since the lap shear area is double that of a simple lap joint, it was necessary to apply twice the load. The tensile loads were applied and recorded using a Hounsfield Tensometer. The quick grips provided with the instrument were used at room temperature while special clamping grips, which had to be designed for use in the liquid nitrogen tank, worked successfully at 77 K.

RESULTS

The results of the lap shear tests, listed in Table 3 for the varnish and epoxies and in Table 4 for the B-stage glass cloth, include the adhesive type, adherend type, lap shear strength at 77 K and 293 K, and average bond thickness. All the epoxy samples tested are listed, and no attempt has been made to average the results. Large variations, in general, can be explained by misalignment of adherends during fabrication. The available small amounts of the Hexcel, Epo-tek, Crest, and Versamid epoxies limited the sample fabrication to two copper and two aluminum samples, which were tested at liquid nitrogen temperatures only.

Many of the aluminum adherends used in the epoxy tests fractured at room temperature because the lap shear strength of the specific epoxy was not known at the time of assembly. The liquid nitrogen shear strength values are valid because the shear strength of the specific epoxy either decreased or remained constant from 293 K to 77 K, whereas the ultimate tensile strength of the aluminum increased. Nonetheless, the room temperature values are still valuable as low minimum shear strengths. Fractured aluminum adherend samples are indicated by a greater than sign (>) indicating that the lap shear strength of the epoxy was higher than the ultimate tensile strength of the aluminum adherend at room temperature. Once the problem was identified thicker aluminum adherends were used.

The highest shear strength of the epoxies was obtained with the Epon 871-828 mixture plus quartz, silane, and glass cloth and using stainless steel adherends. The same epoxy without the glass cloth had about a 35% reduction in shear strength. Only two other epoxies had a shear strength greater than 13.8 MPa (2000 psi): an Epon 815-Versamid 140 epoxy (# 4SS) and the Epo-tek epoxy (# 13A1). The addition of the

Dow Corning Silane Z 6020 to the Epon 871-828 epoxies increased shear strength about ten-fold between room temperature and liquid nitrogen. In most all cases, the addition of glass cloth increased the shear strength from 1% to 37% depending on the epoxy and the adherend type. With a similar testing epoxy and cryogenic environment, the stainless steel-epoxy samples had the highest shear strengths, the aluminum-epoxy next, and copper-epoxy the lowest shear strength.

Average lap shear strengths are shown for the B-stage glass cloth because of the small variation in the values (about 10%) and the large number of samples tested. Room temperature lap shear tests were performed first and the most promising samples were later tested in liquid nitrogen. B-stage glass cloth was used in most of the samples with other insulation materials because it is in this composite adhesive form in which it will most likely be utilized. By itself, the cured B-stage glass cloth is porous when viewed under a microscope, but with the addition of an insulating film between layers of the glass cloth, electrical insulation is guaranteed with little loss in mechanical strength at 77 K.

In general, the copper adherends needed to be abraded or sandblasted to remove the oxidation layer and then wiped with or stored in alcohol until fabrication. Each of the insulations required a particular surface preparation for maximum adhesion to the B-stage glass cloth. Good adhesion was found in all the insulations except G-10 by sandblasting or abrading the insulation surface and then cleaning with alcohol; G-10 required a simple abrasion with water and oven drying for best results. Acetone cleaning in every case greatly reduced the shear strength; in fact, the acetone cleaned G-10 samples fell apart after curing.

The results in Table 4 show that the samples fabricated with the B-stage glass cloth only had the highest strengths at 77 K and 293 K, with the actual strength decreasing about 20% from 293 K to 77 K. The Micarta B-stage glass cloth composite had the next highest values with a similar 20% decrease in strength from 293 K to 77 K. The G-10 and the Kapton composites with B-stage glass cloth had acceptable strengths at 293 K but shear strength increased about 30% from 293 K to 77 K.

CONCLUSIONS

The adhesive with the highest lap shear strength at both room and liquid nitrogen temperatures was a B-stage glass cloth bonded to copper. Shear strengths of 25.65 MPa at room temperature and 20.79 MPa in liquid nitrogen were measured for the B-stage glass cloth.

The highest lap shear strength of the epoxies was an Epon 871-828 formulation with a 17.4 MPa shear strength at 77 K. An Epon 815-Versamid 140 formulation was next with a shear strength of 16.94 MPa at 77 K. Both of these samples used stainless steel for the adherends.

Sophisticated etching solutions and cleaning techniques did not substantially increase the shear strength over simple abrasion and degreasing in trichloroethylene. In relation to coil fabrication, a sandblasting unit with a spray degreaser would be sufficient for surface preparation. It would be necessary to provide an inert atmosphere around the cleaned surface if an extended period of time elapsed between surface preparation and coating or winding to avoid possible oxidation or contaminant buildup.

It is thought that by further increasing the percentage of silane coupling agent, by adding to the epoxy other fillers which would more closely match the adherend thermal contraction coefficient, and by differing the percentage of glass cloth, epoxy combinations with larger shear strengths can be found.

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