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Mechanical Characterization of Selected Adhesives and Bulk Materials at Liquid Nitrogen and Room Temperatures

3. AUTHOR: (List All Authors; if not an ORNL employee, indicate address on a separate line)

C. M. Fitzpatrick, W. C. T. Stoddart

4. PURPOSE: DO NOT ABBREVIATE Titles of Meetings, Journals, Universities

(a) Oral Presentation (Give name, place, and exact dates of meeting)

Paper to be presented at the 7th Symposium on Engineering Problems of Fusion Research, Oct. 25-28, 1977, Hyatt Regency, Knoxville, TN

To be published in program, booklet, brochure, etc. ☐ Yes ☐ No ☒ Not Known

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DEADLINE DATE **October 21, 1977** DIVISION **Fusion Energy** AUTHOR _____

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MECHANICAL CHARACTERIZATION OF SELECTED ADHESIVES AND BULK MATERIALS AT LIQUID NITROGEN AND ROOM TEMPERATURES*

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Summary

This paper presents the results of a series of mechanical tests on selected adhesives and bulk materials. The materials tested are of general interest to designers of magnets for cryogenic service and include several epoxies, a varnish, a B-stage glass cloth, insulation papers, and commercially available fiber-reinforced composites. These tests were performed at room temperature (293 K) and at liquid nitrogen temperature (77 K). The tests include both simple tension tests and lap shear tests with various adherends. The parameters critical to tensile or bond strength were varied as part of the test program. The procedures used to manufacture and test these specimens and the results of the tests are reported in this paper.

Introduction

Organic materials will be used in future superconducting machinery for electrical insulation and/or structural strength. The application of the organic material, whether for structural support, spacing, thermal insulation, or electrical insulation, will determine just how strong the material must be. One particular application of an organic material is as an adhesive to provide good mechanical strength and electrical insulation between turns or layers of the superconducting coils to be fabricated for tokamak type fusion machines. A program was initiated by the Materials Evaluation Group of the Superconducting Magnet Development Program to evaluate the strengths of selected adhesives and bulk samples of organic materials 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 77 K to 4.2 K.

The tests performed were primarily ultimate tensile strength of bulk specimens, and lap shear strength, where the adhesive, adherend, and fabrication procedure were varied. The remaining sections of this paper summarize the important points of specimen fabrication, test procedure, and test results.

Ultimate Tensile Strength Tests

The materials selected for testing in this series were those thought to be useful as potting compounds or as fabricated insulation separators. Some of the materials were available in a directly usable form which could be purchased, while others had to be produced on site due to the parameters which were to be investigated. The materials investigated in these tests included Epon 828, an epoxy resin with a molecular weight affording a moderate room temperature viscosity; Epon 871, an epoxy resin especially designed for increased flexibility; and Epon curing agent Z, a liquid of approximately 20-poise viscosity which may be blended with liquid Epon resins at room temperature.

Five different mixtures of Epon 828, Epon 871, and curing agent Z were used in tensile specimens. The mixtures are given in Table 1. Also tested were Stycast 2850FT (Blue) G-30, G-50, Du Pont 101, Vespel 211, Vespel 92Y77, Tefzel, and various inculation papers.

Table 1

Mixture identification	Epon 828	Epon 871	Curing agent Z
A7135G-20	100%	0%	20 pph
A7135G-22	75%	25%	17.5 pph
A7135G-24	50%	50%	15 pph
A7135G-26	30%	70%	13 pph
A7135G-28	0%	100%	10 pph

Specimen Preparation

Appropriate amounts of each epoxy and curing agent Z were weighed in separate containers. The individual containers were placed in a desiccator, outgassed until entrapped air was pumped out (down to 300-500 torr), and then brought up to atmospheric pressure using nitrogen gas. The components were then poured very slowly down the side of a single container so as to avoid entrapping air. For the same reason, the container was stirred with round rather than overlapping movements. The resulting mixture was then poured into vacuum pouring funnels (see Item A, Fig. 1) and outgassed until bubbling stopped or became very slow, down to 300-400 torr. The specimen molds were prepared by cleaning with a degreaser, drying, and coating with a very thin layer of mold release. The molds were then preheated to about 125°F in order to minimize shrinkage and surface blemishes on the specimens. This process also seems to minimize the seam marks which occur when a split mold is used. The molds were placed in desiccators directly beneath the vacuum pouring funnels and pumped down to approximately 500 torr (see Item B, Fig. 1). The pressure in the vacuum pouring funnel was slowly raised to near atmospheric pressure by releasing nitrogen gas into the funnel (see Item C, Fig. 1). An arrangement of a ground glass valve, a gum rubber hose, and a copper tube (see Item D, Fig. 1) was employed for pouring the mixture from vacuum funnel to mold. The pouring was facilitated (see Item E, Fig. 1) by using an angle in the pouring tubes, a pouring trough screwed on to the top of the mold, and a lever clamped to the pouring tube to maneuver the tube into the proper position. After the pouring, the samples were outgassed in the desiccator for a short time, then removed from the desiccator by valving in nitrogen gas to bring the pressure slowly up to atmospheric pressure. The molds were then placed in an oven to cure at 150°F for 24 hours. The specimens were removed from the molds and checked with a polariscope for residual stresses. If signs of stress were found, specimens were annealed by slowly raising the temperature to slightly above 150°F, then cooling very slowly at an approximate rate of 5°F/hr until room temperature was reached. Specimens were smoothed with a fine emery cloth after annealing. Burnishing tools

*Research sponsored by the Department of Energy under contract with Union Carbide Corporation.

were used to eliminate seam marks or any blemishes that might cause premature failure in testing.

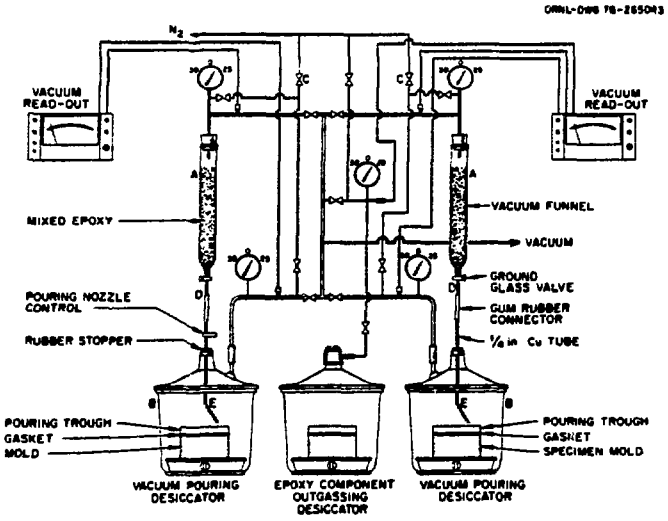

Fig. 1. System for vacuum pouring of epoxy tensile and impact specimens.

Where commercially available material was used, the specimens were simply fabricated from bulk stock.

Ultimate Tensile Strength Testing

The specimens were tested at 293 K and at 77 K (see Fig. 2). The tensile tests were carried out using a motor-driven Houndsfield Tensometer[1] with seven spring beams giving ranges with maximum loads of 2 tons, 1 ton, 1000 lb, 500 lb, 125 lb, and 62.5 lb.

Ultimate Tensile Strength Results

The measured ultimate tensile strengths of the Epon series of tests are presented in Fig. 3. The tests of other bulk materials are reported in Table 2. Additional information regarding these tests and data on measured modulus of elasticity and charpy impact strength may be found in Ref. 2.

Lap Shear Strength Tests

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.

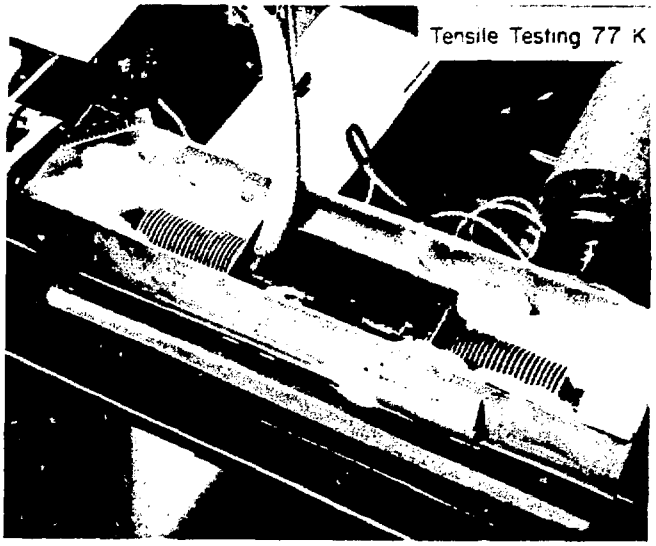

Fig. 2. Liquid nitrogen temperature tensile testing apparatus.

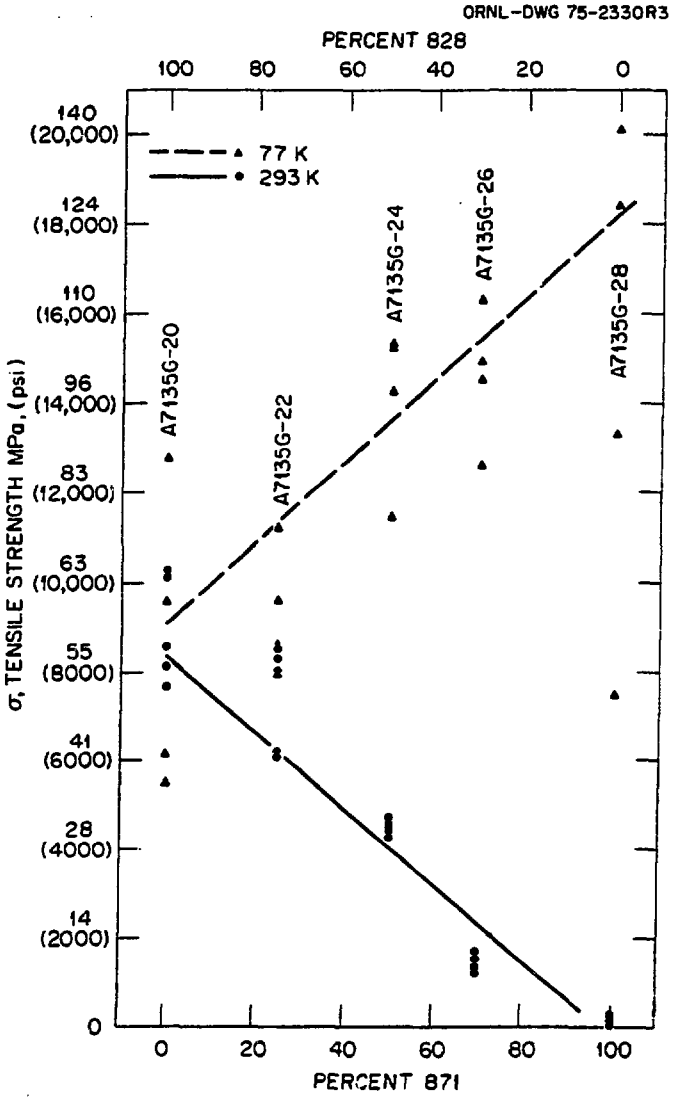

Fig. 3. Effect of constituent on tensile strength.

Table 2. Ultimate tensile strengths of bulk materials

	Ultimate tensile strength, MPa (psi)	
	293 K	77 K
Epon 30% 871, 70% 828	14 (2,000)	105 (15,000)
Stycast 2850FT	25 (7,000)	105 (15,000)
G-30	340 (50,000)	620 (90,000)
G-50	310 (45,000)	550 (80,000)
Du Pont 101	48 (7,000)	130 (18,500)
Delrin	69 (10,000)	140 (21,000)
Vespel 211	62 (9,000)	100 (14,500)
Vespel 92Y77	86 (12,500)	105 (15,000)
Tefzel	32 (4,700)	83 (12,000)
Insulation papers	19 (2,800)	45 (6,500)

Epoxies

The several epoxies used in the shear strength evaluation are listed in Table 3. 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.

Varnish

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 6031 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 gages 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 cure cycle of 360°F @ 50 psi, and the shear strength was still higher than any of the epoxies. The B-stage glass cloth was very easy to use and withstood severe abuse (handling, cutting, bending) with no noticeable decrease in shear strength.

Adherends

The base metals used in these tests were copper ASTM B 152 light cold rolled; stainless steel, 304L ASTM A 240 bright cold rolled; and aluminum ASTM B 209 AA 1100-H14.

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.

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 cure pressure of 210 kPa (30 psi).

The third and simplest of the techniques was required for the B-stage glass cloth sample. The fabrication consisted of the adherend surface preparation, 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 a 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 there was no stringent setup time.

The effects of surface preparation, ranging from sophisticated etching and cleaning solutions to simple abrasion and degreasing, were investigated along with the use of a coupling agent, Dow Corning Z 6020 (which was added to the Epon 871/828 mixture and Epon 815/Versamid 140 mixture). In addition the effects of interleaved glass cloth and a filler, 300-mesh fused quartz, were investigated.

Test Procedure

A double lap joint (see Fig. 4) was chosen for the test configuration for ease in assembly and alignment. With this arrangement, bending 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.

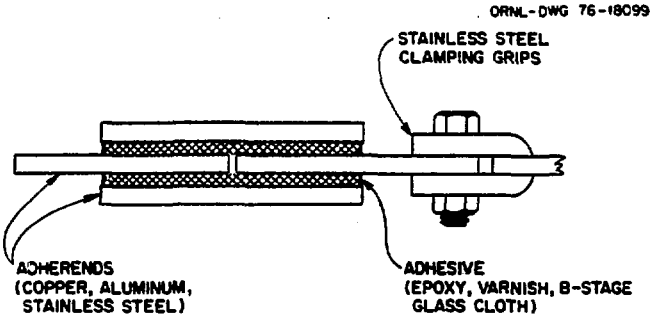

Fig. 4. Typical double lap shear sample with grips.

Lap Shear Testing Results

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 26 MPa at room temperature and 21 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 17.5-MPa shear strength at 77 K. An Epon 815-Versamid 140 formulation was next with a shear strength of 17.0 MPa at 77 K.

Table 3

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 - 13 pph	∿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 - 13 pph 0.5%/wt	∿5 hrs @ 75°F ∿2 hrs @ 150°F	24 hrs @ 150°F	Thin
Stycast 2850FT (Blue) 24 LV Hardener	Emerson and Cummings	Stycast 2850FT 100% 24 LV - 7 pph	1 hr @ 75°F	24 hrs @ 75°F	Medium thick ∿15,000 cps @ 70°F
Stycast 2850FT (Black) #9 Hardener	Emerson and Cummings	Stycast 2850FT 100%; #9 Hardener 3.5 pph	1 hr @ 75°F	24 hrs @ 75°F	Thick 90,000 cps @ 70°F
General Elec. No. 7031	General Electric	100% G.E. Adhesive and 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 Z - 13 pph 0.5% 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
Crest	Crest	100 parts "A" 26 parts "B" (by weight)	∿10 min.	2 hrs @ 250°F	Thick
Epo-Tek 920 FL	Epoxy Technology Inc.	100 parts "A" 3 parts "B" (by weight)	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 40 pph 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 Z - 13 pph 1% Silane	∿5 hrs @ 75°F	24 hrs @ 150°F	Thin
Epon 815 Versamid 140 Silane 6020	Shell General Mills Dow Corning	100 parts 40 pph 2%	4-6 hrs @ 75°F	24 hrs @ 75°F	Thin

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. Because of the extensive amount of data generated in these tests a detailed tabulation is not possible. A complete record of the results may be found in Ref. 3.

Conclusions

The tests presented have been used to screen out materials for further testing. The procedures have provided adequate data on which to base decisions for small-scale design tasks. Multimillion dollar projects like the Large Coil Program require more extensive tests than those reported here. The test program of the National Bureau of Standards has been set up to meet these greater needs.

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1. Hounsfield Tensometer manufactured and marketed by Tensometer Limited, 81 Moorland Road, Croydon, Surrey, England.
2. C. M. Fitzpatrick, "A System for Vacuum Pouring of Epoxy Tensile and Impact Specimens with a Study of the Behavior of these Specimens at 77 K and 293 K," ORNL/TM-5493, Oak Ridge National Laboratory, to be published.
3. K. J. Froelich and C. M. Fitzpatrick, "Lap Shear Strength of Selected Adhesives (Epoxy, Varnish, B-Stage Glass Cloth) in Liquid Nitrogen and at Room Temperature," ORNL/TM-5658, Oak Ridge National Laboratory (December 1976).