D—1

MECHANICAL, ELECTRICAL, AND THERMAL CHARACTERIZATION OF G-10CR AND G-11CR GLASS-CLOTH/EPOXY LAMINATES BETWEEN ROOM TEMPERATURE AND 4 K*

M. B. Kasen, G. R. MacDonald, D. H. Beekman, Jr., and R. E. Schramm

National Bureau of Standards
Boulder, Colorado

INTRODUCTION

The United States magnetic fusion energy program will require large-scale use of industrial high-pressure glass-reinforced epoxy laminates for structural supports and for electrical and thermal insulation in superconducting magnets. This choice is based on such factors as availability, cost, machinability, high strength-to-weight ratio, and low electrical and thermal conductivities. There is an immediate need for a commercial supply of these materials having predictable cryogenic performance. Therefore, the National Bureau of Standards (NBS) has cooperated with the laminating industry and the National Electrical Manufacturers' Association (NEMA) to establish specifications for cryogenic grades of NEMA G-10 and G-11 glass-cloth-reinforced epoxy laminates. These materials, designated G-10CR and G-11CR, meet existing NEMA, as well as federal and military specifications for G-10 and G-11 products. The component and manufacturing specifications have been distributed to all U. S. manufacturers through the NEMA organization. The products are currently available from one manufacturer, and others have shown an interest in their production.

NBS is currently undertaking a mechanical, electrical, and thermal property characterization of the CR-grade materials over the 295- to 4-K temperature range. Contributions to the characterization have also been made by the Massachusetts Institute of Technology (MIT), Los Alamos Scientific Laboratory (LASL), Oak Ridge National Laboratory (ORNL), and the Westinghouse Corporation. In this paper is presented the data base obtained on G-10CR and G-11CR produced for NBS by two manufacturers. The mechanical properties of CR-grade laminates produced with boron-free glass reinforcement were screened in anticipation of improved radiation resistance of this variant.

* Supported by the Office of Fusion Energy, U. S. Department of Energy.

MATERIALS

The basis for both G-10CR and G-11CR is the same glass fabric of silane-finished E glass.* Its plain weave is produced on a conventional loom by interlacing warp (length) threads (16.9 ± 1.2 per cm or 43 ± 3 per in.) and fill (width) threads (12.6 ± 0.8 per cm or 32 ± 2 per in.). The specially made, boron-free E glass has a similar configuration.

The G-10CR matrix is a conventional, heat-activated, amine-catalyzed, bisphenol-A, solid-type epoxy resin chosen for proven performance at cryogenic temperatures. The G-11CR matrix is an aromatic-amine-hardened, bisphenol-A, liquid-type epoxy resin expected to provide improved resistance to radiation damage. The finished products are a natural beige color. Resin weight fraction is 32 to 36% for G-10CR and 28 to 33% for G-11CR. Production details are presented by Benzinger [1].

Two producers, designated manufacturers A and B, supplied sheets of G-10CR and G-11CR in nominal thicknesses of 0.127 cm (0.050 in.), 0.381 cm (0.150 in.) and 2.54 cm (1 in.). Manufacturer B also provided similar panels in the boron-free variant. Manufacturer A produced the materials as a pilot-plant run. Although pilot-plant procedures closely parallel production conditions, the manufacturer subsequently also provided actual production panels for additional testing.

The mechanical tests performed on these materials are summarized in Tables I and II. As noted in Table I and Figs. 1 through 3, the pilot-plant production panels of manufacturer A were used to establish base mechanical properties for tension (warp, fill), compression (warp, fill, normal), and shear (warp, fill). Results of the screening tests on the mechanical performance of material from manufacturer B appear in Table II. These tests were performed only in the warp direction. The pilot-plant production panels of manufacturer A were also used in the study of electrical performance (Table III), thermal conductivity (Fig. 4), and thermal expansion (Fig. 5).

TEST METHODS

Mechanical Tests

Tensile specimens cut from the 0.127-cm sheets were a modified version of the MIL-HDBK 17A specification. The gauge length was 5.4-cm (2.125-in.) long and 1.27-cm (0.5-in.) wide. The specimen width tapered to 2.54 cm (1 in.) in the grip area. Resistance strain gauges cemented directly to the surface in the longitudinal and transverse orientations allowed measurement of Young's modulus and Poisson's ratio. The ultimate strength and elongation were also determined.

Warp and fill compression-strength specimens from the 0.381-cm sheets were 6.99-cm (2.75-in.) long and 0.794-cm (0.313-in.) wide; specimens tested normal to the glass cloth had about the same cross section but were only 2.54-cm (1-in.) long. With end caps cemented in place, the gauge length was 1.91 cm (0.75 in.). The compression method and fixture were described elsewhere [2].

Interlaminar shear strength tests followed two methods. Three-point bending (ASTM D 2344-72) was the simplest and good for comparison or quality checks but provided no real engineering data, since tensile and compressive stresses were also present. The specimens were 2.54-cm (1-in.) long by 0.64-cm (0.25-in.) wide and

* The use of trade names in no way implies endorsement or approval by NBS and is included only to identify the specimen materials completely.

Table I. Mechanical Characterization of G-10CR and G-11CR (Manufacturer A; Pilot Plant)*

Temperature, K	Young's modulus, E, GPa†		Poisson's ratio, ν		Tensile strength, σ^{TU}, MPa†		Compressive strength, σ^{CU}, MPa†			Tensile failure strain, ε^{TU}, %		Shear strength, σ^{St}, MPa† Short beam		Shear strength, σ^{St}, MPa† Guillotine	
	Warp	Fill	Warp	Fill	Warp	Fill	Warp	Fill	Normal	Warp	Fill	Warp	Fill	Warp	Fill
							G-10CR								
295	28.0	22.4	0.150	0.144	415	257	375	283	420	1.75	1.55	60.1	45.2	42.3	
76	33.7	27.0	0.190	0.183	825	459	834	557	693	3.43	2.53	131	93.4	61.3	72.9
4	35.9	29.1	0.211	0.210	862	496	862	598	749	3.67	2.70		105	72.6	78.8
							G-11CR								
295	32.0	25.5	0.157	0.146	469	329	396	315	461	1.82	1.73	71.9	44.9	40.6	
76	37.3	31.1	0.223	0.214	827	580	804	594	799	3.21	2.85	120	92.0	56.5	56.6
4	39.4	32.9	0.212	0.215	872	553	730	632	776	3.47	2.67		89.3	56.2	57.0

* Average of at least three specimens.
† 1 MPa = 145 psi; 1 GPa = 1.45×10^5 psi.

Table II. Mechanical Screening of G-10CR and G-11CR; three batches*

Temperature, K	Young's modulus, E, GPa† Warp	Poisson's ratio, ν Warp	Tensile strength, σ^{TU}, MPa† Warp	Compressive strength, σ^{CU}, MPa† Warp	Tensile failure strain, ϵ^{TU}, % Warp	Shear strength, σ^{SI}, MPa† Short beam Warp	Shear strength, σ^{SI}, MPa† Guillotine Warp
		G-10CR (manufacturer A; production plant)					
295	27.9	0.175	429	374	1.89	57.2	49.7
76	31.4	0.222	878	695	3.54	136	73.6
		G-11CR (manufacturer A; production plant)					
295	30.2	0.175	463	400	1.86	67.6	48.1
76	34.7	0.225	932	631	3.51	111	60.9
		G-10CR (manufacturer B; production plant)					
295	28.5	0.156	419	421	1.62	58.3	42.2
76	34.1	0.219	877	730	3.21	133	61.8
		G-11CR (manufacturer B; production plant)					
295	33.1	0.176	486	450	1.66	65.9	39.5
76	37.3	0.228	975	721	3.47	121	59.4
		G-10CR (manufacturer B; boron free)					
295	24.0	0.185	358	383	1.63	60.2	44.5
76	28.9	0.236	657	749	2.86	133	70.8
		G-11CR (manufacturer B; boron free)					
295	35.0	0.184	477	343	1.58	61.2	41.4
76	40.6	0.233	982	829	3.12	114	58.1

* Average of two specimens.
† 1 MPa = 145 psi, 1 GPa = 1.45×10^5 psi.

came from the 0.381-cm-thick sheets. The guillotine shear method (ASTM D 2733-70) can provide more meaningful values of strength, but it, too, is subject to considerable inaccuracy, unless the results are applied carefully. Data appearing in Tables I and II were obtained with a modified ASTM specimen [saw cuts on opposite surfaces were 0.953-cm (0.375-in.) apart] with side supports established at a level yielding consistent results regardless of specimen thickness. Without this support, an apparent peeling of each side from the shear plane reduced the measured strength by 50%.

Conditioning of all specimens prior to testing (ASTM D 3039-71) was at least 40 hr at 22 ± 2°C and 50 ± 10% relative humidity.

Electrical Tests

Volume resistivity tests conducted by Westinghouse according to ASTM D 257 used 1000-Vdc and 7.62-cm(3-in.)-diameter electrodes with a 0.64-cm (0.25-in.) guard gap. For the electric strength, 60 Hz was applied at 0.5 kV/s rise with 1.27×3.81-cm (0.5×1.5-in.) painted electrodes.

Thermal Tests

Thermal conductivity measurements were conducted by the longitudinal heat-flow method at closely spaced temperatures [3]. The specimens were 1.91-cm (0.75-in.) cubes with a density of 1.904 g/cm^3 for G-10CR and 1.956 g/cm^3 for G-11CR.

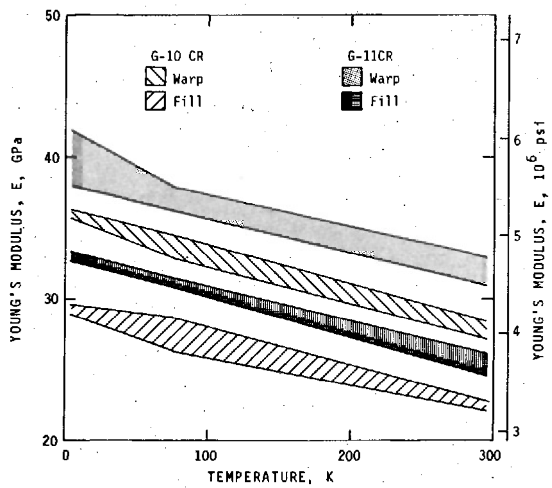

Fig. 1. Young's modulus vs. temperature for G-10CR and G-11CR (manufacturer A, pilot plant). Error bands show upper and lower data spreads.

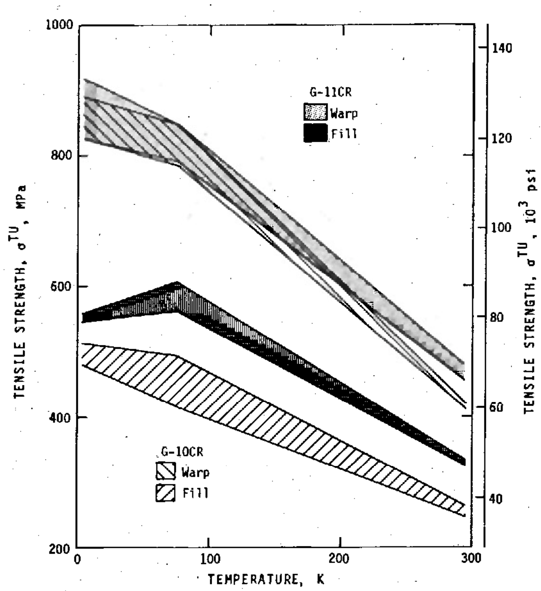

Fig. 2. Tensile strength vs. temperature for G-10CR and G-11CR (manufacturer A, pilot plant). Error bands show upper and lower data spreads.

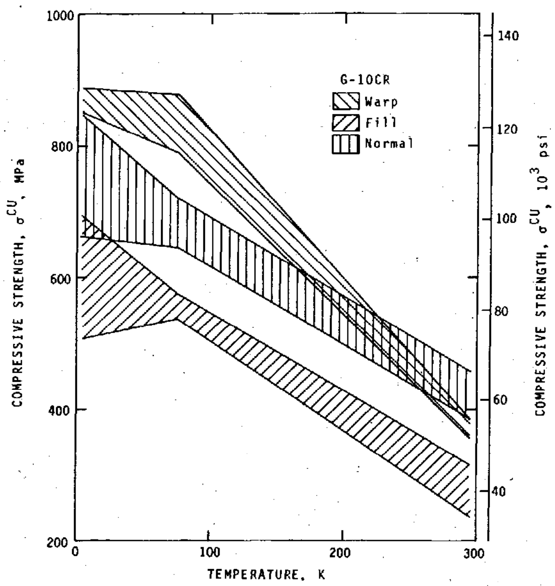

Fig. 3. Compressive strength vs. temperature for G-10CR (manufacturer A, pilot plant). Error bands show upper and lower data spreads. G-11CR displayed similar data spreads.

Table III. Electrical Properties of G-10CR and G-11CR (Manufacturer A; Pilot Plant)

Temperature, K	Thickness, cm	Volume resistivity, Ω·cm	Voltage stress at breakdown, kV/mm
		G-10CR	
295	0.030		48.4
	0.036	8.9×10^{15}	45.7
	0.051	9.3×10^{14}	
77	0.051	1.5×10^{17}	
4	0.036	4.0×10^{17}	48.4
	0.051	4.1×10^{17}	
		G-11CR	
295	0.030	7.3×10^{15}	
	0.051	1.3×10^{15}	
77	0.051	6.1×10^{16}	
4	0.030	2.0×10^{17}	48.0
	0.051	2.0×10^{17}	

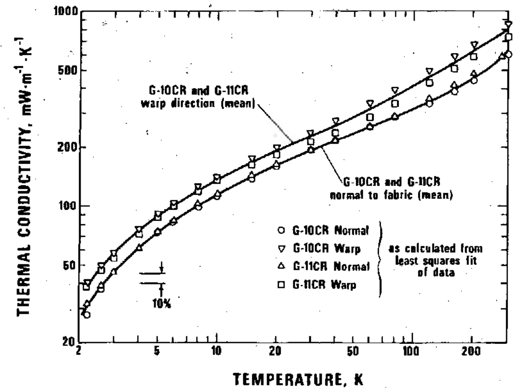

Fig. 4. Thermal conductivity vs. temperature for G-10CR and G-11CR (manufacturer A, pilot plant).

A preliminary test of total contraction between room temperature and 75 K was conducted at the Los Alamos Scientific Laboratory. Later measurements as a continuous function of temperature were performed at NBS with a quartz dilatometer.

RESULTS AND DISCUSSION

Mechanical Tests

The mechanical data in Table I are a characterization of the pilot-plant G-10CR and G-11CR from manufacturer A at all three temperatures and in both fiber directions (also normal direction for compressive strength). These are averages of three or four specimens. The standard deviations among individual tests were about

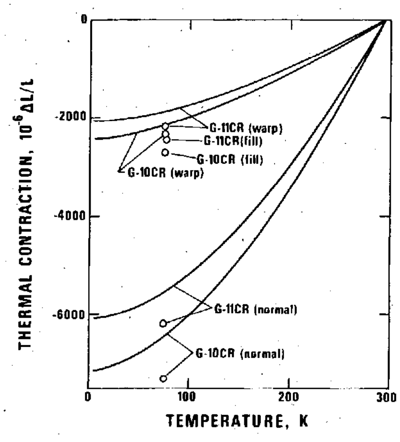

Fig. 5. Thermal Contraction of G-10CR and G-11CR (manufacturer A, pilot plant). The discrete points were from the preliminary tests done at LASL; the continuous curves were measured at NBS.

4% of the average value for the Young's modulus, tensile strength, and shear strengths, and about 9% for the Poisson's ratio, compressive strength, and strain-to-failure. Overall, there was no noticeable change in data scatter with temperature. In Figs. 1 through 3, the shaded areas indicate the extent of upper and lower data spread among tests.

Only two parameters showed any significant temperature-related material differences. G-11CR had a higher modulus in both directions and a higher tensile strength in the fill direction. The guillotine shear-strength test results may indicate some superiority in the G-10CR.

Cooling both materials from room to cryogenic temperatures increased the value of all parameters: Young's modulus about 30%, Poisson's ratio 35 to 40%, tensile and compressive strengths 70 to 130%, ultimate strain about 100% in the warp direction and 60% in the fill direction, short-beam shear strength about 100%, and guillotine shear strength in the range of 50%. G-11CR appears to be slightly less temperature-dependent than G-10CR. The elastic properties, modulus, and Poisson's ratio showed somewhat greater change with temperature for fill than warp directions, whereas the opposite occurred for the ultimate strength and elongation.

Since the fill direction yarn count is approximately 0.75 of the warp direction count, it would seem likely to find the fiber-dependent parameters occurring in roughly this ratio. Fill/warp was about 0.80 for the modulus and ultimate strain, 0.65 for tensile and compressive strengths, and 1.00 for Poisson's ratio. Shear strength is largely a function of matrix and matrix-fiber bonding, so it should be directionally independent. The guillotine tests showed no real difference between orientations, but the short-beam tests yielded a fill/warp property ratio of 0.70; this indicates that flexure produces tensile and compressive, as well as shear, forces. Some material dependence with fill/warp ratio did appear in the tensile and compressive strengths where it was somewhat higher for G-11CR than for G-10CR. The only parameter to display a temperature dependence was the failure strain; the ratio of fill/warp properties decreased in the cryogenic region.

The compressive strengths normal to the cloth plane are valuable data for high-pressure laminates since these materials are likely to be loaded in this direction in many weight-bearing applications. In general, the values seem to be comparable to those in the warp direction and show the same temperature dependence.

The screening data in Table II include only two temperatures and only the warp direction; nevertheless, they do cover composite lots from manufacturers A and B's production plants as well as the boron-free product from manufacturer B.

Some differences exist between values in the two tables. In the discussion following, all comparisons are with the complete characterization data in Table I for manufacturer A pilot-plant specimens. For G-10CR of manufacturer A's production plant, Poisson's ratio runs about 15% high, and the compressive strength at 76 K is 15% low. For G-11CR of manufacturer B's production plant, the tensile strength at 76 K is 18% high.

The majority of differences, however, occur in the boron-free products. In G-10CR the modulus is 15% low, Poisson's ratio is 23% high, tensile strength is 14 to 20% low, and failure strain at 76 K is 17% low. The differences in G-11CR are a 9%-high modulus, and a 19%-high tensile strength at 76 K. None of these differences are really dramatic, and they could be the result of normal experimental scatter and the allowable variations in yarn count and resin content.

Limited data, particularly at cryogenic temperatures, are available for comparison, and they deal mostly with S-glass cloth [4,5]. In general, they show that the present materials display a higher modulus and tensile strength by a factor of about 1/3 and a somewhat higher compressive strength and lower failure strain. Direct comparison of shear strengths is very difficult because of the many experimental variables.

Electrical Tests

Volume resistivity (Table III) showed 1.5 to 2 orders of magnitude increase on cooling to 4 K. Electric strength, however, displayed no temperature dependence and no difference between composite types.

Thermal Tests

Thermal conductivities (Fig. 4) were nearly identical for both materials. Data were about 10% higher for the warp direction than the normal to the reinforcement. Fill direction measurements would likely yield values similar to present results. Uncertainties in measurement are about ±5%. Complete details and analysis will be published later [6].

The thermal contraction data (Fig. 5) show a directional dependence possibly indicating that the epoxy was a dominating factor. A detailed analysis of the continuous data is now in preparation for publication elsewhere [7]. A preliminary measurement of total contraction between room temperature and 75 K conducted at LASL indicated approximately 10% greater $\Delta L/L$.

CONCLUSIONS

The CR-grade glass–epoxy laminates should prove useful in applications that require a commercially available structural or insulating material of predictable performance. These composites do have good mechanical properties at room temperature and below. In this and other laboratories, work continues on mechanical, electrical, and thermal characterization as well as resistance to radiation damage.

ACKNOWLEDGMENT

The authors are grateful to members of the NBS Thermophysical Properties Division who performed the thermal conductivity studies (J. G. Hust) and thermal contraction measurements (A. F. Clark and M. A. Ranney). They also wish to thank G. A. Mullen of the Westinghouse R & D Center in Pittsburgh for the electrical data and J. Harlow and R. I. Schermer of the Los Alamos Scientific Laboratory for the preliminary thermal contraction data.

NOTATION

Roman symbols

E = Young's modulus
L = length

Greek symbols

ε^{TU} = tensile failure strain
ν = Poisson's ratio
σ^{CU} = compressive strength
σ^{SI} = shear strength
σ^{TU} = tensile strength

REFERENCES

1. J. R. Benzinger, in *Advances in Cryogenic Engineering, Vol. 26,* Plenum Press, New York (1980), p. 252.
2. R. E. Schramm and M. B. Kasen, *Mater. Sci. Eng.* **30**:197 (1977).

3. J. G. Hust and J. M. Arvidson, "Thermal Conductivity of Glass Fiber/Epoxy Composite Support Bands for Cryogenic Dewars," unpublished Report 275.03-78-2, National Bureau of Standards, Boulder, Colorado.
4. M. B. Kasen, *Cryogenics* **15**:327 (1975).
5. M. B. Kasen, in *Composite Reliability*, ASTM STP 580, American Society for Testing and Materials, Philadelphia, Pennsylvania (1975), p. 586.
6. J. G. Hust, Thermophysical Properties Division, National Bureau of Standards, Boulder, Colorado, private communication.
7. A. F. Clark, Thermophysical Properties Division, National Bureau of Standards, Boulder, Colorado, private communication.