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INSULATION INTERLAMINAR SHEAR STRENGTH TESTING WITH COMPRESSION AND IRRADIATION*

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ABSTRACT

The Compact Ignition Tokamak (CIT) project identified the need for research and development for the insulation to be used in the toroidal field coils. The requirements included tolerance to a combination of high compression (380 MPa) and shear (83 MPa shear stress) and a high radiation dose (approximately 10^{10} rad). Samples of laminate-type sheet material were obtained from commercial vendors. The materials included various combinations of epoxy, polyimide, E-glass, S-glass, and T-glass. The T-glass was in the form of a three-dimensional (3-D) weave. The first tests were with 50 × 25 × 1 mm samples. These materials were loaded in compression and then to failure in shear. At 345-MPa compression, the interlaminar shear strength was generally in the range of 110 to 140 MPa for the different materials. A smaller sample configuration was developed for irradiation testing (13 by 13 mm). The data before irradiation were similar to those for the larger samples but approximately 10% lower. Limited fatigue testing was also performed by cycling the shear load. No reduction in shear strength was found after 50,000 cycles at 90% of the failure stress. Because of space limitations, only three materials were *chosen for irradiation: two polyimide systems and one* epoxy system. All used boron-free glass. The small shear/compression samples and some flexure specimens were irradiated to 4×10^9 and 2×10^{10} rad in the Advanced Technology Reactor (ATR) at Idaho National Engineering Laboratory (INEL). A lead shield was used to ensure that the majority of the dose was from neutrons. The shear strength with compression before and after irradiation at the lower dose was determined. Flexure strength and the results from irradiation at the higher dose level will be available in the near future.

INTRODUCTION

A number of commercial vendors supply glass-reinforced insulation in the form of large sheets, with sizes up to approximately 3 by 1.2 m available in a thickness of 1 mm. Fifteen samples of material from six vendors were obtained for mechanical testing. The principal test of interest was combined shear and compression. The test configurations are described below. In general, the materials were first screened by testing 25.4- by 50.8-mm samples with varying compressive loads. A smaller test configuration for use with irradiated samples was then developed using 12.7- by 12.7-mm (0.5- by 0.5-in.) samples.

Irradiations at dose levels of 4×10^9 and 2×10^{10} rad are complete. However, testing is still in progress. Preliminary results for combined shear and compression loading for the lower dose level are presented.

The material combinations tested in the first series of screening tests are given in Table I. The term

Table I. Materials tested in first series of screening tests

Material	Resin	Glass type	Vendor
Spaulrad-S	Bismaleimide	S-2	Spaulding[a]
Spaulrad-K	Bismaleimide	Kevlar 29	Spaulding[a]
G-30	Bismaleimide	E	Norplex[b]
3-D	Epoxy	E, 3-D weave	Shikishima[c]
PG2-1	Epoxy	E, 3-D weave	Shikishima
PG1-2	*Epoxy*	*E, 3-D weave*	*Shikishima*
PG3-1	Epoxy	T,[d] 3-D weave	Shikishima
PG5-1	*Bismaleimide*	T, 3-D weave	Shikishima
GB-803	Phenolic	E	NVF[e]
PI-1225	Bismaleimide	E	NVF
G-215	Epoxy	E	Electro-Isola[f]
OH-67	Epoxy	E	ABB[g]
OH-65	Epoxy	E	ABB
(No name)	Bismaleimide	E	ABB

[a] Spaulding Composite Company, Tonawanda, N.Y.
[b] Norplex Division, UOP Inc., La Crosse, Wis.
[c] Shikishima Canvas Co., Ltd., Shiga, Japan.
[d] T-glass (a product of Nitto Boseki Co., Ltd., Nittobo Glass-fiber Division) is a boron-free glass similar to S-glass.
[e] NVF Co., Kennet Square, Pa.
[f] U.S. distributor: Franklin Fibre Lamitex Corp., New Hyde Park, N.Y.
[g] ASEA Brown Boveri Technology, Inc., North Brunswick, N.J.

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"polyimide" has been used in the literature for both pure polyimide systems and for bismaleimide systems, which have a slightly different chemistry with a higher average hydrogen content.

SCREENING TEST CONFIGURATIONS

The screening tests were done at the Material Test Laboratory of Princeton Plasma Physics Laboratory. Figures 1 and 2 show the two test devices. Both use the same test method. Two insulation samples are tested simultaneously. Each sample has a grit-blasted, 2-mm-thick (0.080-in.) Inconel backing piece on each side. These assemblies are placed on each side of a central steel block. The compressive load is applied first through the two outside steel blocks. This load is produced by a hydraulic piston and threaded rods suspended around the samples. The shear load is applied on the central block and reacted by vertical supports on the side blocks. An important feature of this configuration is that shear strain is not coupled to the compressive strain. If the shear strength integrity is lost, the sample will fail catastrophically.

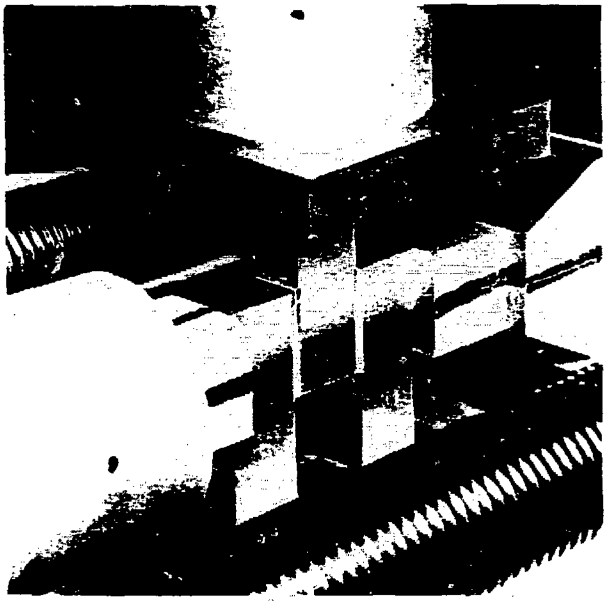

Fig. 1. Biaxial test device for 25.4- by 50.8-mm samples.

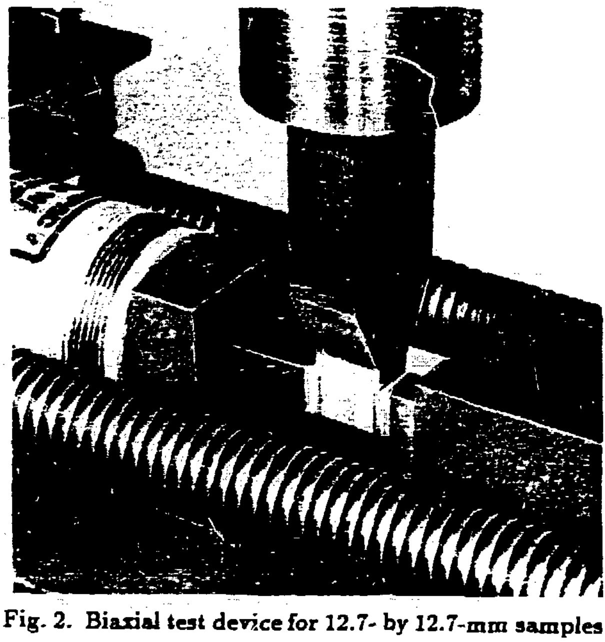

Fig. 2. Biaxial test device for 12.7- by 12.7-mm samples.

Finite element stress analysis showed significant gradients and stress concentrations near the edges of the samples [1,2]. The test method does, however, appear to give a good qualitative comparison between different materials, and at high compression levels the failure mode is frequently pure interlaminar shear, as shown in Fig. 3. The test results given here are in terms of the average stress levels.

Fig. 3. Interlaminar shear failure of Spaulrad-S.

TEST RESULTS

Testing with the larger device was done at average compressions of 69, 207, and 345 MPa (10, 30, and 50 ksi). Two pairs of samples were tested at each level. The compressive load was applied first, and then the shear load was increased until failure. At 69 MPa, all materials failed by slipping at the interface between the Inconel and the insulation. The effective coefficient of friction varied between 0.43 and 0.71 for the different materials. With 207-MPa compression, all of the materials sheared at stresses between 90 and

104 MPa except for Spaulrad-K, which reached only ≃65 MPa. (This material also showed significantly lower shear strength than the other materials at 345-MPa compression and was eliminated from further testing.) Figure 4 summarizes the test results with 345-MPa compression for both size samples. The values shown are the average of two pairs for the larger samples and three pairs for the smaller samples. Both configurations show the same trends, with the smaller samples having approximately 10% lower shear strength. In general, all of the materials demonstrated very high interlaminar shear strengths with the high compressive loads.

Samples of PG3-1 material were also tested in the fill direction. They showed a 9% lower strength.

Spaulrad-S and PG5-1 give a direct comparison of two-dimensional (2-D) vs 3-D weave performance for systems with nearly the same glass fractions and resin systems. The 3-D weave shows ≃15% higher average shear strength with 345-MPa compression. The failure modes are also different; the Spaulrad produced a true interlaminar shear, separating between two of the five layers of glass, while the 3-D weave failure was more of a tearing through the thickness.

Figure 5 shows the shear strength vs compression for Spaulrad-S in both test configurations. Also shown is the result for the test of three pairs of the smaller samples that were cut at 45° to the warp or fill direction. These samples consistently showed an increase in strength (averaging 13%) in this direction. A data point is also included for the single-axis test device described below.

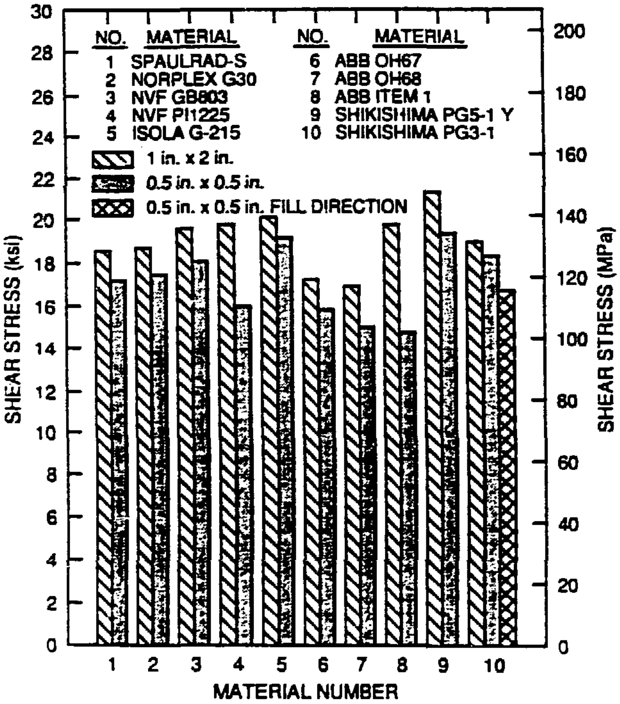

Fig. 4. Average interlaminar shear strengths of sheet materials under 50-ksi compression.

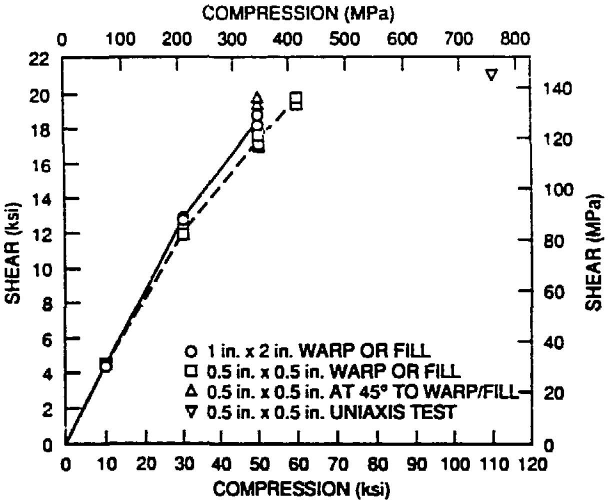

Fig. 5. Spaulrad-S interlaminar shear strength vs compression.

SINGLE-AXIS TEST DEVICE

Small samples were tested in a configuration in which the shear and compressive stress were applied simultaneously with a single-axis test machine, shown in Fig. 6. The concept was to have a sample on each side of a wedge. The angle of the wedge was chosen to give the same ratio of shear to compression as expected in the CIT device. The wedge was restrained in the horizontal direction only by the shear strength of the two insulation samples. The same type of Inconel backing pieces with the grit-blasted finish were used on each side of the samples. In a test of Spaulrad-S with this device, failure occurred at 758-MPa compression and 145-MPa shear. The failure was pure interlaminar shear. The test was repeated once with similar results (8% higher strength). Handling of the samples was difficult, however, and this test configuration was not developed further.

FATIGUE TESTING

Three tests were performed on the samples of Spaulrad-S in which the shear load was cycled at 1 Hz with a constant compressive stress of 345 MPa. The first set of two samples was tested for 3000 cycles with a maximum shear stress of 61 MPa (50% of the static

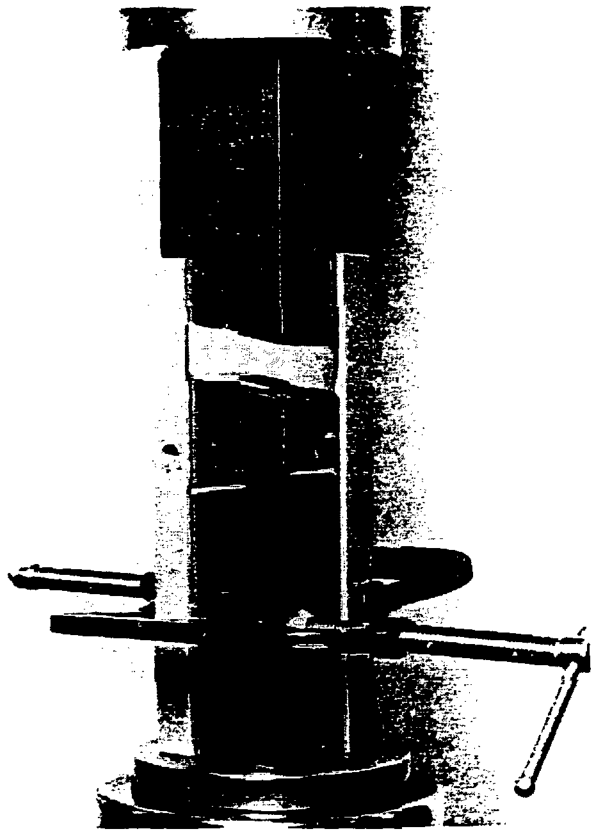

Fig. 6. Single-axis test device with 12.7-by 12.7-mm samples. External clamps are used only for alignment and then removed.

strength) and a minimum of 5 MPa. The samples did not fail, and the maximum shear stress was increased to 91 MPa for 3000 cycles. Another increase was then made to 109 MPa, and 6000 cycles were performed without failure. The shear stress was then increased to produce single-cycle failure, which occurred at 128 MPa, actually slightly higher than the static strength before cycling. A second set of samples produced similar results. A third test was done using a maximum shear stress of 109 MPa. After 48,000 cycles, the test was terminated and a static test was performed. The shear strength was 125 MPa.

A fatigue test was also performed with the single-axis device to cycle both the compressive and shear loads. At 414-MPa compression with 79-MPa shear stress, 18,000 cycles were performed without failure. With this material, it appeared that the only significant effect of cyclic shear stress at these levels was to seat the samples better with the grit-blasted Inconel surfaces.

IRRADIATION TESTING

Materials

The materials selected for irradiation testing are described in Table II. A primary consideration was to avoid including E-glass, which contains boron, because insulation using glass with boron performs poorly when exposed to a typical reactor thermal neutron flux [3,4]. Spaulrad-S and PG5-1 were chosen because both use a bismaleimide resin, which has shown good radiation resistance. Shikishima has also reported improved radiation resistance with 3-D weave materials [5], and a direct comparison can be made with a 2-D system. The third material used an epoxy resin with an aromatic amine hardener.

Irradiation Facility

Samples were irradiated in ATR at INEL (operated by EG&G). This facility has been used previously for irradiation and testing of insulation, including Spaulrad-S [3]. Two aluminum sample capsules were

Table II. Rad-I test materials

	Material		
	Spaulrad-S	PG5-1	PG3-1
Resin	Bismaleimide (Kerimid 601)	Bismaleimide triazine	Bisphenol-A
Hardener	None	None	Aromatic amine
Fiber			
Material	S-2 glass	T-glass	T-glass
Preparation	Silane	Epoxy silane	Epoxy silane
Form	Fabric	3-D weave	3-D weave
Resin, wt %	26	33	29
Glass, vol. %			
Warp direction		57	57
Fill direction		34	34
Thickness direction		9	9

fabricated with an inside diameter of 23.6 mm. Each capsule contained eighteen 12.7 × 12.7 × 1.0 mm shear/compression samples and six 50 × 5 × 1 mm flexure specimens of each material. The samples were packed with aluminum foil and powder for heat conduction to the outer surface of the cylinder to limit the peak temperature to <340 K. The lengths of the capsules were sized to limit the pressure due to outgassing, based on the outgassing rates quoted in Ref. [6] and dose levels of 10^{10} and 5×10^{10} rad. The capsules were contained within a 41.4-mm-thick cylindrical lead shield. This shield was fabricated in order to limit the gamma dose compared to the neutron dose. The shield and capsule assembly was placed in a 127-mm-diam "I-hole" such that one capsule was near the top of the core and the other was on the midplane. The upper capsule was removed after one 14-day fuel cycle; the second, after two 14-day cycles.

Radiation Dose Levels

The calculation of neutron dose is difficult and depends on many factors, including the spectrum, element material percentages, and the range of recoil particles [7]. Preliminary calculations for CIT give a peak gamma dose to a bismaleimide resin (Kerimid 601) of 2.2×10^9 rad and a neutron dose of 9.0×10^9 rad, assuming that there is 4.1 wt % of hydrogen in the resin and that all of the recoil particle energy is deposited in the resin.

The dose levels for the two capsules in ATR were calculated by M. Jacox at INEL using a Monte Carlo code and will be compared to flux wire measurements. The average fast (>1 MeV) fluence was 6×10^{17} n/cm^2, and the average total fluence was 1.6×10^{19} n/cm^2 for capsule 1. The dose levels were as follows:

	Dose (rad)	
Resin	Gamma	Neutron
Spaulrad-S	1.46×10^9	2.60×10^9
PG5-1	1.53×10^9	2.72×10^9
PG3-1	1.66×10^9	3.28×10^9

TEST RESULTS

At this time only shear/compression testing has been done on the samples from capsule 1 and on a group of control specimens.

The small shear/compression test device and procedures were modified slightly for use in the hot cell at INEL. The Inconel backing pieces and insulation samples were first bonded together on one outside edge so that the three pieces could be handled as one assembly. The steel blocks were modified with vacuum holes to hold the assembly during installation and alignment.

The test results are given in Table III. In all tests, a compression of 345 MPa was applied and then the shear load was increased to failure. No significant loss of shear strength was observed after irradiation.

Table III. Shear/compression test results

	Shear stress (MPa) with 345-MPa compression			
	Set 1	Set 2	Set 3	Average
Spaulrad-S				
Unirradiated	90[a]	124	127	125
Capsule 1	127	127	132	129
PG5-1				
Unirradiated	138	131	135	135
Capsule 1	130	136	134	134
PG3-1				
Unirradiated	121	127	121	123
Irradiated	120	119	117	119

[a] One of the Inconel backing pieces on this sample was reversed so that the smooth surface, instead of the grit-blasted face, was facing the insulation. This sample was not included in the average.

Fatigue Testing

Fatigue testing was performed with a constant 345-MPa compression and a shear load cycling at 5 Hz from approximately 14 MPa to 90% of the lowest static shear stress at failure for the material. Six tests were planned for each material: three for a control group and three for a group irradiated in capsule 1. All of the Spaulrad-S and PG5-1 samples survived 30,000 cycles without failing. The peak cyclic shear stress on the Spaulrad-S samples was 112 MPa; on the PG5-1 samples, it was 117 MPa. The three unirradiated epoxy fatigue specimens failed during the cyclic testing after 300, 900, and 1400 cycles at peak loads of 108-, 108-, and 105-MPa shear stress. Testing of the irradiated PG3-1 was postponed until additional control specimens could be tested at lower stress levels. The results of the tests performed on the Spaulrad-S and PG5-1 samples are as follows:

	Shear stress after cycling (MPa)		
	Sample 1	Sample 2	Sample 3
Spaulrad-S			
Unirradiated	123	125	127
Capsule 1	131	135	133
PG5-1			
Unirradiated	130	132	129
Capsule 1	128	132	128

Length and Weight Measurements

The 18 shear/compression samples of each material were stacked, weighed, and measured to determine thickness and weight before and after irradiation. The epoxy PG3-1 developed a twist out of plane after irradiation. Figure 7 shows this stack after irradiation. The measured increase in length is due to this twisting and not to swelling. The results were as follows:

	Weight (g)	Thickness (mm)
Spaulrad-S		
Unirradiated	5.80602	20.91
Capsule 1	5.7812 (−0.4%)	20.74 (−0.8%)
PG5-1		
Unirradiated	5.40832	18.66
Capsule 1	5 4172 (+0.2%)	18.61 (−0.3%)
PG3-1		
Unirradiated	5.19228	18.34
Capsule 1	5.1229 (−1.3%)	19.38 (twisted)

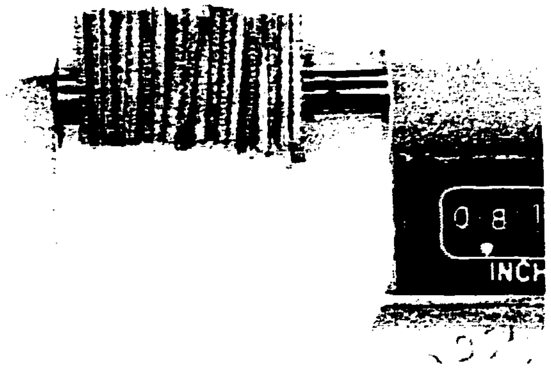

Fig. 7. Shear/compression samples after irradiation to 4×10^9 rad.

CONCLUSIONS

All of the commercial glass-reinforced insulation sheet materials demonstrated high interlaminar shear strength (above 100 MPa) when tested in combination with 345 MPa of compression. The Spaulrad-S and Shikishima PG5-1 systems with boron-free glass and bismaleimide resin systems have shown no reduction in strength, statically or in fatigue, when tested in combined shear and compression after irradiation at room temperature to a total dose level of 4×10^9 rad with over 60% of the dose from neutrons. The aromatic amine hardened epoxy PG3-1 showed some effects, such as darkening and warping, from the irradiation, but static strength was nearly the same. Further testing is needed for the epoxy system to verify the fatigue limits.

ACKNOWLEDGMENT

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