

**Annex to**  
**Design Requirements and Guidelines Level 1 (DRG1)**

**Structural Material Database**

**Article 5. Qualification of Irradiated Insulation  
Materials (Document No. 2)**

## Experimental Results and Discussion

The results from all tests have been presented and discussed in a series of five Updates issued during the time period 10 October 1993 through 30 June 1995. The Updates are included in this section. Several technical papers<sup>15-21</sup> have been written that present selected results of this program.

### ITER Insulation Irradiation Program – Update I (10 October 1993)

The first irradiated specimens of the ITER insulation program have been tested. Following irradiation at 4 K in the EC Garching fission reactor, the specimens were warmed and stored at room temperature for about three months, shipped to the United States, and tested at 4 K by Composite Technology Development (CTD).

Specifically, the specimens were irradiated at 4 K for periods of 9, 17, and 30 h  $\pm$  5 min. At the in-core, low-temperature capsule, the thermal neutron flux was  $28 \times 10^{16}$  n/m<sup>2</sup>/s, and the gamma dose rate was 700 Gy/s. The fast neutron flux ( $\geq 0.1$  MeV) was  $29 \times 10^{16}$  n/m<sup>2</sup>/s. The three irradiation runs of the first test series were conducted in June 1993; the specimens were tested in the United States in September 1993.

The specimens were a short-beam shear type, 25 mm  $\times$  6.3 mm  $\times$  3.2 mm. Short-beam shear tests were conducted at 4 K; from these tests, the interlaminar shear strength and flexural modulus were measured. Sections of one specimen from each material were cut to produce compression specimens, 3.2 mm  $\times$  6.3 mm  $\times$  6.3 mm. Compression tests were conducted at 4 K, and compressive strength and modulus were measured.

The materials were S-2-glass reinforced in the as-received (AR) condition—coated with epoxy-compatible silane, and in the desized (DS) condition—stripped of the coating by heating at 400°C for 4 h. Three epoxy-base resin systems were included in this test series:

1. A flexibilized DGEBA epoxy system formulated by CTD (CTD-101K) that is suitable for vacuum-pressure impregnation (VPI) of magnet coils.
2. A DGEBA epoxy system formulated by Shell (Shell 826) that is also suitable for the VPI process.
3. A TGDM epoxy system formulated by CTD (CTD-112P) that can be used as a prepreg resin.

The following table summarizes the material components of the resin systems.

Materials for Insulation Systems

Label	Material	Trade Name (Supplier)	Fabrication Method	
			Process*	Cure Schedule
DGEBA	diglycidyl ether of bisphenol A epoxy, anhydride curing agent	Shell 826 (Shell)	VPI	150°C/4 h
flex. DGEBA	flexibilized diglycidyl ether of bisphenol A epoxy, anhydride curing agent	CTD-101K (Composite Technology Development)	VPI	135°C/1.5 h
TGDM	tetraglycidyl diaminodiphenyl methane epoxy, amine curing agent	CTD-112P (Composite Technology Development)	PP	177°C/2 h
AR glass	S-2 glass, 6781, 8-harness satin weave, as received with silane finish	S-2 glass (BGF Industries)		
DS glass	S-2 glass, 6781, 8-harness satin weave, desized at 400°C/4 h	S-2 glass (BGF Industries)		

\* VPI = vacuum-pressure impregnation; PP = prepreg (preimpregnation)

The results, which are presented in Figures I-1 through I-5, show that

1. The shear strength is very sensitive to low-temperature irradiation, probably controlled by fiber/matrix-interface failure mechanisms.
2. The flexural modulus is also sensitive to irradiation; it decreases about 50% at  $3.1 \times 10^{22}$  n/m<sup>2</sup>.
3. The through-thickness compressive strength is rather insensitive to irradiation and is most influenced by fiber volume fraction.
4. The through-thickness elastic moduli of the DGEBA epoxy systems are sensitive to irradiation level, and this sensitivity probably reflects the contribution of the resin matrix. The elastic modulus of the TGDM epoxy system is nearly independent of irradiation.
5. The ductility in compression remains above 5% at all irradiation levels for all resin systems.
6. Typical resin systems suitable for use in the fabrication and insulation of the TF coils cannot withstand fast neutron fluences much above  $1 \times 10^{22}$  n/m<sup>2</sup> and maintain mechanical integrity under significant shear stress.

These results represent 3 irradiation runs. A total of 20 runs are in progress and will be reported periodically during the following nine months. Inorganics, as well as other organics, are included in this test program. Electrical barriers, such as plasma-sprayed inorganics, polyimide films, and mica are also included in many insulation systems within this program.

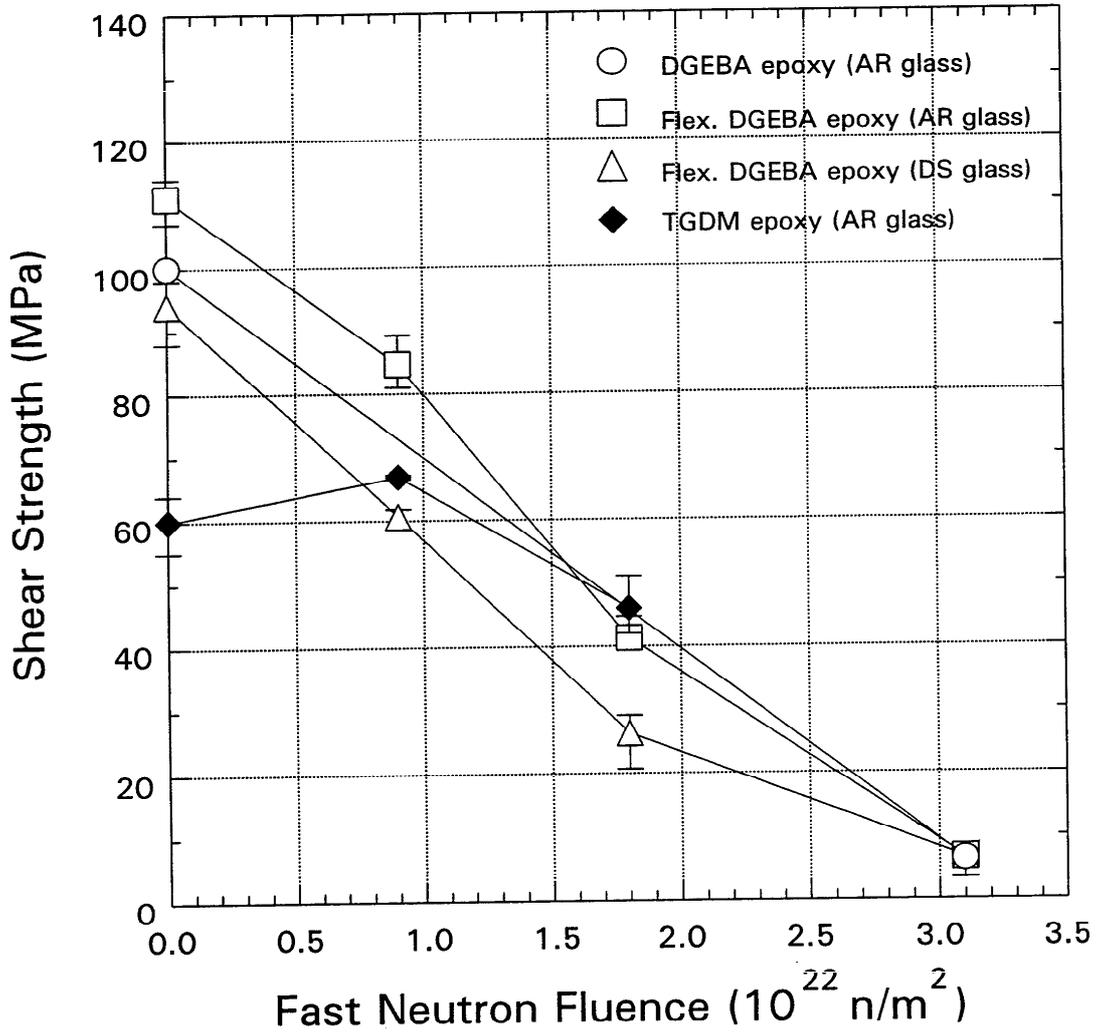


Figure I-1. The interlaminar shear strength from short-beam shear tests at 4 K as a function of fast neutron fluence at 4 K.

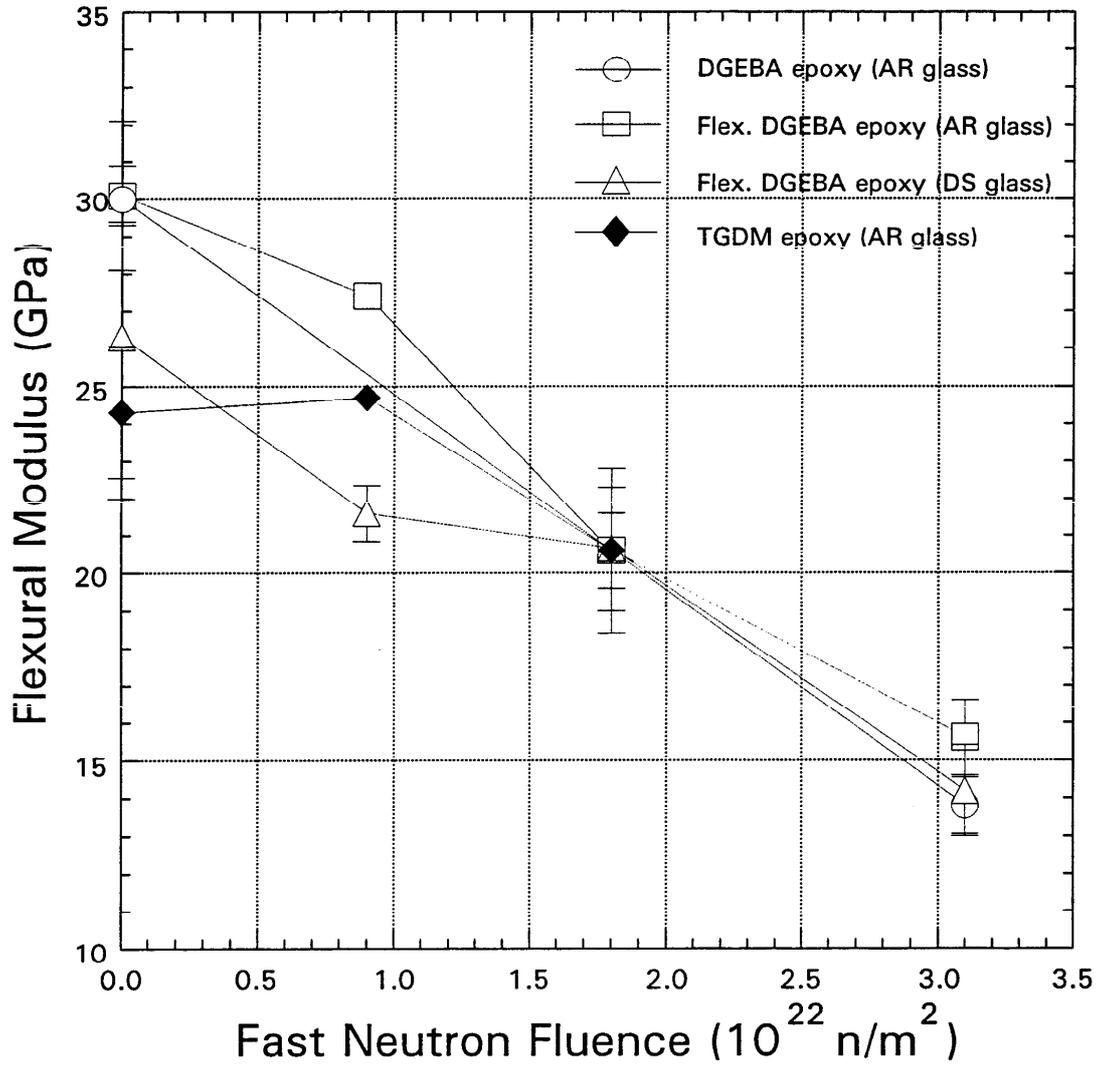


Figure I-2. The flexural modulus from short-beam shear tests at 4 K as a function of fast neutron fluence at 4 K.

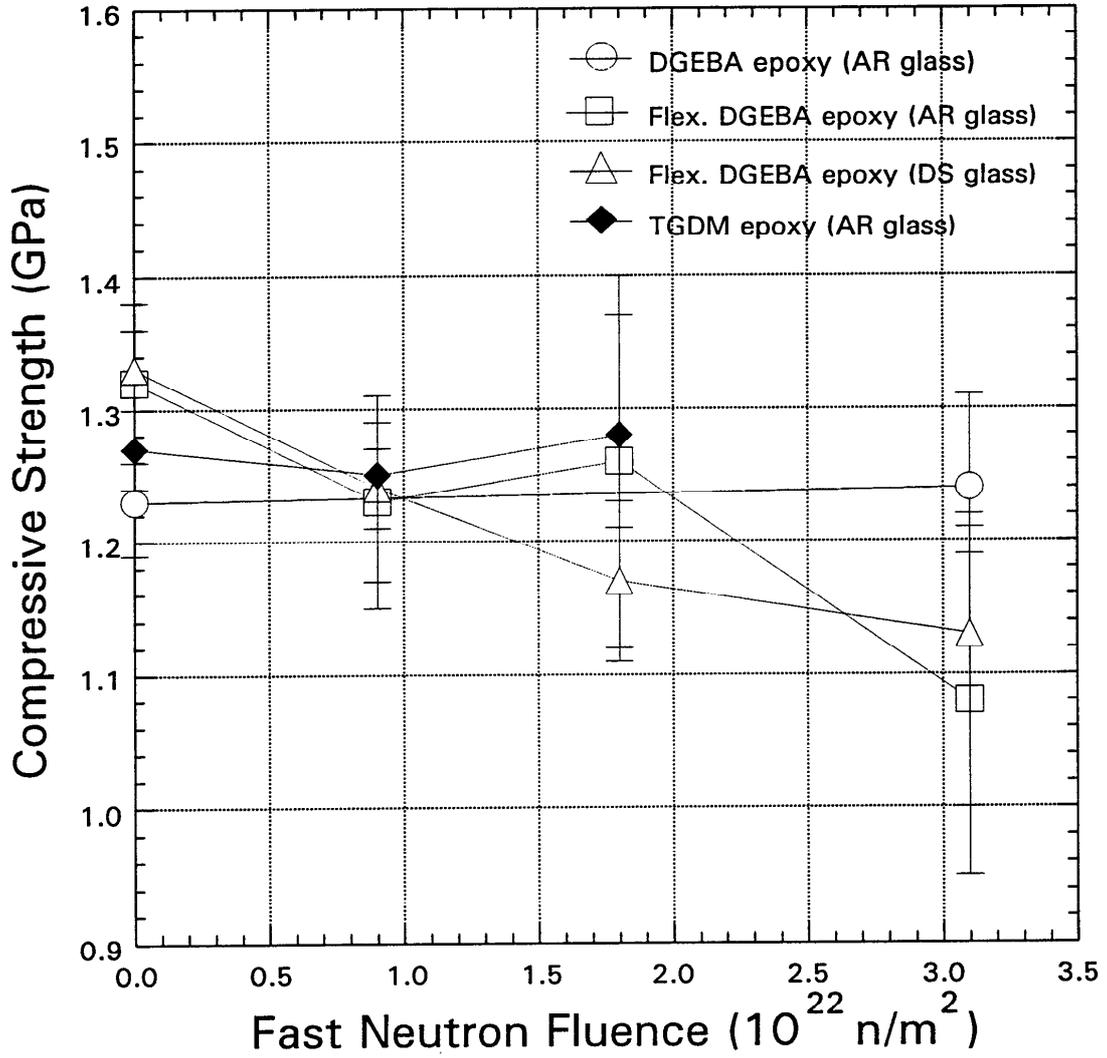


Figure I-3. The compressive strength at 4 K versus fast neutron fluence at 4 K.

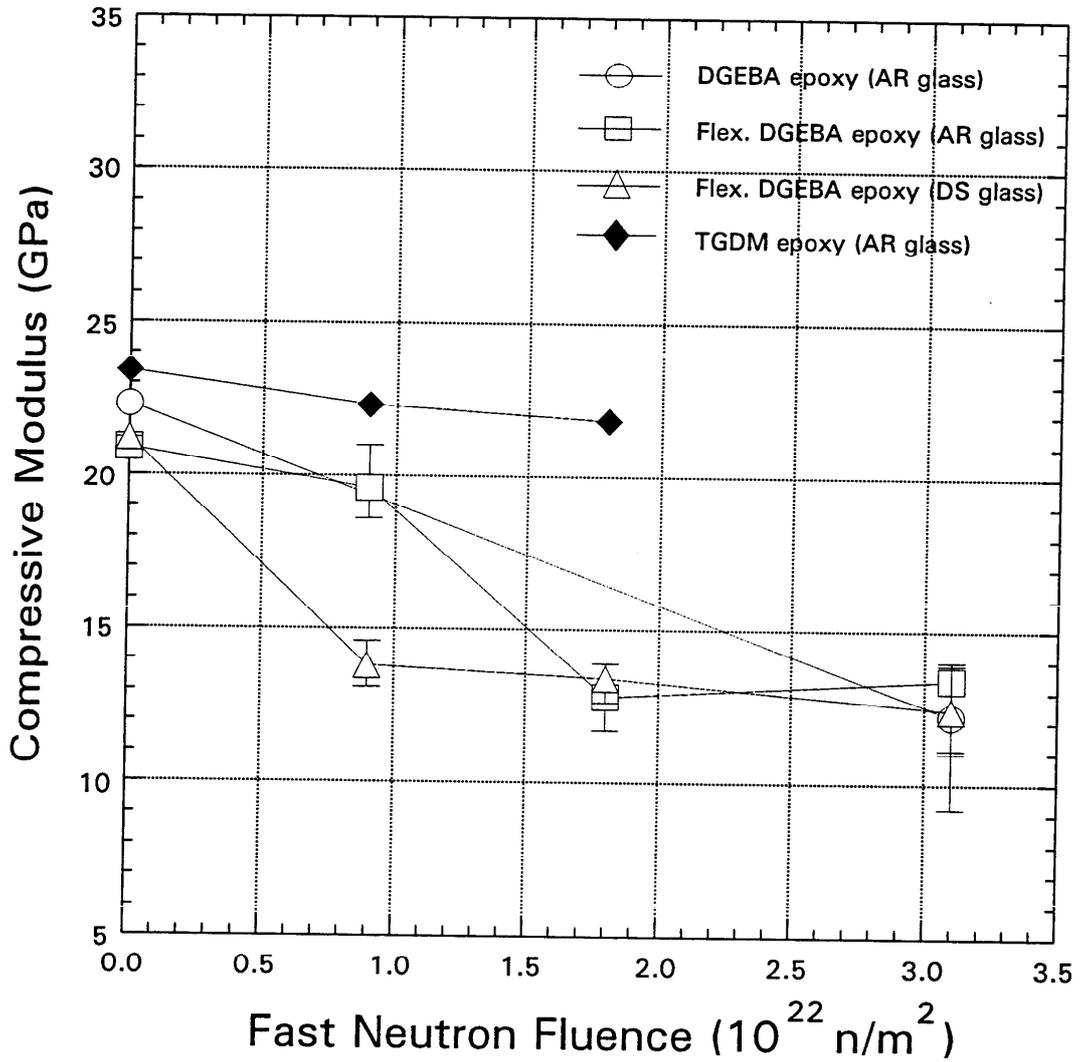


Figure I-4. The compressive modulus at 4 K versus fast neutron fluence at 4 K. These values have been adjusted upward from Appendix B data based on more recent calibrations of displacement of the load fixture.

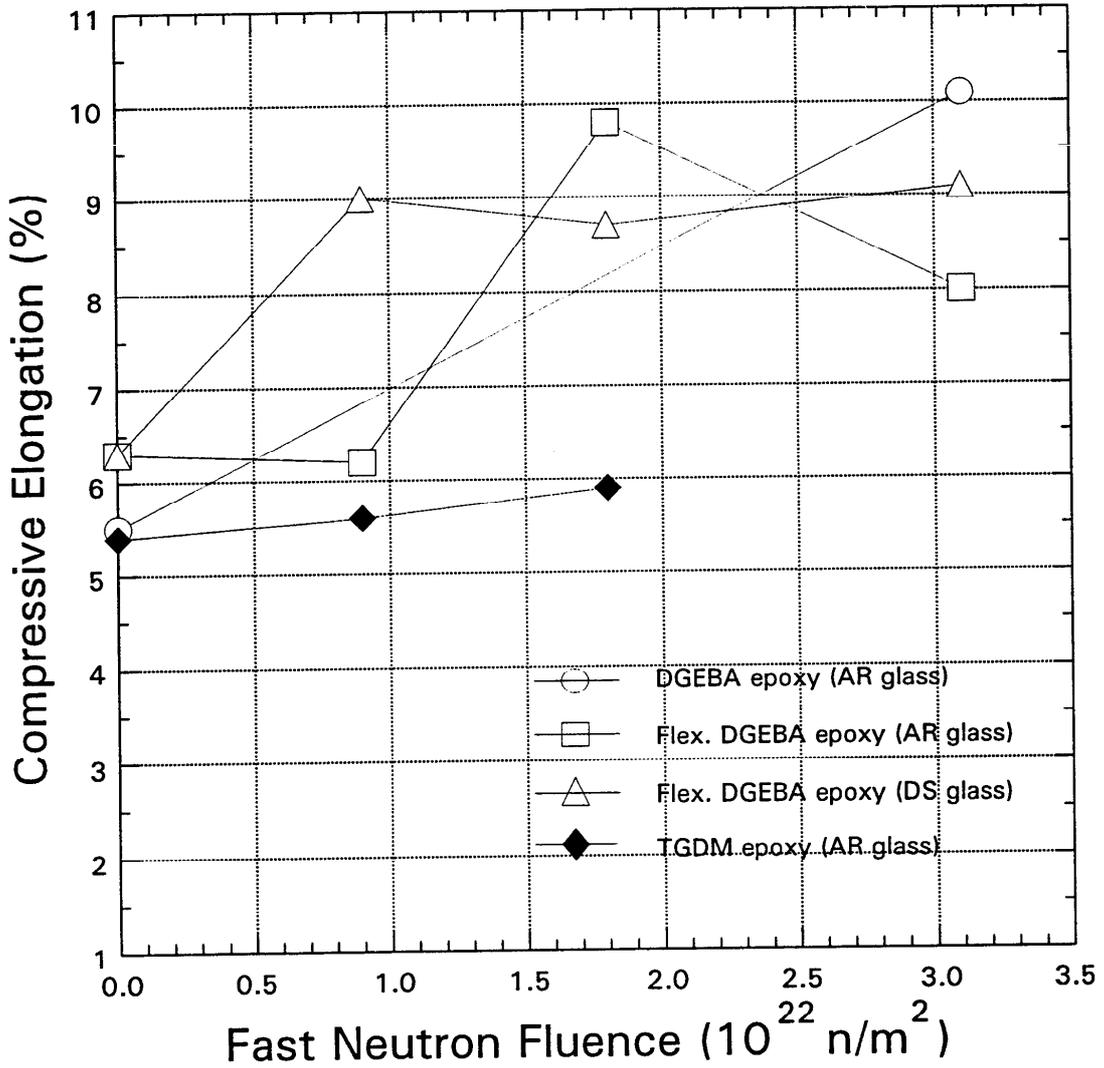


Figure I-5. The compressive elongation to failure at 4 K versus fast neutron fluence at 4 K. These values have been adjusted from Appendix B data based on more recent calibrations of displacement of the load fixture.

## ITER Insulation Irradiation Program – Update II (20 June 1994)

The second set of irradiated specimens of the U.S. ITER insulation program has been received and tested in the United States. Following irradiation to a fast neutron fluence of  $1.8 \times 10^{22}$  n/m<sup>2</sup> with an associated gamma dose of about  $2.1 \times 10^7$  Gy at 4 K in the EC Garching fission reactor, the specimens were warmed and stored at room temperature for about three months, shipped to the United States (Boulder, Colorado), and tested at 4 K at Composite Technology Development.

All materials were reinforced with S-2 glass (satin weave) in two conditions: (1) as-received (AR), coated with an epoxy-compatible silane and (2) desized (DS), with the coating removed by heating at 400°C for 4 h. The resin systems in this test series were preimpregnated and tested with and without barriers. They were a TGDM (tetrafunctional) epoxy, CTD-112P; a polyimide, AFR-700; and a bismaleimide, CTD-220P. The barriers were mica, polyimide film, and a combination of mica and polyimide film. All materials are summarized in Table II-1. The data for the flexibilized DGEBA systems were obtained during the first test series (see Update I) and are included for comparison.

The irradiated specimen dimensions were 25 mm long  $\times$  6.3 mm wide  $\times$  3.2 mm thick. Three to four short-beam shear tests per material were conducted at 4 K with these specimens. Application of three-point bend loading was normal to the plane of the glass-weave and barrier. Shear strength and flexural modulus were measured from these tests.

Sections of one specimen from each material were cut to produce compression specimens, 3.2 mm thick  $\times$  6.3 mm  $\times$  6.3 mm. The compressive strength ( $\sigma_c$ ) and modulus ( $E_c$ ) were measured, and the strain to failure ( $\sigma_c/E_c$ ) was calculated with the assumption of a linear stress-strain relationship. Compressive strain was measured with one 25-mm gage length, strain-gage extensometer fastened to each load platen.

Test results are presented in Figures II-1 through II-6. From the first test series (Update I, 10 October 1993), data for the flexibilized DGEBA epoxy for the same irradiation fluence are included to facilitate comparison of all systems.

Table II-1. Materials for Insulation Systems

Label	Material	Trade Name (Supplier)	Fabrication Method	
			Process *	Cure Schedule
DGEBA	diglycidyl ether of bisphenol A epoxy, anhydride curing agent	Shell 826 (Shell)	VPI	150°C/4 h
flex. DGEBA	flexibilized diglycidyl ether of bisphenol A epoxy, anhydride curing agent	CTD-101K (Composite Technology Development)	VPI	135°C/1.5 h
TGDM	tetraglycidyl diaminodiphenyl methane epoxy, amine curing agent	CTD-112P (Composite Technology Development)	PP	177°C/2 h
PI	polyimide	AFR-700 (Hexcel)	PP	150°C/1 h + 310°C/1 h
BMI	bismaleimide	CTD-220P (Composite Technology Development)	PP	177°C/1 h + 250°C/ 6 h
AR glass	S-2 glass, 6781, 8-harness satin weave, as received with silane finish	S-2 glass (BGF Industries)		
DS glass	S-2 glass, 6781, 8-harness satin weave, desized at 400°C/4 h	S-2 glass (BGF Industries)		
M mica	calcined, epoxy binder, S-2-glass fabric	MM 553 (Midwest Mica)		
IM mica	calcined, epoxy binder, S-2-glass fabric	IMI 498-37B (Insulating Materials)		
PI	polyimide film, 0.025 mm thick, relatively amorphous structure	Kapton HA (DuPont)		

\* VPI = vacuum-pressure impregnation; PP = prepreg (preimpregnation)

### Short-beam shear tests

The unirradiated and irradiated interlaminar shear strength and flexural (tensile) modulus are compared in Figures II-1 and II-2, respectively. Those systems with the highest interlaminar shear strengths (polyimide and flexibilized DGEBA epoxy) were most sensitive to the irradiation. After irradiation, the insulation system with the highest interlaminar shear strength (64 MPa) was the polyimide resin. Many TGDM epoxy and bismaleimide systems, following irradiation, had interlaminar shear strengths from 40 to 50 MPa. Those insulation systems with mica barriers exhibited the lowest interlaminar shear strengths (20–25 MPa).

The flexural moduli (Figure II-2) represent tensile-like properties and are not of engineering significance, but they may be used for material comparison. In systems that contain mica, the flexural moduli strongly decrease following irradiation. Most other systems show small increases or decreases following irradiation. The flexural moduli increase with increasing span ratio ( $l/t$ , where  $l$  is the length of the flexing specimen under the pivots and  $t$  is the specimen thickness). The span ratio of all specimens was 5, except for the bismaleimide and TGDM (desized) systems whose span ratio was 6; consequently, these two systems exhibited larger flexural moduli. Thus, Figure II-2 should not be used to compare the relative stiffness of these two systems with those of the other systems.

### Compression tests

Comparisons of the irradiated and unirradiated compressive properties at 4 K are presented in Figures II-3 through II-5. Within data scatter, there were no significant effects of irradiation on the compressive strength, except for those of insulation systems containing DS S-2 glass: The DGEBA- (Update I) and TGDM-epoxy-based DS S-2-glass systems showed about a 10% decrease of compressive strength following irradiation.

The compressive moduli of most insulation systems did not change following irradiation. In three systems (TGDM epoxy, TGDM epoxy/polyimide film, and TGDM epoxy/polyimide film/mica), the moduli apparently increased following irradiation. However, these systems also had larger data scatter than most other systems. In two systems [flexibilized DGEBA epoxy/S-2 glass (AR and DS)], the moduli decreased significantly—about 40% (reported in Update I).

The compressive elongation to failure (calculated as  $\sigma_c/E_c$  with the assumption of a linear  $\sigma$ - $\epsilon$  curve) of all insulation systems did not degrade substantially following irradiation and, in the case of many systems, actually increased. Notice that the elongations are large, in most cases about 5%.

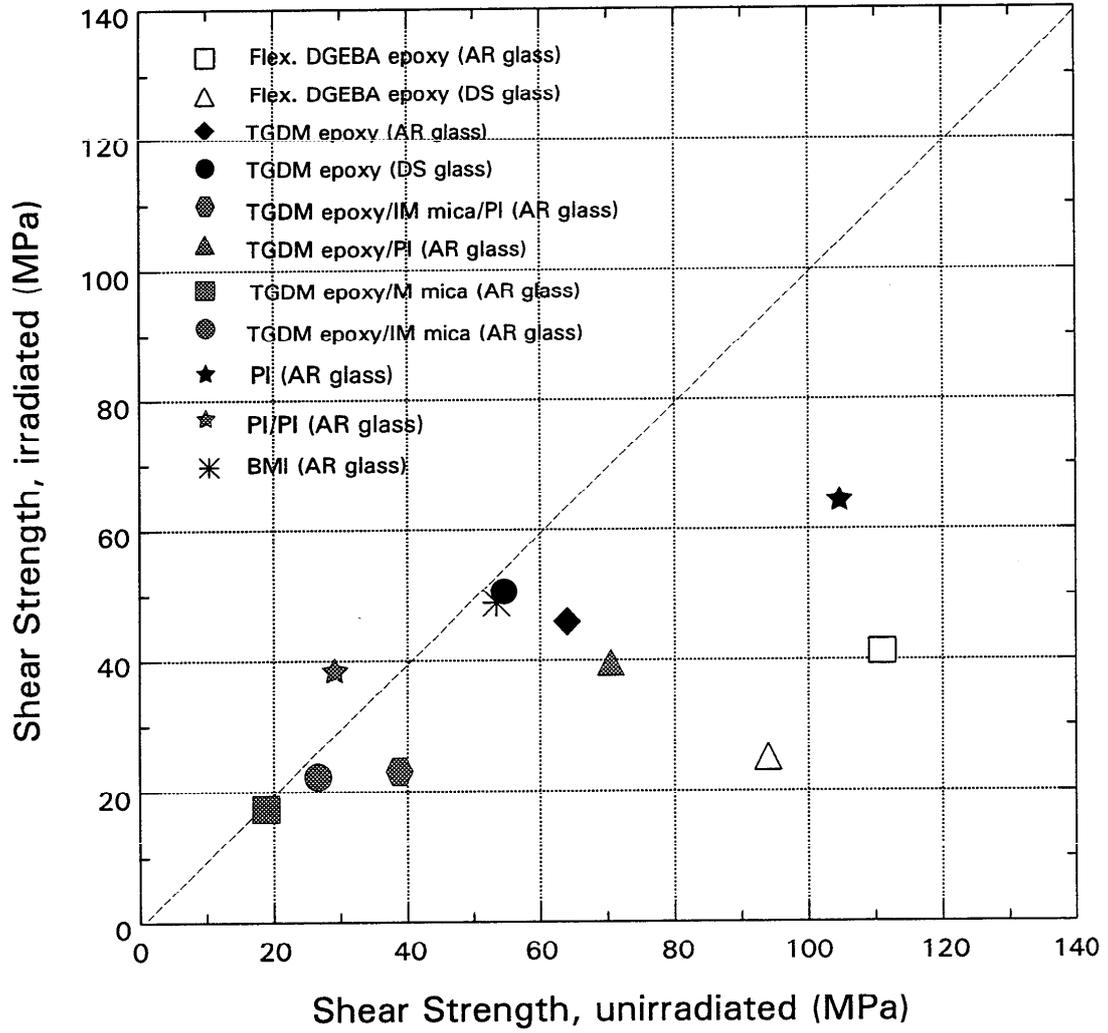


Figure II-1. Irradiated ( $1.8 \times 10^{22} \text{ n/m}^2$ ) versus unirradiated interlaminar shear strength at 4 K, measured with short-beam shear tests.

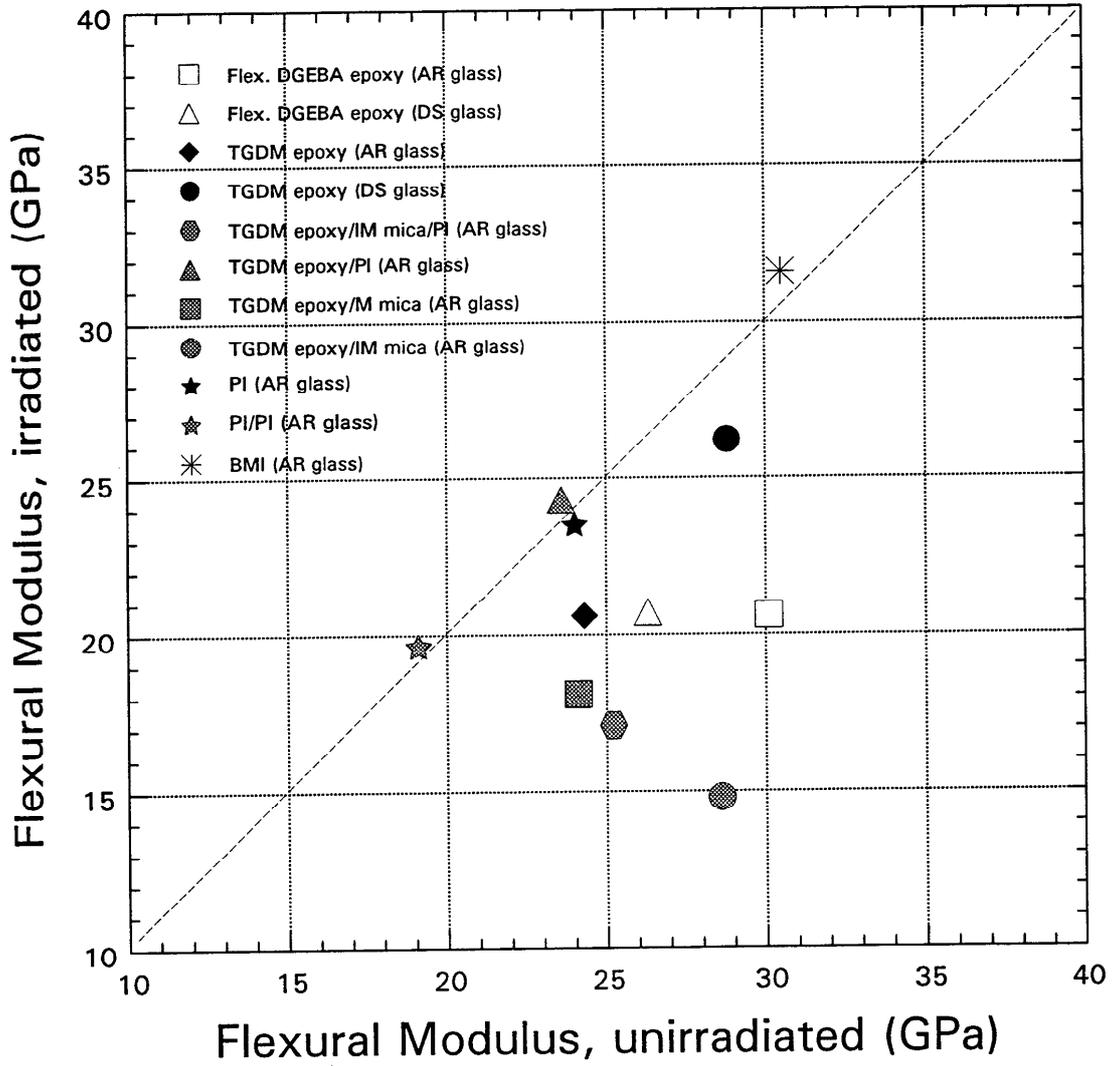


Figure II-2. Irradiated ( $1.8 \times 10^{22} \text{ n/m}^2$ ) versus unirradiated flexural modulus at 4 K, measured with short-beam shear tests.

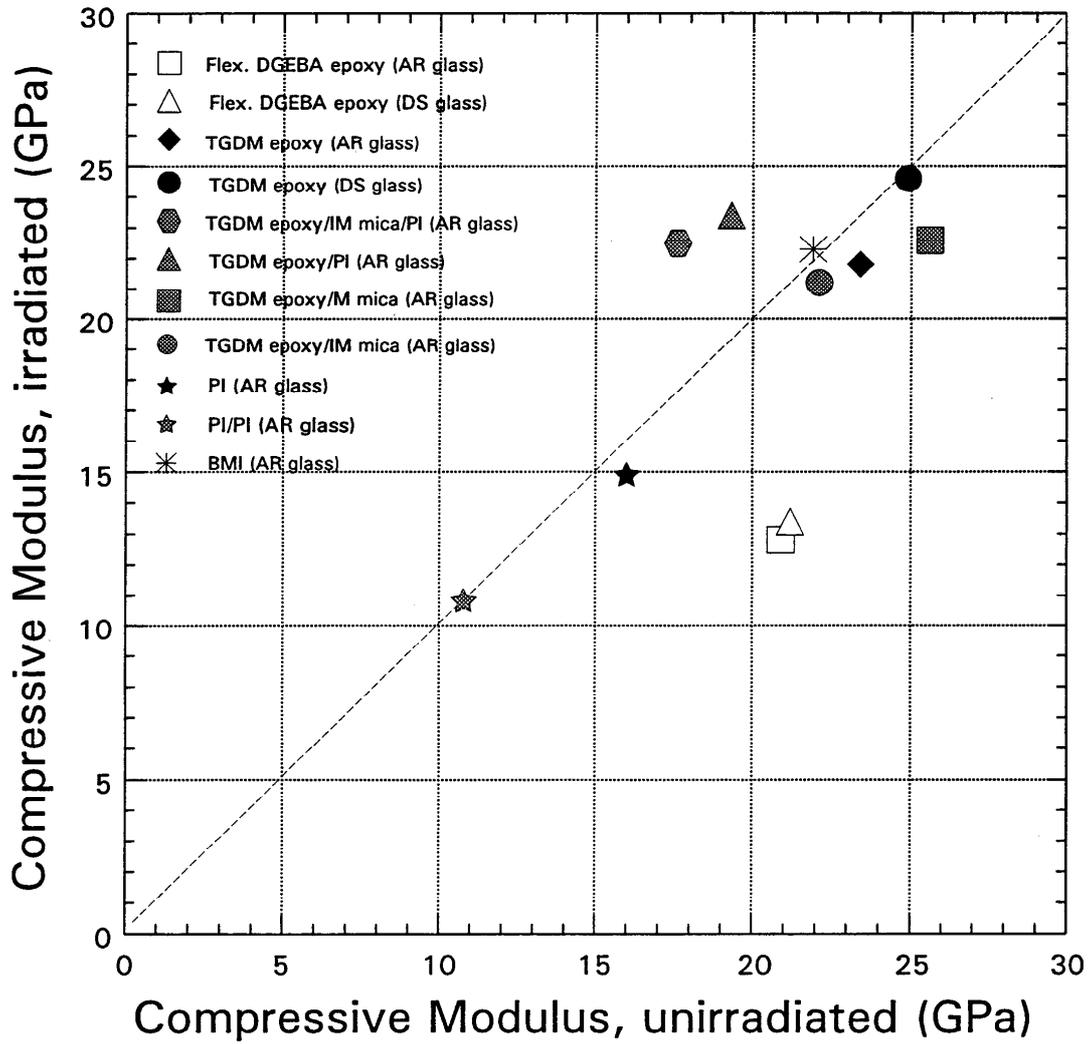


Figure II-3. Irradiated ( $1.8 \times 10^{22} \text{ n/m}^2$ ) versus unirradiated compressive modulus at 4 K. Some data points are adjusted from Appendix B data based on more recent calibration of displacement loading.

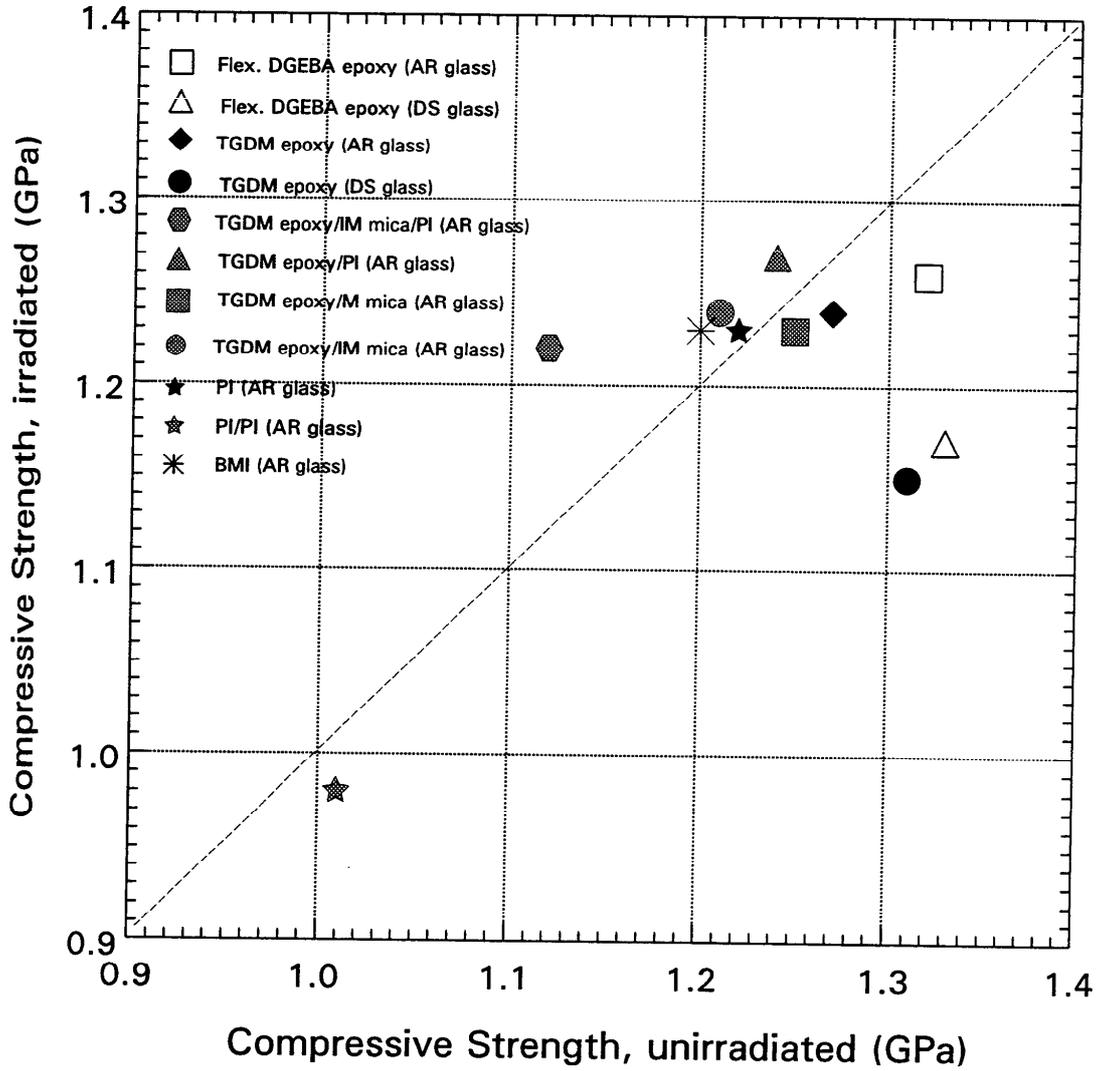


Figure II-4. Irradiated ( $1.8 \times 10^{22} \text{ n/m}^2$ ) versus unirradiated compressive strength at 4 K.

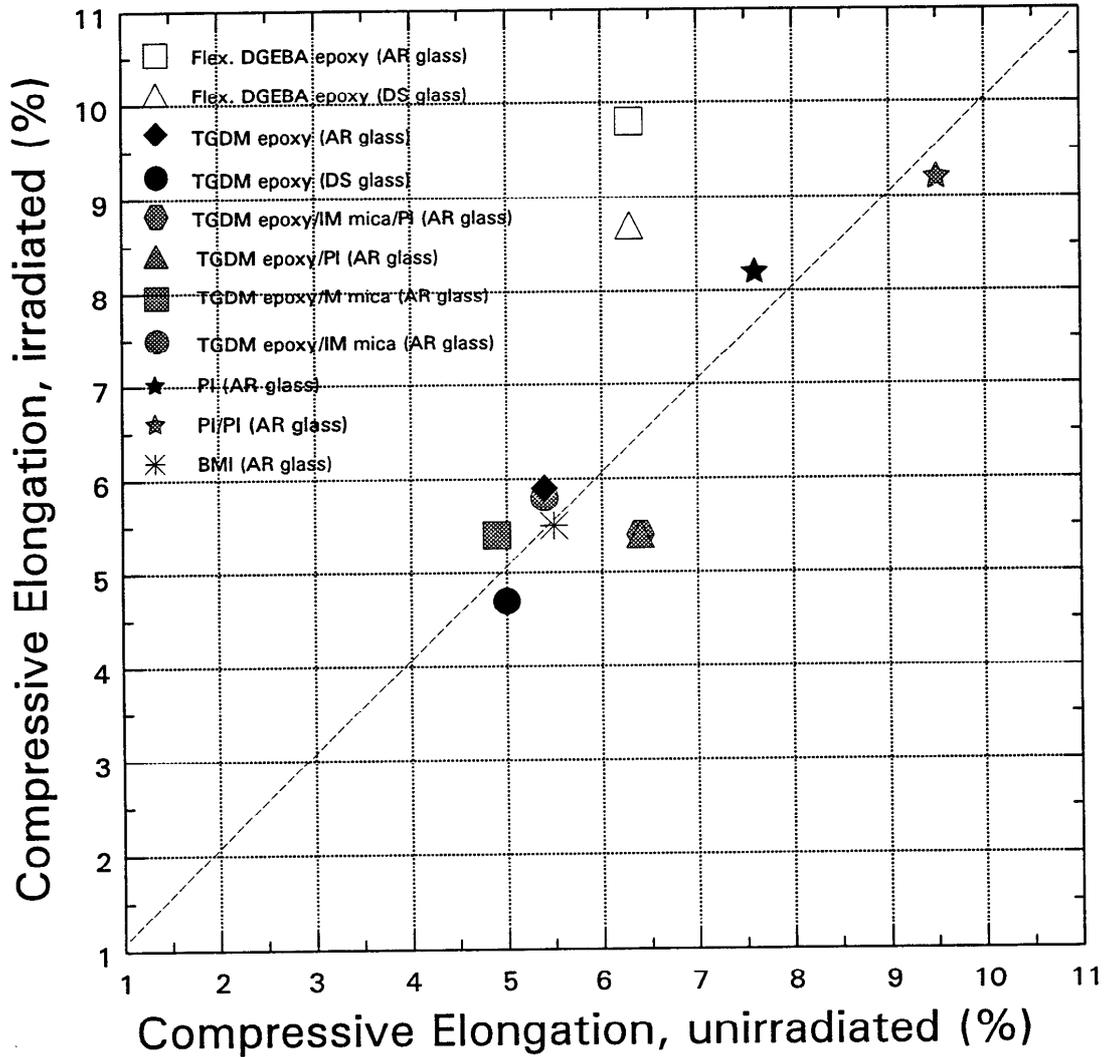


Figure II-5. Irradiated ( $1.8 \times 10^{22} \text{ n/m}^2$ ) versus unirradiated compressive elongation to failure at 4 K. Some data points are adjusted from Appendix B data based on more recent calibration of displacement loading

### **Comparison of irradiated shear and compressive strengths**

In Figure II-6, the strengths of irradiated specimens measured by short-beam shear and compression tests at 4 K are compared. By using these combined strengths as a basis for evaluation, the insulation systems without barriers (mica, polyimide film) are best. However, the properties of the TGDM epoxy/polyimide film (barrier) system are comparable to those of systems without barriers and are superior to those of other systems with barriers.

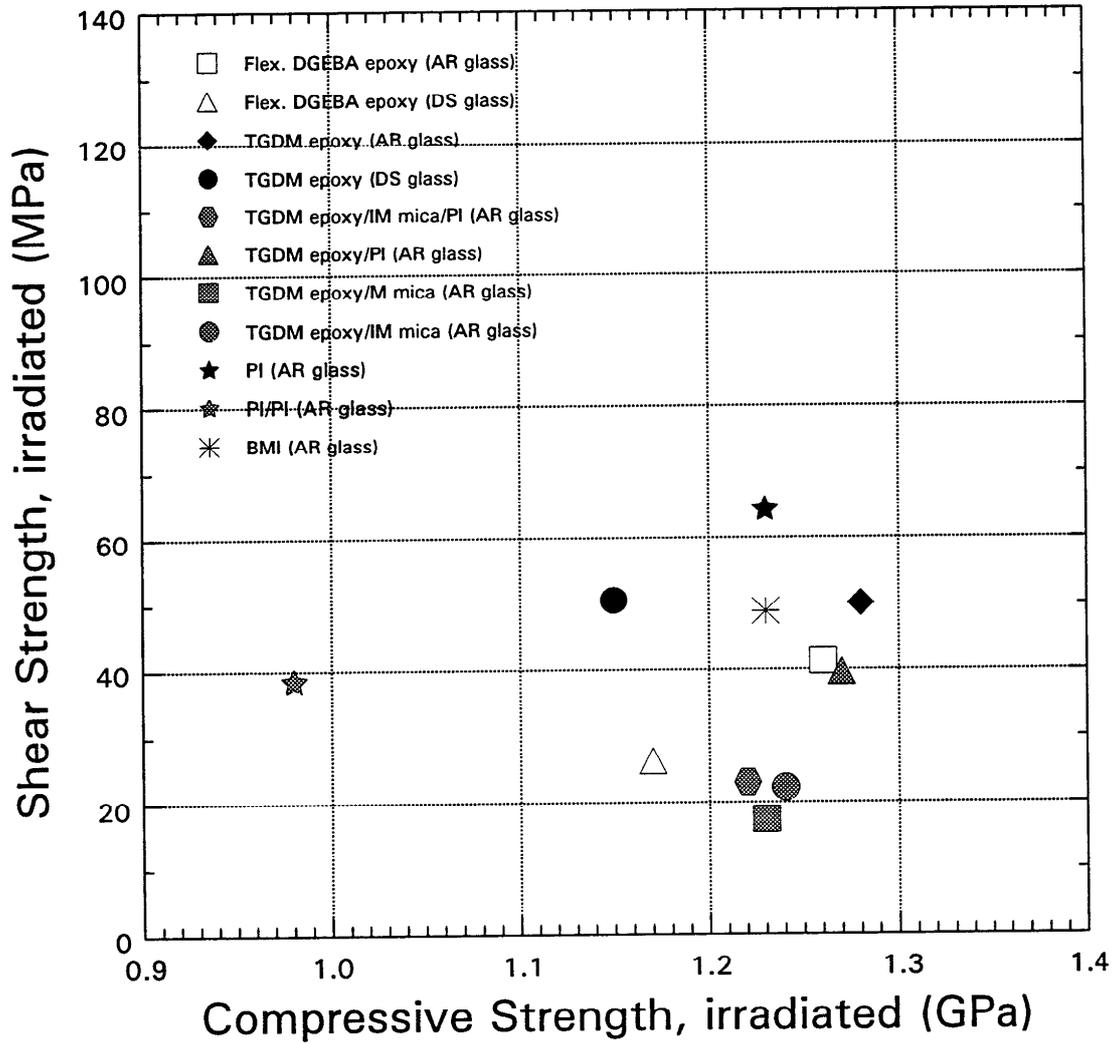


Figure II-6. Irradiated shear strength versus irradiated compressive strength at 4 K for all systems (fast neutron fluence =  $1.8 \times 10^{22}$  n/m<sup>2</sup>).

## ITER Insulation Irradiation Program – Update III (4 August 1994)

Although the primary purpose of the U.S. insulation irradiation program is to assess the effects of irradiation on the mechanical integrity of candidate TF-coil insulation systems, other parameters were also measured. This update reports on the effects of low-temperature irradiation on the mass, dimensions, and dielectric breakdown of these candidate ITER TF-coil insulation systems. This information was obtained from the first and second sets of irradiation runs, from which we determined the effects of irradiation on shear and compressive properties reported in Update I (10 October 1993) and Update II (20 June 1994).

In the EC Garching fission reactor, specimens were irradiated at 4 K, then warmed to room temperature and stored for about three months. Specifically, the specimens were irradiated at 4 K for periods of 9, 17, and 30 h  $\pm$  5 min. At the in-core, low-temperature capsule, the thermal neutron flux was  $28 \times 10^{16}$  n/m<sup>2</sup>/s, and the gamma dose rate was 700 Gy/s. The fast ( $\geq 0.1$  MeV) neutron flux was  $29 \times 10^{16}$  n/m<sup>2</sup>/s.

In the United States, the mass and dimensions were measured at room temperature, and electrical tests were performed at 76 K. The mass and dimensions of short-beam shear specimens [25 mm  $\times$  6.3 mm (in-plane)  $\times$  3.2 mm (through-thickness)] were measured following irradiation and storage (still enclosed in the aluminum foil that had been used to package the specimens in the reactor capsule) at room temperature in a lead-shielded container. Measurement uncertainty of the mass was about 2 parts in  $10^4$  (0.02%) and of the length, about 5 parts in  $10^3$  (0.05%). Electrical breakdown tests were conducted on 13-mm-diameter disc specimens with thicknesses varying from 0.025 mm (PI film) to 0.5 mm (composites) that had been stored at room temperature in an identical lead-shielded container for a similar length of time.

The materials are summarized in the accompanying table. Measurement results are described below.

### Mass

The vacuum-pressure-impregnated epoxy (DGEBA) resins lost mass during the irradiation process. This mass loss probably occurred during warm-up when the free radicals (produced from the effects of radiation ionization) recombined as gaseous products and escaped from the specimen. The amount of mass loss was dependent on the amount of radiation; it is plotted versus gamma dose and fast neutron fluence in the Figure III-1. However, a typical, more radiation-resistant, preimpregnated resin, TGDM, actually gained a small amount of mass from the irradiation, presumably from recombination of gaseous products in the air with free radicals that were still attached to molecular chains. This small mass increase probably would not occur within a vacuum or helium environment.

Materials for Insulation Systems

Label	Material	Trade Name (Supplier)*	Fabrication Method	
			Process <sup>†</sup>	Cure Schedule
DGEBA	diglycidyl ether of bisphenol A epoxy, anhydride curing agent	Shell 826 (Shell)	VPI	150°C/4 h
flex. DGEBA	flexibilized diglycidyl ether of bisphenol A epoxy, anhydride curing agent	CTD-101K (CTD)	VPI	135°C/1.5 h
TGDM	tetraglycidyl diamminodiphenyl methane epoxy, amine curing agent	CTD-112P (CTD)	PP	177°C/2 h
PI	polyimide	AFR-700 (Hexcel)	PP	150°C/1 h + 310°C/1 h
BMI	bismaleimide	CTD-220P (CTD)	PP	177°C/1 h + 250°C/6 h
AR glass	S-2 glass, 6781, 8-harness satin weave, as received with silane finish	S-2 glass (BGF Industries)		
DS glass	S-2 glass, 6781, 8-harness satin weave, desized at 400°C/4 h	S-2 glass (BGF Industries)		
M mica	calcined, epoxy binder, S-2-glass fabric	MM 553 (Midwest Mica)		
IM mica	calcined, epoxy binder, S-2-glass fabric	IMI 498-37B (Insulating Materials)		
PI film (HA)	polyimide film, 0.025 mm thick, relatively amorphous structure	Kapton HA (du Pont)		
PI film (H)	polyimide film, 0.025 mm thick	Kapton H (du Pont)		
VRI mica	calcined, epoxy binder, S-2 glass fabric	~VRI 366.63 (Von Roll Isola)		
PI (320)	polyimide	CTD-320P (CTD)	PP	300°C/1 h
DGEBA-1P (PP)	diglycidyl ether of bisphenol-A epoxy, anhydride curing agent; one-part system	CTD-1PFS (CTD)	PP	150°C/2 h + 175°C/2 h
PI (HPL)	polyimide	Spaulrad S (Westinghouse)	HPL	
DGEBA/TGDM	DGEBA/TGDM blend, cyanoguen-idine curing agent	JDL552 (J.D. Lincoln)	PP	177°C/1 h + 150°C/2 h + 200°C/2 h

\* CTD = Composite Technology Development, Inc.

<sup>†</sup> VPI = vacuum-pressure impregnation; PP = prepreg (preimpregnation); HPL = high-pressure laminate

## Dimensions

For short-beam shear specimens, the changes in in-plane length, width, and through-thickness are shown in the Figures III-2 and III-3. The through-thickness changes are primarily resin dominated, but the in-plane dimensional changes are constrained by the S-2 glass, which is relatively insensitive to radiation effects.

A possible explanation for the relatively large increases in the through-thickness following irradiation is that a portion of the gas formed by free-radical recombinations stays within the material. This concentration of retained gas increases the porosity of the resin with attendant increases in volume.

Consideration of both mass and length changes leads to the conclusion that in the TGDM resin system there are fewer free-radical recombination products than in the DGEBA resin systems. This reduced formation of gaseous by-products results in retention of mass, in no shrinkage, and in small increases of in-plane and through-thickness dimensions. In contrast, there may be more gaseous by-products formed in the DGEBA epoxy systems. This leads to loss of mass, increased porosity, and increased length in the resin-dominated through-thickness.

Three apparent anomalies in the data are

1. The flexibilized DGEBA epoxy (AR glass) systems show no through-thickness changes after a gamma dose of  $2.5 \times 10^7$  and  $8.4 \times 10^7$  Gy (fast neutron fluence of 0.9 and  $3.1 \times 10^{22}$  n/m<sup>2</sup>), but an increase in thickness after a gamma dose of  $4.7 \times 10^7$  Gy. This increase only at the intermediate radiation level is difficult to explain. Perhaps it reflects different environments, such as differences in humidity, during the three-month storage at room temperature.
2. The through-thickness length changes following irradiation at the highest level ( $8.4 \times 10^7$  Gy – gamma;  $3.1 \times 10^{22}$  n/m<sup>2</sup> – fast neutron) are, in general, less than those following irradiation at the two lower levels for the DGEBA resin systems. These differences may reflect changes in the storage environment or an unknown chemical change due to the irradiation.
3. The distinctions in change of length for the DGEBA epoxy with as-received (AR) and desized (DS) S-2-glass reinforcement suggest (a) a beneficial chemical interaction between the irradiation products of the silane finish and the DGEBA or (b) more extensive segregation of the radiation-induced products to the fiber–matrix interface. The changes in length of the AR-glass specimens are considerably less than those of the DS-glass specimens, suggesting the formation of fewer gaseous products.

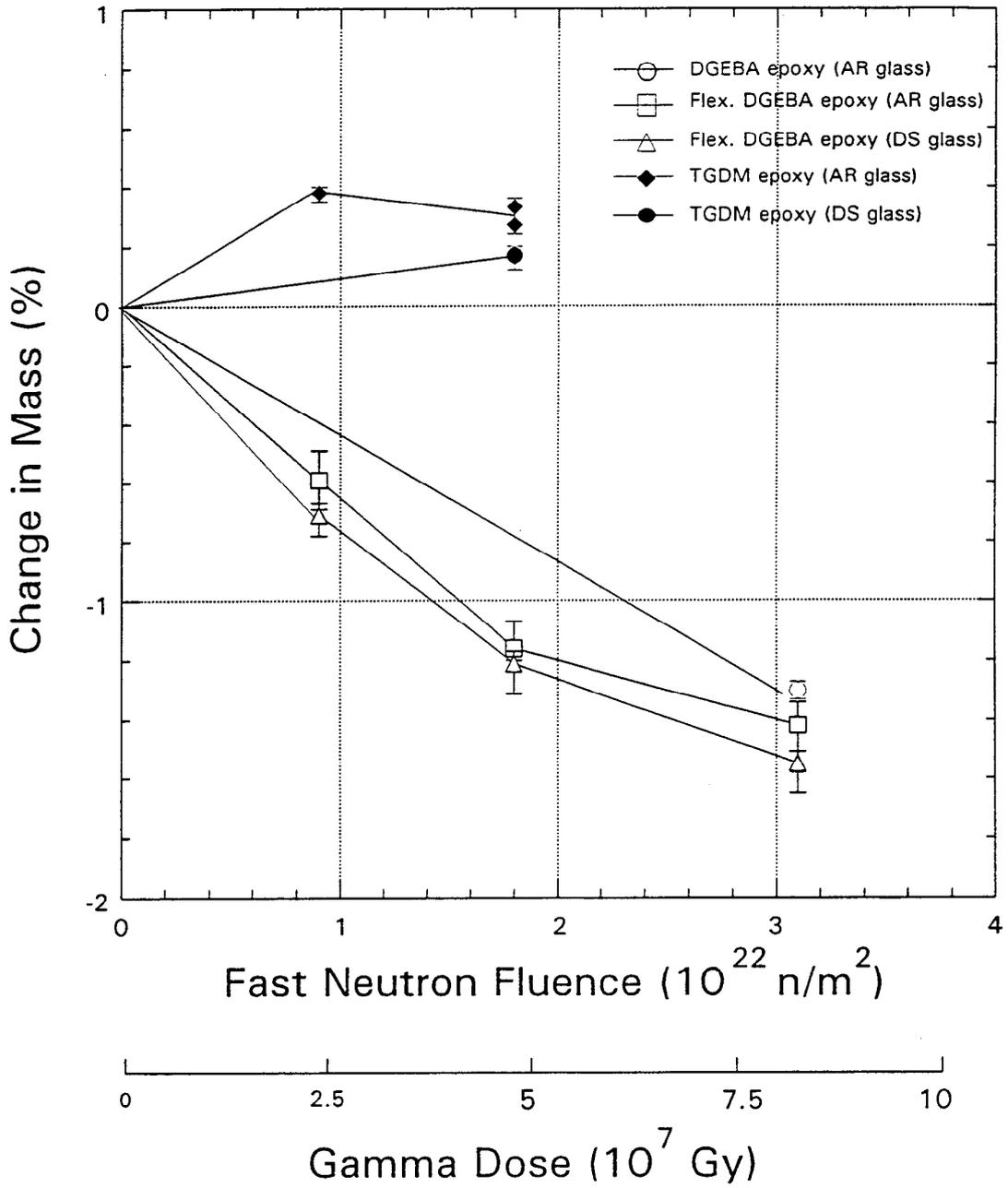


Figure III-1. Change in mass as a function of irradiation at 4 K of selected candidate TF-coil insulation systems.

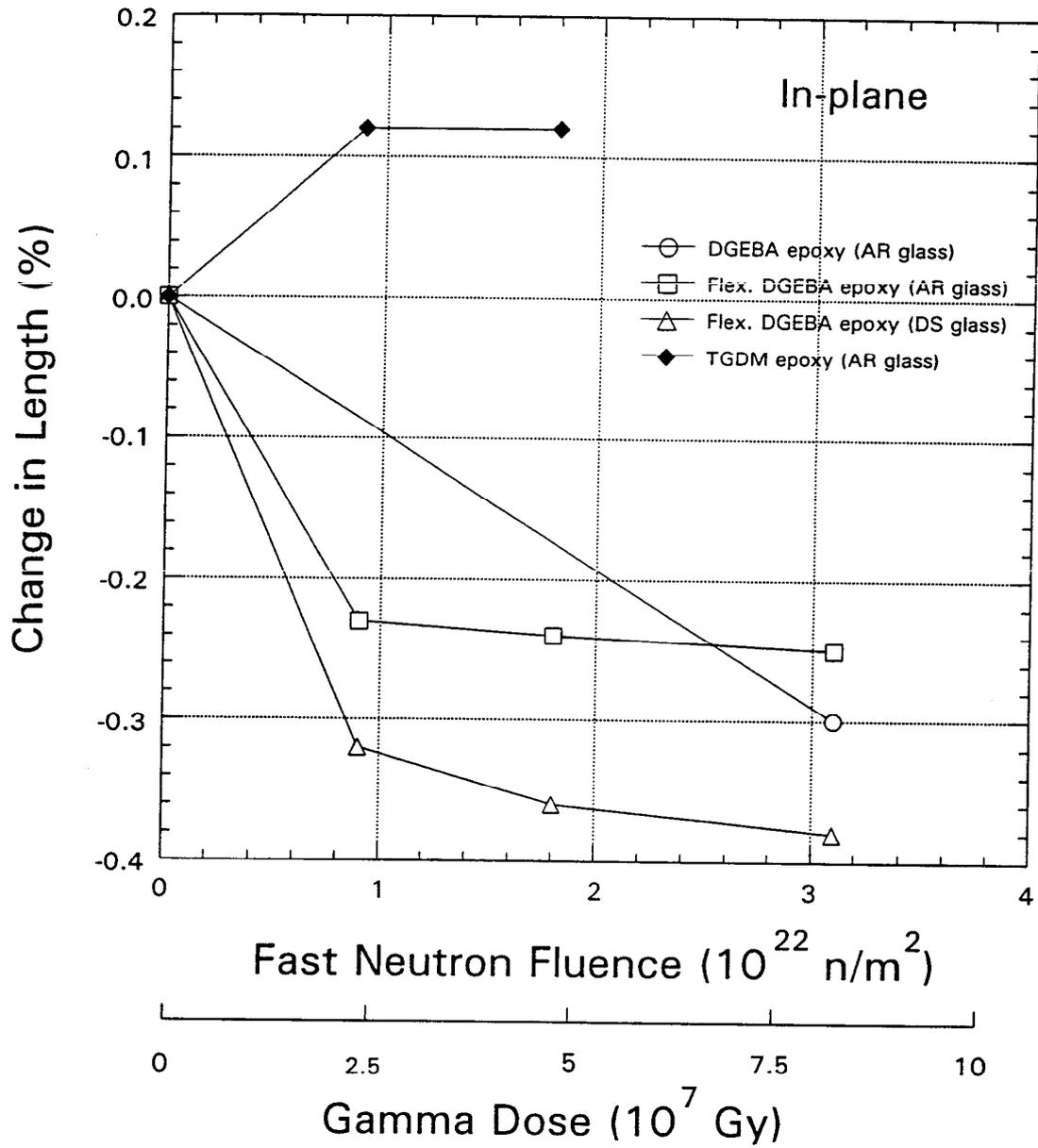


Figure III-2. Average of change in length of in-plane length of short-beam shear specimens following irradiation at 4 K.

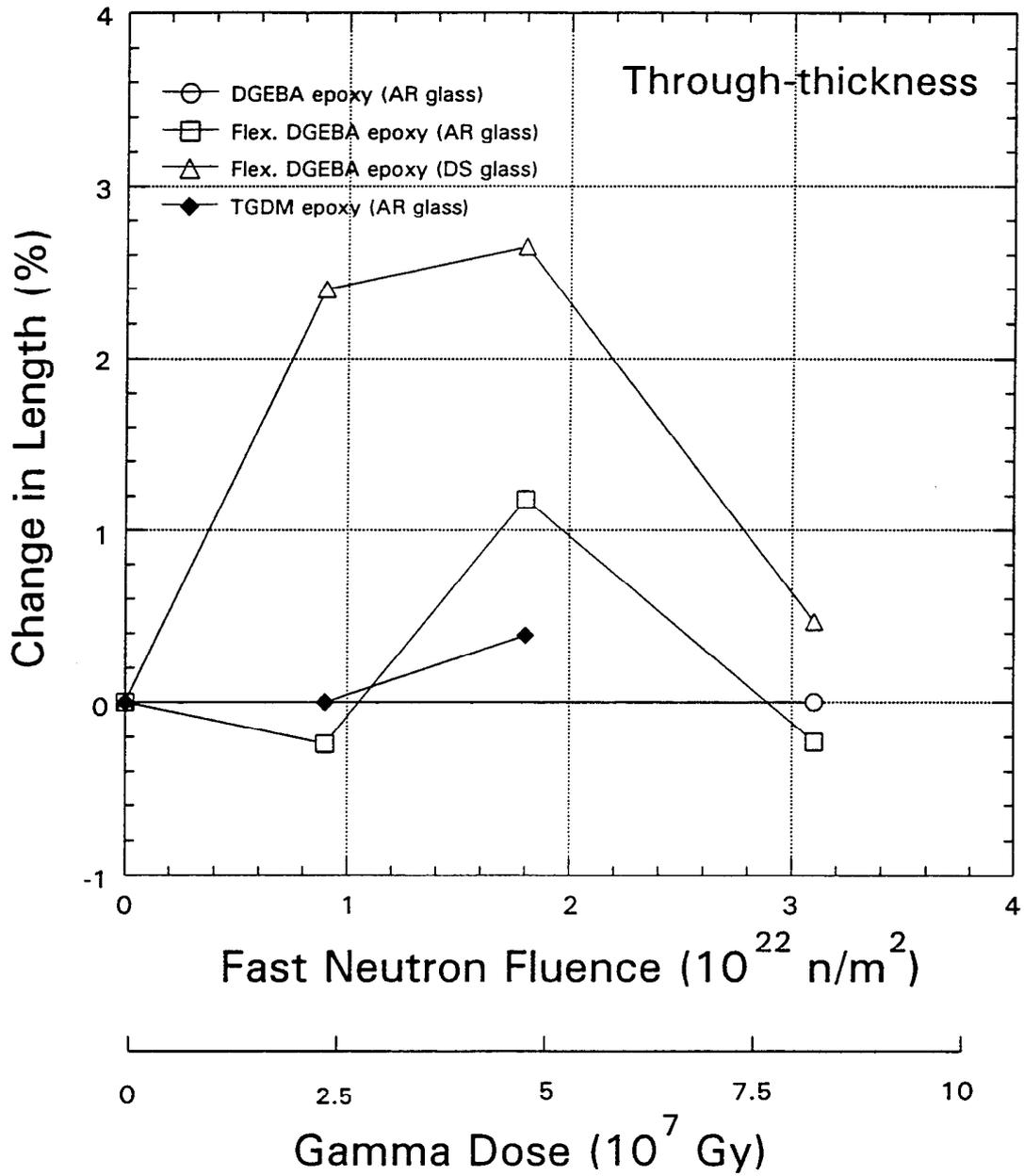


Figure III-3. Average of change in length of through-thickness length of short-beam shear specimens following irradiation at 4 K.

## Electrical properties

The electrical-strength test method is adapted from ASTM Standard Test Methods D149-81 and DD3755-79. The electrodes had a tapered, conical shape with a 2.5-mm (0.10 in) diameter flat. Tests were performed at 76 K with the specimen fully immersed in liquid nitrogen, which also served as a dielectric fluid. A Hipot 120-kVdc dielectric test system was used to supply the voltage, monitor the current, and detect breakdown. The step-by-step voltage test method was used, with 5-s hold times and 2-kV steps. Voltage was increased until breakdown occurred through the specimen. Breakdown is generally confirmed when reapplication of the test voltage results in a substantially lower breakdown voltage, usually through the same damaged location. Retests were performed to verify breakdown. Specimens were also examined visually or microscopically after testing to confirm breakdown.

Breakdown voltage does not scale linearly with material thickness, so dielectric-strength data reports must also include the specimen thickness. In many materials, breakdown voltage scales more closely with the square root of thickness. Therefore, the electrical tests reported here also include a parameter termed the *electrical strength constant*, which is the breakdown voltage normalized by the square root of thickness; its units are kilovolts divided by millimeters to the 1/2 power.

The electrical-strength-constant data at 76 K for irradiated and unirradiated specimens are shown in Figure III-4. Except the polyimide-film specimens, all these specimens included S-2-glass reinforcement (nominally 50 vol%). In Figure III-5, the same data are plotted in terms of dielectric strength for irradiated and unirradiated conditions. The materials that were most sensitive to radiation are the DGEBA and TGDM resin/glass and mica/glass systems. Specimens that included either polyimide film or mica barriers were neither the most sensitive or the most resistant to radiation. The least deterioration was experienced by the polyimide 320, the DGEBA/TGDM blend combined with glass, the TGDM/glass/mica, and the PI film materials. Polyimide film materials clearly have the highest dielectric strength and electric strength constants following irradiation. But, recall that these films were not reinforced with glass.

Sufficient inconsistencies occur in this data set to suggest that material selection should not be based on these data. For example, the polyimide film is clearly superior to the mica in the absence of a VPI or PP resin/glass. However, when these two materials are inserted as barriers within a resin/glass insulation, most of the mica barrier systems are superior to the polyimide film systems. The reason for these apparent anomalies may lie in specimen preparation or handling; they may not depend on the material system.

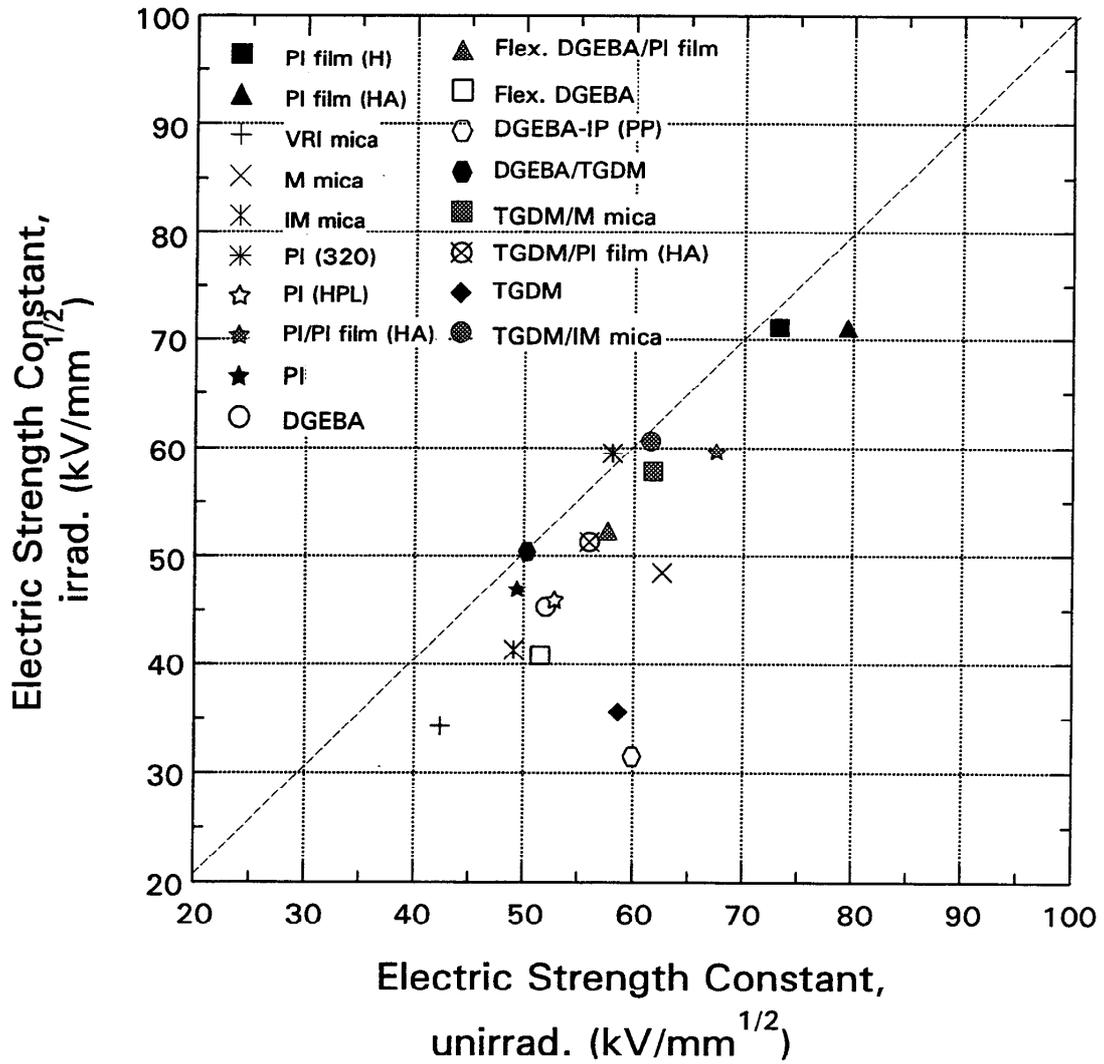


Figure III-4. Irradiated versus unirradiated electric strength constant of selected electrical barriers, resin/S-2 glass composites, and resin/S-2 glass/barrier composites.

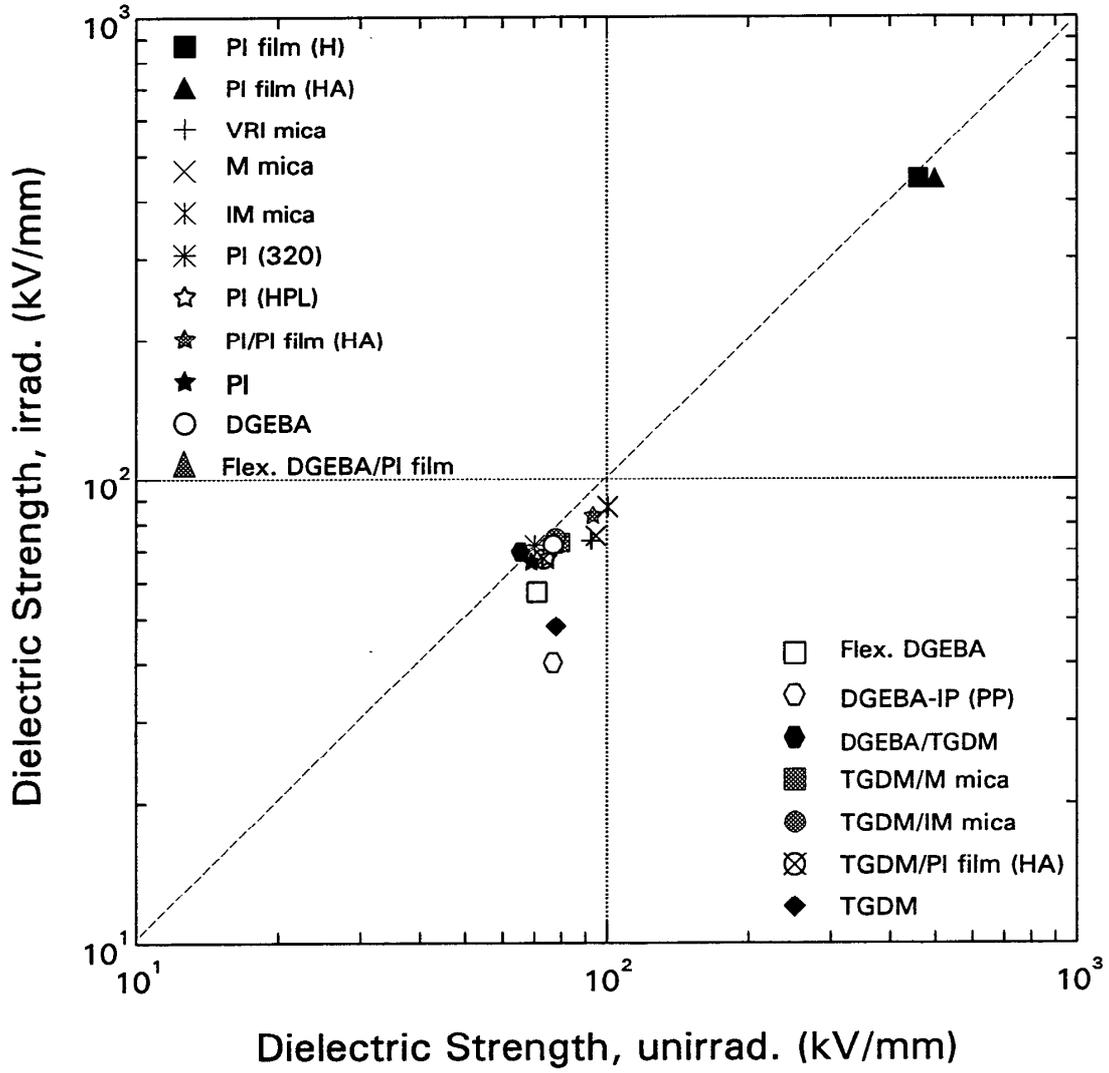


Figure III-5. Irradiated versus unirradiated dielectric strength of selected electrical barriers, resin/S-2 glass composites, and resin/S-2 glass/barrier composites.

## ITER Insulation Irradiation Program – Update IV (3 January 1995)

This update reports the shear/compressive properties of irradiated sandwich-type specimens. Earlier updates had reported the effects of irradiation on shear and compressive properties (I – 10 October 10 1993; II – 20 June 20 1994) and on mass, dimensions, and dielectric breakdown (III – 4 August 1994).

### Irradiation conditions

In the EC Garching fission reactor, specimens were irradiated at 4 K, then warmed to room temperature and stored for about eight months. Specifically, the specimens were irradiated at 4 K for a period of 17 h  $\pm$ 5 min. At the in-core, low-temperature capsule, the thermal neutron flux was  $28 \times 10^{16}$  n/m<sup>2</sup>/s, and the gamma dose rate was 700 Gy/s. The fast (>0.1 MeV) neutron flux was  $29 \times 10^{16}$  n/m<sup>2</sup>.

### Test technique

A test fixture (shown in Figure IV-1) to produce combined shear and compressive forces at predetermined ratios and to provide unrestrained strain for both shear and compression was used. This fixture had been developed previously and used extensively in static tests.<sup>4,7-11</sup> Ratios of shear force to compressive force were established by adjusting the angle of the specimen relative to the applied compressive force (see Figure IV-1). Small angles (relative to the direction of the applied force) produced relatively high shear forces; large angles produced relatively high compressive forces. By conducting tests over a large range of angles, an effective envelope of shear and compressive strengths can be developed that defines the strength of a material under combined shear and compressive forces.

All tests were conducted in liquid helium (at 4 K).

A sandwich type of specimen was used, consisting of 316 stainless steel/bonded insulation system/316 stainless steel. Specimens were tested at two angles: 45° and 75°. A description of the preparation of sandwich-style specimens is provided in our other papers on shear/compression testing;<sup>4,7-11</sup> their dimensions are given in Figure IV-1. In general, for each material, 3 tests were conducted at 45° and two tests were conducted at 75°.

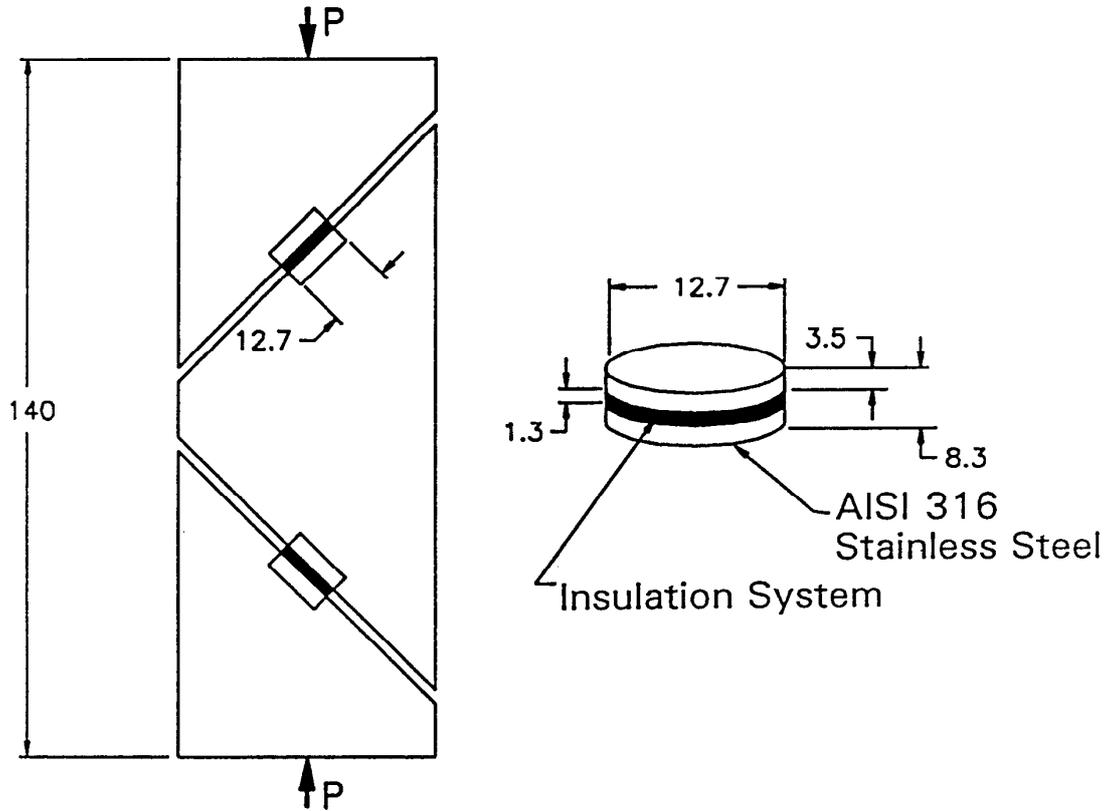


Figure IV-1. Shear/compression test fixture and sandwich-type specimen. Test angles were  $45^\circ$  and  $75^\circ$  with respect to the vertical. Dimensions are in millimeters.

## Materials

The insulation system components (resin, reinforcement, barrier) that were included in this program are identified in Table IV-1. Two layers of mica were used. These barrier layers were centered and inserted between alternating layers of glass fabric, as shown schematically in Figure IV-2. All insulation systems tested in this program contained about 50 vol.% S-2 glass.

The inorganic coatings were plasma sprayed. All coatings (plasma sprayed and painted) were attached to just one of the two 316LN chips that were used to prepare the sandwich-type specimens (see Figure IV-3). Coating thicknesses are provided in Table IV-1. Additional discussion of all materials is given in earlier reports.<sup>10,11</sup>

The relatively large thickness of the coatings affected the number of glass plies required to maintain about 50 vol% glass. In the absence of a coating, a total of 6 plies of

Table IV-1. Materials for Insulation Systems

Label	Material	Trade Name (Supplier)*	Fabrication Method	
			Process <sup>†</sup>	Cure Schedule
<i>Resins</i>				
DGEBA	diglycidyl ether of bisphenol A epoxy, anhydride curing agent	Shell 826 (Shell)	VPI	150°C/4 h
flex. DGEBA	flexibilized diglycidyl ether of bisphenol A epoxy, anhydride curing agent	CTD-101K (CTD)	VPI	135°C/1.5 h
TGDM	tetraglycidyl diamineodiphenyl methane epoxy, amine curing agent	CTD-112P (CTD)	PP	177°C/2 h
PI	polyimide	AFR-700 (Hexcel)	PP	150°C/1 h + 310°C/1 h
<i>Reinforcements</i>				
S-2 glass	S-2 glass, 6781, 8-harness satin weave, as received with silane finish	S-2 glass (BGF Industries)		
DS glass	S-2 glass, 6781, 8-harness satin weave, desized at 400°C/4 h	S-2 glass (BGF Industries)		
<i>Electrical Barriers</i>				
IM mica	calcined, epoxy binder, S-2-glass fabric, 0.36 mm thick	IMI 498-37B (Insulating Materials)		
PI film (HA)	polyimide film, 0.025 mm thick, relatively amorphous structure	Kapton HA (du Pont)		
<i>Electrical Coatings</i>				
PI	polyimide, 0.31 mm thick	Pyre ML (du Pont)	painting	
Al <sub>2</sub> O <sub>3</sub>	alumina, 0.34 mm thick	—	plasma sprayed	
Zr <sub>2</sub> O <sub>2</sub> /8Y <sub>2</sub> O <sub>3</sub>	zirconia/yttria, 0.60 mm thick	—	plasma sprayed	
MgAl <sub>2</sub> O <sub>4</sub>	spinel, 0.29 mm thick	—	plasma sprayed	

\* CTD = Composite Technology Development, Inc.

† VPI = vacuum-pressure impregnation; PP = prepreg (preimpregnation); HPL = high-pressure laminate

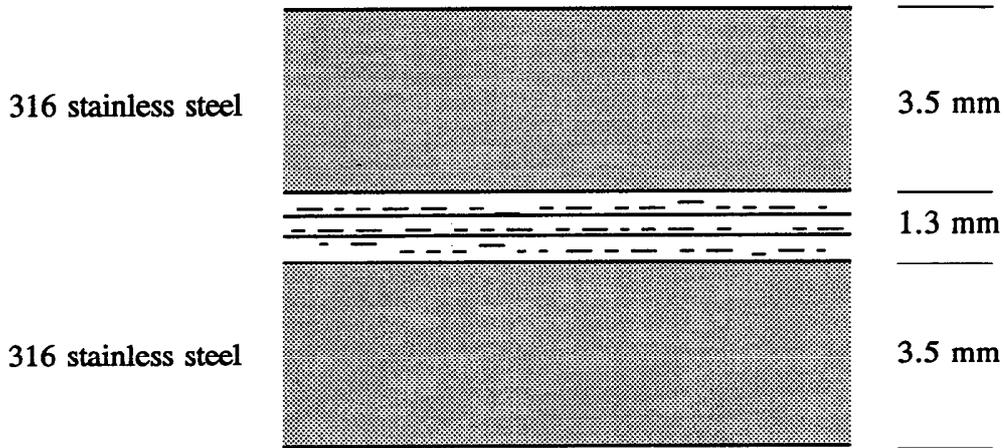


Figure IV-2. Schematic of sandwich-type specimens with mica barriers: 4 plies of S-2 glass, satin weave; 2 layers of mica, backed with 1 layer of S-2 glass, satin weave, each 0.36 mm thick; 50 vol% glass.

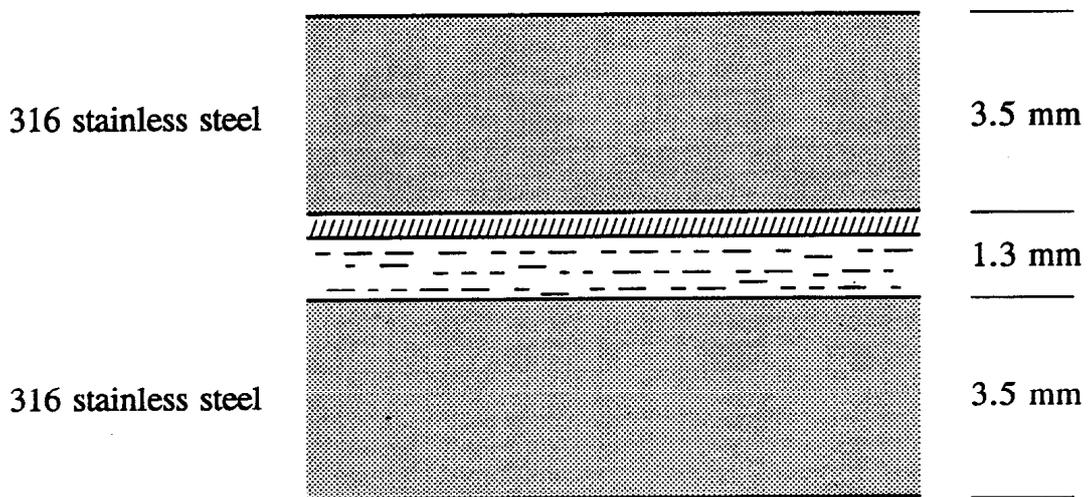


Figure IV-3. Schematic of sandwich-type specimens with a coating: 6 plies of S-2 glass, satin weave; 1 layer of either plasma-sprayed inorganic coating or polyimide paint, 0.29–0.60 mm thick; 50 vol% glass.

the S-2-glass satin weave were used to attain a nominal 50 vol% in all specimens. The number of S-2-glass plies incorporated in coated specimens were alumina – 5, spinel – 5, zirconia/yttria – 4, and polyimide – 6. In the mica specimens, the 2 layers of mica barriers contained the same S-2-glass satin weave as a backing material; therefore, 3 additional layers of weave were required.

## Results and discussion

The average shear strengths for each material at each test angle (45°, 75°) are plotted in Figure IV-4. The shear strength, instead of the compressive strength, is used since the failure mode of all specimens that are summarized in Figure IV-4 was shear. Most specimens experienced interlaminar failure within the insulation system; however, a few specimens failed at equivalent loads at the steel/insulation interface. Materials that exhibited premature (adhesive) failure at the metal/insulation interface during specimen-handling procedures are

- polyimide (Pyre ML): 3 of 5 specimens broke during transport from Europe
- TGDM/IM mica: 2 of 5 specimens broke during transfer to the test rig
- flex. DGEBA/spinel coating: 1 of 5 specimens broke during transfer to the test rig

The same data are presented in Figure IV-5 as a function of fast neutron fluence. From these figures, several observations are apparent:

1. The plasma-sprayed coatings of alumina and zirconia/yttria are very beneficial in improving the radiation resistance of the flexibilized DGEBA epoxy system. At 45°, the shear strengths of the irradiated specimens with these coatings were about 60% higher, and at 75°, about 35% higher than those of the other irradiated specimens.
2. The plasma-sprayed spinel coating does not improve the radiation resistance of the flexibilized DGEBA epoxy system.
3. The flexibilized DGEBA appeared to be more radiation resistant than the DGEBA system under the combined shear/compressive loads of these tests. Yet, in Update I (October 10, 1993), we reported equivalent shear strengths (as measured by short-beam shear tests) for both systems at the fast neutron fluence of  $3.1 \times 10^{22} \text{ n/m}^2$ .
4. The mica system with TGDM epoxy was considerably weaker than the other systems in both the 45° and 75° tests, despite the fact that the irradiated and unirradiated data were essentially the same at 75°.

The combined shear strengths and the associated compressive stresses for each system are plotted in Figure IV-6. This figure clearly indicates the relative strengths of the strongest alumina- and zirconia/yttria-coated systems, the weakest mica systems, and the medium-strength flexibilized DGEBA and spinel-coated systems.

From Updates I and II, it is possible to construct or estimate shear/compressive strength envelopes for these systems. Owing to their similar properties, the data for the alumina and

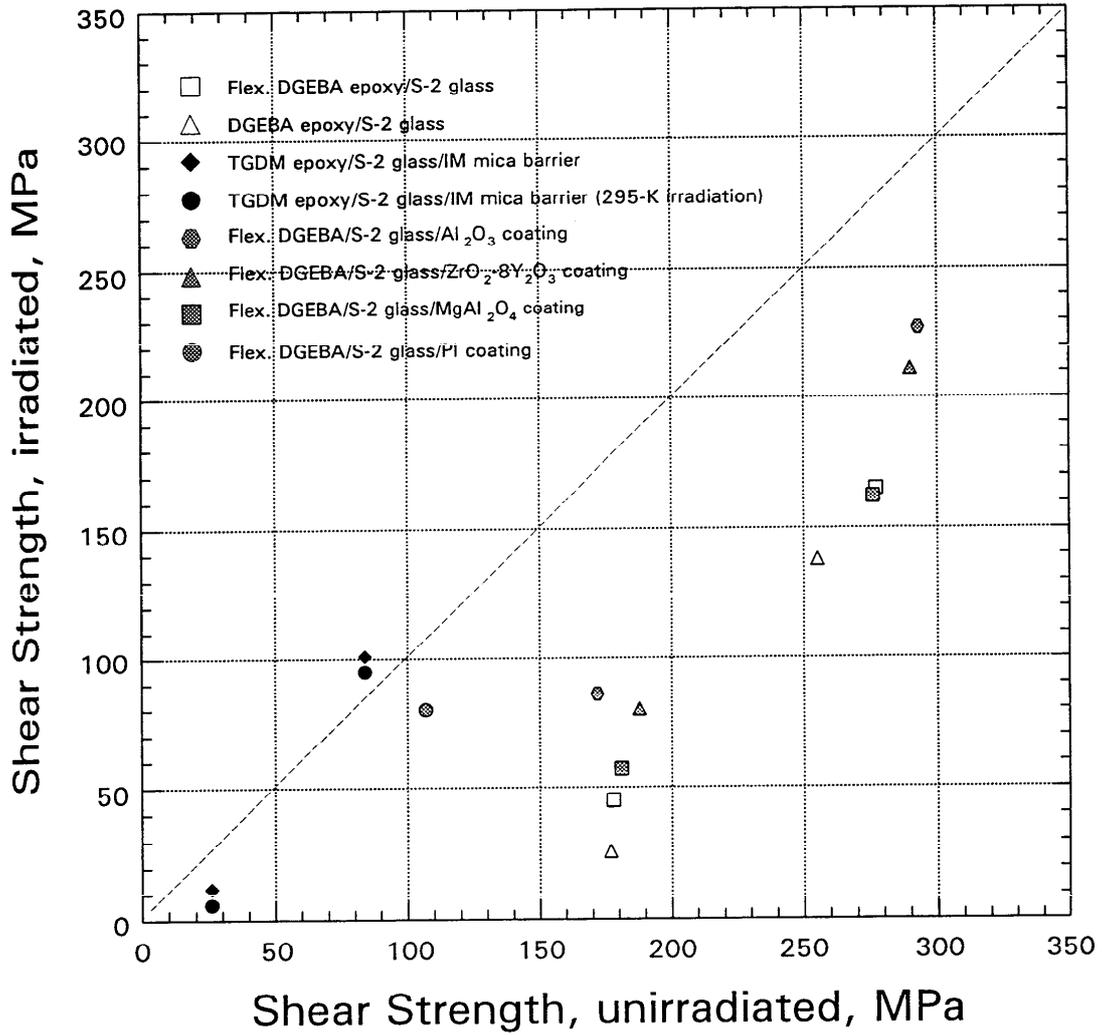


Figure IV-4 Irradiated ( $1.8 \times 10^{22}$  n/m<sup>2</sup> fast neutron fluence at 4 K) versus unirradiated shear strength (at 4 K) measured under combined shear/compressive loads at 45° and 75°.

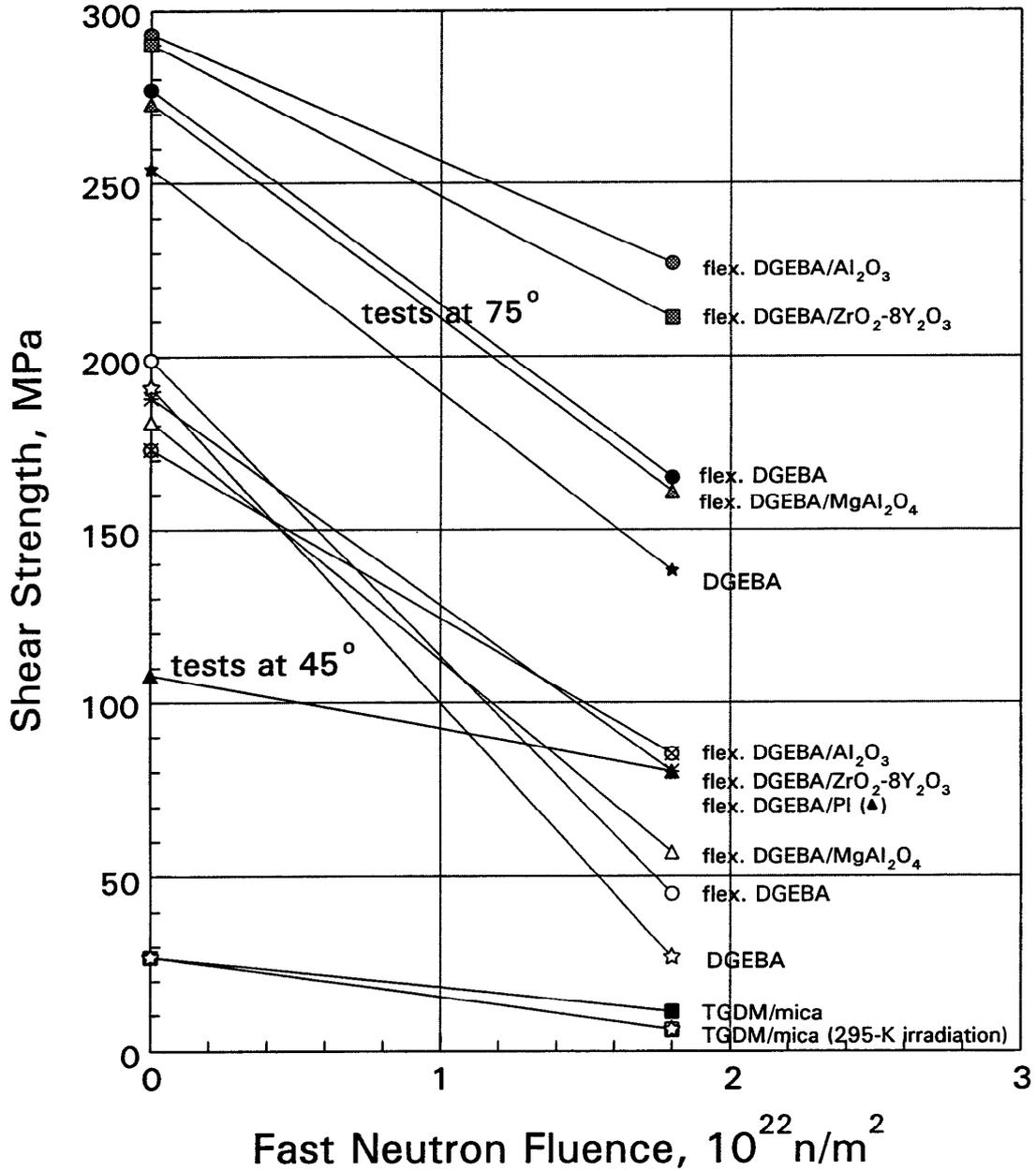


Figure IV-5. Shear strength (at 4 K) versus fast neutron fluence (at 4 K). Shear strength was determined under combined shear/compressive loads.

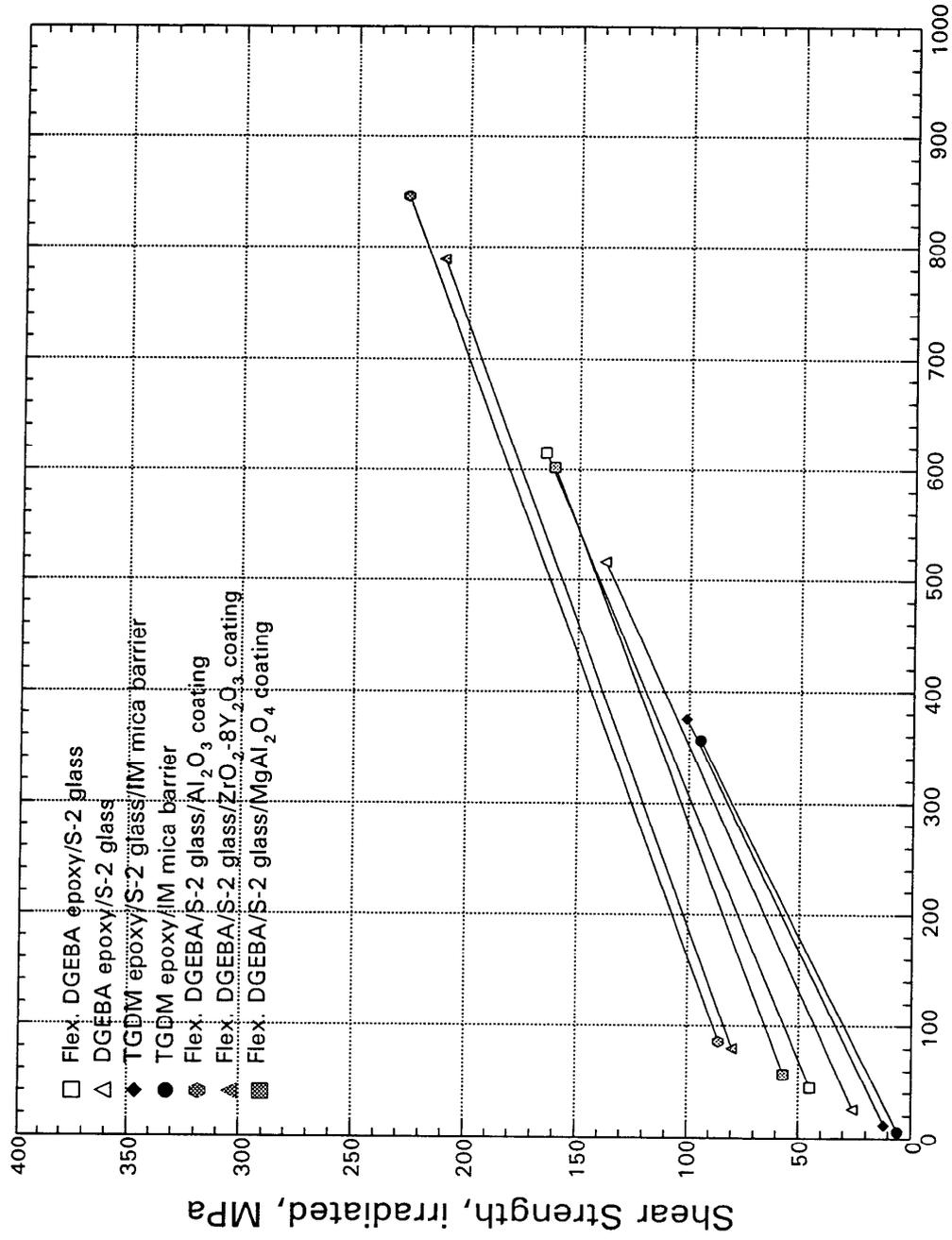


Figure IV-6. Shear/compressive data (at 4 K) following irradiation to a fast neutron fluence of  $1.8 \times 10^{22}$  n/m<sup>2</sup>.

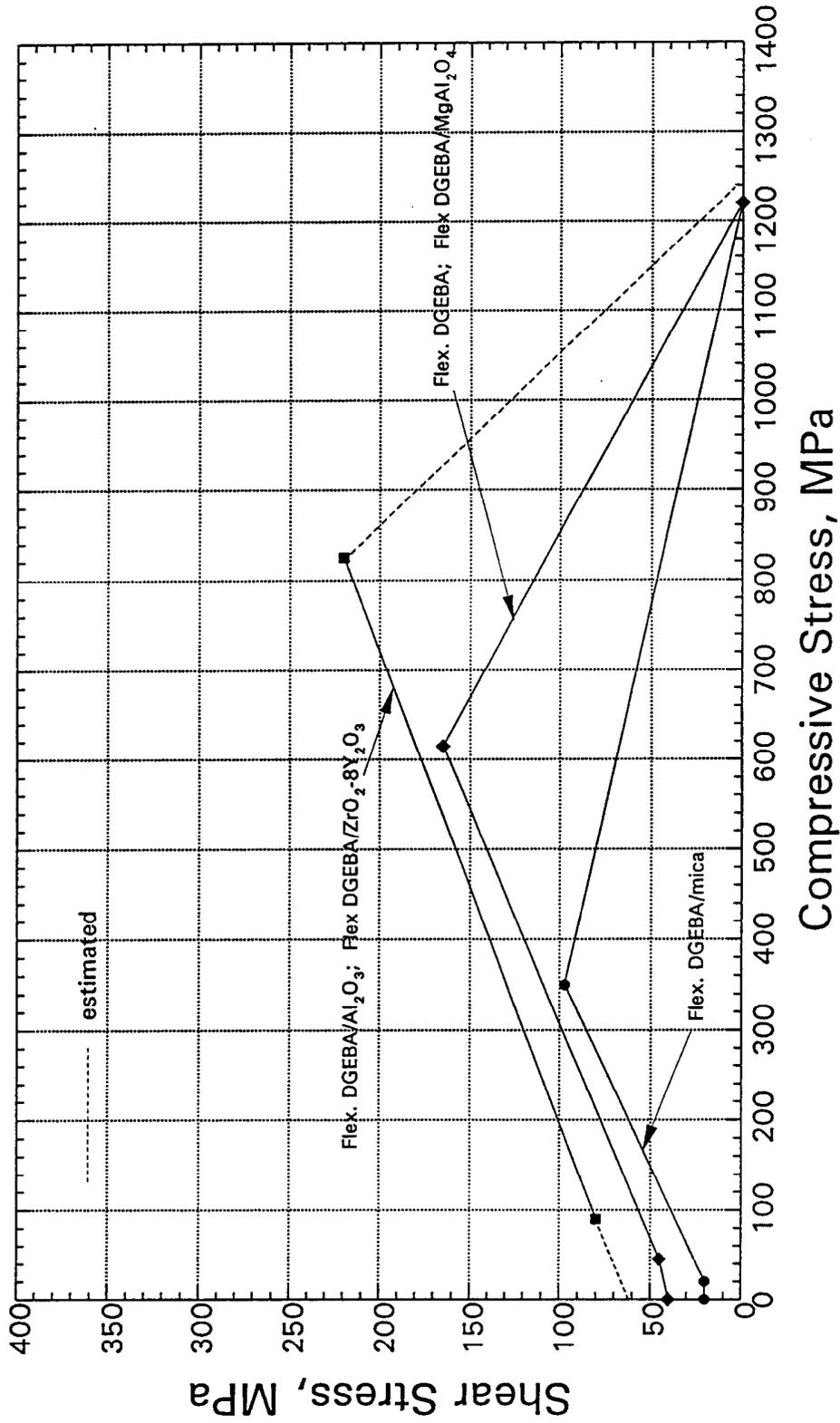


Figure IV-7. Measured and estimated shear/compressive strengths (at 4 K) of three classes of insulation systems based on irradiation to  $1.8 \times 10^{22}$  n/m<sup>2</sup>.

zirconia/yttria systems, the flexibilized DGEBA and spinel-coated systems, and the 4- and 295-K irradiated mica system are combined in three general curves in Figure IV-7. These curves depict the strengths at the irradiation level of  $1.8 \times 10^{22}$  n/m<sup>2</sup>. The pure shear and compressive strengths of irradiated alumina- and zirconia/yttria-coated systems are estimated because the strengths of these systems have been measured only in the unirradiated condition.

Several variables may account, at least partially, for the beneficial effects of the alumina and zirconia/yttria plasma-sprayed coatings:

1. The estimated porosity of the plasma-sprayed coatings is 30%. The pores provide internal space that may act as a sink for the considerable gas released from the flexibilized DGEBA during warm-up following irradiation at 4 K.
2. The inorganic structure may assist in absorbing evolved gas molecules. The relatively higher density of the spinel structure could partially account for its smaller effect on radiation sensitivity.
3. The zirconia/yttria coating is considerably thicker (0.60 mm) than the spinel coating (0.29 mm) and the alumina coating (0.34). The thickness of the entire insulation system within the sandwich-type specimen is 1.3 mm. Thus, the zirconia/yttria coating occupies a much more significant portion (about 45%) of the insulation system of the specimen than the other two coatings.

### **ITER Insulation Irradiation Program – Update V (30 June 1995)**

This update reports the shear/compressive properties of additional irradiated sandwich-type and short-beam shear specimens that had been stored ten months at 76 K prior to testing (the major distinction of this test series). Earlier updates had reported the effects of irradiation on shear and compressive properties (I – 10 October 1993; II – 20 June 1994), on mass, dimensions, and dielectric breakdown (III – 4 August 1994), and on shear/compressive properties (IV – 3 January 1995).

#### **Irradiation conditions**

In the EC Garching fission reactor, specimens were irradiated at 4 K for a period of  $17 \text{ h} \pm 5$  min. At the in-core, low-temperature capsule, the thermal neutron flux was  $28 \times 10^{16}$  n/m<sup>2</sup>/s, and the gamma dose rate was 700 Gy/s. The fast (>0.1 MeV) neutron flux was  $29 \times 10^{16}$  n/m<sup>2</sup>/s. The total fast neutron fluence was estimated at  $1.8 \times 10^{22}$  n/m<sup>2</sup>. After irradiation, the specimens were stored at 77 K for about ten months, then warmed to room temperature and stored for about one month before being shipped to the United States, where they were tested within one month.

### Test techniques

A test fixture (shown in Figure V-1) to produce combined shear and compressive forces at predetermined ratios and to provide unrestrained strain for both shear and compression was used to test 10 irradiated specimens. This fixture had been developed previously and used extensively in static tests.<sup>4,6,8-11</sup> Ratios of shear force to compressive force were established by adjusting the angle of the specimen relative to the applied compressive force (see Figure V-1). Small angles (relative to the direction of the applied force) produced relatively high shear forces; large angles produced relatively high compressive forces. By conducting tests over a large range of angles, a failure envelope of shear and compressive strengths can be determined that defines the strength of a material under combined shear and compressive forces.

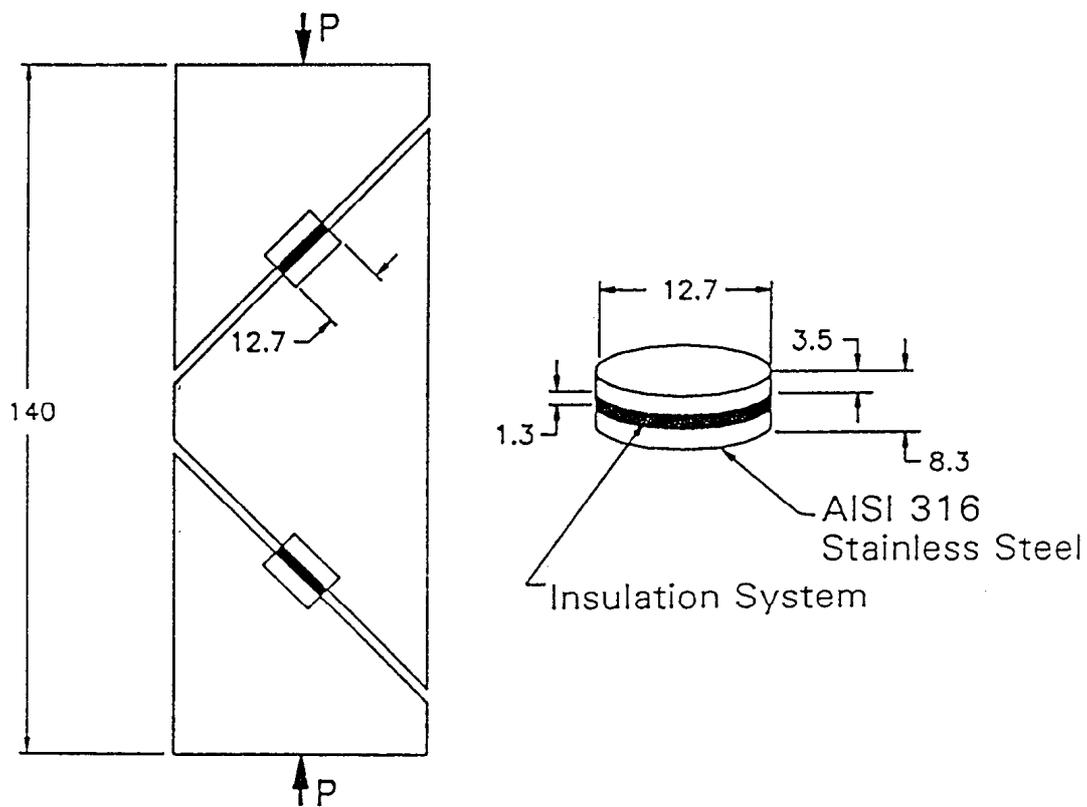


Figure V-1. Shear/compression test fixture and sandwich-type specimen. Test angles were 45° and 75° with respect to the vertical. Dimensions are in millimeters.

In this test series, all specimens were irradiated at 4 K to a fast neutron fluence of  $1.8 \times 10^{22}$  n/m<sup>2</sup>, stored in liquid nitrogen, warmed to room temperature, shipped to the United States, and then tested at 4 K in liquid helium. A load rate of 0.5 mm/min was used for static tests and a frequency of 10 Hz and an *R*-ratio of 0.1 was used for fatigue tests.

The sandwich-type specimen consisted of 316 stainless steel/bonded insulation system/316 stainless steel. Specimens were tested at two angles: 45° and 75°. A description of the preparation of sandwich-style specimens is provided in our other papers on shear/compression testing,<sup>4,6,8-11</sup> their dimensions are given in Figure V-1. In general, for each material, 3 tests were conducted at 45° and two tests were conducted at 75°.

The short-beam shear specimen dimensions were 25 mm long × 6.3 mm wide × 3.2 mm thick. Three to four short-beam shear tests of each material were conducted at 4 K with these specimens. Application of three-point bend loading was normal to the plane of the glass-weave and barrier. Shear strength and flexural modulus were measured from these tests.

Sections of one specimen from each material were cut to produce compression specimens, 3.2 mm high × 6.3 mm × 6.3 mm. The compressive strength ( $\sigma_c$ ) and modulus ( $E_c$ ) were measured, and the strain to failure ( $\sigma_c/E_c$ ) was calculated with the assumption of a linear stress-strain relationship. Compressive strain was measured with one 25-mm gage-length strain-gage extensometer fastened to each load platen.

## Materials

The insulation system components (resin, reinforcement, barrier) that were included in this program are identified in Table V-1. Three layers of polyimide film (Kapton HA) were used. These barrier layers were centered and inserted between alternating layers of glass fabric, as shown schematically in Figure V-2. All insulation systems tested in this program contained about 50 vol.% S-2 glass.

Table V-1. Materials for Insulation Systems

Label	Material	Trade Name (Supplier)*	Fabrication Method	
			Process†	Cure Schedule
<i>Resins</i>				
DGEBA	diglycidyl ether of bisphenol A epoxy, anhydride curing agent	Shell 826 (Shell)	VPI	150°C/4 h
flex. DGEBA	flexibilized diglycidyl ether of bisphenol A epoxy, anhydride curing agent	CTD-101K (CTD)	VPI	135°C/1.5 h
TGDM	tetraglycidyl diaminodiphenyl methane epoxy, amine curing agent	CTD-112P (CTD)	PP	177°C/2 h
<i>Reinforcements</i>				
S-2 glass	S-2 glass, 6781, 8-harness satin weave, as received with silane finish	S-2 glass (BGF Industries)		
DS glass	S-2 glass, 6781, 8-harness satin weave, desized at 400°C/4 h	S-2 glass (BGF Industries)		
<i>Electrical Barrier</i>				
PI film (HA)	polyimide film, 0.025 mm thick, relatively amorphous structure	Kapton HA (DuPont)		

\* CTD = Composite Technology Development, Inc.

† VPI = vacuum-pressure impregnation; PP = prepreg (preimpregnation)

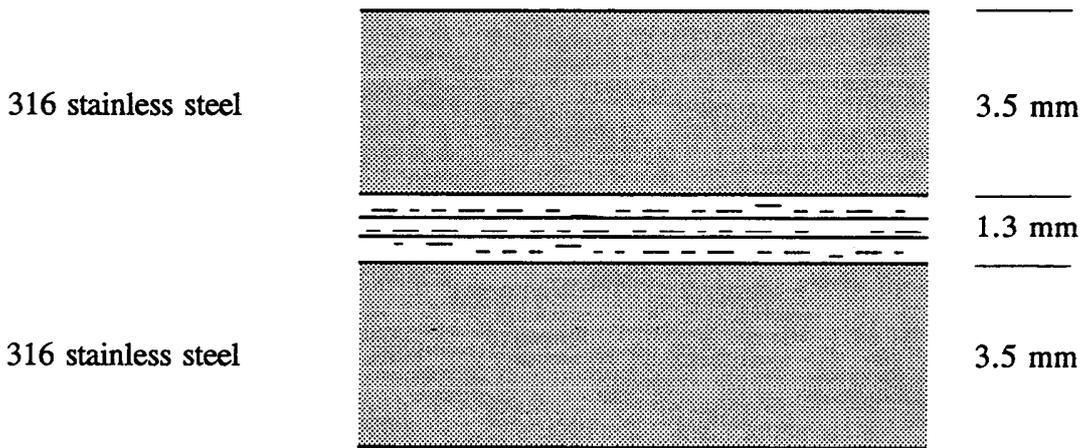


Figure V-2. Schematic of sandwich-type specimens with polyimide film barriers: 6 plies of S-2 glass, satin weave; 3 layers of polyimide film (Kapton HA); 50 vol% glass.

**Results**

*Shear/Compression – Static Tests*

Two insulation systems were tested in this series: (1) vacuum-pressure impregnated (VPI) flexibilized DGEBA (CTD-101K)/50 vol% S-2 glass and (2) prepregged (PP) TGDM (CTD-112P)/50 vol% S-2 glass/polyimide film (Kapton HA). Previously, the flexibilized DGEBA–VPI system had been tested only after storage at room temperature. The results of these tests are presented in Table V-2.

These data are also plotted in Figure V-3 (Figure IV-4 of Update IV). Note that the flexibilized DGEBA values were less in this test series; the shear strength of 75° tests fell from about 160 MPa to 111 MPa. The TGDM system had not been tested previously, but following irradiation, its shear strength was slightly more than that of the flexibilized DGEBA system. Despite the presence of the Kapton film barrier, the mechanical strengths at 4 K of the TGDM system were much less sensitive to radiation than those of the DGEBA systems (without a barrier).

Table V-2. Shear and Compressive Strengths

System	Test Angle (°)	Shear Strength			Compressive Strength			Failure Mode <sup>‡</sup>
		Average (MPa)	S.D.* (MPa)	C.V. <sup>†</sup>	Ave (MPa)	S.D.* (MPa)	C.V. <sup>†</sup>	
flex. DGEBA	75	111	1.6	0.01	415	5.9	0.01	C
TGDM/pol yimide film	45	84	7.3	0.09	84	7.3	0.09	A/K
	75	116	13.5	0.12	431	50.2	0.12	A/K

\* S.D. = standard deviation

† C.V. = coefficient of variation

‡ C = cohesive failure; A/K = adhesive failure at Kapton

*Shear/compression–fatigue tests*

A limited number of fatigue tests at 4 K of irradiated and unirradiated specimens of the VPI flexibilized DGEBA system (stored at 77 K, then 295 K) were conducted at the 75° test angle. Data from these tests, combined with the 76-K fatigue-test data at 75°, are presented in Figures V-4 and V-5 for shear and compressive stresses, respectively.

Included in this data set are the results from two earlier fatigue tests at 75° (labeled *irradiated, stored at 295 K*) that were conducted on the same insulation system and exposed to the same radiation level ( $1.8 \times 10^{22}$  n/m<sup>2</sup>). However, these specimens from the earlier

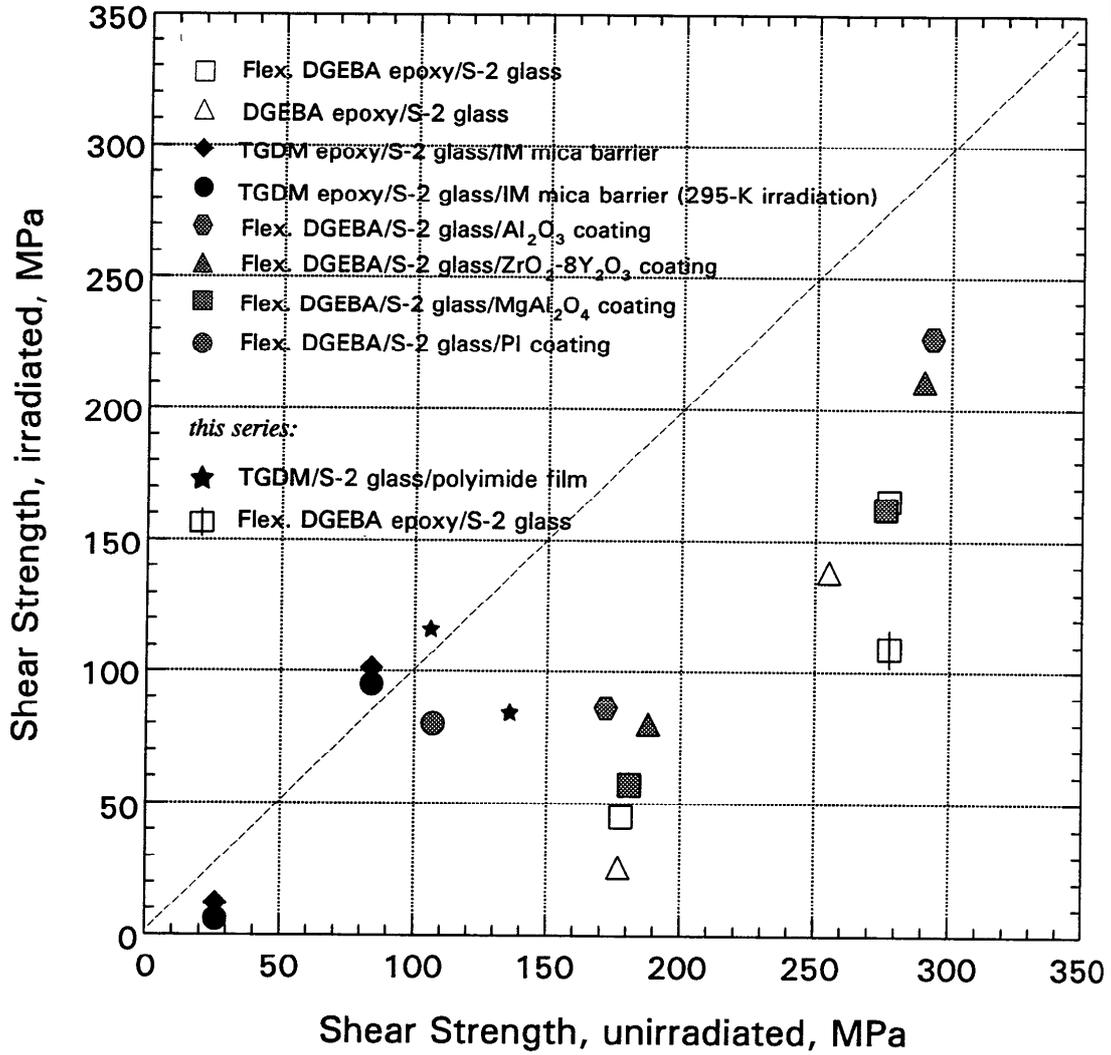


Figure V-3. Irradiated ( $1.8 \times 10^{22}$  n/m<sup>2</sup>) fast neutron fluence at 4 K) Versus unirradiated shear strength (at 4 K) measured under combined shear/compressive loads at 45° and 75°.

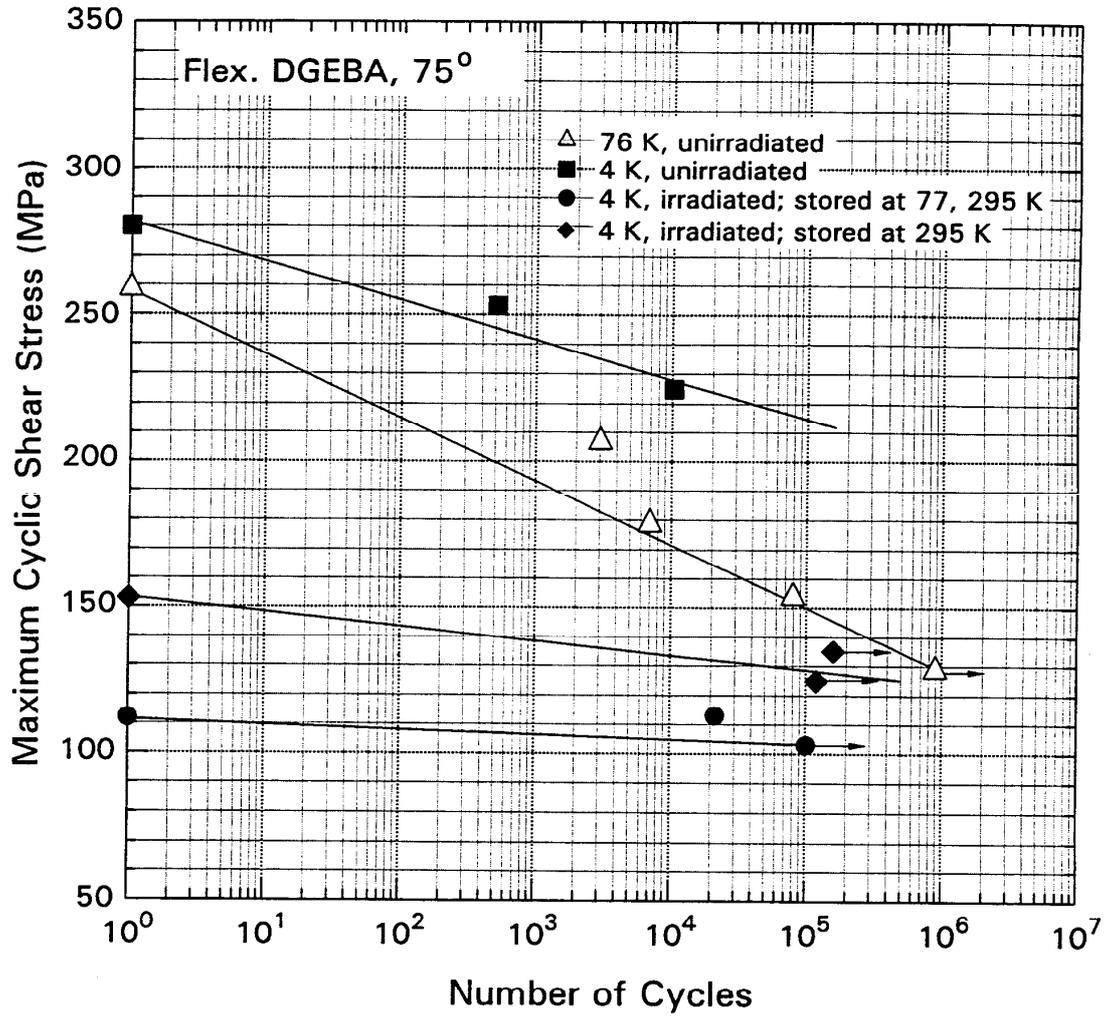


Figure V-4. Fatigue *S-N* curves of flexibilized DGEBA epoxy/S-2 glass at 76 and 4 K under combined shear/compressive stress, plotted versus compressive stress.

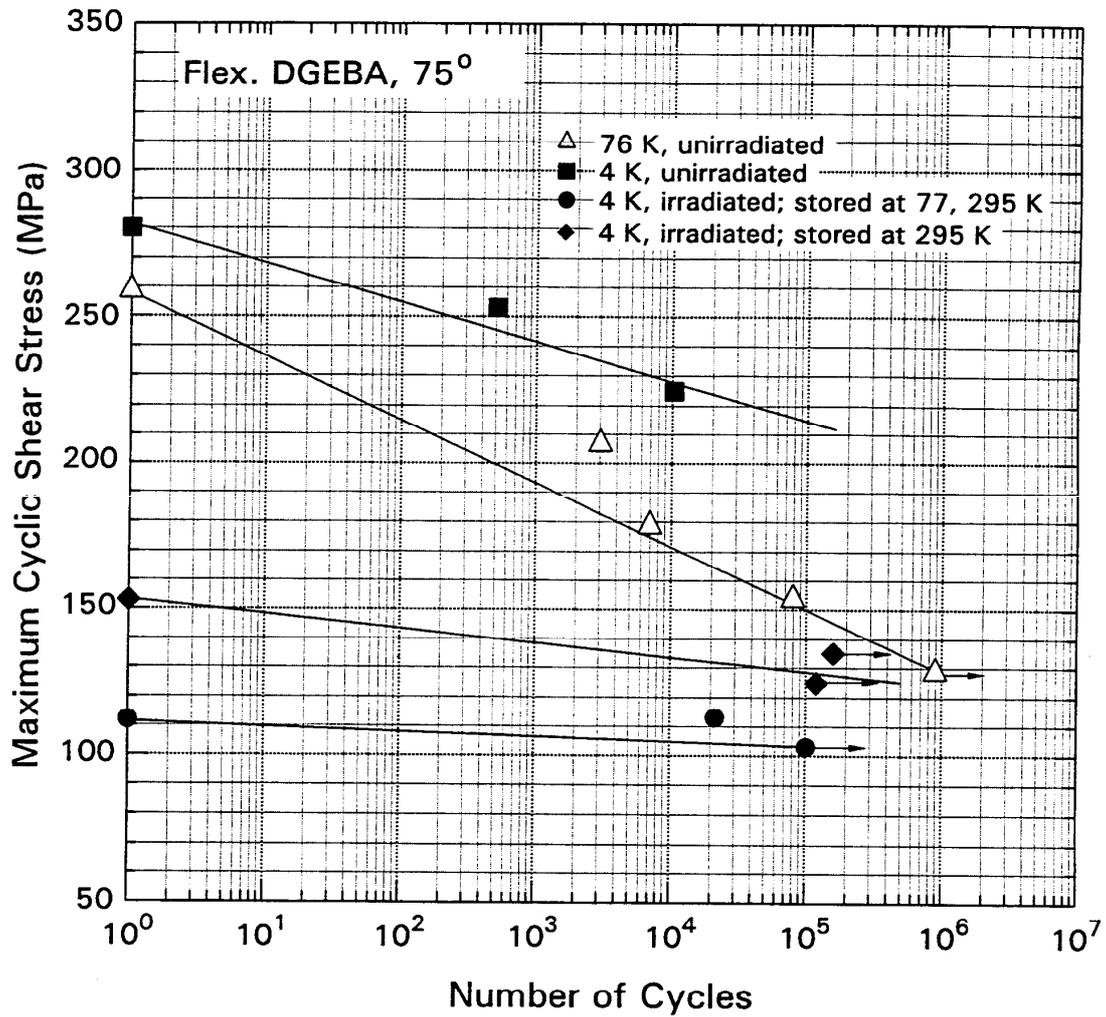


Figure V-5. Fatigue strengths of flexibilized DGEBA epoxy/S-2 glass at 76 and 4 K under combined shear/compressive loading, plotted versus maximum cyclic shear stress.

run were warmed to room temperature and stored. Notice that both the static and fatigue strengths of these specimens were larger.

The fatigue strengths of unirradiated specimens were higher than those of irradiated specimens. However, the dependence of fatigue life on cyclic stress of the irradiated specimens was much lower. Remarkably, the S/N curves of irradiated specimens are practically flat.

Another interesting factor illustrated by these very limited data is the difference between the S-N curves at 76 and 4 K. The fatigue life is apparently less dependent on cyclic stress at 4 K than at 76 K. This observation clearly requires more fatigue tests of unirradiated specimens at 4 K for confirmation. The results of these tests will be reported elsewhere.

*Compression tests*

Compression tests of unirradiated (control) and irradiated specimens were conducted at 4 K. The results are summarized in Table V-3. These data are plotted in Figures V-6 through V-8 and compared with earlier results.

In general, irradiation reduced the compressive strengths and moduli, but since it affected the moduli more than the strength, strain-to-failure values were larger for irradiated specimens. The most likely explanation for these effects is that compressive fracture paths must propagate through glass plies, and the glass is much less sensitive to radiation than the epoxy matrix. The through-thickness compressive modulus, however, is very dependent on the resin matrix.

Table V-3. Compressive Strength and Modulus of Unirradiated and Irradiated Specimens

System	Irradiation Level (fast neutron fluence, n/m <sup>2</sup> )	Strength			Modulus			Strain		
		Average (MPa)	S.D. (MPa)	C.V.	Average (GPa)	S.D. (GPa)	C.V.	Average	S.D.	C.V.
flex. DGEBA (desized S-2 glass)	1.8 × 10 <sup>22</sup>	1200	60	0.05	16.8	0.4	0.02	7.15	0.25	0.04
	0	1330	40	0.03	23.7	0.3	0.01	5.62	0.25	0.05
flex. DGEBA	1.8 × 10 <sup>22</sup>	1200	50	0.04	20.6	0.5	0.03	5.84	0.37	0.06
DGEBA (desized S-2 glass)	1.8 × 10 <sup>22</sup>	1120	40	0.03	16.8	1.2	0.07	6.71	0.66	0.10
	0	1370	10	0.01	25.1	0	0	5.45	0.06	0.01

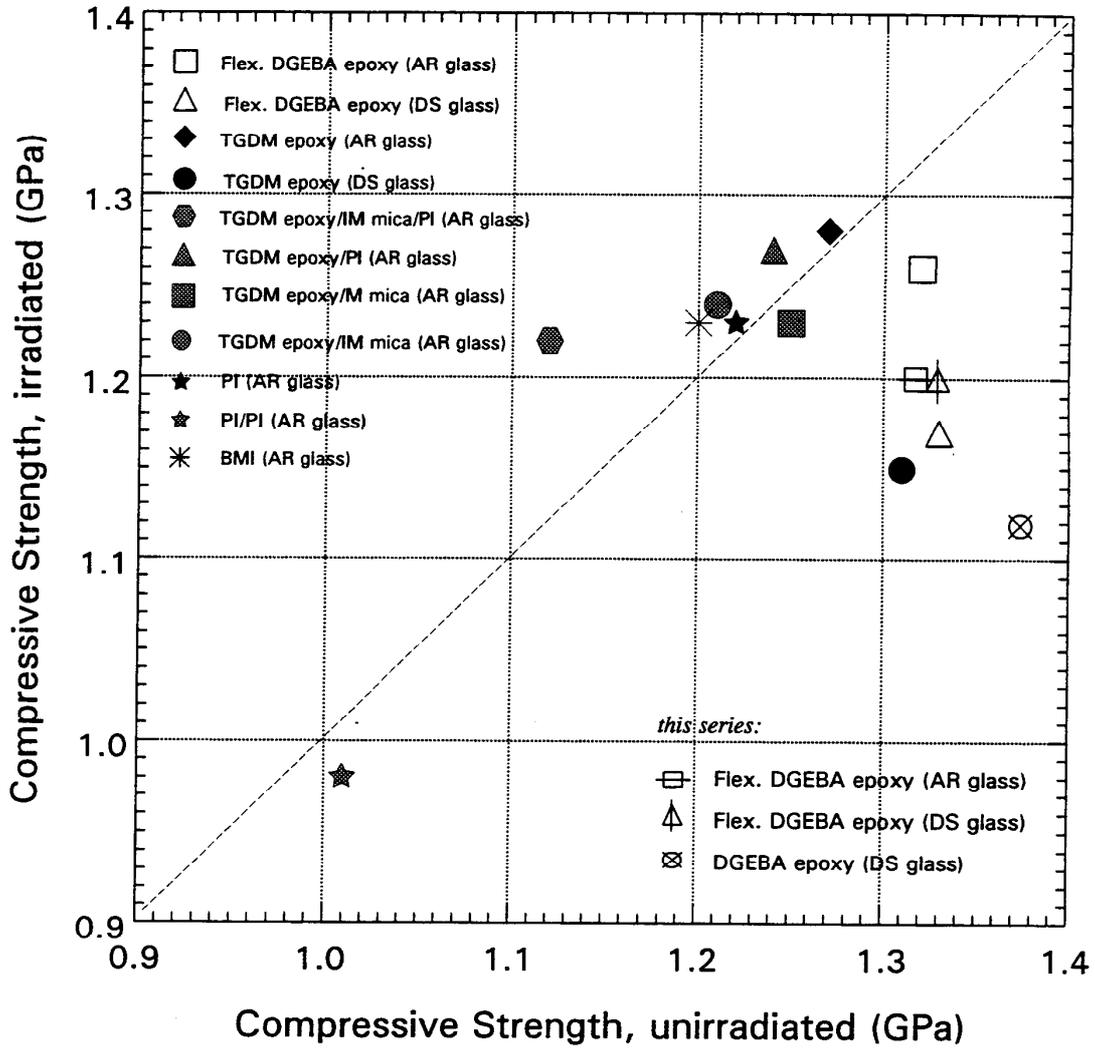


Figure V-6. Irradiated ( $1.8 \times 10^{22} \text{ n/m}^2$ ) versus unirradiated compressive strength at 4 K.

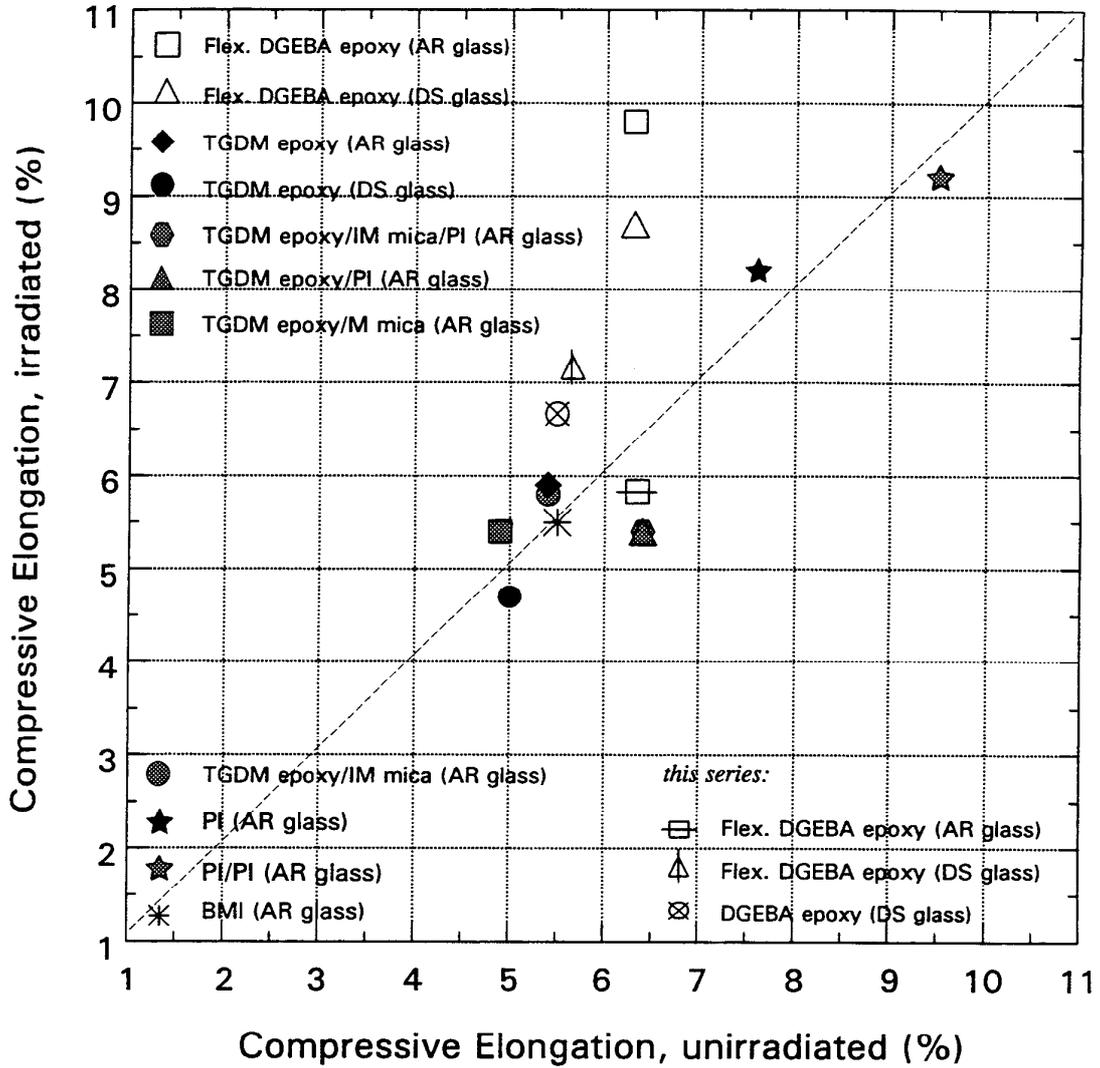


Figure V-7. Irradiated ( $1.8 \times 10^{22} \text{ n/m}^2$ ) versus unirradiated compressive elongation to failure at 4 K.

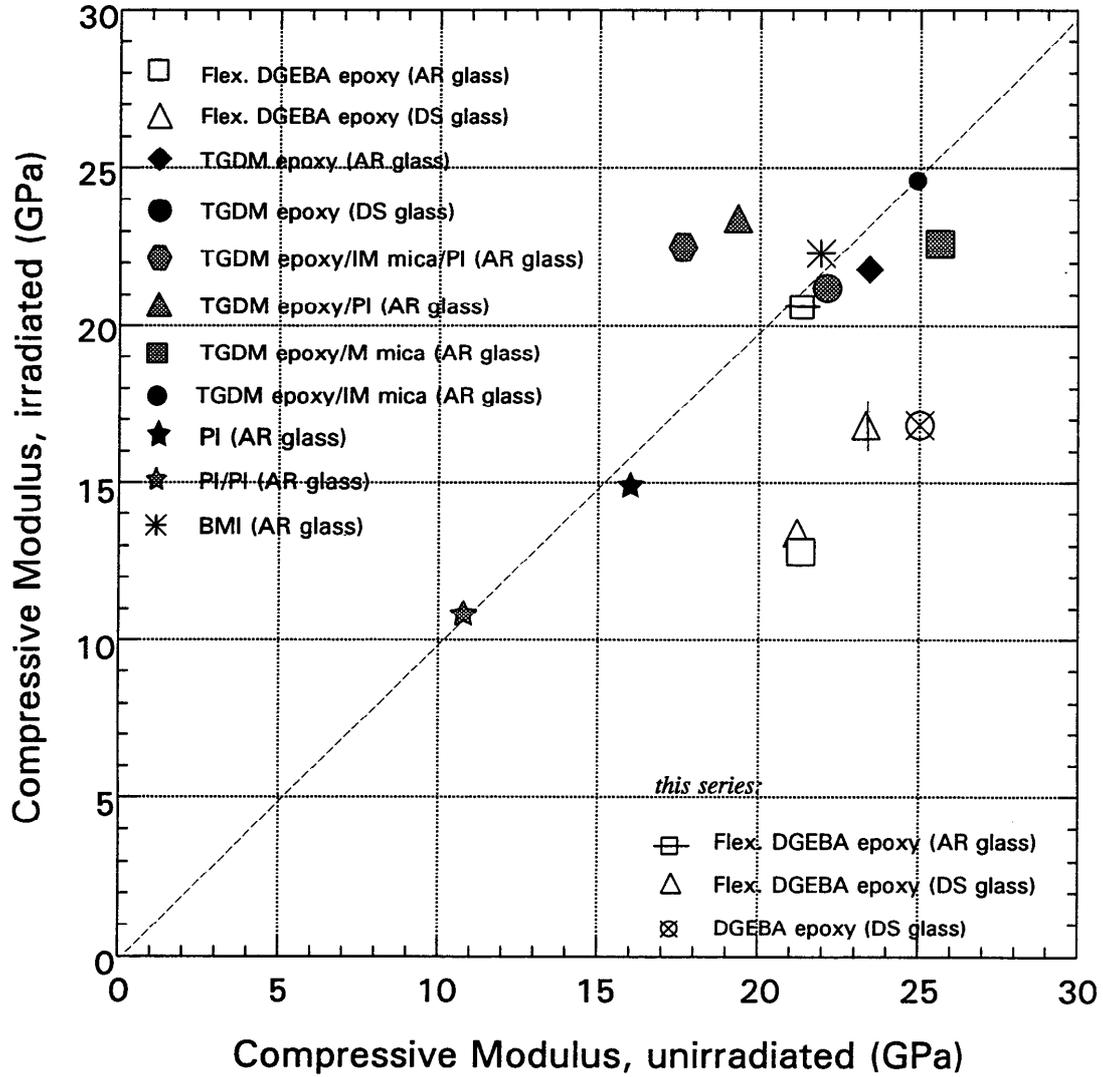


Figure V-8. Irradiated ( $1.8 \times 10^2 \text{ n/m}^2$ ) versus unirradiated compressive modulus at 4K. Some data points have been adjusted from Appendix B data owing to recent calibration of the displacement of the load platens.

Short-beam shear tests

The results of the short-beam shear tests at 4 K of irradiated specimens (1.8 ( 1022 fast neutrons/m<sup>2</sup>) are summarized in Table V-4. Quite obviously, the use of desized S-2 glass seriously degraded the interlaminar shear strength of insulation systems after irradiation. The results are compared with earlier data in Figures V-9 through V-11.

Table V-4. Shear Strength and Flexural Modulus of Irradiated Specimens

System	Shear Strength			Flexural Modulus		
	Average (MPa)	S.D. (MPa)	C.V.	Average (MPa)	S.D. (GPa)	C.V.
flex. DGEBA (as-received S-2 glass)	31.4	0.2	0.00	24.8	0.6	0.02
flex. DGEBA (desized S-2 glass)	10.6	1.3	0.12	16.8	1.7	0.10
DGEBA (desized S-2 glass)	8.1	0.4	0.04	16.9	2.3	0.13

Measurements of mass and dimensions

Changes in mass and dimensions following irradiation (at 4 K) and storage (at 77 and 295 K) of short-beam shear specimens are summarized in Table V-5. These data are compared with previous results in Figures V-12 through V-14. This set of data supports the following conclusions: DGEBA-type epoxy systems lose weight and in-plane length but increase in the through-thickness direction. The decrease in weight is probably caused by the evolution and escape of gas formed by free-radical recombination. The increase in through-thickness is caused by trapped evolved gas that forms in newly created or expanded pores.

Table V-5. Changes in Mass and Dimensions

System	Test	Change in Dimension (%)		
		Through-thickness	In-plane	Mass
flex. DGEBA (AR glass)	S/C	+5.8		
TGDM (AR glass)	S/C	+2.3		
flex. DGEBA (AR glass)	SBS	+2.65	-0.08	-0.43
flex. DGEBA (DS glass)	SBS	+1.34	-0.24	-0.64
DGEBA (DS glass)	SBS	+1.08	-0.28	-0.88

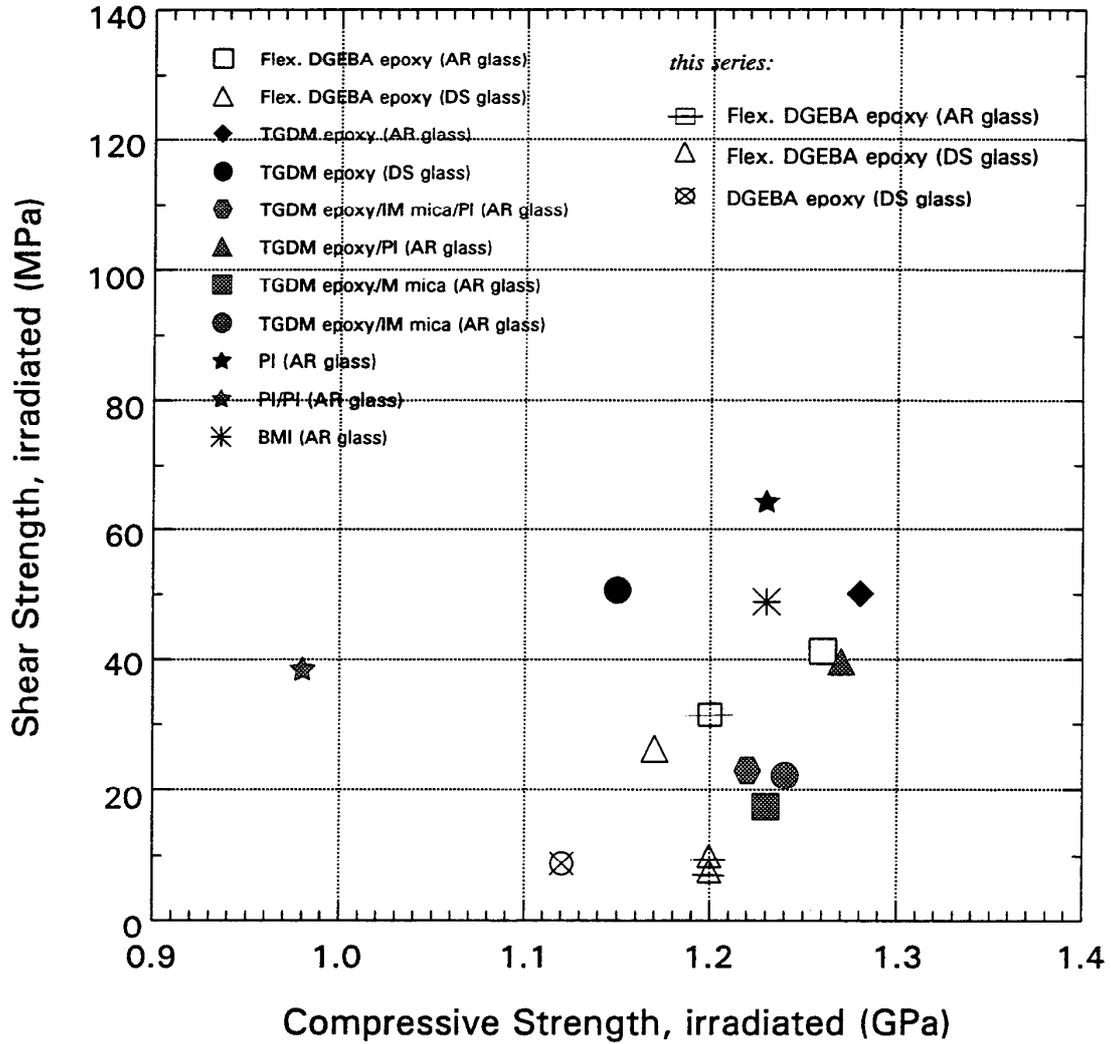


Figure V-9. Irradiated shear strength versus irradiated compressive strength at 4 K for all systems (fast neutron fluence =  $1.8 \times 10^{22}$  n/m<sup>2</sup>).

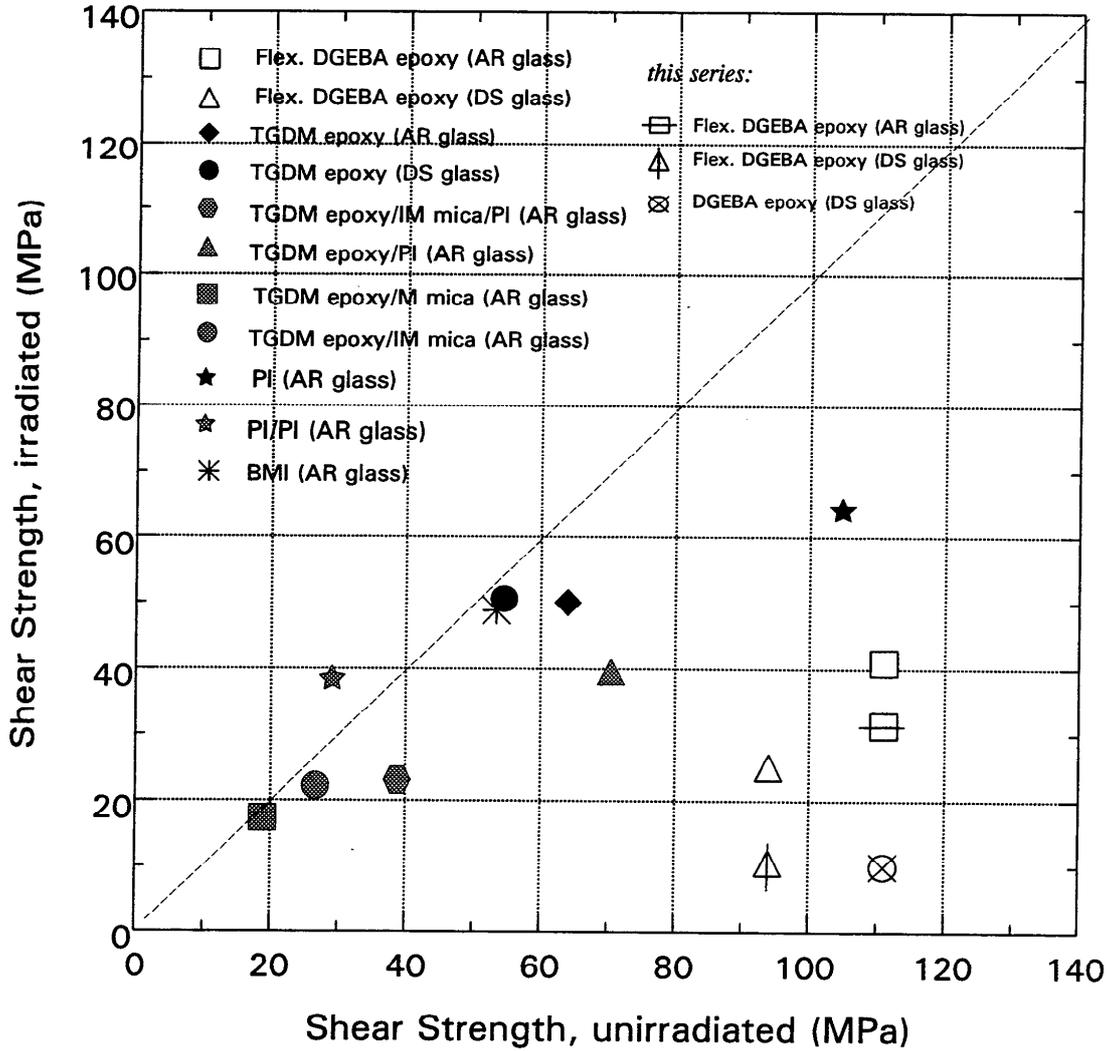


Figure V-10. Irradiated ( $1.8 \times 10^{22} \text{ n/m}^2$ ) versus unirradiated interlaminar shear strength at 4 K, measured with short-beam-shear tests.

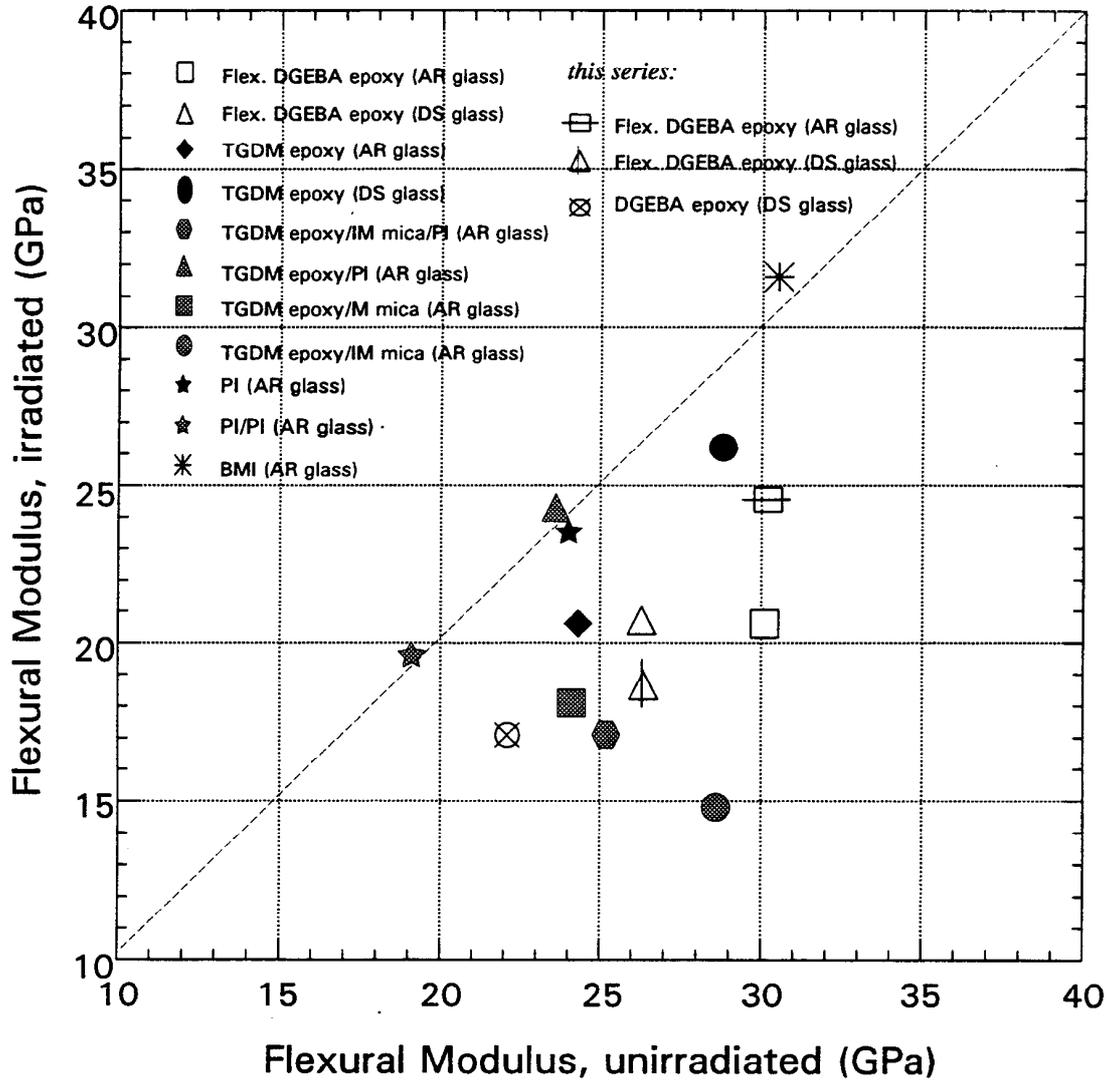


Figure V-11. Irradiated ( $1.8 \times 10^{22} \text{ n/m}^2$ ) versus unirradiated flexural modulus at 4 K, measured with short-beam-shear tests.

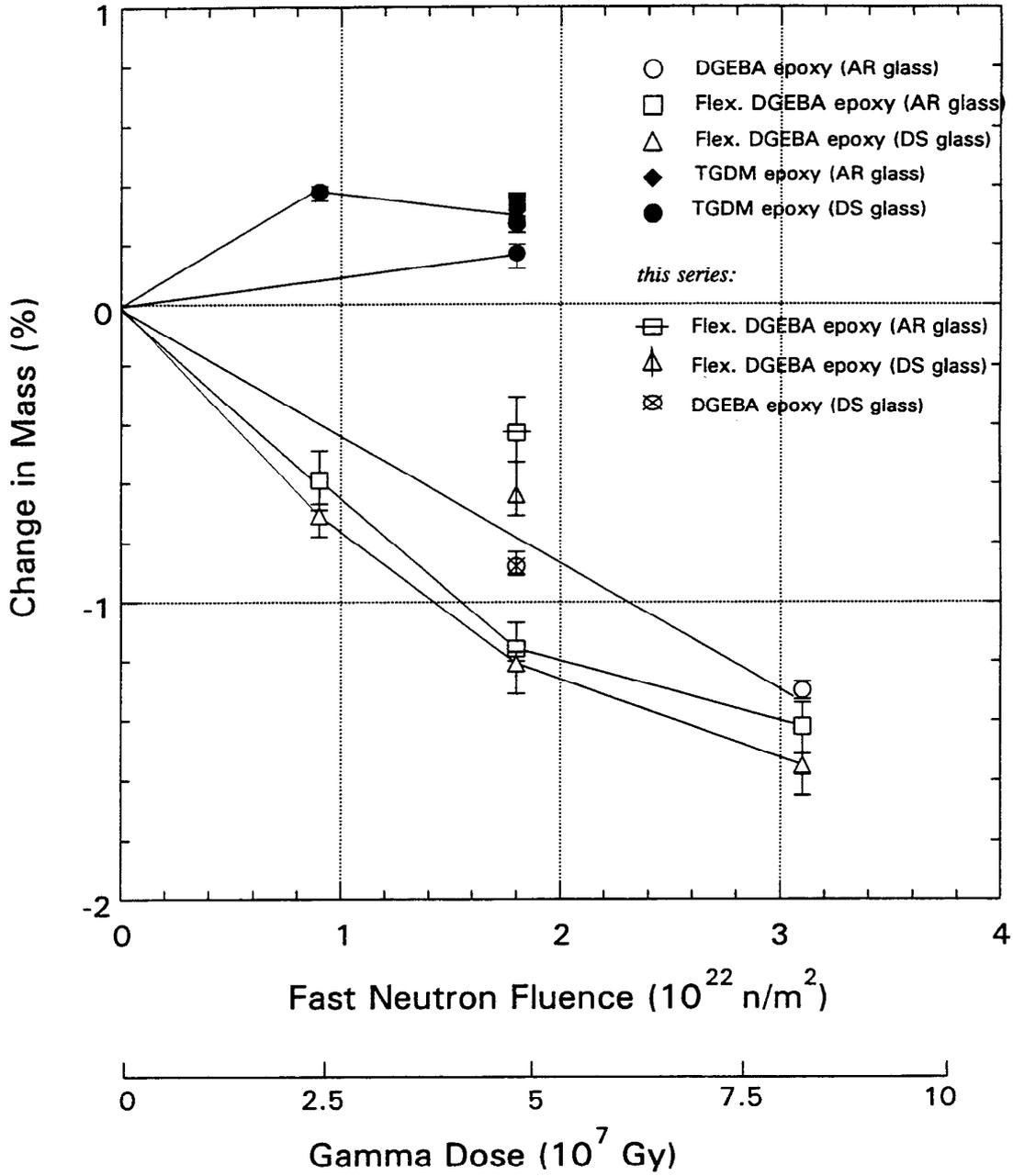


Figure V-12. Change in mass as a function of irradiation at 4 K of selected candidate TF-coil insulation systems.

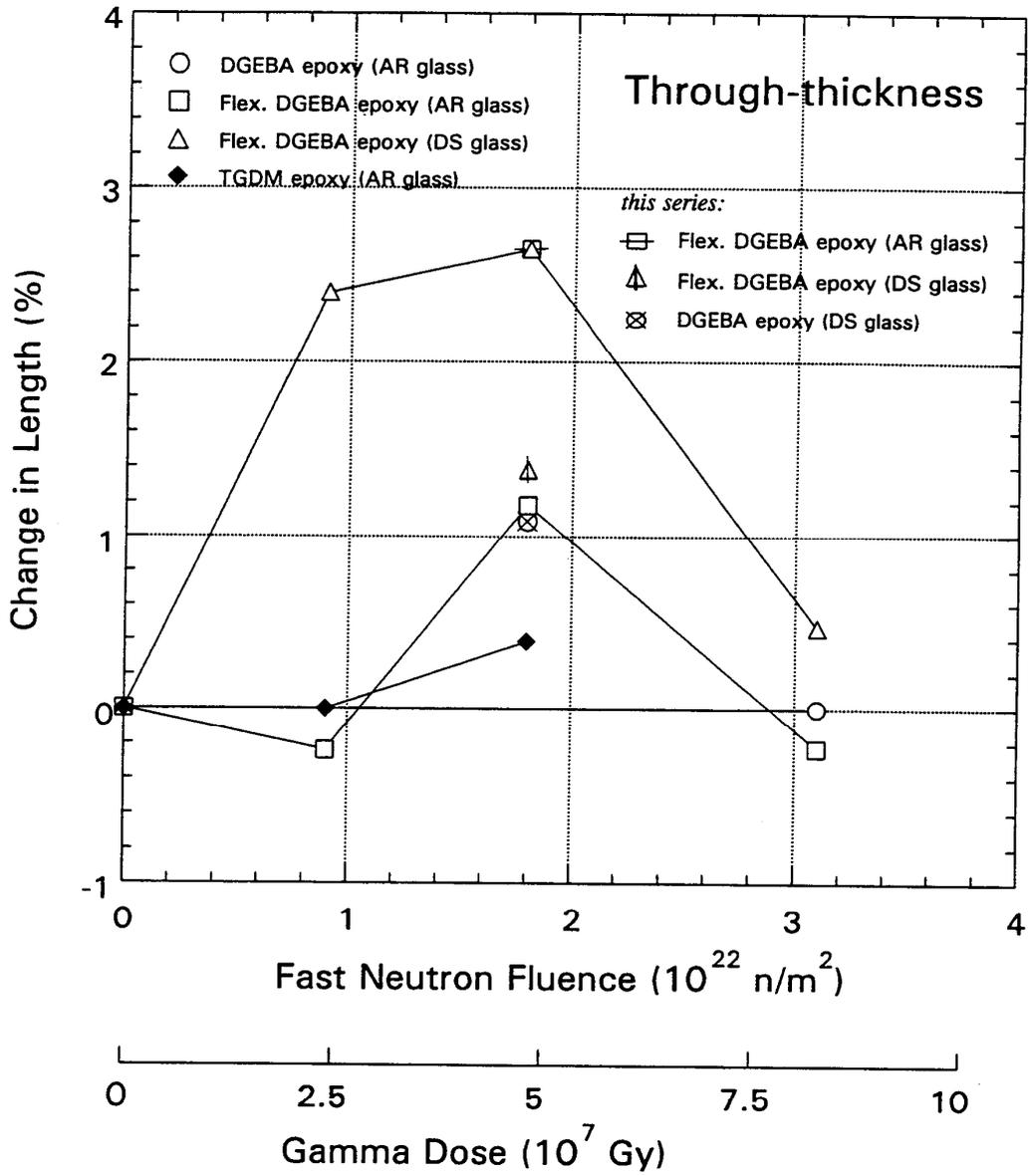


Figure V-13. Average of change in through-thickness length of short-beam shear specimens following irradiation at 4 K.

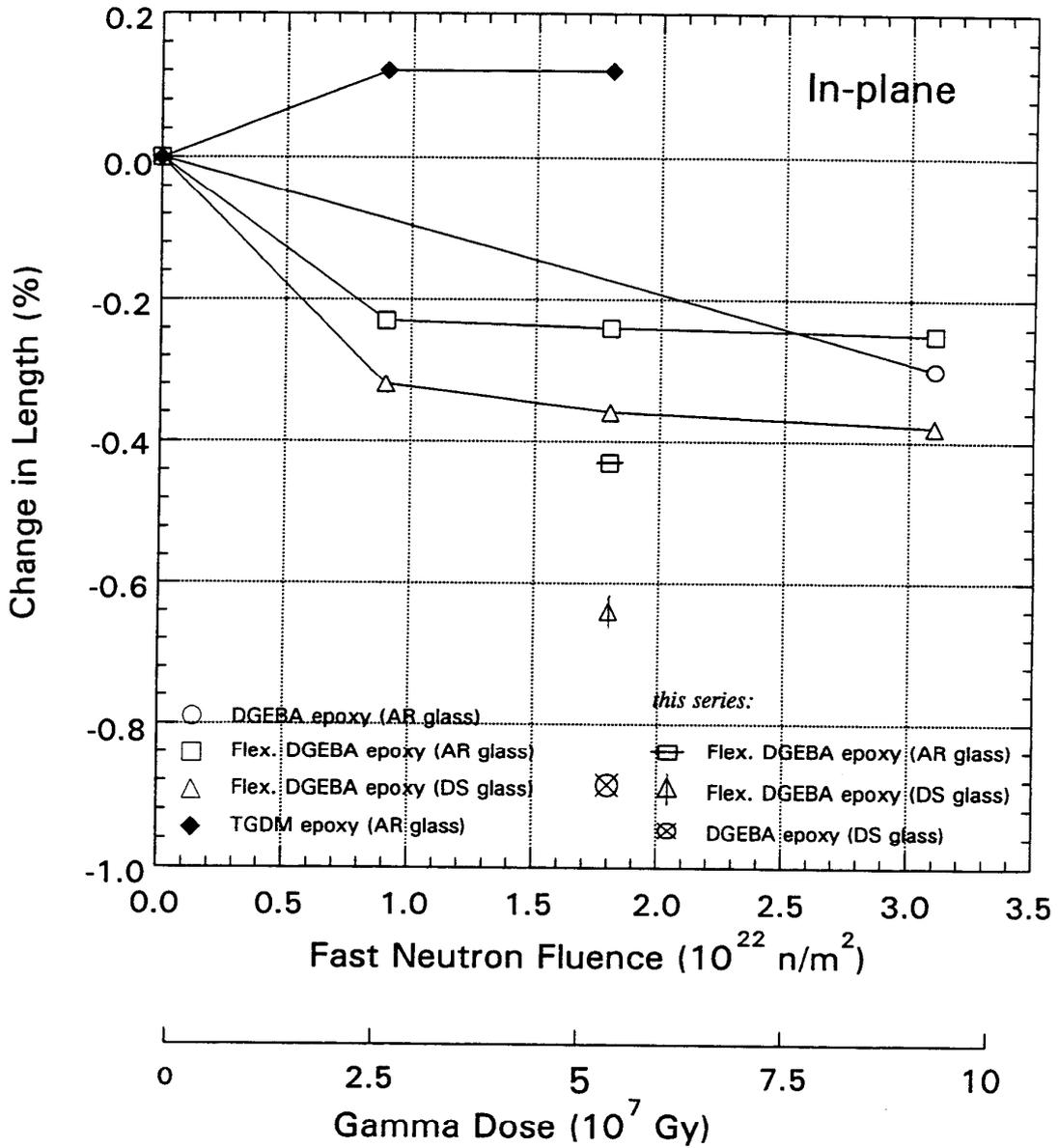


Figure V-14. Average of change in in-plane length of short-beam shear specimens following irradiation at 4 K.

The through-thickness data for the shear/compression specimens may be less precise. Measurement changes across the entire specimen were required, including across 1.3 mm of insulation and 7 mm of 316LN. The percentage change was calculated by assuming that the entire change of length occurred within the 1.3-mm-thick insulation system. The larger changes in the shear/compression specimens may reflect less surface area per unit volume of the specimen. Less surface area per unit volume may inhibit the ability of gas (formed from free-radical particles) to escape during the warm-up and room-temperature holding process. These data must be checked with more tests or measurements, since volume changes as high as 5% will seriously affect strength levels of the insulation systems during subsequent magnet operation.

## Summary

The prepreg TGDM/S-2 glass polyimide film insulation system was relatively insensitive to radiation; shear strengths (determined in 45° shear/compression tests) decreased only about 25% after irradiation to  $1.8 \times 10^{22}$  n/m<sup>2</sup>.

The VPI DGEBA/S-2 glass insulation systems were more sensitive to radiation; shear strengths (determined in 45° shear/compression tests) decreased more than 50% after irradiation to  $1.8 \times 10^{22}$  n/m<sup>2</sup>.

The fatigue life of an irradiated VPI insulation system was much less dependent on the cyclic stress than in the unirradiated condition. At  $10^6$  to  $10^7$  cycles, the fatigue strengths of irradiated and unirradiated specimens of one VPI system (flex. DGEBA/S-2 glass) tested at 75° appeared to be equivalent, but more data are needed to generalize this finding.

Systems that contained desized S-2 glass had higher sensitivity to radiation than systems that contained a silane glass finish compatible with epoxy resins.

Of the specimens tested for this report, the shear strengths of specimens that had been stored at 77 K for ten months were lower than those of specimens that had been stored only at room temperature. The explanation for this difference is not clear.

In accord with results of earlier measurements, after irradiation the DGEBA epoxy specimens lost weight, increased in the through-thickness dimension, and decreased slightly in the in-plane dimension. The mass and dimensions of the TGDM epoxy system were relatively insensitive to radiation.

## **Irradiation at Room Temperature**

A limited number of specimens were irradiated at room temperature in an in-core reactor position. At room temperature, the ratios of thermal neutron fluence and gamma dose to fast neutron fluence increase by factors of 4 and 2.5, respectively. Thus, for the same fast neutron fluence of  $1.8 \times 10^{22}$  n/m<sup>2</sup>, the estimated total dose for 4-K irradiation is  $6.1 \times 10^7$  Gy, and for 300-K irradiation, it is  $12.9 \times 10^7$  Gy; in each case only the contributions from fast neutrons and gamma rays are considered.

The short-beam shear strengths at 4 K of 4- and 300-K irradiated specimens (both to a fast neutron fluence of about  $1.8 \times 10^{22}$  n/m<sup>2</sup>) are compared in Figure 13. In all materials, the shear strengths of the specimens irradiated at 300 K were lower than the shear strengths of the specimens irradiated at 4 K. This result must be attributed to the damage from the increased gamma dose and thermal neutron fluence.

The compressive strengths at 4 K of 4- and 300-K irradiated specimens do not differ significantly. In general, the compressive moduli at 4 K of specimens irradiated at 295 K are about 10% lower than those measured on specimens irradiated at 4 K. However, the compressive moduli of the flexibilized DGEBA epoxy composite specimens were quite different: the average modulus of specimens irradiated at 300 K was 25.4 GPa; that of specimens irradiated at 4 K was 14.6 GPa. The reason for this large disparity is not clear.

Shear/compression specimens of one insulation system (TGDM/mica) were irradiated at both temperatures and tested at 45° (1 specimen each) and 75° (2 specimens each). The shear strength under 45° testing of the specimen irradiated at 300 K was 6 MPa, half that (12 MPa) of the specimens irradiated at 4 K, and, at 75°, the average shear strengths were 100 and 95 MPa for the 4- and 300-K irradiated specimens, respectively.

Adding the room-temperature irradiation data to Figure I-1 of Update I (p. 28), a plot of short-beam shear strengths versus total fast neutron fluence (see Figure 14) is informative. The shear strengths of the DGEBA epoxy specimens fall well within the data scatter; however, the shear strengths of the TDGM epoxy specimens that were irradiated at 300 K are much too low. Apparently, the data are inconsistent. Either the DGEBA epoxy data are sensitive primarily to fast neutron irradiation and the TGDM epoxy is sensitive to all types of irradiation (gamma, thermal, and fast neutron), or the scatter of data between the two sets of irradiation experiments is too great to discuss the distinctions between 4-K and 300-K irradiation or between the types of irradiation, with regard to their effects on interlaminar shear strength.

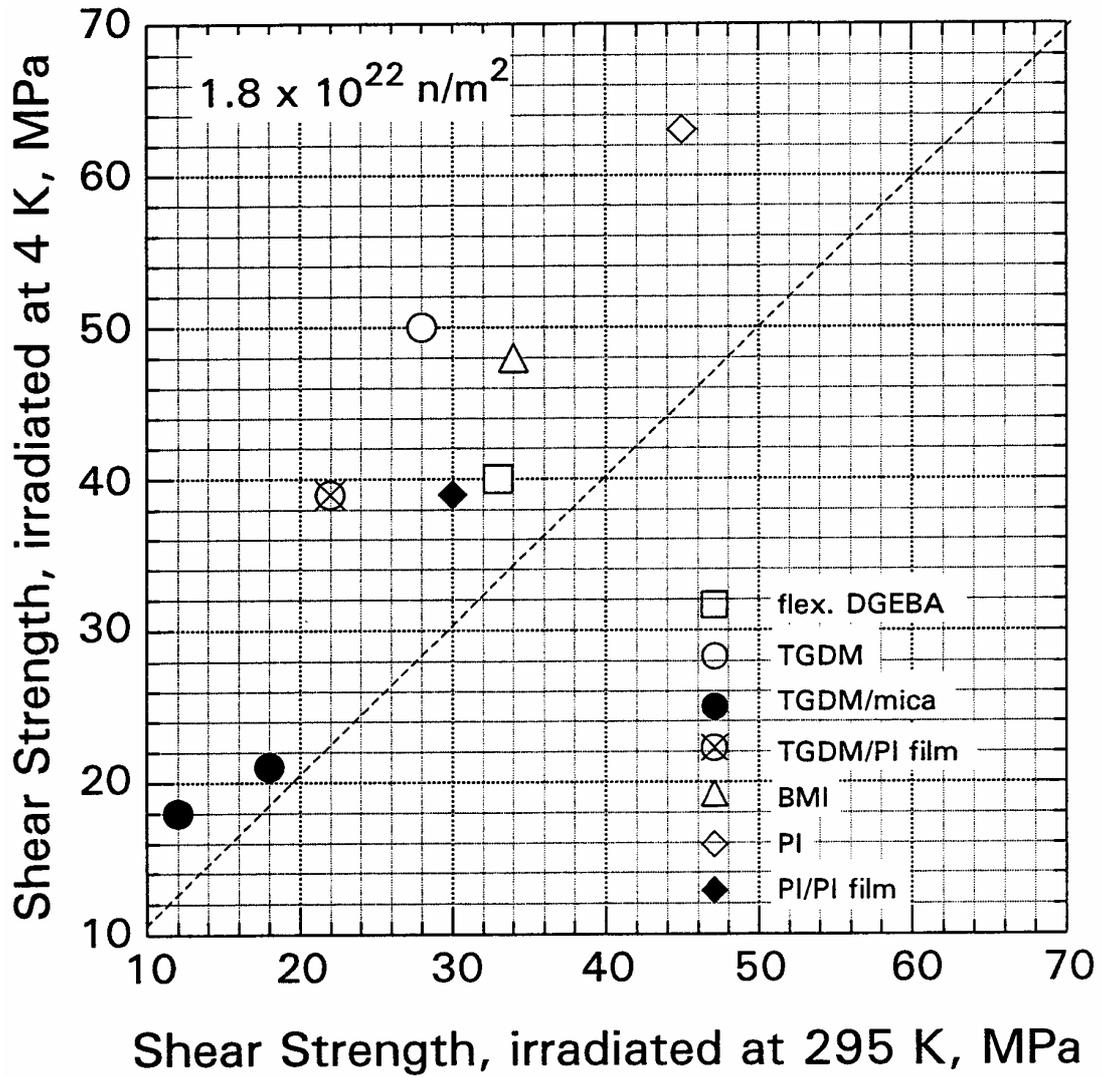


Figure 13. Short-beam shear strengths at 4 K of specimens irradiated at 4 and 300 K.

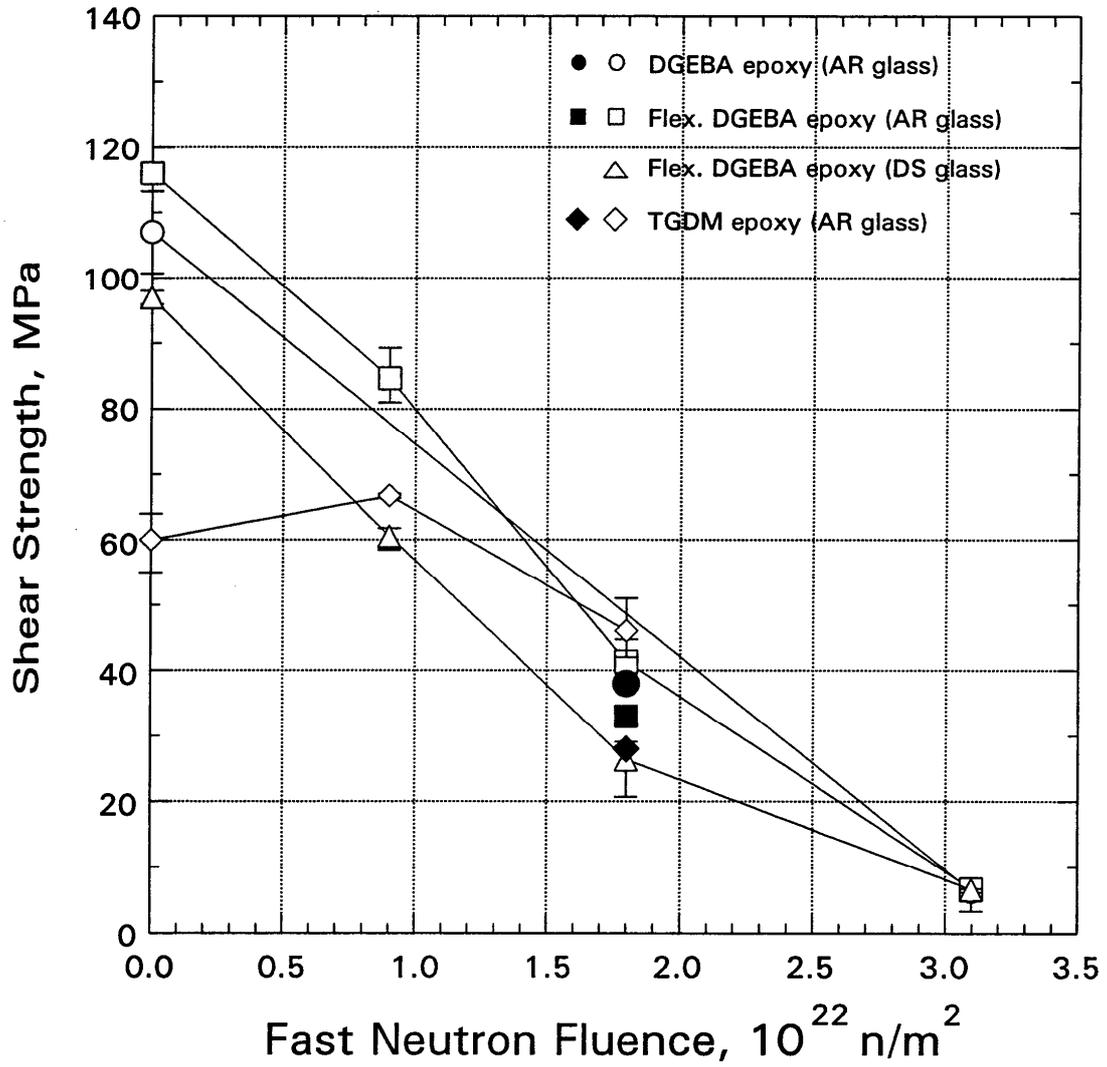


Figure 14. Interlaminar shear strengths at 4 K versus fast neutron fluence for specimens irradiated at 4 K (open symbols) and at 300 K (solid symbols).

## Summary

The radiation sensitivity of electrical insulation materials and properties are summarized in this section. The results are analyzed with two objectives in mind: (1) select the best materials for insulation systems used in a radiation environment and (2) determine how the properties of these materials at low temperatures are altered following exposure to neutron and gamma radiation.

### Materials

Resin and electrical-barrier systems were evaluated in this program. In all specimens, reinforcement was provided by S-2 glass, 6781 satin weave, 50 vol%.

### Resins

Various properties are sensitive to the strength of resins in laminate composite systems. The failure modes in the short-beam shear and shear/compressive tests were interlaminar cracks that ran through the resin or through the resin/glass interface. The compressive elastic modulus and mass and dimensional changes from irradiation are also dominated by contributions from the resin, since the S-2 glass is not sensitive to radiation.

From the data reported in Updates I through V, the sensitivities to radiation of the resin systems included in this study are ranked in Table 5. This ranking agrees with conclusions from past studies<sup>1,2</sup> and the ongoing evaluation of the gas evolved from irradiated and warmed resin systems.<sup>22-24</sup> (References 22 and are included in Appendix A.)

### Barriers

A number of candidate electrical barrier systems were also evaluated: polyimide film (Kapton grade HA), mica, and plasma-sprayed and painted inorganic and organic coatings. The incorporation of a barrier affected the shear strengths and flexural modulus, but not the compressive properties. After irradiation, the voltage breakdown of all barrier systems were essentially unchanged from unirradiated values. With regard to mechanical properties, the barriers are ranked and their characteristics are compared in Table 6.

The fracture mode of all specimens with plasma-sprayed coatings was interlaminar shear failure of the epoxy/glass composite. Two plasma-sprayed coatings,  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2/\text{Y}_2\text{O}_3$ , actually improved the resistance of the adjacent flexibilized DGEBA/S-2 glass composite: The relatively high porosity (~30%) of the coatings may have acted as a sink for the gases that evolved from the resin during warm-up. The  $\text{MgAl}_2\text{O}_4$  (spinel) did not affect the radiation resistance of the epoxy/glass composite.

Table 5. Relative Sensitivity of Resin Systems to Radiation

Rank	Changes in 4-K Properties after $1.8 \times 10^{22}$ n/m <sup>2</sup> Fast Neutron Fluence plus $1 \times 10^7$ Gamma Dose
Highest sensitivity to radiation: flex. DGEBA and DGEBA epoxies (VPI resins)	<ul style="list-style-type: none"> <li>• &gt;50% reduction in shear strength</li> <li>• 40–50% reduction in compressive modulus</li> <li>• &gt;75% loss of shear/compressive strength (45°)</li> <li>• 0.4–1.5% reduction in mass</li> <li>• 0.25–0.35% reduction in in-plane length</li> <li>• 1–5.8% increase in through-thickness dimension</li> </ul>
Lowest sensitivity to radiation: TGDM epoxy (PP, HPL resin)	<ul style="list-style-type: none"> <li>• 20% reduction in shear strength</li> <li>• 5–10% reduction in compressive modulus</li> <li>• 25% loss in shear/compressive strength (45°)</li> <li>• no change in mass</li> <li>• 0.1% increase in in-plane length</li> <li>• 0.1–0.4% increase in through-thickness dimension</li> </ul>
bismaleimide and polyimide (PP, HPL resins)	<ul style="list-style-type: none"> <li>• no change in shear strength*</li> <li>• no change in compressive modulus</li> </ul>

\* Except for specimens of polyimide resin with epoxy-compatible silane glass sizing; they exhibited a 40% reduction of short-beam shear strength.

Table 6. Relative Sensitivity of Barriers to Radiation

Rank	Characteristics after Irradiation to $1.8 \times 10^{22}$ n/m <sup>2</sup> Fast Neutron Fluence
Highest sensitivity to radiation: mica	<ul style="list-style-type: none"> <li>• lowest absolute values of shear strength</li> <li>• 10–50% reduction in shear strength</li> <li>• 25–50% reduction in flexural modulus</li> <li>• 50–80% reduction in shear/compressive strength (45°)</li> </ul>
Intermediate sensitivity to irradiation polyimide film	<ul style="list-style-type: none"> <li>• 40% reduction in shear strength</li> <li>• no change in flexural modulus</li> <li>• 10–20% reduction in shear/compressive strength (45°)</li> </ul>
Lowest sensitivity to radiation: inorganic coatings	<ul style="list-style-type: none"> <li>• shear/compressive strength higher than DGEBA/S-2 glass desized specimens</li> </ul>

## Reinforcement

Throughout this program, boron-free S-2 glass was used for resin reinforcement. However, the effects of desizing the glass (removal of its epoxy-compatible silane finish) were studied. The results of tests following irradiation are consistent: the shear strengths and the shear/compressive strengths of desized glass specimens were always less than those of the as-received (sized) S-2 glass specimens. The changes in mass and dimensions of desized glass specimens were always slightly larger than the changes in as-received specimens. Thus, in a radiation environment, silane-finished S-2 glass is preferable to the desized S-2 glass.

## Properties

The loss of mechanical integrity of irradiated glass/epoxy composites depended on the extent of degradation of the resin system. The boron-free S-2 glass with epoxy-compatible silane finish retained essentially all of its mechanical strength after irradiation in a fission reactor (neutron, gamma radiation) at 4 K. The resins deteriorated, however, and the amount of degradation depended on the type of resin and the magnitude of the fast neutron fluence and total dose.

The composites evaluated for the electrical insulation of superconducting magnets were laminates with plies of glass weave for reinforcement; in some cases, electrical barriers (such as polyimide film) were interspersed between the glass plies. Some mechanical properties of laminated composites are relatively insensitive to resin characteristics. For example, in compression tests, the specimens' fracture path in the through-thickness direction tended to be at 45° to the applied force as it passed through layers of glass ply. Thus, the through-thickness compressive strength is relatively independent of irradiation.

Interlaminar shear or shear/compressive strengths, however, strongly depended on the state of the resin and, thus, on irradiation. The compressive modulus in the through-thickness direction also decreased significantly with increasing irradiation. Since the through-thickness compressive strength remained insensitive to radiation and the compressive modulus decreased following irradiation, it is surprising that the compressive elongation to failure in the through-thickness direction increased with increasing irradiation.

Evidence suggests two principal reasons for the deterioration of resin systems under irradiation. The mass of the specimens decreased and their through-thickness dimension expanded after irradiation. From these characteristics, it is clear that radiation ionization effects produced gas that formed primarily during warm-up from 4 K (hydrogen gas may actually form from free radicals at 4 K). This gas either evolved from laminate-free surfaces or remained trapped within the composite laminate, which caused the resin to swell. Other, ongoing experiments are documenting the amount of evolved gas from neat resins as a function of irradiation dose.<sup>22-24</sup>

Another likely source of mechanical degradation of the resin is displacement damage due to fast neutrons. Other research<sup>1,2</sup> has identified that the mechanical properties of polymers degrade more after fast neutron radiation than after gamma radiation at equivalent dose levels.

Thus, both ionization (gamma, neutron) and displacement (neutron) damage contribute to the loss of mechanical strength and stiffness of some resin systems, but not all. Multifunctional chain epoxies and polyimide resins are superior to difunctional chain epoxies. A parallel study<sup>22-24</sup> is assessing radiation resistance based on different resin types, cure schedules, mixing ratios, accelerators, and antirads. The results of this study have demonstrated that, with proper selection of resins, the radiation sensitivity of insulation systems can be substantially improved. In the following subsections, the study results are summarized for the key characteristics: shear/compressive strength, compressive strength, dielectric strength, mass, and dimensions.

### **Shear/compressive strength**

For the VPI DGEBA resins and the PP TGDM resin with a polyimide film barrier, the static shear/compression test results of irradiated specimens compared with those of unirradiated specimens are summarized in Figures 15 and 16. They have been reported in Updates II and V. For the DGEBA resin system, shear/compressive fatigue data as a function of test angle have been reported in another paper.<sup>25</sup> Clearly the radiation sensitivity of the DGEBA epoxy resin system is much higher than that of the TGDM system, and, at approximately equal levels of applied shear and compressive forces (test angle = 45°), the TGDM system has much higher shear strengths (~80 MPa) than the DGEBA system (~45 MPa). From current ITER design analyses for both TF and SC coils, the maximum shear stresses (from global analyses) range from 20 to 35 MPa, with associated compressive stresses less than 300 MPa. Thus, both types of insulation systems can withstand these static stress magnitudes.

During operation, however, fatigue strengths and the effects of resin swelling must be considered.

### **Shear/compressive fatigue strength**

The available fatigue data on irradiated specimens are contained on pages 67 and 68 (Figures V-4 and V-5 of Update V) of this report. All data were obtained under cyclic loading at a specimen angle of 75° (combined shear and compressive forces). The *S-N* curves of irradiated specimens apparently are much flatter (i.e., the fatigue life is much less dependent on applied cyclic stress) than those of unirradiated specimens. At 10<sup>6</sup> cycles, the fatigue strengths of both irradiated (tested at 4 K) and unirradiated DGEBA epoxy systems (tested at 76 K) were nearly equivalent. However, notice the apparent disparities between the 76- and 4-K *S-N* curves for unirradiated specimens, which are also shown in these figures.

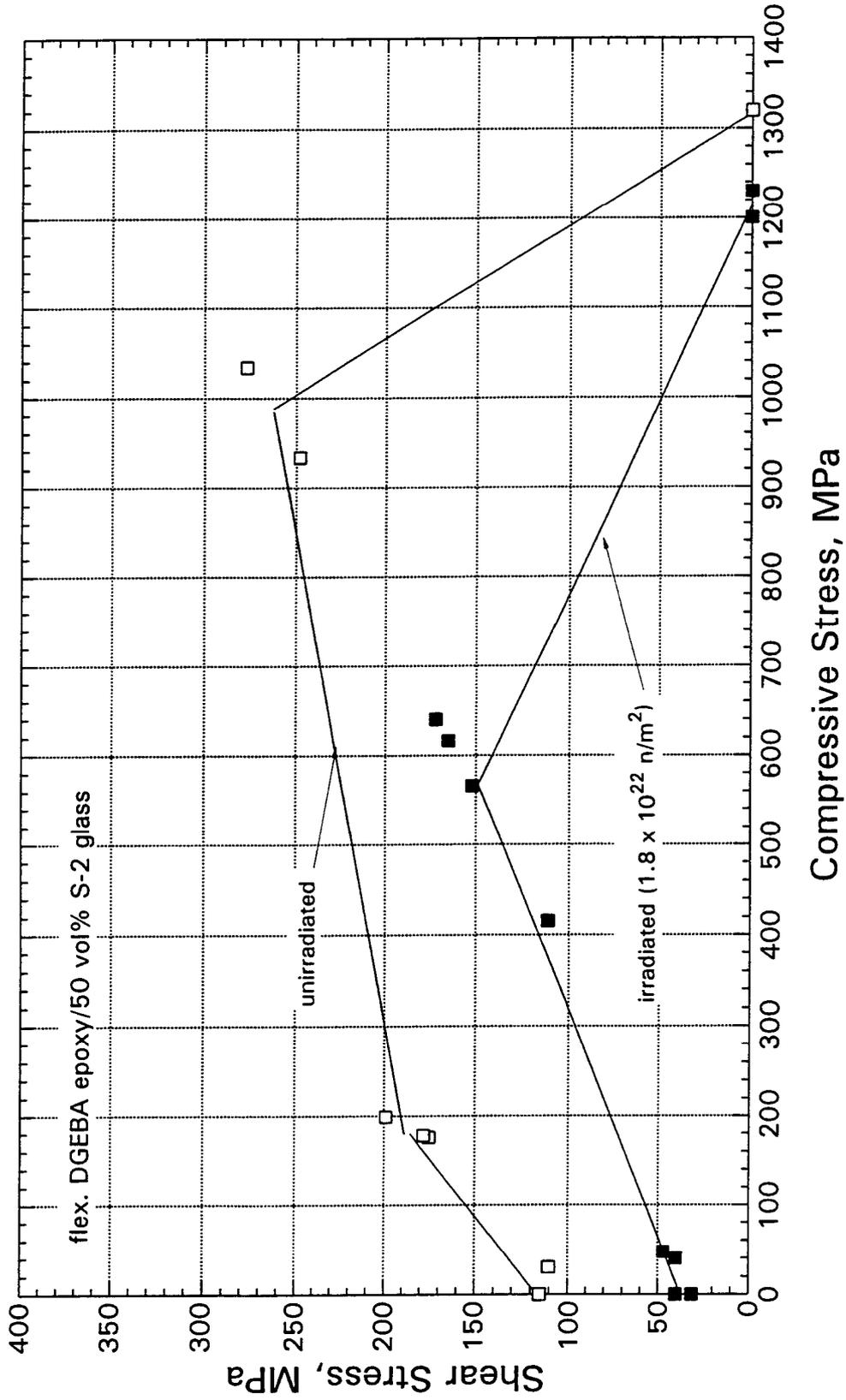


Figure 15. Static shear/compressive properties at 4 K of flexibilized DGEBA epoxy/50 vol% S-2 glass sandwich specimens.

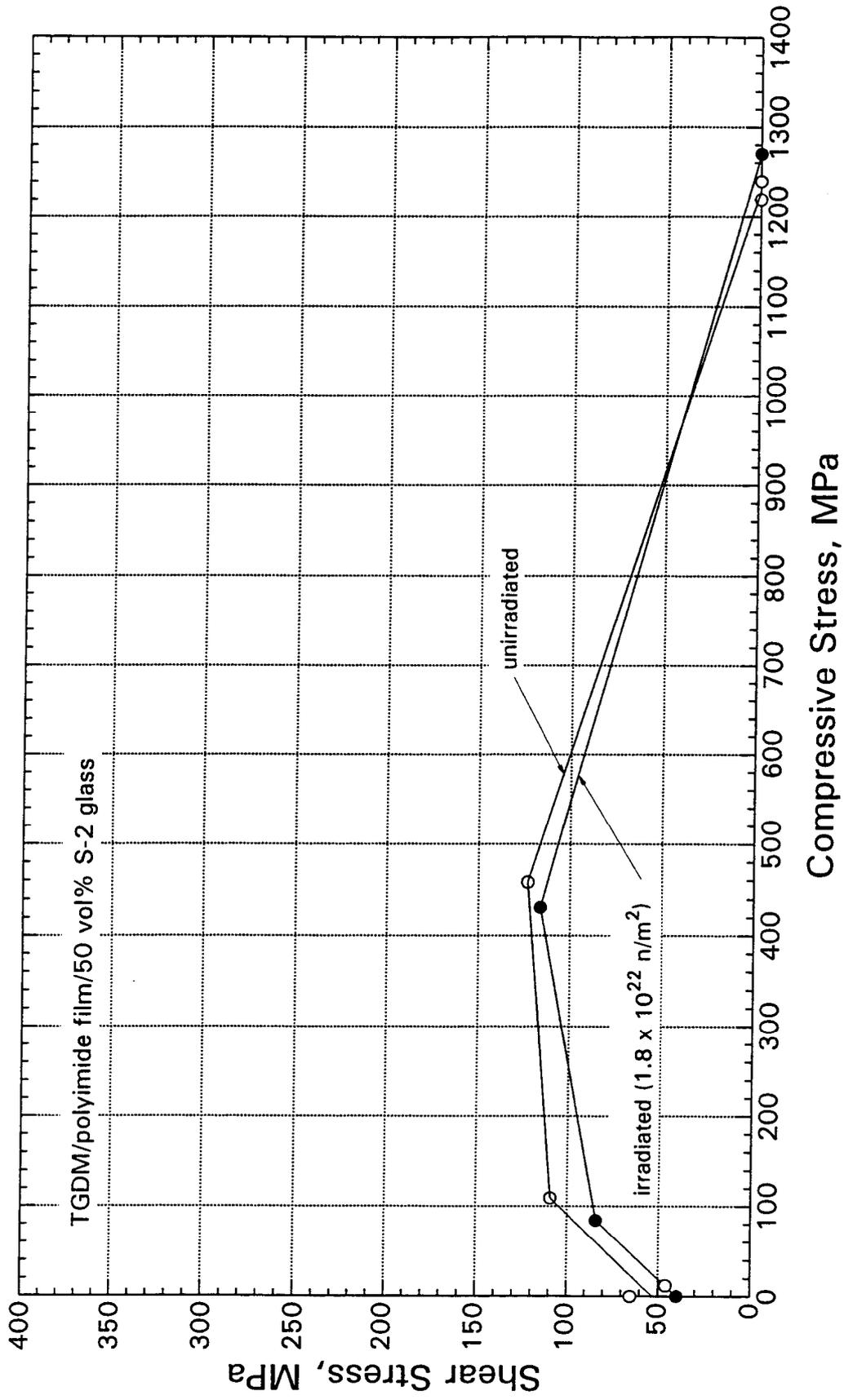


Figure 16. Static shear/compressive properties at 4 K of TGDM epoxy/polyimide film/50 vol% S-2 glass.

More measurements are necessary, and will be forthcoming, to verify this relatively strong temperature dependence. If this lower dependence of fatigue life at 4 K on cyclic stress is verified, then at  $10^6$  cycles, the fatigue strengths of irradiated specimens will be about 40% less than those of irradiated specimens of the DGEBA system.

In Figure 17 the estimated fatigue strength after  $10^6$  cycles is included with the static shear/compression data and with the unirradiated fatigue-strength data at 76 K. Over the range of compressive stresses to about 250 MPa, the limiting allowable stresses from the strengths of irradiated specimens and from 10 times the operational lifetime ( $10^6$  cycles) are almost equivalent. At layer compressive stresses, the fatigue life produces lower allowable shear/compressive stresses. When one considers that the expected maximum fast neutron fluence on the in-board side of the TF coils is now expected to be  $2.5 \times 10^{21}$  n/m<sup>2</sup>, the maximum possible safety factor (to account for hot spots and other uncertainties) on fast neutron fluence is expected to be 4. Thus, the use of data plotted in Figures 15 through 17 for a fast neutron fluence of  $1.8 \times 10^{22}$  n/m<sup>2</sup> for design purposes would be conservative.

### **Compressive properties**

In the through-thickness direction, the compressive modulus declines, the compressive strength remains nearly constant, and, therefore, the compressive strain-to-failure increases. The amount of change of the compressive modulus with irradiation depends on the type of resin system; the results for several systems have been reported in Updates I and V (see especially Figures V-6–V-8 of Update V, pp. 70–72), and they are summarized in Table 7.

Remarkably, the strain to failure at 4 K following irradiation is very high, from 5.5 to 9.7% for all resin systems. On the basis of their through-thickness compressive properties, listed in Table 7, all resin systems appear acceptable. Only the large decrease in compressive modulus of the DGEBA resin system suggests its strong sensitivity to radiation.

### **Dielectric strength**

Static voltage breakdown was measured on irradiated discs (13 mm in diameter, 0.025–0.5 mm thick) and reported in Update III (see Figure III-5, p. 51) and discussed by Schutz et al.<sup>16</sup> Insulation systems that included electrical barriers did not have a significant ( $\leq 5\%$ ) change of dielectric strength after exposure to  $1.8 \times 10^{22}$  n/m<sup>2</sup> at 4 K. The dielectric strengths of DGEBA and TGDM systems without electrical barriers were 15 to 40% less following irradiation.

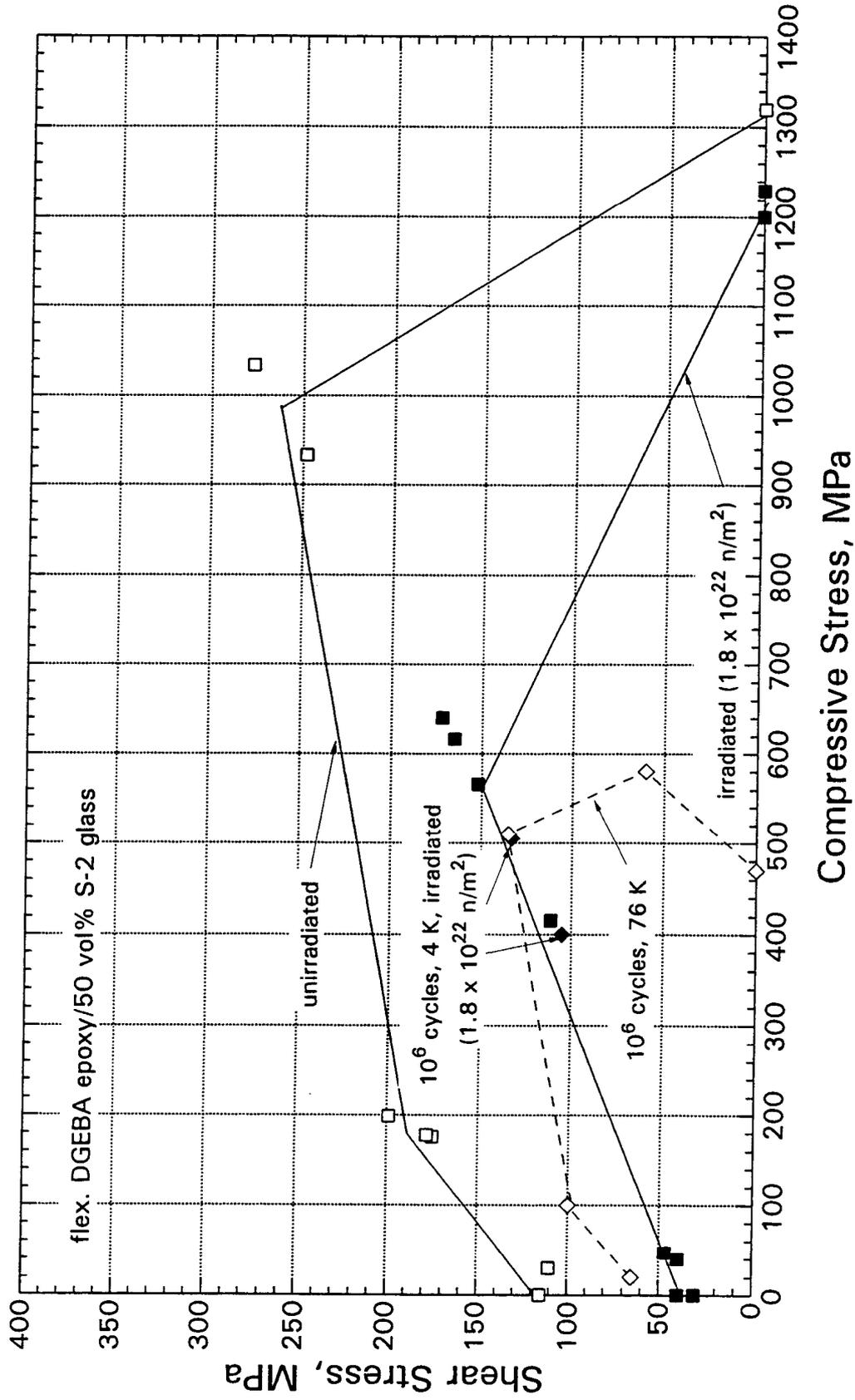


Figure 17. Static and shear/compressive fatigue strengths at 4 K of flexibilized DGEBA epoxy/50 vol% S-2 glass.

Table 7. Through-Thickness Compressive Properties at 4 K

Resin System	Strength (GPa)	Modulus (GPa)	Strain to Failure (%)
DGEBA/S-2 glass*			
unirradiated	1.32	21	6.3
irradiated <sup>†</sup>	1.23	13	9.7
TGDM/S-2 glass*			
unirradiated	1.28	23	5.4
irradiated <sup>†</sup>	1.28	22	5.9
Bismaleimide/S-2 glass*			
unirradiated	1.22	22	5.5
irradiated <sup>†</sup>	1.22	22	5.5
Polyimide/S-2 glass*			
unirradiated	1.23	16	7.6
irradiated <sup>†</sup>	1.23	15	8.2

\* Irradiated to  $1.8 \times 10^{22}$  n/m<sup>2</sup> fast neutron fluence at 4 K; stored at room temperature.

<sup>†</sup> 50 vol% S-2 glass, satin weave, with silane finish.

### Mass and dimensions

During warm-up from 4 K, radiation-induced free radicals combine to form gas molecules in resin systems. The type and amount of gas formed are being studied in a separate project.<sup>22-24</sup> Results from this project indicate that the rate of gas formed from ionization effects in DGEBA-epoxy-resin powder samples is 1.3 cm<sup>3</sup>/g/MGy and in TGDM epoxy resin powder, it is 0.43 cm<sup>3</sup>/g/MGy.

Some of the gas that is formed in bulk specimens during warm-up following irradiation escapes from the surface; some is trapped within the solid. The accumulated trapped gas within the resin of the composite produces expansion; laminate composite structures expand in the through-thickness direction. Gas evolved from the solid reduces its mass. Table 8 summarizes the results of this study, which were reported in Updates III and V.

The ratio of the difference of through-thickness expansion of the DGEBA and TGDM epoxy systems is about 3, which corresponds closely to their ratio of evolved gases. Large increases in the thickness of DGEBA epoxies cured with aliphatic amines have been reported previously.<sup>26</sup>

Notice the very large expansions measured for these resins in confined bulk specimens (sandwich type, consisting of 13-mm-diameter stainless steel/composite/stainless steel). Following irradiation, more measurements of dimensional changes must be made to ensure that these changes represent gas-evolution-induced porosity within the specimen and not

Table 8. Changes in Mass and Dimensions Induced by Irradiation at 4 K  
( $1.8 \times 10^{22}$  n/m<sup>2</sup> fast neutron fluence)

Material	Specimen Type	Average Change (%)		
		Mass	Through-thickness	In-plane
DGEBA	laminate (all composite)	-1.2	+1.2	-0.2
epoxy/50 vol% S-2 glass	sandwich (SS/composite/SS)	—	+5 (+1.5 to +12.3)	—
TGDM epoxy/50 vol% S-2 glass	laminate (all composite)	+0.3	+0.4	+0.1
	sandwich (SS/composite/SS)	—	+2 (0 to +5.7)	—

absorbed moisture or gas interactions at ambient conditions. For instance, consider that the through-thickness expansion following warm-up may be as high as 0.8% if we assume a total fluence of  $2.5 \times 10^{21}$  n/m<sup>2</sup>, maximum DGEBA expansion of 6% at  $1.8 \times 10^{22}$  n/m<sup>2</sup> (the range in Table 8 is a maximum of 12.3%), and a linear dependence of expansion on neutron fluence. (Actually, from the report of Evans,<sup>24</sup> gas-evolution rates are higher at lower fluences.) Then, if the magnet structure remains fixed, the increased compressive stress on the insulation would be about  $0.8\% \times 25 \text{ GPa} = 200 \text{ MPa}$  (25 GPa is the approximate through-thickness compressive modulus of the insulation system). Thus, radiation-induced resin expansion may approximately double the compressive stress on the insulation, unless more radiation-resistant resins are used in the TF coils.

### Future Research and Development

A second-phase irradiation program is planned that also uses the 4-K facilities of the Garching fission reactor. For this program, a new shear/compressive test fixture has been developed for in-situ testing,<sup>27</sup> and systems selected for radiation resistance from the results of this program will receive additional testing. The distinctions between testing cold with no warm-up to room temperature and testing cold after warm-up to room temperature will be investigated.

Other variables leading toward identification of worst-case conditions include irradiation of specimens while they are under an applied stress and irradiation with very fast neutrons (5 to 14 MeV). These variables have been identified by Simon<sup>1,2</sup> as conditions that may result in additional degradation of resin properties. A recent study determined that the effects of applied stress result in higher creep velocities in epoxy-based systems.<sup>28</sup> To date, no ITER-related research and development is planned on these subjects.

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