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**Radiation Effects on Organic Insulators
for Superconducting Magnets
Annual Progress Report for Period
Ending September 30, 1978**

R. H. Kernohan
R. R. Coltman, Jr.
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OAK RIDGE NATIONAL LABORATORY
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R. H. Kernohan, R. R. Coltman, Jr., C. J. Long*

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CONTENTS

	Page
Abstract.....	1
I. Introduction.....	2
II. Experimental Results.....	3
A. Materials Specifications.....	3
B. Specimen Fabrication and Tests.....	4
C. Irradiation Conditions.....	5
D. Irradiation Procedure.....	6
III. Postirradiation Results from the First Experiment.....	7
IV. <i>In-Situ</i> Electrical Measurements During the Second Experiment.....	11
V. Results and Discussion of Postirradiation Tests for 2 x 10 ⁹ Rads.....	17
A. General Appearance.....	17
B. Weight Changes.....	19
C. Electrical Measurements.....	21
D. Mechanical-Strength Tests.....	23
E. Auxiliary Tests.....	27
F. Gas Analysis.....	29
VI. Conclusions.....	31
VII. Acknowledgments.....	32
VIII. References.....	33

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ABSTRACT

Postirradiation tests on specimens irradiated at 4.9 K to a dose of 2×10^8 rads showed no significant changes. The details of the materials involved, specimen preparation, testing procedures, and the *in-situ* results of electrical-resistivity measurements are given in the prior annual report ORNL-TM-6193.⁽¹⁾

A similar experiment, but for a ten-times-greater dose (2×10^9 rads) at the same irradiation temperature, was performed during fiscal year 1978. Improved specimen-preparation and testing techniques were used, and some significant changes in physical properties were observed at this dose. These changes tend to restrict the use of some of these materials. Radiation-induced gaseous-breakdown products were analyzed and found to contain principally H_2 but also other inorganic and organic compounds.

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I. INTRODUCTION

A program was initiated at ORNL to study the effects of irradiation at liquid-He temperature on the properties of organic insulators. The impetus for the program was derived from the need to understand the irradiation behavior of materials that may be used in the construction of large superconducting coils which provide magnetic containment for the plasma in a fusion reactor. The use of such materials, especially hardenable epoxies, provides an economical method for producing not only electrical insulation but mechanical support needed in coil construction. Although many studies on the effects of radiation on organic insulators do exist, there are very few studies on the simultaneous effects of radiation and low temperatures.

In the first experiment carried out for this program, 5 *in-situ* electrical-resistivity and 50 other specimens for postirradiation tests were exposed to a gamma-ray dose of 2×10^8 rads at 4.8 K. The results of this experiment, which have been reported,^(1,2) showed that, except for certain interesting effects related to the ambient ionizing radiation in the irradiation facility and contamination of the chamber atmosphere, there were no changes in electrical properties outside the scatter of the data.

Progress in the conceptual design of Tokamak fusion reactors has led to the consideration of higher plasma power densities, and hence higher irradiation dose rates to superconducting coils and their

components than had first been considered.⁽³⁾ In addition, recent calculations by Santoro et al.⁽⁴⁾ show that without additional protective shielding certain necessary blanket and shield penetrations will lead to still higher dose rates at some magnet locations. For these reasons, a second experiment was carried out to a ten-times-greater dose (2×10^9 rads) on an experiment assembly very similar to the first, but with a total of 80 specimens. After irradiation at 4.9 K and warm-up to room temperature, all mechanical-properties testing was done in liquid nitrogen, in contrast to the first experiment where such tests were done at room temperature. Measurements after warm-up are considered appropriate, since periodic annealing is expected during the operating life of fusion-reactor magnets. Further, the possibility is considered that some additional damage might result during warm-up after irradiation as a result of the diffusion of defects or reactive damage products previously frozen in. This report presents the results of postirradiation tests in the first experiment and all results from the second experiment,

II. EXPERIMENTAL RESULTS

A. Materials Specifications

As before, the materials chosen for study were those presently in use for magnet construction and already known to have good radiation resistance at room temperature. For a further discussion of the materials' qualities, see Ref. 1.

The materials tested in the second experiment were:

1. Stycast 2850 Blue FT Epoxy, with 7% 24 LV hardener, Emerson and Cuming, Inc., an inorganically filled room-temperature-curing epoxy;
2. EPON 828 epoxy, 20 pph Z curing agent, 0.5% Z6020 Silane couplant, Shell Chemical Company, filled with 40 wt.% 400-mesh SiO_2 (good strength, but requires 80°C cure, followed by 150°C post-cure treatment);
3. EF-527 B-stage, Synthane-Taylor Corporation, a partially cured glass-cloth-epoxy composite (good mechanical properties, but requires curing at 175°C under pressure);
4. National Electrical Manufacturers Association (NEMA) G-10 high-pressure glass-epoxy laminate (mechanical tests) and G-10CR, Spaulding Fibre Company (electrical tests);
5. Nomex paper, type 410, DuPont, aromatic polyamide sheet;
6. Kapton F film, DuPont, polyimide film frequently used in magnets;
7. Formvar and Omega varnish-insulated #18 copper wires;
8. Aluminized Mylar thermal superinsulation.

B. Specimen Fabrication and Tests

Because of space limitations and gamma-ray heating in the irradiation cryostat, it was necessary to use miniaturized specimens. Standard specimen designs were scaled down to fit present conditions. Electrical-resistance specimens (both *in-situ* and postirradiation types) and electrical-breakdown specimens were prepared and tested as described in Ref. 1. The devices used to test mechanical properties were modified to allow all such tests to be made at liquid-nitrogen temperature. Specimens

for flexure-strength tests (0.16 x 0.32 x 5 cm) which employ 3-point bending with 2.5 cm between supports were the same as Ref. 1, while lap-shear-strength specimens were slightly modified (a sandwich of three 0.64 x 0.64-cm Cu plates and two adhesive layers, each 0.33 mm thick) over the Ref.-1 design to reduce the mass of copper. In addition, compression-strength-test specimens (0.64-cm-diam. x 1.27-cm-long cylinders) were added to the present experiment.

Other tests which also utilize some of the specimens mentioned above include changes in weight, color, and electrical-dissipation factor. The integrity of Mylar superinsulation and two types of varnish coatings on commercial copper wire was also examined. After fabrication, all specimens were immersed at least twice in liquid helium to insure that thermal shock would cause no damage. In most cases, three similar specimens for each test were irradiated, and three similar control specimens were held for comparison purposes. The control specimens were subjected to the same temperature cycling as the irradiated specimens.

C. Irradiation Conditions

The experiment assembly, which was nearly identical to that described in Ref. 1, was irradiated at 4.9 K in the ORNL Low-Temperature Irradiation Facility (LTIF), which receives a highly thermalized neutron flux originating in the ORNL Bulk Shielding Reactor. Neutron capture gamma rays produced in the cadmium shield surrounding the assembly added to the ambient gamma level to provide an intense gamma-ray flux which was by far the principal damaging radiation received by the samples within. In a separate test, the gamma-ray intensity was measured by substituting

electrical for gamma-ray heating in an aluminum cylinder at constant temperature and fixed ambient heat-exchange conditions. A value of 38 mW/g or 1.4×10^7 rads/h was measured.

The specimens were arranged in two groups as before: one for the *in-situ* measurement of resistance changes during the irradiation, and the other for the postirradiation measurement of mechanical and electrical properties. Prior to the initial cool-down of the LTIF, the sample chamber was evacuated to less than 0.05 torr each of several times after back-filling with clean dry helium. During irradiations, the specimens were immersed in liquid helium. At all elevated temperatures, the helium gas pressure in the sample chamber of the LTIF was held at 350 torr. During irradiation, specimen temperatures were monitored using the liquid-helium vapor pressure. Elevated temperatures were monitored with a Cu-Constantan thermocouple centrally located in the specimen assembly.

In-situ electrical-resistance measurements were made with a commonly used guard-ring circuit (see Section IV). To minimize the influence on resistance calculations of electrical leakage caused by ionizing radiation present in the LTIF sample chamber, the current in a circuit having no sample was always subtracted from the current measured in a normal sample circuit before calculating resistance values. For further details, see Ref. 1.

D. Irradiation Procedure

The experiment assembly for the second experiment was first irradiated at 4.9 K to a dose of 0.6×10^9 rads and then warmed to 305 K. The gaseous contents of the chamber were then evacuated to about 1 torr through

a liquid-He cold trap where all materials except He were frozen out for future mass-spectrographic analysis. The irradiation cryostat was again cooled down, and a second irradiation of 1.5×10^9 rads was performed at 4.9 K, making the total dose 2.1×10^9 rads. During warm-up after each irradiation, the guard-ring current was monitored. After the sample chamber was evacuated through the liquid-He cold trap to obtain a gas sample as before, the chamber was recharged to 350 torr. The specimen remained in this atmosphere at 305 K for 35 days to allow for the decay of the now radioactive assembly. Another gas sample was then taken as before, after which the assembly was removed and the specimens were recovered for post-irradiation testing.

III. POSTIRRADIATION RESULTS FROM THE FIRST EXPERIMENT

Results on the *in-situ* electrical-resistivity specimens after the 2×10^8 rads irradiation are given in Ref. 1.

Measurements on the other specimens were taken about 2 months after the irradiation. There was very little change in the appearance of the irradiated specimens. No cracks or blemishes were evident. Irradiated EPON 828 and B-stage glass cloth were slightly more yellow than their controls, and the irradiated G-10 and Fireban fiberglass specimens were somewhat more yellow-brown than their controls. The others, including aluminized Mylar, showed no change in appearance.

All control and irradiated specimens were weighed on an analytical balance after being stored in a desiccator for three weeks. Changes in weight due to irradiation were not measurable (< 0.2 mg in samples weighing

from 62 mg to 500 mg). One type of sample, Fireban 1011 (a fiberglass of the FR-4 type; i.e., modified to be fire retardant), was found to be about 30 times more radioactive than the other types.

The average results of postirradiation electrical-resistivity measurements are given in the top half of Table I. These values are 10 to 20 times higher than those measured for the *in-situ* specimens in Ref. 1. The differences may be attributed to (1) stray ionization either from the radioactivity around or in the *in-situ* specimens or induced by the reactor, or (2) leakage in the extremely long lead wires going to the *in-situ* specimens. Nevertheless, the postirradiation tests indicate that there was little deterioration in the materials tested.

The results of the electrical-breakdown tests are shown at the bottom of Table I. It should be noted that only one set of specimens (Nomex 410) yielded true breakdown through the material. For all other specimens, the electric arc passed over the surface at discharge, so that the results shown are indicative of surface flashover rather than electrical breakdown. They therefore represent minimum breakdown fields for these materials. The actual flashover voltage for both epoxies and the Fireban 1011 specimens was the same; i.e., about 13 kV.

The results of the mechanical tests are shown in Table II. The three irradiated lap-shear specimens of B-stage glass cloth exhibited the greatest scatter in results, the values in shear strength being 26, 28, and 37 mPa. No distinction is made between a failure of the material itself and a failure in the adhesive bond to the copper. Examination of the specimen after the tests showed that both events occurred, with adhesive-bond failure predominating slightly. The scatter in the

TABLE I
 POSTIRRADIATION ELECTRICAL MEASUREMENTS AT ROOM TEMPERATURE
 Irradiation Dose 2×10^8 rads at 4.8 K

Specimen	Resistivity, ρ ($10^{15} \Omega\text{-cm}$)					
	2850 FT Epoxy	EPON 828 Epoxy	B-Stage Glass Cloth	Fireban 1011 Fiberglass	Nomex 410	Kapton F
Control	2	1	3	1	5	5
Irradi- ated	2	1	2	1	4	8

	Electrical Breakdown, E (kV/mm)					
Control	14	25	41	21	39	80
Irradi- ated	14	24	43	20	37	81

TABLE II
POSTIRRADIATION MECHANICAL MEASUREMENTS AT ROOM TEMPERATURE

Irradiation Dose 2×10^8 rads at 4.8 K

(Each value is the average of three tests.)

	Specimen	2850 FT Epoxy		B-Stage Class Cloth	
		kpsi	MPa	kpsi	MPa
Shear Strength	Control	1.8	12	5.2	36
	Irradi- ated	2.15	15	4.3	30

	Specimen	2850 FT Epoxy		G-10 Fiberglass		Fireban 1011	
		kpsi	MPa	kpsi	MPa	kpsi	MPa
Flexure Strength	Control	12	83	57	390	54	370
	Irradi- ated	12	83	58	400	61	420

flexure-test results shown in the lower part of Table II was rather low, the greatest deviation being only 8% from the average. The flexural modulus for both control and irradiated 2850 FT epoxy material was about 1.35×10^6 psi (9 GPa); the modulus for the fiberglass materials in both control and irradiated conditions was about 2.4×10^6 psi (17 GPa).

In conclusion, the materials chosen for the first experiment withstood the irradiation dose of 2×10^8 rads at 4.8 K in liquid helium with almost no deterioration in the properties studied.

IV. *IN-SITU* ELECTRICAL MEASUREMENTS DURING THE SECOND EXPERIMENT

The arrangement for measuring the resistivity of four selected specimens during irradiation was the same as that in the first experiment (Ref. 1) with one major exception. Whereas previously the entire sample chamber was grounded and served as a return circuit for the current through each resistivity specimen, the central support tube in the second experiment was electrically isolated and served alone as the current return. This isolation of the specimen current gave resistivity values higher than the first experiment by a factor of about 10, although the values still were not as high as those measured on control and irradiated specimens in a bench-test rig after irradiation. The method for measuring resistivity was adapted from the ASTM method.⁽⁵⁾ The circuit is shown in Fig. 1.

The resistivity values in the first row of Table III were measured with the specimens in place in the LTIF sample chamber, before irradiation and with the reactor off and 3 meters away. The values in the second row

RESISTANCE MEASURING EQUIPMENT (SCHEMATIC)

Fig. 1.

TABLE III

IN-SITU ELECTRICAL-RESISTIVITY MEASUREMENTS AT ROOM TEMPERATURE $(10^{15} \Omega\text{-cm})$

	2850 FT Blue Epoxy	EPON 828 Epoxy	Fiberglass G-10CR	Nomex 410 Aramid
Before Irradiation	1	1.5	0.7	3.5
One Month After 2×10^9 rads	0.35	0.75	0.5	1.5

were taken just before the specimen assembly was removed from the sample chamber 35 days after the irradiation. At this time the reactor was also 3 meters away in the reactor pool, but operating at full power. Based on past experience, we believe the lower values in the second row can be accounted for by stray ionization rather than any permanent radiation-damage effects on the electrical properties of the specimens.

The values in Table III were obtained by measuring the current through each specimen with a picoammeter when 100 volts dc was applied between the top and bottom electrodes attached to the disc-shaped specimen (~ 12.5 mm diam and $\sim .25$ mm thick); see Fig. 1. The currents used to determine the values in Table III were of the order of one picoampere. After the temperature was lowered to 3.5 K, the currents changed very little. When the reactor was brought in place next to the LTIF, the currents increased to the vicinity of 100 pA (resistivities $\sim 10^{13} \Omega\text{-cm}$).

With the reactor at a power level of 1.2 MW, the currents further increased to between 1500 and 10,000 pA (resistivities $\sim 10^{11} \Omega\text{-cm}$), and the liquid-He temperature rose to 4.9 K. The dose rate at 1.2 MW power is about 10^7 rads/hr. Thus, while ionizing radiation may affect the resistivity of an insulator, it may be concluded that even for the high dose rate used in this experiment the resistivity of the materials remains high enough for use in many practical applications.

The method used for measuring the resistivity⁽⁵⁾ (see Fig. 1) employs a center electrode and a surrounding concentric guard-ring electrode. Both are at the same potential with the central electrode carrying current through the specimen, while the guard ring carries currents around the edge of the circular disc. The guard-ring current at 100 volts potential was less than 0.5 μA before and after irradiation with the reactor 3 meters away from the irradiation facility. With the reactor operating in place, the guard-ring current increased to about 35 μA .

After an irradiation of about 75 hours ($\sim 6 \times 10^6$ rads), the reactor was shut down and moved away, and then a slow warm-up to room temperature was started. During this time, the specimens in the sample chamber were maintained in He gas at a pressure of 350 torr (47 kPa). The guard-ring current was steady at 0.5 μA at 100 V until a temperature of about 200 K was reached. The current then began to increase rapidly, and when the capsule reached room temperature it was 23 μA with only 25 V applied. A similar phenomenon had occurred in the first experiment.⁽¹⁾

At this point, the gas in the sample chamber was pumped out through an evacuated liquid-He cold trap. After the sample chamber was purged with He gas twice, the guard-ring current at room temperature returned to

less than $0.5 \mu\text{A}$ at 100 V. During the period that the guard-ring current was high, resistance measurements of the specimen gave spurious results. After the specimens were cooled to 3.5 K in order to continue the irradiation, the specimen resistivities returned to their preannealed values.

At the end of the irradiation (2×10^9 rads), the annealing process was repeated, and the same phenomenon occurred. Specimen resistivities were measurable until about 20 K, at which temperature spurious results were obtained. We recognize the possibility that this behavior can be related to hydrogen evolved from the specimens. The guard-ring current started to increase at about 175 K. A graph of the guard-ring current vs temperature is shown in Fig. 2. After room temperature was reached, the sample chamber was again evacuated through a liquid-He-cooled trap to .065 torr (9 Pa) and refilled with He gas to a pressure of 350 torr (47 kPa). The guard-ring current then returned to its normal low value.

Specimen resistivities gradually increased during the next month by a factor of 4 or 5 due to decay of radioactivity. The final values are given in the last row of Table III. At this time, the sample chamber was again evacuated through a liquid-He-cooled trap, and the specimens were removed from the irradiation sample chamber. The results of the sample-chamber gas analyses are described in Section V-F.

A subsidiary test on a pair of #30 (0.25-mm diam) Formvar-insulated copper wires was made during the irradiation. The pair was tightly twisted with 4 or 5 turns over about a 1-cm length located in the irradiation capsule. The resistance measured in place was $3 \times 10^{14} \Omega$ at room temperature, $5 \times 10^{14} \Omega$ at 3.5 K, $1 \times 10^{11} \Omega$ at 4.9 K with reactor at

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Fig. 2. Guard-ring current during warm-up after irradiation at 4.9 K for 2×10^9 rads.

power, and $2 \times 10^{13} \Omega$ at room temperature one month after the irradiation to 2×10^9 rads. Further discussion on insulation-coating tests are in Section V-E.

V. RESULTS AND DISCUSSION OF POSTIRRADIATION TESTS FOR 2×10^9 RADS

Specimens were removed from the lower part of the irradiation capsule 35 days after the end of the irradiation. These and control specimens which received the same temperature cycling as the irradiated specimens (i.e., immersing in liquid He, warming to room temperature, etc.) were stored in a desiccator until measurements were made. All measurements of a given type were made on pairs of control and irradiated specimens on the same day under similar conditions. Between measurements of each type, the specimens were stored in the desiccator.

A. General Appearance

Nearly all of the specimens were darker in color, as can be seen in the photograph of the voltage-breakdown specimens shown in Fig. 3. However, aluminized Mylar, Kapton F, and the Formvar- and Omega-coated wires did not change in color. The irradiated 2850 FT-epoxy specimens turned a darker blue. The greatest color change was in the originally tan EPON 828 specimens. The irradiated specimens turned a very dark brown. The B-stage glass-cloth and G-10CR fiberglass samples turned from white to a golden brown, with the former showing a few carbonized areas near the periphery. Nomex 410 turned only slightly darker in color. The irradiated Kapton electrical-breakdown specimens developed flakes on the surface,

ELECTRICAL-BREAKDOWN SPECIMENS

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Fig. 3. Color changes due to irradiation at 4.9 K for 2×10^9 rads.

indicating delamination. The aluminized Mylar samples retained the circular shape of the cadmium liner and proved quite brittle. One piece, approximately 2.5 x 4 cm, came out nearly whole with two pieces torn out, but a similar piece the same size fractured into about ten parts.

The Kapton F material consists of .075 mm of Kapton coated on each side with 0.025 mm of Teflon. The results of both the first and second experiments indicate that the onset of significant damage in Kapton F and Mylar occurs between 2×10^8 and 2×10^9 rads by irradiation at low temperature in liquid helium.

B. Weight Changes

All of the specimens were weighed on an analytical balance seven days after the irradiated specimens were removed from the irradiation assembly. During this period, the specimens were stored in a desiccator jar containing a solid granular drying agent. Although all specimens were weighed, only the specimens that contained a minimum of metal were considered as useful in making weight-change comparisons between control and irradiated specimens. These combined weight changes are shown in Table IV. The greatest total change was in Kapton F, a result that is consistent with the appearance change noted in Section V-A. The Kapton F specimens with the greatest change (14% loss) were those with the greatest exposed surface; i.e., the electrical-breakdown specimens.

The two irradiated aluminized Mylar specimens weighing 60 mg appeared to lose 0.3 mg (0.5% loss), but this result may be incorrect because some small pieces of the irradiated material may have been lost during removal from the capsule.

TABLE IV
CHANGES IN COMBINED WEIGHT OF ELECTRICAL BREAKDOWN, FLEXURE, AND COMPRESSION SPECIMENS
(mg)

Specimen		2850 FT Epoxy	EPON 828 Epoxy	B-Stage Glass Cloth	G-10 Fiber- glass	Nomex 410	Kapton F
Control	Total Wt.	4043	2391	680	3452	535	738
	Change	+7.6	+8.5	+2.2	+5.5	+10.4	+0.4
	% Change	+1.19	+3.5	+3.2	+1.6	+1.9	+0.5
Irradiated to 2×10^9 rads	Total Wt.	4028	2567	680	3455	535	738
	Change	-7.8	+3.9	+4.8	+6.4	+10.2	-41.6
	% Change	-1.19	+1.5	+7.0	+1.9	+1.9	-5.6

The general gain in weight of the control specimens may have been the result of a hygroscopic effect. These specimens were immersed in liquid He several times and allowed to warm up to room temperature in the upper part of a long dewar. Frost may have enveloped the control capsule at this time, resulting in moisture absorption. Nomex 410, in particular, is sensitive to hygroscopic conditions.⁽⁶⁾ The most significant weight loss is that for Kapton F. Small losses were also observed in 2850 FT epoxy and G-10 materials, but all other materials showed no significant change.

C. Electrical Measurements

The average electrical resistivity of two each postirradiation and control specimens pairs is shown in the top part of Table V. The greatest effect was in B-stage glass cloth, where the resistivity decreased by several orders of magnitude with this irradiation dose. These values, however, are somewhat higher than those given in Table I for the smaller dose because of improvements in both specimen-preparation and measurement techniques. It should be noted that to determine resistivity values only very small currents are used, so that it is doubtful whether the decrease in resistance by about a factor of 2 in both epoxy specimens is significant. However, the changes are in line with those of the *in-situ* measurements on the two epoxies shown in Table III.

Measurements of the electrical-dissipation factor, $\tan \delta$, were made on the electrical-breakdown specimens, using a capacitance bridge and associated equipment at 1000 Hz. No significant difference was found between control and irradiated specimens, except for the irradiated B-stage

TABLE V

POSTIRRADIATION ELECTRICAL MEASUREMENTS AT ROOM TEMPERATURE

Irradiation Dose 2×10^9 rads at 4.9 K

Specimen	Resistivity, ρ ($10^{15} \Omega\text{-cm}$)					
	2850 FT Epoxy	EPON 828 Epoxy	B-Stage Glass Cloth	Fiber- glass G-10CR	Nomex 410	Kapton F
Control	14	10	12	7	13	15
Irradiated	5	6	$[5 \times 10^{10}]$	7	14	19
	Electrical Breakdown, E (kV/mm)					
Control	33	24	31	22	38	70
Irradiated	32	23	11	22	39	57

glass cloth, which showed a change too large to measure with our equipment. Nomex 410 and Kapton F had values of .008 for $\tan \delta$, while all others were about .025.

The results of electrical-breakdown tests are given in the bottom part of Table V. As previously described in Section III, these are really minimum breakdown values, because all tests resulted in surface flashovers, except for B-stage glass cloth and Nomex 410, which were true breakdowns. Only the B-stage glass cloth showed a significant loss of electrical strength. The change in Kapton F is not as great as one might expect from the wrinkled condition of the Teflon surface layer. The breakdown

value for 2850 FT epoxy is larger than that shown in Table I, because the specimen thickness for the second experiment was only about 40% of that of the first experiment.

D. Mechanical-Strength Tests

All mechanical-strength tests were made with specimens immersed in liquid nitrogen (77 K), and the results are shown in Table VI. Results of the shear tests are shown in Fig. 4. One of the 2850 FT-epoxy specimens failed just as the stress was being applied. Of the three adhesive materials, the filled EPON 828 epoxy decreases the least at this radiation dose. The characteristic failure occurred at the adhesive bond with the metal in most instances.

On the left side of Fig. 5, the results of flexure tests on specimens $3.2 \times 1.6 \times 51$ mm are shown graphically. The specimens were tested in 3-point flexure with a span of 25.4 mm and a span/thickness ratio of 16. The end supports of the span had a 1.6-mm radius of curvature. The effect of this irradiation dose on G-10 fiberglass was drastic, but the filled EPON 828 epoxy withstood the irradiation with no change. Values of the flexural modulus are shown in Table VII.

The results of compression tests on small cylinders (12.7 mm long, 6.3 mm diameter) are displayed on the right side of Fig. 5. Again, the EPON 828 epoxy withstood the radiation dose well. The G-10 compression cylinders were not made of material from the same manufacturer as that used for the electrical and flexure specimens. While the G-10 showed the largest proportional changes, its postirradiation values for the compression and flexural strength were comparable to those of the other materials.

TABLE VI. MECHANICAL-PROPERTY MEASUREMENTS

Material	Lap Shear Strength (MPa)		3-Point Flexure Strength (MPa)		Compressive Strength (MPa)	
	Individual Values	Mean	Individual Values	Mean	Individual Values	Mean
Stycast 2850 Control	22.8, 59.7, 58.9	46.8	259, 266, 238	254	500, 489	495
Stycast 2850 Irradiated	18.7, 19.8	19.2	137, 162, 132	143	322, 273	298
EPON 828 Control	53.4, 55.9, 47.4	52.2	207, 152, 315	225	511, 479	495
EPON 828 Irradiated	28.4, 44.3, 28.1	33.6	263, 315, 270	283	465, 483	474
G-10 Control			921, 860, 805	862	602, 837	769
G-10 Irradiated			229, 176, 168	191	225, 237	231
EF 527 Control	94.4, 58.4, 53.2	68.7				
EF 527 Irradiated	16.7, 10.6, 9.1	12.1				

ORNL-DWG 78-19090

Fig. 4. Changes in shear strength due to irradiation at 4.9 K for 2×10^9 rads.

C = CONTROL SPECIMENS

I = SPECIMENS IRRADIATED TO 2×10^9 R

Fig. 5. Changes in flexure and compression strength due to irradiation at 4.9 K for 2×10^9 rads.

TABLE VII
FLEXURAL MODULUS

at 77 K

Irradiation Dose 2×10^9 rads at 4.9 K

	2850 FT Epoxy		828 EPON Epoxy		G-10 Fiberglass	
	10^6 psi	10^9 Pa	10^6 psi	10^9 Pa	10^6 psi	10^9 Pa
Control	3.6	25	1.6	11	2.7	19
Irradiated	3.6	25	1.5	10	1.9	13

In addition to the cylindrical compression cylinders, a number (n) of 12.7-mm-diam circular disks of 0.25-mm Nomex 410 (6), 0.25-mm B-stage glass cloth (6), and 0.125-mm Kapton F (12) were stacked between matching copper disks to give a total nominal thickness of about 3 mm. Stack thicknesses were found to be unchanged after a compressive stress of 25.5 kpsi (175 MPa) was applied at 77 K. Further, subsequent examination of the irradiated disks showed no fracture of the material.

E. Auxiliary Tests

An attempt was made to study the effect of a compressive stress on materials during irradiation at 4.9 K. Washer-shaped specimens 12.7 mm in diam, 1.5 mm thick with a central hole large enough to accommodate an 8-32 screw, were fabricated from each material. Allen-head 8-32 stainless-steel

screws 11 mm long were inserted through four Belleville washers in parallel and then a specimen. The assembly was fastened with a specially designed flanged nut. The screws were tightened with a torque wrench to 40 in.-lb., which deformed the Belleville washers to a flattened position. According to the Belleville washer manufacturer's specification, the stress on each specimen was ~ 2000 psi (1.4 MPa). Each assembly was relatively bulky and heavy (6.2 g), so that only four were irradiated — both epoxies, G-10, and Nomex 410. None of the four specimens cracked or otherwise deteriorated during the irradiation. Accurate measurement of dimensional changes indicated that deformation occurred in only the 2850 FT-blue-epoxy specimen, whose diameter increased 3%.

Two lengths (~ 18 cm each) of Formvar-insulated and two lengths of Omega-insulated No. 18 AWG copper wires were also irradiated in the second experiment. Both control and irradiated samples were returned to their source, Westinghouse Research and Development Center, for ASTM tests of dielectric strength and scrape abrasion.⁽⁷⁾ The dielectric strength was measured with the pairs of wires twisted together for 16 turns over their lengths. The scrape-abrasion test indicates the number of cycles to failure under a load of 700 g on a 0.41-mm-diameter needle. The average results, along with those obtained on other controls not sent to Oak Ridge, are given in Table VIII. The lower values obtained on the irradiated samples in both tests are not very different from those obtained on samples irradiated to comparable doses at room temperature.⁽⁸⁾

TABLE VIII
WIRE-INSULATION TESTS⁽⁷⁾

2×10^9 rads

(Average Values at Room Temperature)

	Dielectric Strength (kV)		Scrape Abrasion (cycles to failure)	
	Formvar	Omega	Formvar	Omega
Mfg. Controls (no thermal cycling)	5.9	12.0	32	50
Control	3.0	10.0	35	59
Irradiated	1.8	4.0	12	21

F. Gas Analysis

During the initial warm-up after irradiation, the sample chamber was carefully observed for small pressure and temperature surges. The total specimen weight loss of only 50 mg, along with the relatively large volume of the sample-chamber system, is consistent with the fact that no such surges were observed.

The results of the gas analyses taken approximately one-third of the way through the irradiation, at the end of the irradiation, and 35 days after the end of the irradiation are given in Table IX. All samples were taken by evacuating the sample chamber (at room temperature) through

TABLE IX
ANALYSIS OF SPECIMEN-CHAMBER GAS
(wt %)

Sample	1	2	3
Dose (rads)	6.3×10^8	2.1×10^9	2.1×10^9 *
H ₂	58.9	93.7	35.8
CH ₄	1.7	0.3	29.9
H ₂ O	21.7	3.2	3.1
N ₂ and CO	9.0	1.4	18.2
CO ₂	6.9	0.7	8.9
Other Organics	1.4	~0.2	1.8
Fluorocarbons	0.2	~0.5	2.0

All gas samples taken by evacuating through cold trap in liquid He with chamber at room temperature after the dose indicated.

*Gas sample taken 35 days after end of irradiation.

a cold trap immersed in liquid helium. The values in the table have been normalized so as not to include He.

The three analyses were quite different. At the end of the irradiation, hydrogen predominated, while high fractions for carbon compounds were noted after the samples had remained in the irradiation cryostat for a month after the irradiation. From the changes in appearance and weight

loss discussed in Sections V A and B, it would appear that a large contribution to the gases came from the Teflon coating on the Kapton F specimens. In the presence of ionizing radiation, these products may account for the unusual *in-situ* electrical behavior at higher temperatures described in Section IV. The lower relative H₂ content in Col. 3 of Table IX is consistent with a higher diffusion rate out of the specimen material of H₂ than the other species.

VI. CONCLUSIONS

The materials tested all withstood an irradiation dose of 2×10^8 rads satisfactorily, but some degradation in properties appeared after a dose of 2×10^9 rads.

The electrical properties of all materials, while changed by the irradiation, remained suitable for their intended use, even at the higher dose. We suggest that B-stage glass cloth should be eliminated as a candidate material in magnet design, not only because of the degradation in electrical properties at higher irradiation doses, but because this material requires a temperature of 175 °C under some pressure to cure in place.

In spite of the appreciable change in color, the SiO₂-filled EPON 828 epoxy showed less overall deterioration in electrical and mechanical properties than all other materials up to a dose of 2×10^9 rads.

Higher values of shear strength than those we measured may be possible by using a coarser grit than the #400 emery paper used here, for preparing the metal surface for the adhesive. Couplant materials can be

added to epoxies to increase adhesive strength, but these may change the radiation resistance of the epoxy.

Materials such as Mylar and Teflon have been known to deteriorate when irradiated in air to doses on the order of 10^6 to 10^7 rads.⁽³⁾ The Mylar specimens and the Teflon coating on Kapton F withstood 2×10^8 rads in liquid helium, but deteriorated at 2×10^9 rads. Serious consideration should be given to the use of Kapton H (no Teflon coating). Thin aluminized Kapton H, if it can be fabricated, could be used as a superinsulation in preference to aluminized Mylar.

It would also be advisable to examine the radiation resistance and strength of a type of fiberglass material fabricated with EPON 828 epoxy.

For wire insulation in a proposed fusion-magnet design, it appears that Omega-type insulation would be superior to Formvar.

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