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ORGANIC MATERIALS FOR FUSION-REACTOR APPLICATIONS

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ORGANIC MATERIALS FOR FUSION REACTOR APPLICATIONS*

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Organic materials requirements for fusion reactor magnets are described with reference to the temperature, radiation, and electrical and mechanical stress environment expected in these magnets. A review is presented of the response to gamma-ray and neutron irradiation at low temperatures of candidate organic materials; i.e. laminates, thin films, and potting compounds. Lifetime-limiting features of this response as well as needed testing under magnet operating conditions not yet adequately investigated are identified and recommendations for future work are made.

1. INTRODUCTION

Organic insulation is specified for superconducting coils in fusion reactors because of its lower material and fabrication costs compared to alternate forms of insulation.

The major part of the insulation system in a superconducting magnet will comprise a high strength glass-reinforced polymer composite material, of which the National Electrical Manufacturers Association (NEMA) G-10CR is typical. Such materials differ in two important respects from most other metallic and non-metallic reactor components. The most important difference is that the properties of organic materials and glasses are degraded by the neutrons and gamma-rays present in fusion magnets. As a result, the radiation response of organic insulation will depend upon the mix of neutron and gamma-ray intensities which differ for various reactor designs.¹⁻⁴ In contrast, the remaining important components of a superconducting magnet, the superconductor and the copper or aluminum stabilizer, are sensitive only to neutron radiation. In both cases, the serious damage is to the electrical properties; irradiation of superconductors

results in decreased critical current density and critical temperature, while in the stabilizer materials lifetime is limited by an increase in resistivity. A second difference is that structural degradation of the organics occurs at lower neutron doses than those for the other classes of magnet materials.⁵ Comparison of magnet lifetimes, based on available data for degradation of these components in calculated magnet irradiation environments, shows that either or both the stabilizer or the epoxy-based insulators are critical.^{6,7}

A detailed understanding of the stress, temperature and radiation conditions imposed on superconducting magnet insulators, and of the response of candidate materials to these conditions is the key to the successful engineering of these large magnet systems. In the following, we review the current range of insulator requirements and operating conditions, and the response of currently proposed materials to these conditions. Finally, we discuss perceived needs for the development and testing of successful insulation systems.

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2. OPERATING CONDITIONS AND INSULATOR REQUIREMENTS

Magnets planned for fusion reactors fall into two categories: water-cooled resistive magnets and superconducting magnets. The former are specified for applications such as divertor coils inside the shield, and in the inner part of the MARS axicell magnet. In these cases, superconducting magnets cannot be used for reasons of excessive nuclear heating and radiation damage.⁸ Generally, copper conductors and ceramic insulation are specified for these conditions.⁹

Superconducting magnets are specified for ohmic heating coils, most equilibrium field coils, and the toroidal field coils in tokamaks. In addition, most magnets in reactors based on the mirror configuration (Yin-Yang, transition, solenoid coils and the outer part of the axicell coils) are also superconducting. In all such cases, organic insulation is specified. For these applications, the maximum dose to the insulation which is specified for development purposes is 0.5 to 1×10^9 Gy.¹⁰

Doses specified in designs for reactor configurations which have appeared in recent years vary widely, but are typically below the development goal. Such specifications generally fall in the range of 10^7 to 10^8 Gy for reactors as listed in Table 1,¹⁰⁻¹³ typically based on calculations for G-10CR.

TABLE 1
Recent design specifications for dose to magnet insulation

Machine	Dose (Gy)	Reference
INTOR	6.8×10^6	11
FED	1×10^7	10
INTOR	5×10^7	12
MARS	5×10^7	13

The radiation at the magnet is a mix of

neutrons and gamma rays, which depends on the location in the magnet and the nature of the shield. Calculations show that, typically, 55 to 80% of the deposited energy in the insulator results from neutrons, the balance from gamma rays.²⁻⁴ This has two consequences. First, for a specified neutron dose, the deposited energy in organics increases with increasing hydrogen content. In addition, fiberglass fabricated from E-glass, which contains boron, will suffer further damage due to the ^{10}B fission (n, α) reaction, if significant thermal or epithermal neutrons are present. Secondly, defect production mechanisms differ between neutrons and gamma rays, posing the possibility that details of the radiation mix may affect the nature of the damage.^{5,14,15}

Designers agree that magnets may periodically be warmed to room temperature to anneal damage in the stabilizer. The maximum number of such cycles which would be economically feasible is of the order of 5. The effect of such cycling on the accumulation of damage in the insulator must be considered.

Insulation in the magnet is used to separate windings from each other and from the case. Although designs differ in detail, a common feature is that forces produced by the magnetic fields must be transmitted by the insulation to the case. In addition, in pool-boiled magnets the insulation is machined to provide passages for liquid helium. An example of the latter is shown in Fig. 1.¹⁰

Compressive stresses arise in insulation from magnetic forces as the winding layers are thrust out against one another. The magnitude of the stress depends on the magnetic field strength, the design for limiting the cumulative load, and a stress multiplier if the insulation is machined as in Fig. 1. Compression stresses so generated are estimated to be 275-400 MPa^{10,16} (40 to 60 KSI), and it has been suggested that a safety factor of 3 is

FIGURE 1
Sectional views of insulation system for pool-boiled magnets¹⁰

appropriate.¹⁰ Besides the compression load resulting from in-coil fields, a sideways thrust is generated by interactions between coils; for example, between the toroidal and poloidal field coils of a tokamak. These forces generate shear stresses in the insulator the magnitudes of which are not currently well known. These stresses too will be subject to some design control.

Additionally, mechanical stresses arise from a variety of other effects, including bending stresses in unsupported regions (e.g. as in Fig. 1), or in regions of less than perfect packing of insulation and winding. In the final analysis, such stresses, particularly those due to imperfections, may dominate the mechanical stress picture.

Stresses arise from both steady state and pulsed forces. Lifetime cycles may be as few as ~1000 cycles,¹⁷ while pulsing the EF coils in INTOR results in 7×10^5 design lifetime cycles.¹² Hence response of the insulator to fatigue conditions will be a criterion for its acceptance.

Although they are not yet fully defined, the mechanical requirements are severe and seem likely to define the major lifetime-limiting

property of the insulation. The electrical requirements, though not as stringent, are nonetheless important. Maximum voltages amount to a few hundred volts between turns, and a thousand or more at the terminals or between the windings and case.^{13,17} Maximum voltages would be expected to occur during a serious disruption, or from turn-to-turn shorts under normal operation,¹⁸ and the feared result would be breakdown of the insulation. Speculation exists that helium breakdown would occur before insulator breakdown.¹⁸

Designs for fusion magnets utilize several types of organic insulation. High strength materials such as G-10CR, as noted previously, are a conspicuous component of the insulation system. Often, insulating film materials are used for ground insulation around the periphery of the case. A third component which may be required is a potting compound for gluing things in place.¹⁹ Finally, superinsulation composed of many closely-spaced layers of aluminized film may be used in the vacuum insulation system.

3. MATERIALS AND RADIATION DAMAGE MECHANISMS

A large number of materials in the three

TABLE 2
Prominent insulators considered for use in fusion magnets

NO.	NAME	DESCRIPTION	APPLICATION
1	G-10CR	Epoxy matrix laminate, aliphatic amine cure	High strength laminates
2	G-11CR	Epoxy matrix laminate, aromatic amine cure	
3	Spaulrad (Spaulding Fibre)	Polyimide (Kerimid) resin matrix laminate, analogue of G-10CR	
4	NP-530	Same as above	
5	Epon 828 (Araldite)	DGEBA with various fillers, reinforcements, and curing agents	Potting compounds, candidate laminating resins
6	Aracast	Bisphenol A resin modified with hydantoin resins	
7	Nomex	Polyamide (nylon) film	Paper-type insulation
8	Mylar	Polyester film	Slip-plane insulation and superinsulation
9	Kapton (Vespel)	Polyimide film (bulk version of the same polymer)	Same as above

classes described above, have undergone some evaluation for fusion reactor magnet service. Table 2 lists some of the more prominent types which have been considered along with their intended application. More complete listings of radiation resistant plastics are available, for example in References 1 and 20.

The first four materials in Table 2 are well characterized high performance laminates with epoxy or Kerimid-601 polyimide matrix, and E-glass reinforcement. Since E-glass contains ~3 wt % boron which has a high (n, α) cross section for thermal and epithermal neutrons leading to fission and concomittant damage, interest has developed in variants with S-glass (boron-free) reinforcement. While the thermal neutron flux in fusion magnets will probably be low, the epithermal population in the neutron energy spectrum may be high enough (depending

upon reactor design) to produce significant damage by ^{10}B fission. Of these four materials, the first two display the best starting properties, while the latter two, decidedly inferior to start, exhibit good retention of properties after irradiation.

Resins based upon diglycidyl ether of bisphenol A (DGEBA) are very common, and can be modified in many ways to give various properties. Because manufacturers blend resins for various purposes, it is advisable to consider particular brands since results may vary between products of different manufacturers. Epon 828 resin with various curing agents, fillers, or reinforcements has received considerable attention. Araldite is a similar resin from a different manufacturer, and Aracast is a modified epoxy from that manufacturer. Fillers* which have been considered

*The expressions "fillers" and "reinforcements" distinguish between primarily inorganic, particulates and high-aspect-ratio fibers which may be added to plastics to modify their properties.

include SiO_2 ²¹⁻²³ and mica²³⁻²⁴ while epoxies reinforced with E-glass,²⁵⁻²⁶ S-glass²⁶ and carbon²⁷ fibers have been investigated.

Mylar and Kapton are candidates for slip-plane insulation, or in metallized form, for thermal superinsulation. Vespel is a bulk version of Kapton film. Properties of these materials are well known, and considerable radiation effects data are available.

Both the organic matrix and the filler or reinforcement in these materials may undergo radiation damage in the dose range of interest, although effects in carbon fibers are small. The organics are damaged by both neutrons and gamma rays. The onset of degradation for many materials is in the low 10^6 Gy absorbed dose range,^{25,26} with degradation noted in mechanical properties as we describe later. More radiation-resistant organics, such as polyimides, tolerate doses at least an order of magnitude greater.²⁸ It is possible that even more resistant organics may be developed. Ultimately, the radiation resistance of the filler or reinforcement rather than the resin may impose a limit on the useful dose range.

Silica glass and muscovite mica undergo radiation damage by radiolysis. Silica compacts upon reactor neutron irradiation with observable density changes at less than 1×10^{19} nvt and the largest change, ~3%, at $\sim 4 \times 10^{19}$ nvt.²⁹ To find the range of deposited energy related to this fluence range requires neutron spectrum data we do not have. For a fission reactor spectrum, however, we estimate roughly that the range is less than 10^9 Gy specified for development purposes. Glasses of other compositions exhibit both positive and negative density changes of similar magnitude upon irradiation.³⁰ Mica also undergoes radiolysis, which results in substantial swelling when irradiated in the same dose range.³⁰ The effect of these volume changes on the properties of organic composites requires careful

evaluation, as well as an experimental study of the reinforced (E-Glass and S-Glass) materials now in use.

The foregoing recognizes the role of the composite nature of laminated glass-reinforced insulation on its radiation response. Thus, if more radiation resistant composites are developed, then a resin/reinforcement mismatch in radiation response, due for example to shrinkage of the reinforcement, may define the limit to irradiation tolerance. Klabunde and Coltman³¹ have shown that a separation occurs at the interface between epoxy and glass fibers in G-10CR and G-11CR and have proposed that the separation may occur as irradiation-induced off-gases preferentially migrate to the interface. They point out that tensile stress generated in the same location by differential expansion on warming to room temperature after irradiation would contribute to the separation. We speculate here that further contributions to the interfacial stress may result at high dose from irradiation damage to the reinforcement.

The composite nature of such materials is important in another way. Hurley et al.²⁶ have discussed the effect of compositional heterogeneity on damage deposition in G-10CR. It was noted that such materials are composed of bundles of organosilane treated glass fibers interspersed with epoxy, and the fiber bundles, themselves more widely spaced than the individual fibers, are also interspersed with epoxy. When aminosilane is used, the fiber bundles are locally rich in hydrogen, making the interfacial areas preferred sites for energy deposition under neutron irradiation. Damage is produced over a few 10 's of μm range by hydrogen knock-ons. In contrast, the glass fibers absorb less energy from neutron bombardment than the surrounding organic material, further complicating our understanding of these spatial effects.

As we have noted above, both neutrons and gamma rays damage the component phases of the

organic insulation; details of the interactions differ and may, hence, give rise to phenomenological differences in the ensuing damage. Neutrons interact primarily by elastic collisions with nuclei, resulting in a scattered neutron of lower energy and an energetic ion. The maximum energy (since momentum and energy are conserved) which can be imparted by the neutron decreases as the mass of the struck nucleus increases. Thus, the highest possible energy transfer is to hydrogen and that can equal the energy of the impinging neutron. Inelastic collisions can occur when the neutron is captured by the struck nucleus. Capture of neutrons by boron in E-glass is an example of the latter, and it is followed by fissioning of the unstable nucleus yielding ${}^7\text{Li}$ and ${}^4\text{He}$ recoil nuclei. Thus, neutron bombardment results in energy transfer to charged particles, in organic materials, primarily a proton. Gamma rays, with typical mid-range energies, interact with electrons to produce an energetic Compton electron and another photon, of reduced energy.

Both gamma rays and neutrons, then, result in the internal generation of energetic charged particles, electrons and ions, respectively. These, in turn, lose energy by ionization and excitation. These events are similar, whether produced by Compton electrons (gamma rays) or ions (neutrons). The latter, however, tend to produce tracks of greater defect density, than do electrons. These differences may be important in controlling the type of radiation damage observed for differing mixes of neutron and gamma ray intensities.^{1,20} Such effects too must be considered in evaluating experimental data obtained from samples irradiated in differing spectra.

Differences in the nature of the damage produced by neutrons and by gamma rays have been observed in a few organics^{14,15} but they have not been investigated in materials

proposed for fusion reactors under appropriate irradiation conditions. Pluym and Van de Voorde¹⁴ found an increase in main-chain scissions in polymethylmethacrylate (PMMA) for neutron over gamma-ray irradiation, but were unable to detect differences in changes in strength in two epoxy systems after irradiation under the same conditions.

4. RADIATION EFFECTS ON PRIME CANDIDATE MATERIALS

Although many organic materials are considered for magnet insulation (Table 2, Refs. 1, 20, 32, 33), interest in the U. S. fusion program has centered on the materials 1 to 5 and 9 in Table 2. All of these materials have been irradiated at 4.2 K with subsequent testing at 77 K. In addition, samples based on Epon 828, and on Vespel have been irradiated and tested at 4.2 K without intervening warmup. In this section, we briefly review the pertinent findings for these materials. Some of these materials have been irradiated and tested under other conditions which we do not review. The reader is directed to references 1, 20, 32, and 33 for information on some of these other studies.

4.1. G-10CR

G-10CR is a high strength laminate in the unirradiated condition, but loses strength rapidly upon irradiation with both gamma rays and neutrons. Longitudinal flexure strength was reduced 15% after neutron irradiation²⁶ to 2.48×10^6 Gy and 40% after gamma irradiation²⁵ to 3×10^6 Gy. Both of these contained E-glass, and the latter irradiation had additionally an equal dose from ${}^{10}\text{B}$ fissions. Results for a laminate containing boron-free S-glass however, agreed more closely with the neutron results. These two materials lost strength rapidly upon further gamma-ray irradiation, as shown in Fig. 2, with the strengths of G-10CR and G-10CR BF falling to half their

FIGURE 2

The results of flexure strength tests at 77 K on glass-fabric filled epoxies after gamma-ray irradiation at 4.9 K and warmup to 307 K.

initial strengths at 3.5 and 5.5 MGy respectively. Both neutron and gamma-ray irradiation tests were made at 77 K after irradiation in liquid helium and warmup to room temperature.

4.2. G-11CR

G-11CR exhibits improved radiation tolerance in both gamma-ray and neutron irradiation tests.^{25,26} After irradiation to 2.31×10^6 Gy in a neutron source, no reduction in flexure strength was noted. This result is consistent with the gamma-ray data. The strength of G-11CR was reduced by 50% after gamma-ray

irradiation to approximately 6.2×10^6 Gy; a substantial improvement over G-10CR. These results confirm the expected superiority of G-11CR in terms of intrinsic radiation stability resulting from its aromatic structure.

Electrical properties of G-10CR and G-11CR do not change as markedly after irradiation as do the mechanical properties, up to gamma-ray doses of 10×10^6 Gy. Results of Coltman et al.,³⁴ summarized in Table 3, show the dependency of resistivity and dielectric breakdown strength, measured at room temperature, on imposed gamma-ray dose. Sharp reductions in both property values occurred between 0.24 and 1×10^8 Gy. Room temperature bulk breakdown strength for G-10CR and G-11CR has been reported as high as 78 kV/mm,²⁶ indicating that the reduction at 1×10^8 Gy may be to ~10% of the starting value. Whether or not these changes are of concern are not now known. Certainly, the postirradiation breakdown strengths and resistivities are large compared to electrical stresses. However, the extrapolation of values measured on small samples to the large areas involved in the magnets should be carefully considered.

4.3. Spaulrad and Norplex NP-530

Spaulrad, manufactured by Spaulding Fibre Co., and Norplex NP-530 are similar laminates of E-glass and a polyimide (Kerimid) matrix. Both materials showed little decrease in

TABLE 3

Electrical measurements at room temperature after irradiation at 5 K (each result is the average of three measurements)

DOSE (10^8 Gy)	RESISTIVITY ($10^{13} \Omega\text{-m}$)			DBS (kV/mm)*		
	Epon 828	G-10CR	G-11CR	Epon 828	G-10CR	G-11CR
Control	20	8.2	4.4	33*	23*	24*
0.24	5.5	8.5	2.8	34*	23*	23*
1.0	8.1	0.64	0.14	31 ¹	8 ³	10 ³

*The superscripts indicate the number of samples exhibiting volume breakdown.

strength when irradiated in a gamma source up to 2×10^7 Gy.²⁸ In this dose range, the epoxy matrix laminates had lost most of their strength.

Spaulrad, the stronger of the two polyimide matrix materials, lost 40% of its initial flexure strength (1000 MPa) at 10^8 Gy, while MP-530 declined about 35%, from an initial value of 680 MPa, tested at 77 K. Although these results do indeed indicate substantial (order of magnitude) improvement in dose resistance of the polyimides, their properties are initially inferior to those of the epoxy matrix laminates, and it remains to be seen whether resistance to fatigue, multiaxial, and interlaminar shear stresses of the polyimides will meet the requirements of the application. Preliminary evidence of extensive damage (eg. delamination) has been observed in the latter materials during flexure testing.³⁵

4.4. Epoxy resins

Epon 828 resin, a product of the Shell Oil Co., is based on diglycidyl ether of bisphenol A, and irradiation tests on materials with a variety of hardeners, fillers, and reinforcements have been made. Samples of this resin cured with aliphatic amine, aromatic amine, or acid anhydride were irradiated in a nuclear reactor at 4.2 K by Takamura and Kato,²⁷ and tested at 77 K and at 4.2 K. Sharp reductions in compression strengths were observed after a combined gamma ray and neutron irradiation equivalent to a dose of 1.1×10^7 Gy. Irradiation-induced strength loss was greatest for aliphatic amine-hardened material.

The same investigation studied the effect of both glass and carbon fiber uniaxial reinforcement of acid anhydride-hardened material. Initial compression strength was much higher and was unaffected by temperature between 77 K and 4.2 K, with carbon fiber-reinforced material the stronger of the two. The loss of

strength due to irradiation was small at both temperatures, but larger at 4.2 K than at 77 K. In another experiment, aromatic and aliphatic amine hardeners were used and the testing was conducted in flexure after a dose of 7.1×10^6 Gy. This experiment suggested an irradiation-induced weakening of the fiber-matrix interface.

Epon 828 resin with the Shell "Z" hardener (blended aromatic amine),³⁶ 40 wt.% SiO_2 filler, and 0.5 wt.% 3-(2-Amino-ethylamino) propyltrimethoxy silane has been neutron irradiated to 2.2×10^6 Gy²⁶ and gamma irradiated to 10^8 Gy in the BSR.³⁴ At the low neutron dose no important mechanical property changes were observed, although yield stress in compression tests appeared to increase slightly.²⁶ Results for higher dose gamma irradiations revealed behavior similar to polyimide matrix materials, with a large decrease in mechanical strengths occurring between 1.0 and 10×10^7 Gy.³⁴

4.5. Films

Nomex, Mylar and Kapton have been irradiated at 4.2 K and evaluated after doses up to 1 to 10×10^7 Gy. Mylar was found to be too weak to handle after doses as low as 6×10^6 Gy. Nomex and Kapton were found qualitatively to be useful after 10^8 Gy,³⁴ although small decreases in strength have been measured after 1.1×10^7 Gy.²⁷ Vespel, the bulk form of Kapton, has shown little deterioration in mechanical properties after irradiation to 1.1×10^7 Gy and compression testing, both at 4.2 K.²⁷

5. PROBLEM AREAS AND FUTURE WORK

The response to irradiation of the materials listed in Table 2 and described in the foregoing, suggests a number of problem areas and topics for future research. These include additional characterization of materials, development of a fundamental understanding of

damage mechanisms and development of new materials.

In the work which has been described, samples of candidate materials have been irradiated in cryogenic sources (mostly gamma sources) and then characterized by means of comparatively simple (i.e. flexure and compression) mechanical tests. However a few studies have suggested that problems may occur at even lower doses in tests involving either fatigue or multiaxial stresses. For example, Conway³⁷ has observed radiation effects in tests of G-11CR at ambient and 77 K after room temperature irradiation in a gamma source. Testing was in tensile fatigue. Effects at this dose in simpler tests would not be expected. Further, Wang and Chim³⁸ have shown that multiaxial (tension-torsion) fatigue testing results in more significant mechanical degradation than does uniaxial fatigue alone, in G-10CR. These results emphasize the need for irradiation testing of this kind.

Further efforts needed on materials of current interest include: continuation of studies characterizing failure in unirradiated materials,³⁹ characterization of neutron damage effects, characterization of failure mechanisms after irradiation, and more extensive testing at 4.2 K with and without warmup to room temperature. Results of Takamura and Kato^{39,40} have suggested that results of testing at 4.2 K may be different from those at 77 K, and that intervening room temperature warmup may also affect the results.

A fundamental understanding of radiation effects is needed in order to extrapolate behavior to new radiation environments and to predict how new materials may be developed better to withstand the fusion environment. The basis for differentiation between neutron and gamma effects and initial results of a phenomenological comparison between results for the same materials after irradiation in IPNS

and BSR have been discussed. Further work on analytical studies using model compounds has been proposed.³⁵

Development of new materials is a long term goal which is made desirable by the poor performance of materials like G-10CR and rendered promising by results such as the improvement noted for the polyimide matrix laminates. Studies of glass-reinforced Epon 828 systems have revealed radiation resistance superior to G-10CR and G-11CR which is not yet understood. Development of new laminates is an ambitious task which requires consideration of matrix, interface, and reinforcement.

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