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Abstract

Available low-temperature neutron and gamma irradiation data for organic insulating materials are collected and compared with room temperature data. Only the most promising polymers in terms of mechanical strength for magnet insulation are taken into account. For characterization and comparison of different materials the 75 % dose is used, i.e. the dose, where the mechanical strength is reduced by 25 %, and 75 % is retained. For room temperature special prepared polyimide and epoxy materials reinforced with glass fibre retained 75 % of the mechanical strength up to a dose of 7×10^7 Gy. For 5 K irradiation the best epoxy material retained the 75 % dose up to 1×10^7 Gy, the best polyimide material up to 1×10^8 Gy.

Neutronen- und Gammabestrahlungseffekte in organischen Materialien für Fusionsmagnete

Zusammenfassung

Verfügbare Tieftemperaturdaten für Neutronen- und Gammabestrahlungen an organischen Isolationsmaterialien wurden gesammelt und mit Zimmertemperaturbestrahlungsdaten verglichen. Nur die von der mechanischen Festigkeit her vielversprechendsten Polymere für Magnetisolationen wurden berücksichtigt. Zur Charakterisierung und zum Vergleich der verschiedenen Materialien wird die 75-%-Dosis benutzt, d. h., die Dosis, bei der die mechanische Festigkeit um 25 % reduziert ist und noch 75 % verbleiben. Speziell hergestellte glasfaserverstärkte Polyimide und Epoxy-Proben behielten 75 % ihrer mechanischen Festigkeit bis zu einer Dosis von 7×10^7 Gy. Für 5 K Bestrahlung behielt das beste Epoxy-Material 75 % seiner Festigkeit bis etwa 1×10^7 Gy, während das beste Polyimid-Material bis zu 1×10^8 Gy 75 % seiner Festigkeit behielt.

Table of Contents

1. Introduction

The plasma of a fusion reactor contained by magnetic fields emits neutron and gamma radiation. Before reaching the superconducting magnets, the level of radiation is reduced by blanket and shield materials and by geometrical factors. The reduction factor must be in the order of 10^6 to 10^8 to meet essential criteria for the magnetic field producing superconducting coils operated at 4 K. One criterion is that the radiation-induced degradation of the properties of all materials used in the magnets must be sufficiently small to guarantee acceptable performance over the reactor lifetime. Another criterion is that the refrigeration power necessary to remove nuclear heat in the magnets must be less than a given small fraction of the total power produced by the fusion reactor. In a reactor design, a cost-minimizing trade-off must be made between materials cost and power cost.

We limit us to the most promising polymers investigated until now in radiation environments. These polymers are used in coils operating at cryogenic temperatures. Ceramic insulation materials are not considered in this report. Those materials are used for near-room-temperature copper coils exposed to intense radiation fields.

There are a lot of reports on radiation of organic insulators as seen in the reference list. All reports were used and searched through, but not all reports are cited.

2. Function of Insulation in Fusion Reactor Magnets

2.1 General operation conditions

In table 2.1 general operation conditions are listed for magnets used in fusion reactors. In this report the radiation effects on organic materials are discussed. During the lifetime of the magnets a radiation dose of (10^7 -10^9) Gy (1 Gy = 100 rad) is expected.

The electrical and thermal insulation material is used, in the form of potting resin, flexible sheets or rigid plates. The insulation separates electrically the turns and the layers or pancakes of the magnet coil from each other and from the magnet case (ground potential). It transfers the magnetic forces from the windings to the case. Moreover, it can provide (in bath cooled magnets) channels for the liquid helium coolant to remove heat from the conductor surface and to transport it to the (gas) outlet of the magnet connected to the refrigeration system. The thermal insulation is used in the cryostat to decrease the heat influx from higher temperature levels (LN_2 or room temperature). A common material is aluminized Mylar.

Since a fusion reactor has to have a high reliability and availability, the mechanical and electrical properties and loads of the insulation has to be known in order to estimate the lifetime. Additionally, the neutron and gamma radiation tends to degrade mechanical and electrical properties. Therefore,the change of properties due to irradiation must be known as a function of dose and taken into account during design to guarantee sufficient performance at the end of the designed magnet life. Table 2.2 shows the kind and the order of magnitude of load levels for the insulation in a superconducting fusion reactor magnet. Tensile, compressive and shear stresses are generated by magnetic, thermal and pressure loads. They act together or independently. Therefore, there may be large relative motions, large with respect to the insulator dimensions. Cyclic loads in pulsed reactor require the knowledge of fatigue properties at 4 K.

Compared with other components of a superconducting magnet (superconductor, stabilizer) the organic insulators behave differently since they can suffer damage from both neutrons and gamma rays, and the basic physical effects are different.

Table 2.1: General operation conditions for insulation materials in fusion reactor magnets (1 Gy = 100 rad)

Probable Materials	Function	Temperature Range	Radiation Load
Ceramics	Electrical insulation for near first wall use		
	- active and passive plasma control coils	300 K - 800 K	(1 - 10) MWy/m^2
	- normal conducting insert coils for high field choke coils	300 K - 400 K	(1 - 10) MWy/m^2 ($\lesssim 1.4 \times 10^{22}$ N/m^2)
Organics (or ceramics?)	Electrical insulation for normal conducting magnets (far from plasma)	300 K - 400 K	(10^7 - 10^9) Gy
	Electrical insulation for superconducting magnets (far from plasma)	4 K	(10^7 - 10^9) Gy
Organics	Thermal insulation for superconducting magnets	4 K - 300 K	(10^7 - 10^9) Gy
(aluminized organics or aluminum) with fiber glass spacers	- heat radiation shield	4 K - 300 K	(10^7 - 10^9) Gy
(Organics)	- magnet supports	4 K - 300 K	(10^7 - 10^9) Gy

Table 2.2: Order of magnitude of load levels for the insulation in a superconducting fusion reactor magnet

	Order of Magnitude
- Loads during manufacturing	?
Loads during handling	?
Loads during transportation	?
- Loads during environmental accidents (earthquakes, explosions)	?
- Thermal stresses during cooldown	
- Static mechanical loads due to large Lorentz forces in toroidal and mirror configurations	
. tensile stress	(100 - 400) MPa
. compressive stress	(100 - 400) MPa
. interlaminar shear stress	(10 - 30) MPa
. interturn and interlayer shear stress	
. dimensional changes	percent
- Time dependent mechanical loads	
. twisting moments (bending moments) due to time varying magnetic fields in toroidal configurations	1 MNn
. cyclic pulsed pressure load	100 MPa
. cyclic interlaminar shear stress	$(10^{4}$ - 30) MPa
. fatigue effects	$(10^{4} - 10^{6})$ cycles
- Electrical loads	
High voltages during charging and especially during rapid discharging of the magnets	
. turn-to-turn	(1 - 3) V/turn
. layer-to-layer	(100 - 200) V/layer
. conductor-to-case (ground)	(3000 - 5000) V
- Radiation loads	
. neutrons	$(10^{18} - 10^{19})$ n/cm^2
. gammas	$(10^{7} - 10^{9})$ Gy
. nuclear heating	10^{-4} W/cm^3

2.2 Insulation Failure

The irradiation environment in a fusion magnet imposes the most severe limitation on the insulation because irradiation tends to weaken the mechanical properties of polymers and hence to induce brittle fracture resulting in magnet failure. Mechanical insulation failure is most often the case in a magnet. Other failure modes in a fusion superconducting magnet are loss of stability, electrical shorts and arcs, failure of the cryogenic system, conductor fracture and failure of the structural support system. But the insulation seems to be the weakest component, especially in a radiation environment.

The mechanical and electrical load in a magnet can cause crushing, fretting due to motions, void production, delamination and perforation. Additionally, time dependent effects amplify the failure modes cited above. Such effects are creep (time dependent plastic deformation), ageing (age dependent fracture strength) or generally fatigue. All these effects, together with the radiation effects are acting together at the same time.

3. Radiation Damage in Polymers

3.1 Mechanism of Radiation Damage in Polymers

3.1.1 General

The damage mechanisms in organic materials are different for neutrons and gamma rays, respectively. In metals where displacement damage is the most important damage mechanism the types of radiation are less important. In cases where excitation and ionization are the main features, as it is for organic materials, the types of radiation must be considered.

There is some evidence to suggest that the amount of ionization is proportional to the absorbed energy

> and is independent on the ionizing source (such as gammas or fast neutrons),
> but the spatial variation will certainly depend on the source of irradiation.

It appears to be impossible to estimate (or predict) theoretically the effects of radiation on the various mechanical and electrical properties of insulating materials, because the processes involved are so complex, e.g. not only the dose, but also the dose rate has been found to be important in some insulator irradiations.

3.1.2 Damage by Neutrons

Neutrons produce primary knock-on atoms as they do in other materials. These recoil and cause secondary reactions as ionization and excitation of electrons, and displacements of other atoms. The hydrogen content in the insulator is particularly important, because neutrons transfer a considerable amount of energy to hydrogen atoms due to nearly the same mass.

The monoenergetic neutrons born in the plasma will be moderated during their penetration of the blanket and shield and reach the magnet with a broad polyenergetic spectrum. Beside the elastic scattering producing a recoil atom and

electron excitation as final reaction products nuclear reactions are possible. The neutron-gamma reaction produces either a recoil atom and gamma rays or in a β-emitting process a recoil atom and an electron. Another process is important for glass filled insulation materials. If the glass contains boron, then a neutron-alpha reaction with $^{10}_{7}B$ is possible which ends up with an alpha particle, an electron and a ^{7}Li-recoil atom. It should be mentioned that the main damage process is however the elastic scattering of the neutrons on the atoms of the chain (H, C, O, N) leading to the liberation of ions, especially of hydrogen ions. These reactive species remain dormant until warm up /45/.

3.1.3 Damage by Gamma Rays

Gamma rays interact with inorganic materials via the photoelectric and Compton effect, and pair production.

Photoelectric effect: the gamma energy is sufficient to free an atomic electron; the result is a free electron and a positive charged ion. The energy transfer to the ion is approximately zero.

Compton effect: the gamma energy is sufficient to free an atomic electron and to change in a gamma with lower energy. The result is another gamma, a free electron and a positive charged ion. The energy to the ion is aproximately zero.

Pair production: an electron and a positron are produced by a gamma together with a mass as recoil partner. In this process the gamma energy must be greater than the twice of the electron rest energy ($\geq$ 1.02 MeV).

The freed electrons interact with others producing again ionization and excitation.

3.1.4 Overall Effects

All these atomic and interatomic processes produced by neutrons and gamma rays can result in:
- cross-linking, i.e. formation of chemical bonds between chains and
- chain scission, i.e. a fracture of the polymer molecule.

Additionally occurring secondary processes are:
- unsaturation, i.e. formation of double bonds in the molecular chain.
- gas evolution, i.e. dissociation of small side molecules from the polymeric chain.
- reactions with the environment, e.g. oxidation and decarboxylation.

Cross-linking leads to an increase of the molecular weight of a polymer, of the hardness, tensile strength and elastic moduli, and of the softening temperatures. It decreases the solubility and the elongation to failure and tends to destroy cristallinity. The materials eventually degrade as the material becomes embrittled and evolves gases.

Chain scission produces the opposite effects on the properties and leads eventually to a soft gummy or tar-like material.

These radiation effects in the organic insulators influence the design and operation of fusion magnets. The most apparent effects are changes in mechanical and electrical properties and in the dimensions, the outgassing and the release of stored energy during postirradiation warmup.

3.2 The Problem of Fluence-Dose Conversion

There are several ways to quantify the interaction of radiation with materials:
- use the fluence of the particles (particles/m^2) at a given energy or energy spectrum,
- calculate the energy deposited by the radiation (Gy, (or rad)) or the power per unit volume (W/m^3).
- calculate the number of events which occur in the irradiated material, e.g. displacements per atom (dpa).

- calculate production rates or percentages of the products, e.g. % transmutation products (ppm).

Usually, for organic polymers the fluence or the energy deposited by radiation is used. The energy deposition and energy deposition rates are different for different particles and radiation source intensities. For the same energy absorption the changes in material properties resulting from neutron irradiation may be different from those due to gamma irradiation due to the different damage mechanisms. Therefore, the conversion rates must be precisely calculated for each irradiation experiment. Also thermal and fast neutrons damage the material differently as discussed. Absorption of thermal neutrons and succeeding nuclear reactions lead to different damage effects compared with fast neutrons where the energy is transferred by elastic scattering with the nuclei. The transferred energy is given by

$$E_t = (4M / (M+1)^2)\, E_i$$

where M is the mass of the nucleus and E_i is the energy of the incident neutrons. For hydrogen is $E_t = E_i$, for oxygen $E_t = 0.21\, E_i$ and for carbon $E_t = 0.28\, E_i$. The multiplication of the collision cross-section, the number of atom and neutron fluence gives the maximum transferred energy per unit volume which is a rough estimate of the absorbed energy. As a rule of thumb a dose-fluence conversion relation of

$$1 \text{ rad} \approx 10^9 \text{ n/cm}^2 \quad (E \geqslant 0.1 \text{ MeV})$$

is obtained. ($1 \text{ rad} = 10^{-5}$ J/g $= 10^{-2}$ Gy) /19/.

A detailed analysis has to be made for each radiation source and for each experimental arrangement in order to get reliable data. Some examples are given in order to show the wide spread of the conversion factors /see e.g. 36, 54/:

dose-fluence relations:

- 1 rad $\approx$ 0.6×10^{9} n/cm^2 for the spectrum of the Intense Pulsed Neutron Source in ANL (Argonne National Laboratory)

- data in Chapt. 7 of /36/: (or Chapt. 6 in Ref. /54/)
  1 rad = 1×10^{10} n/cm^2 for Al_2O_3 (ceramics)
  1 rad = 0.5×10^{10} n/cm^2 for MACOR (ceramics)
  1 rad = 0.3×10^{10} n/cm^2 for boron free polyimide fiber
  1 rad = 0.26×10^{10} n/cm^2 for boron free G-10
  1 rad = 0.23×10^{10} n/cm^2 for G-10.

  1 rad = 0.27×10^{10} gammas/cm^2 (E_{av} = 0.8 MeV)

- for epoxy composites /36, 54/:

  1 rad/s = 0.2×10^{10} gammas/cm^2 s

  3 rad/s due to the ^{10}B (n,α) ^{7}Li reaction, where the high energy α's produce a damage cascade area.
  (Glass epoxy with 60 % E glass with 10 % B_2O_3)

  1.5 rad/s due to beta-emission processes in the glass content.

It should be noted that there is a big uncertainty in the determination of the dose in organic polymers and also in the reproducibility of the data. The radiation resistance of a polymer depends strongly on the exact molecular formula, which is usually not known (because it is a secret of the manufacturer). An example for the conversion of fluence values into rads the calculated values for the low temperature irradiation thimble VT 2 in the IPNS are given in Table 3.1 /76/:

Table 3.1: Conversion factors from total neutron fluence to absorbed dose for the low temperature irradiation thimble VT 2 in IPNS /76/

Compound	Conversion factor (10^{-9} rad/n/cm^2)	Weight fractions (%)
Epoxy	1.049	H(6.3), C(66.2), O(14.3), N(8.4), S(4.8)
Polyimide	0.890	H(5.1), C(73.4), O(11.5), N(10.1)
E-glass [a]	0.300	B(2.3), Ca(13.3), O(48.0), F(0.3), Mg(2.0),
	/0.203/	Al(7.8), Si(25.7), Na(0.4), Fe(0.2)
Carbon	0.206	C(100.0)
Alumina	0.095	Al(47.9), O(47.7), Si(4.4)
Silane [b]	1.327	H(8.5), C(45.7), O(33.8), Si(11.9)

a) Values in brackets are contributions due to boron in E-glass. The weight fractions are for Kanebo KS-1210.

b) γ-glycidoxypropyltrimethoxysilane.

4. Radiation Data of Polymers

4.1 Polymers under Consideration

Organic polymers are divided into three classes: thermosets, thermoplastics and elastomers.

Thermosets, e.g. epoxy and polyester resin, are utilized by mixing the resin with a curing agent which then react chemically together. The molecules of the curing agent tie the moleculus of the resin together forming linear and cross-linked chains. The mixture is eventually hard, insoluble and infusible. When heated it does not soften or melt, but at a temperature of about 500 K it begins to degrade.

Thermoplastics generally consist of linear molecule chains of much greater molecular weight and length than thermosets. Cross-linking is very weak. They soften and melt when heated. Examples for thermoplastics are polyethylene, polypropylene, and polymethylmethacrylate. Thermoplastics can have high cristallinity or they can be amorphous or glassy.

Elastomers, e.g. natural and synthetic rubber are similar in structure to thermoplastics, but they have much greater elastic strains at room temperature because their molecules are capable of greater mobility.

Relatively low radiation doses can produce in thermoplastics or elastomers a significant decrease in the molecular chain length by scisson or a significant increase of the cross-linking density. Both effects cause remarkable changes of the mechanical properties of the polymer. In the highly cross-linked relatively low molecular weight thermosets these effects are much less distinct, i.e. the mechanical properties of the thermosets are rather less affected by nuclear radiation than those of thermoplastics and elastomers. Thermosets were used as magnet insulation materials filled with glass fibre fabric (GFF) as reinforcer. Therefore the radiation effect on the filler must be included. These composites first became of interest for cryogenic applications during the space programmes of the 1960's /49/.

The mechanical properties of the most organic polymers begin to degrade at doses of about 10^4 - 10^5 Gy (10^6 - 10^7 rad). Only very few polymers are useful at doses greater than 10^7 Gy (10^9 rad). The latter are suitable for the use in fusion reactors.

From fission reactor development and high energy particle accelerators irradiation data are available. Mechanical and structural properties were studied on the conditions of tension, compression, flexure, and shear before and after irradiation at various temperature levels (room temperature, 77 K, 20 K, and 4 K). Also the fatigue behaviour is needed to know for pulsed fusion magnets. In addition, the gas evolution, the weight loss and/or dimensional changes, radioactivity, and the color and appearance changes give indications on the overall behaviour of the insulation material in a fusion magnet. Moreover electrical and thermal properties have been studied. Electrical failure occurs usually through the degradation of the mechanical properties rather than the intrinsic electrical properties. Therefore, mechanical data can be used to judge the probable suitability of a material.

4.2 Room Temperature Radiation Data for Reinforced Polymers

The influence of gamma and neutron irradiation at room temperature on the mechanical, thermal, and electrical properties of polymers has been studied already since the 50's. There are several reports /e.g. 6, 11, 34, 35, 58, 69/, where an overview is given for room temperature irradiation data.

This report summarizes the available data for candidate magnet insulation materials. Such materials have ultimate tensile, compression or flexure strengths of about 400 MPa (and higher) and interlaminar shear strengthes of about 50 MPa at room temperature. At low temperatures (4 K and 77 K) these mechanical properties are higher by a factor of two to three. Materials of special interest are epoxy-and polyimide based reinforced materials.

The tables 4.2-1 to 4.2-6 summarize the materials, suppliers and ultimate flexure strength without radiation for materials used in the investigations at CERN. Only materials with about 400 MPa (and higher) are taken into account. The figures 4.2-1 to 4.2-6 show the dependence of the ultimate flexure strength normalized to the value without radiation in percent. Table 4.2-7 gives the doses for the materials to reduce the ultimate flexure strength by 25 %.

Table 4.2-1: Polyimide-based materials

No.	Material	Supplier	Ultimate Flexure Strength without Radiation (MPa)
P1	Polyimide + glass fibre	ISOLA[1)]	426.7 ± 71.6 (100 % ± 16.8 %)
P2	KINEL 5.504 (polyimide)	Rhône-Poulenc[2]	375.7 ± 18.6 (100 % ± 5 %)
P3	KERIMID 601 (glass fibre 181E) (Bis-maleimide amine)	Rhône-Poulenc[2)]	503.3 ± 42.5 (100 % ± 8.5 %)

1) ISOLA, Fabrique Suisse d'Isolants, Breitenbach, Switzerland.

2) Rhône-Poulenc, Genève, Switzerland.

Table 4.2-2: Polyester-based materials

	Polyester	Supplier	Ultimate Flexure Strength without Radiation (MPa)
PO1	Polyester isophtalic + taffeta material	SNPE[3)]	466.0 ± 8.8 (100 % ± 1.9 %)
PO2	CEVOLIT 1413 (polyester resin + glass fibre)	CELLPACK AG[4)]	301.2 ± 5.9 (100 % ± 2 %)
PO3	G-ETRONAX PM (polyester laminate + 40 % glass fibre)	Elektro-ISOLA[5)]	197.2 ± 17.7 (100 % ± 9 %)

3) SNPE, Société Nationale de Poudres et Explosifs, Paris, France.

4) CELLPACK AG, Wohlen/Aargau, Switzerland.

5) Elektro-ISOLA AIS, Veigle, Denmark.

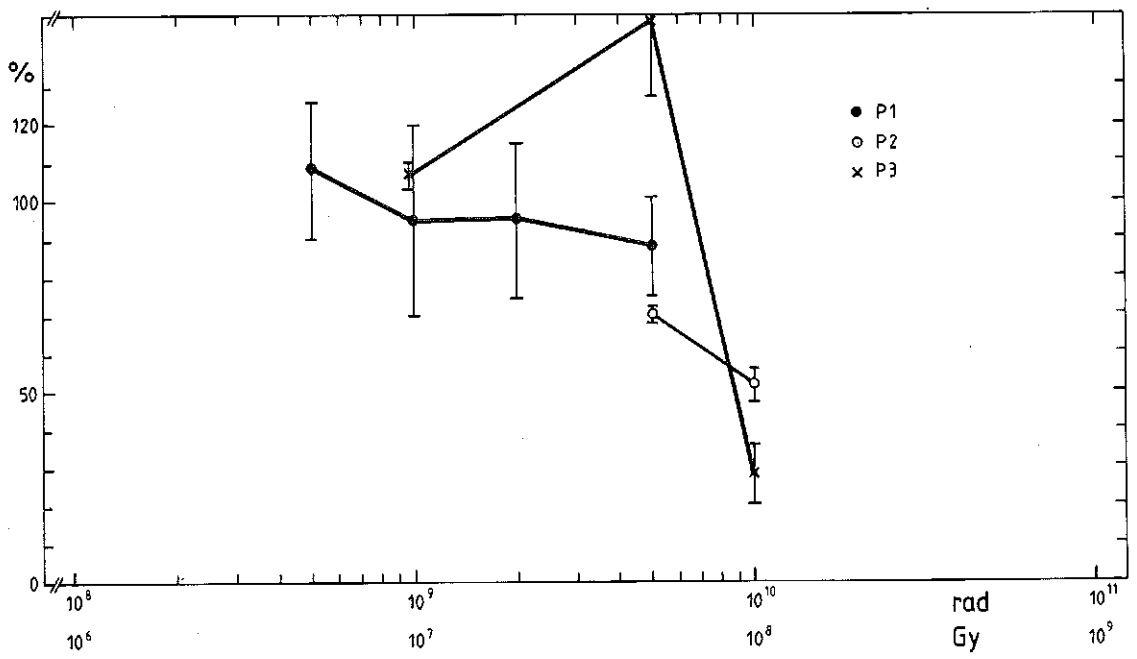

Fig. 4.2-1: RT-irradiation with reactor neutrons. CERN-data for polyimide materials /35/.
(% ultimate flexure strength vs dose)
(P1, P2, P3 according to table 4.2-1)

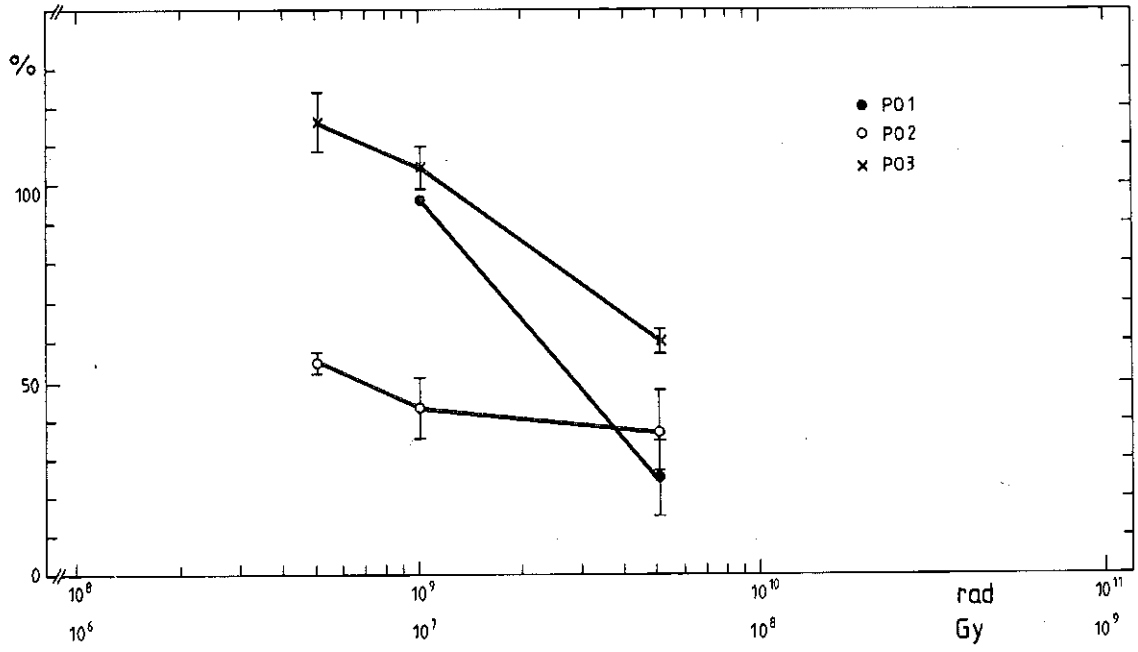

Fig. 4.2-2: RT-irradiation with reactor neutrons. CERN-data for polyester materials /35/.
(% ultimate flexure strength vs dose)
(PO1, PO2, PO3 according to table 4.2-2)

Table 4.2-3: Epoxy Novolac-Materials

	Material	Supplier	Ultimate Flexure Strength without Radiation (MPa)
N1	EPIKOTE 154 + MNA[a] + glass tape	MICAFIL[6]	441.4 ± 18.6 (100 % ± 4.2 %)
N2	EPIKOTE 154 + DDS[b] + glass fabric	MICAFIL	430.6 ± 11.8 (100 % ± 3 %)
N3	EPIKOTE 154 + DDM[c] + glass fabric	MICAFIL	351.2 ± 14.7 (100 % ± 4.2 %)
N4	Epoxy Novolac 438 + HY 906[d] glass fabric	ISOLA[1]	458.1 ± 74.6 (100 % ± 16.3 %)
N5	Epoxy Novolac 431 + MNA + accelerator	ISOLA	464.0 ± 74.6 (100 % ± 16.1 %)
N6	Epoxy Novolac 431 + BF_3MEA[e] complex	ISOLA	363.9 ± 15.7 (100 % ± 4.4 %)
N7	VETRESIT 14; EPIKOTE 827 + DDS + glass tissue	MICAFIL	445.4 ± 3.9 (100 % ± 1 %)

6) MICAFIL, Zürich, Switzerland.

a) MNA = Methyl nadic anhydride (Hardener).

b) DDS = Diaminodiphenyl sulfone (Aromatic hardener).

c) DDM = Diaminodiphenyl methane (Aromatic hardener).

d) HY 906 = Acid anhydride (Hardener).

e) BF_3MEA = Boron trifluoride monoethylamine BF_3-NH_2-CH_2-CH_3.

Table 4.2-4: Epoxy resin materials

No	Epoxy resin materials	Supplier	Ultimate Flexure Strength without radiation (MPa)
E1	BIRAKRIT-Epoxy fibre-glass laminate type 2370.4	UOP[7)]	546.4 ± 27.5 (100 % ± 5 %)
E2	BIRAKRIT-Epoxy fibre-glass laminate type 2372.2	UOP	508.2 ± 46.1 (100 % ± 9 %)
E3	VETRONIT EPG 10	ISOLA[1)]	510.1 ± 21.6 (100 % ± 5 %)
E4	VETRONIT EPG 11	ISOLA	350.2 ± 6.9 (100 % ± 2 %)
E5	Epoxy, type EGS 102	Ferrozell[8)]	538.6 ± 4.9 (100 % ± 1 %)
E6	ISOVAL 11 + charge 2569 (epoxy resin with glass)	ISOVOLTA[9)]	364.9 ± 10.8 (100 % ± 3 %)
E7	SAMICATHERM 366.28.02 Epoxy resin + Mica[f)] + glass tape	ISOLA	224.6 ± 7.8 (100 % ± 3.5 %)
E8	Epoxy resin + Mica + glass tape	CIBA-GEIGY[10)]	299.2 ± 19.6 (100 % ± 7 %)

7) UOP, Bisterfeld & Stolting GmbH, Egerpohl/Wipperfürth, West Germany.

8) Ferrozell-Geschw. Sachs & Co. mbH, Augsburg, West Germany.

9) ISOVOLTA, Vienna, Austria.

10) CIBA-GEIGY, Basel, Switzerland.

f) MICA = $K_2O \cdot 3Al_2O_3 \cdot 6SiO_2 \cdot 2H_2O$.

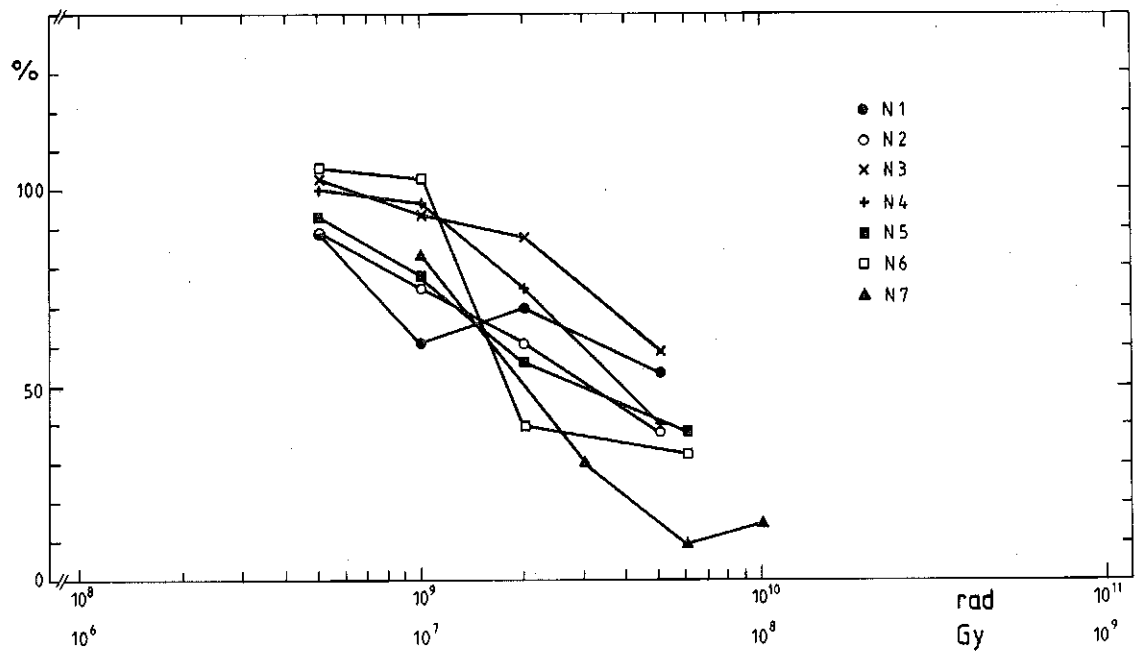

Fig. 4.2-3: RT-irradiation with reactor neutrons. CERN-data for epoxy NOVOLAC materials /35/. (% ultimate flexure strength vs dose) (N 1 etc according to table 4.2-3)

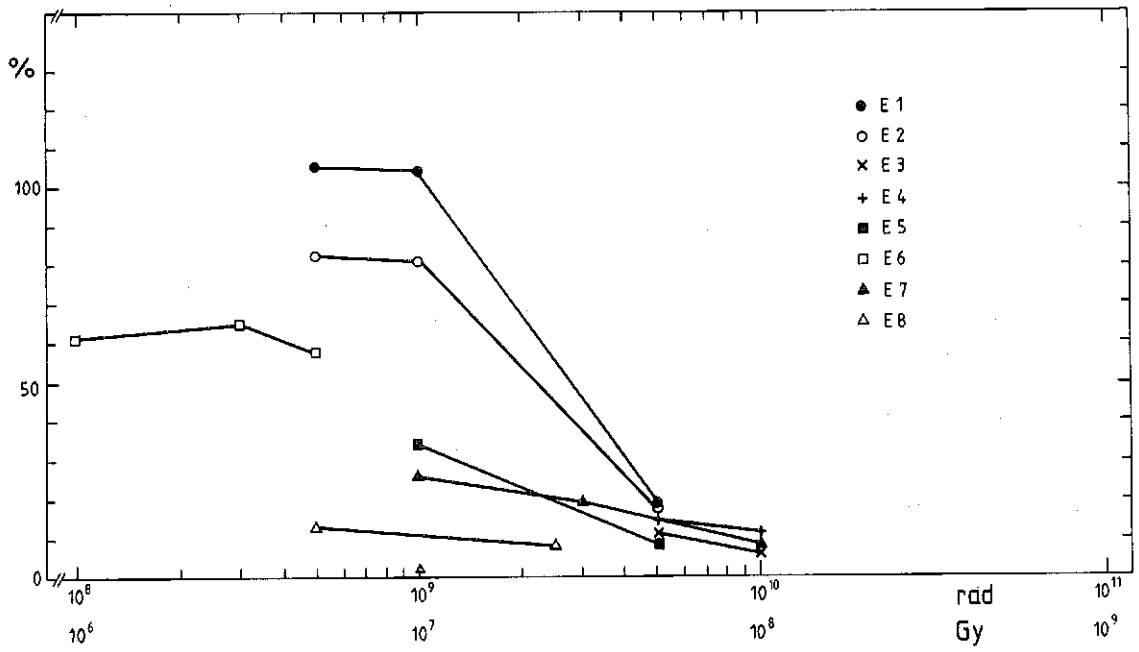

Fig. 4.2-4: RT-irradiation with reactor neutrons. CERN-data for epoxy resin materials /35/. (% ultimate flexure strength vs dose) (E 1 etc according to table 4.2-4)

Table 4.2-5: Epoxy resin (Araldite) materials

	Araldite - Materials	Supplier	Ultimate Flexure Strength without radiation (MPa)
A1	MY 740(100)[g] + MNA(80) + DMP 30(0.5)[h] + glass	PLESSEY[11]	283.5 ± 7.8 (100 % ± 2.0 %)
A2	MY 745[i] + HY 906 + XB 2687[j] + glass	ALSTOM[12]	333.5 ± 56.9 (100 % ± 17 %)
A3	Magnet coil resin type B reinforced with fibre-silanized glass tape type 1 (Base: TGDM[k] + MNA + other components) (cut parallel to fibre)	BBC Baden[13]	421.8 ± 83.4 (100 % ± 20 %)
A4	Araldite F(100) + MNA(80) + DMNA(0.5) + filler	Rutherford Workshop[14]	312.9 ± 2.9 (100 % ± 1 %)
A5	Araldite F + MNA + filler	LINTOTT[15]	436.5 ± 55.9 (100 % ± 13 %)

11) PLESSEY Company Ltd. Ilford, Essex, England.

12) ALSTOM, Belfort, France.

13) BBC, Baden, Switzerland.

14) Rutherford Workshop, Rutherford, England.

15) LINTOTT Engineering Ltd., Horsham, Sussex, England.

g) MY 740 = unmodified epoxy resin based on Bisphenol A.

h) DMP 30 = accelerator.

i) MY 745 = modified epoxy resin based on Bisphenol A.

j) XB 2687 = accelerator.

k) TGDM = Tetraepoxypropyl-methylene-dianiline.

Table 4.2-6: Orlitherm (R) - Materials

No	Araldite F [CY 205]	Supplier	Ultimate Flexure Strength without radiation (MPa)
01	Magnet coil resin Orlitherm (R) reinforced with fibre-silanized woven glass type 1 and mica-paper tape	BBC Baden	224.6 ± 11.7 (100 % ± 5 %)
02	same as 01 without mica-paper tape, 12 h, 165 °C	BBC Baden	510.1 ± 11.8 (100 % ± 2.5 %)
03	Orlitherm (R) reinforced with glass woven tape type 2 with a special silane finish, 12 h, 165 °C	BBC Baden	450.3 ± 24.5 (100 % ± 5.5 %)
04	Orlitherm (R) reinforced with glass woven tape type 2 with a special silane finish and mica-paper tape, 5 h, 135 °C + 6 h, 160 °C	BBC Baden	264.9 ± 9.8 (100 ± 4 %)
05	Orlitherm (R) reinforced with a fibre-silanized woven glass tape type 3, 5 h, 135 °C + 6 h, 160 °C	BBC Baden	563.1 ± 25.5 (100 % ± 5 %)
06	Orlitherm (R) reinforced with a fibre-silanized woven glass tape type 3 and mica-paper tape, 5 h, 135 °C + 6 h, 160 °C	BBC Baden	223.7 ± 19.6 (100 % ± 9 %)
07	Orlitherm (R) reinforced with a sandwich tape built up of a fibre-silanized woven glass tape type 32 and a polyimide film, 16 h, 140 °C	BBC Baden	379.6 ± 44.7 (100 % ± 12 %)

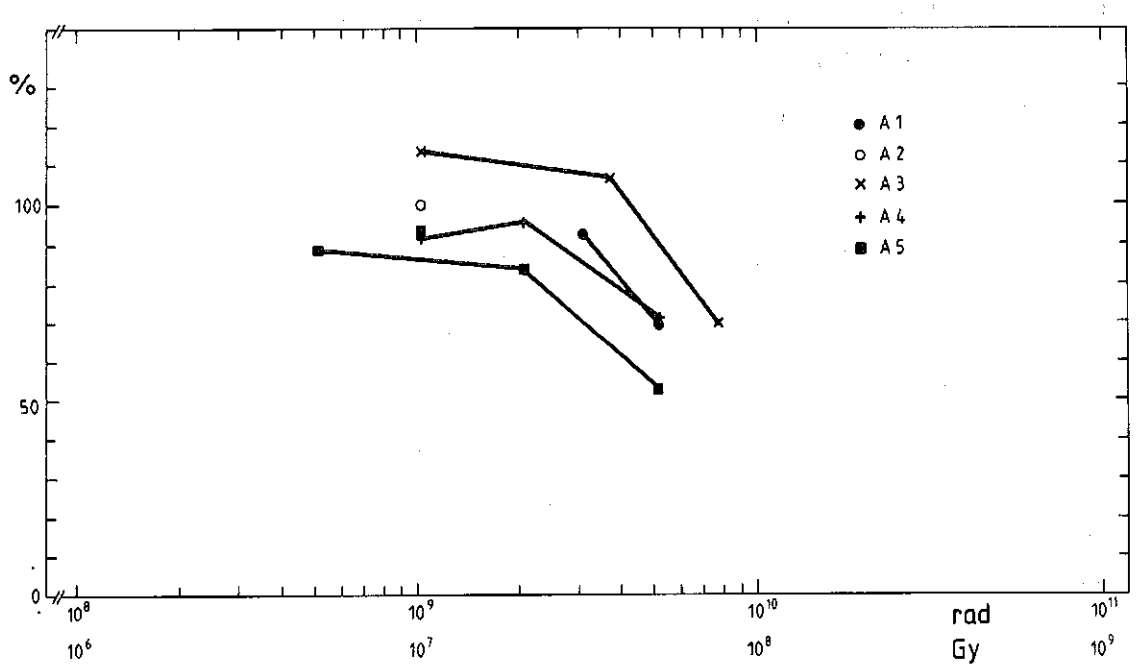

Fig. 4.2-5: RT-irradiation with reactor neutrons.
CERN-data for Araldite materials /35/.
(% ultimate flexure strength vs dose)
(A 1 etc according to table 4.2-5)

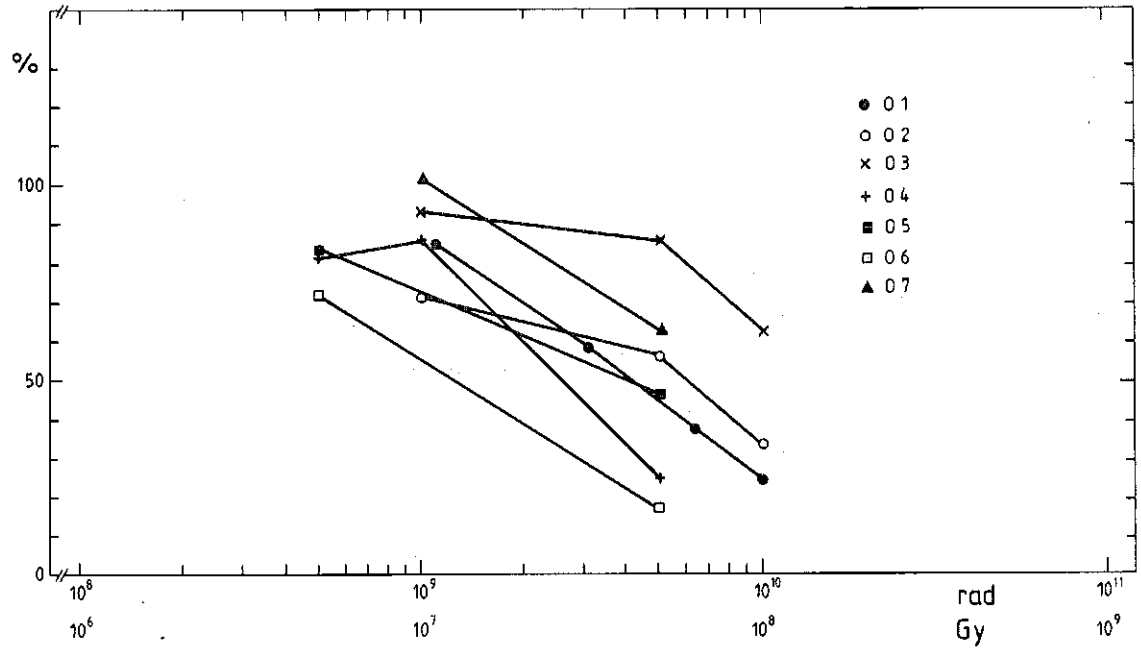

Fig. 4.2-6: RT-irradiation with reactor neutrons. CERN-data for Orlitherm Ⓡ-materials. (% ultimate flexure strength vs dose)
(O 1 etc according to table 4.2-6)

Table 4.2-7: Doses to reduce the ultimate flexure strength (UFS) by 25 % for the materials of Tables 4.2-1 to 4.2-6. RT-irradiation

Material	75 % Dose (Gy) [to reduce UFS by 25 %]
P1	5×10^7
P2	4×10^7 (extrapolated)
P3	7×10^7
P01	1.5×10^7
P03	3×10^7
N1	7×10^6
N2	10^7
N3	3×10^7
N4	2×10^7
N5	10^7
N6	1.3×10^7
N7	1.2×10^7
E1	1.7×10^7
E2	1.2×10^7
A1	4.5×10^7
A2	10^7
A3	6.5×10^7
A4	4.5×10^7
A5	2.6×10^7
01	1.6×10^7
02	7×10^6 (extrapolated)
03	7×10^7
04	1.3×10^7
05	8×10^6
06	4.5×10^6 (extrapolated)
07	3×10^7

Extensive studies on room and low temperature irradiation on five specially prepared specimens are reported in /74, 76, 77/. Two of the samples had epoxy resins as matrix and were reinforced one with E-glass and the other with carbon fibres; two others had a polyimide (Kerimid 601) matrix reinforced again with E-glass and carbon fibres. The fifth sample consisted of unidirectional alumina fibres in an epoxy resin. The samples were irradiated at the Intense Pulsed Neutron Source (IPNS) (ANL) at room temperature and 5 K. Tests of mechanical properties were performed at room temperature. Maximum absorbed doses of the matrix resin were about 3.5 MGy at room temperature and 4.65 MGy at 5 K. Only small changes in the Young´s modulus, shear modulus and ultimate strength were found. But these data are not usable as magnet design data because the mechanical tests were performed at room temperature. Moreover, materials to be used as magnet insulators are expected to withstand more than 5 MGy.

4.3 Low Temperature Radiation Data for Reinforced Polymers

Low temperature radiation data are very sparse for 4 K irradiation, but there is for 77 K and 20 K a suitable data base. Most of the data are gathered in CERN laboratory, in Japan (Liquid Helium Temperature Loop at the JRR-3 Reactor at JAERI, Tokai-Mura), and in USA (ORNL, ANL, LASNL, LLNL, and others).

Table 4.3-1 shows some selected data for several materials. The data stem from Evans and Morgan /51/. The data show that upper 75 %-doses don't exist for several materials.

American and Japanese data are given in the following figures: Fig. 4.3-1 to 4.3-5 for G-10 CR, G-10 CR(BF), G-11 CR, Epikote 828 (Epon 828), and Stycast 2850 FT. These materials base on epoxy resins. These data don't agree with the data in table 4.3-1. The reason for the disagreement is not known.

G-10 CR consists of a high molecular weight epoxy of the diglycidyl ether of bisphenol-A type (DGEBA) with an aliphatic amine catalyst. The E-glass fabric reinforcement is Style 7628 plain wave with a silane coupling agent.

G-11 CR is similar in construction to G-10 CR except that it is formulated with a lower molecular weight DGEBA epoxy and is cured with an aromatic amine which contains the benzene ring.

Epon 828 (Epikote 828) and Stycast 2850 (Blue) are in organically filled epoxies.

It must be mentioned that commercial available polymers are constantly being modified by the manufacturerts and that differences in their composition and in the dose rates and environments to be employed in a fusion reactor might significantly alter their behaviour. Therefore, existing information on permanent effects should be used as a guide to selecting promising commercial materials.

Table 4.3-2 shows the chemical compositions of G-10 and polyimide fiber glass.

Table 4.3-3 shows typical compositions of glass used as reinforcement for cryogenic laminates.

Table 4.3-1: Low Temperature Radiation Data
/according to Evans and Morgan, 51/

Material	Property	T_{irr} (K)	T_{test} (K)	Initial Value (MPa)	75 % Dose (Gy)
Polyimide (Kapton)	Tensile strength	77	77	245	$>1.3 \times 10^6$
Polyimide (Kapton)	Tensile strength	20	20	325	1.3×10^6
Polyimide (Kapton)	Tensile strength	5	77	320	$>9.0 \times 10^6$
Epikote 191/DDM	Flexure strength	77	77	211	$>1.0 \times 10^7$
Epikote 828/ Polyamide	Tensile strength	5	77	510	3.0×10^6
Epikote 828/K61B	Tensile strength	5	77	390	$>3.0 \times 10^6$
MY 745/HY 905/ DY 063/EPN 1139	Flexure strength	77	295	204	$>1.0 \times 10^7$
Epikote 828/DDM/ Epikote 154	Flexure strength	77	77	167	$>7.9 \times 10^6$
NEMA G-10 CR	Flexure strength	77	4.9	862	$>1.0 \times 10^7$
Stycast 2850	Flexure strength	77	4.9	254	1.0×10^7
Epon 828/Z/Silica	Flexure strength	77	4.9	225	$>1.0 \times 10^6$

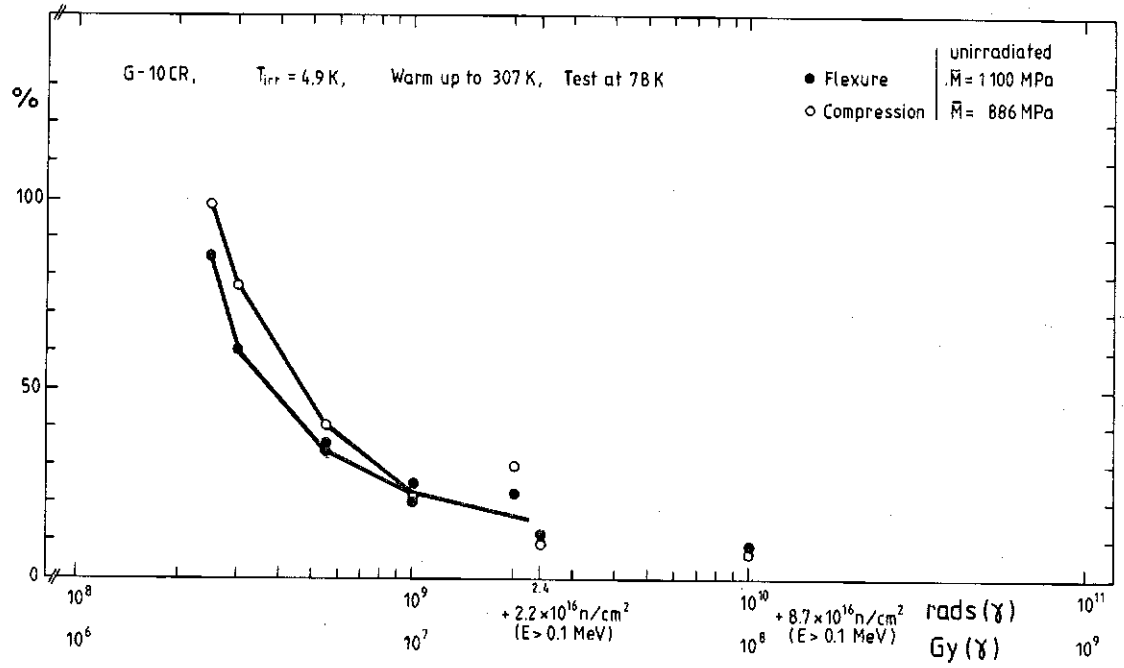

Fig. 4.3-1: Oak Ridge data for G-10CR.

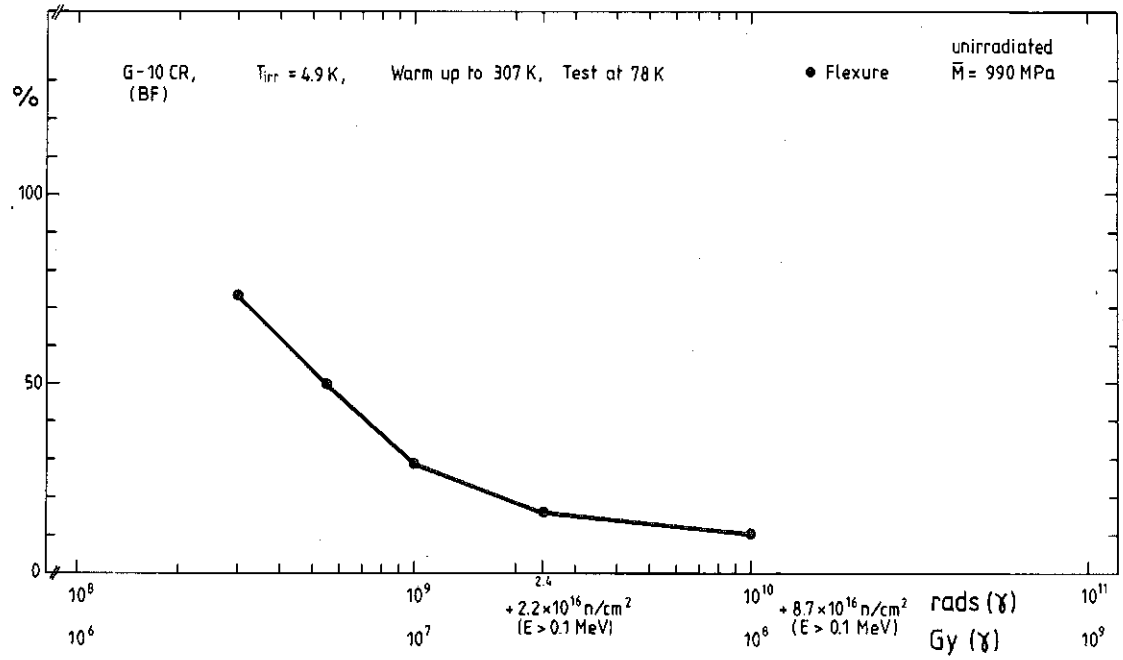

Fig. 4.3-2: Oak Ridge data for boron free (BF) G-10CR.

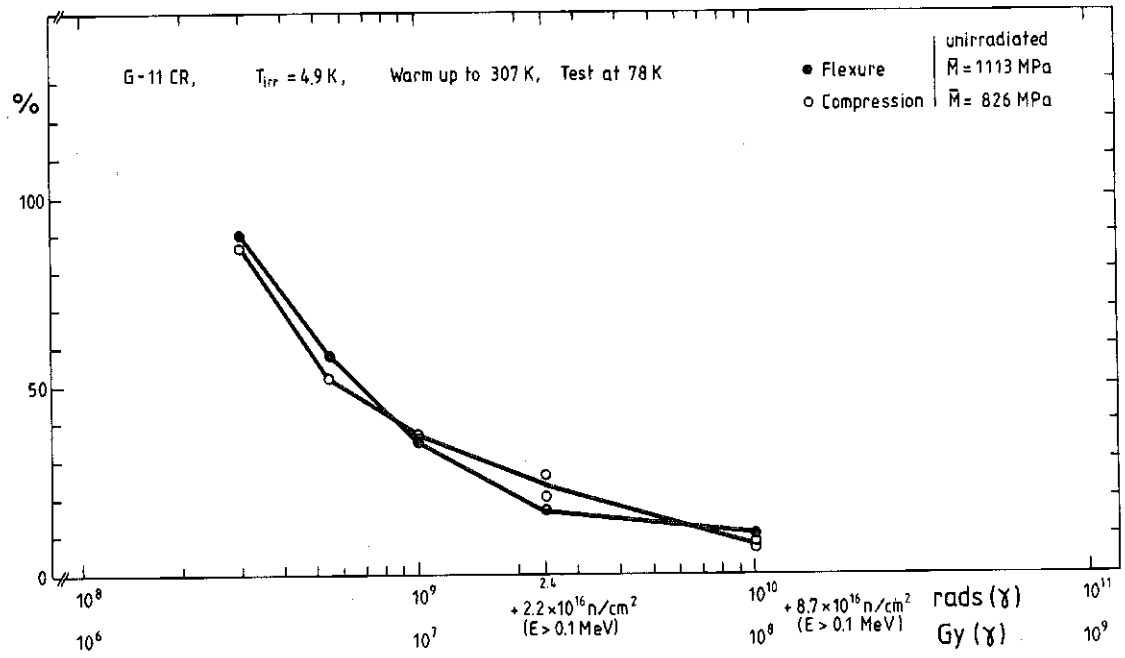

Fig. 4.3-3: Oak Ridge data for G-11CR.

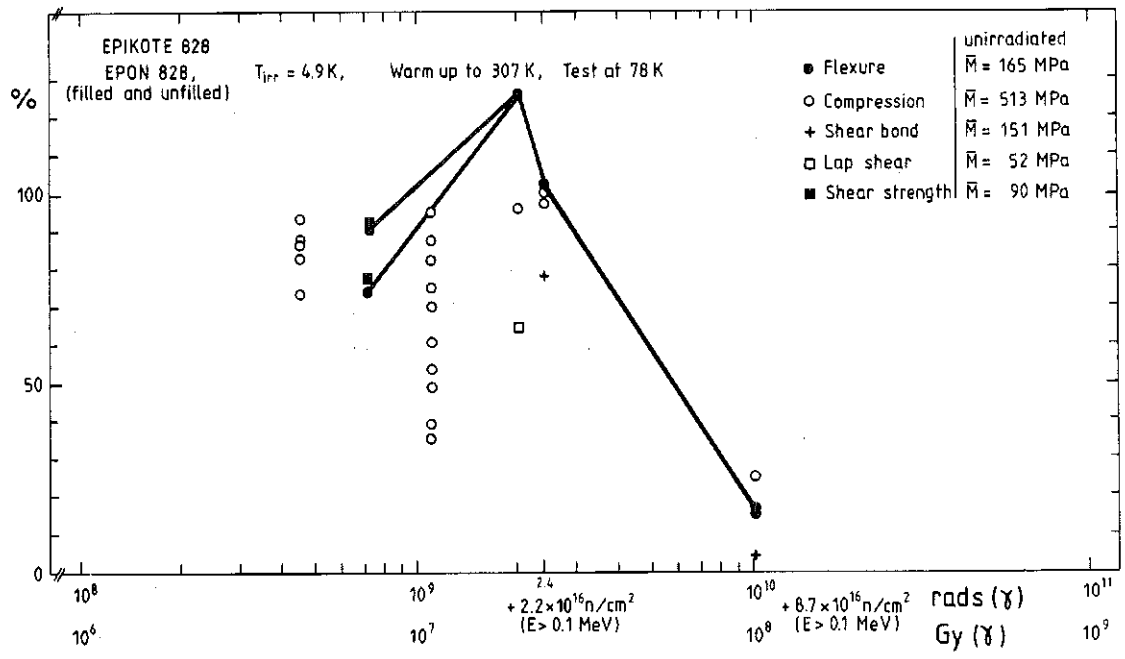

Fig. 4.3-4: Oak Ridge data for (filled and unfilled) Epikote.

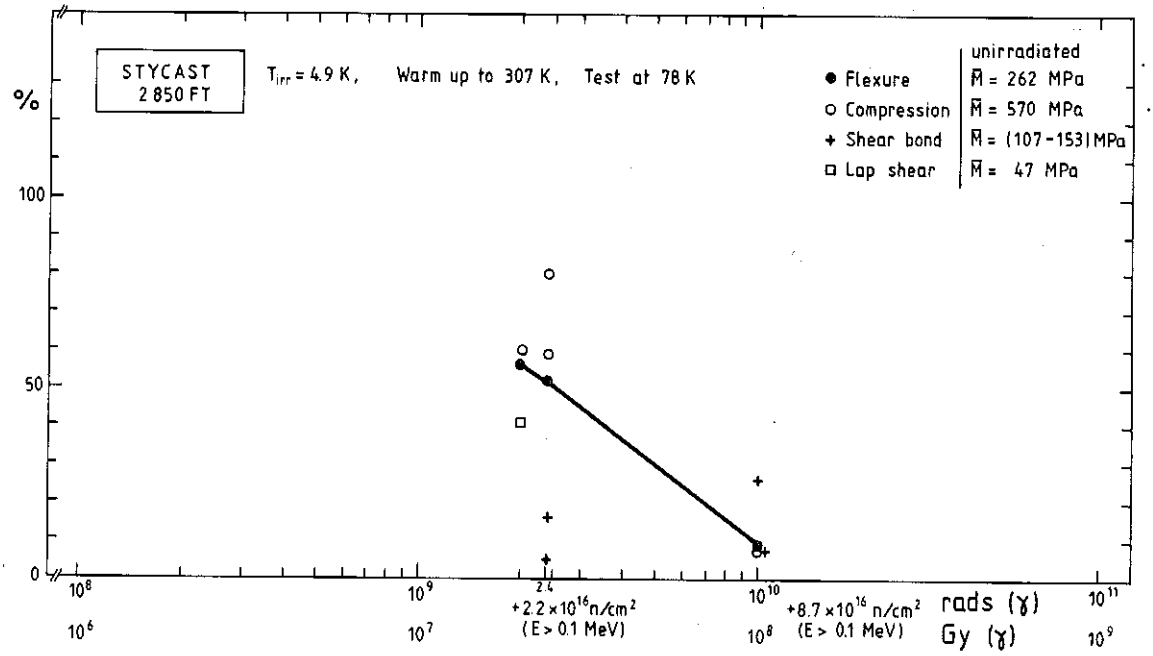

Fig. 4.3-5: Oak Ridge data for Stycast 2850FT.

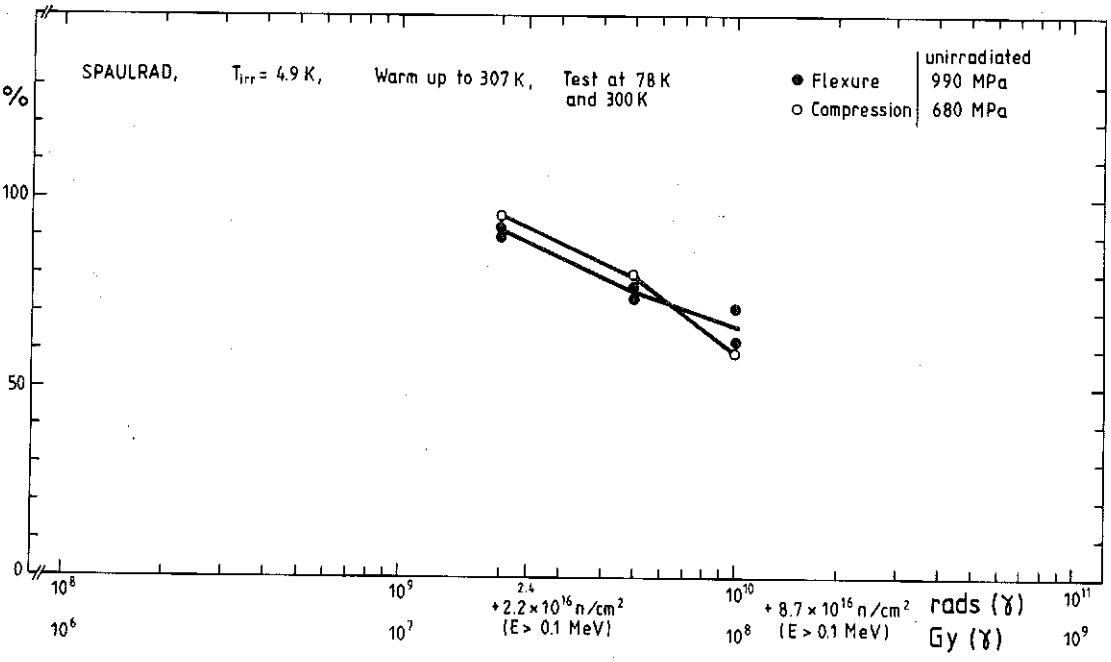

Fig. 4.3-6: Oak Ridge data for SPAULRAD (polyimide).

Table 4.3-2: Chemical Compositions of G-10 and Polyimide Fiberglass (in weight %) (BF = Boron Free) /36/

Element	G-10*	G-10 BF	Polyimide-Fiberglass (BF)
H	2.02	2.02	1.56
B	1.74	-	-
C	21.8	21.8	28.2
N	1.0	1.0	3.13
O	39.41	37.86	35.05
F	0.07	0.077	0.066
Na	0.312	0.343	0.294
Mg	1.9	2.09	1.79
Al	5.19	5.7	4.89
Si	17.7	19.4	16.7
Ca	8.76	9.6	8.23
Fe	0.098	0.108	0.09

*) Bulk densities reported in the literature for G-10 varies from 0.95 g/cm^3 (unfilled) to 1.8 g/cm^3 (filled).

Table 4.3-3: Typical Compositions of Glass used as Reinforcement for Cryogenic Laminates (weight %) /49/

	Glass Designation		
	E	S-901/S-2*	R**
SiO_2	54.4	65.0	60.0
Al_2O_3	14.4	25.0	25.0
CaO	17.5		9.0
MgO	4.5	10.0	6.0
B_2O_3	8.0		
$Na_2O + K_2O$	0.5		
Fe_2O_3	0.4		
F_2	0.3		

*) Owens/Corning Corporation., Toledo, Ohio.

**) Vetroflex Division, Saint-Gobain Industries, Neuilly-sur-Seine, France.

The Figs. 4.3-6 to 4.3-8 show irradiation data for the polyimide based materials Spaulrad, Norplex and Vespel (bulk form of Kapton). Compared with the epoxy based materials the radiation resistance is at least by a factor of 5 higher. Generally polyimide-based insulation materials are more expensive than epoxy-based materials by a factor of 3 to 4 /54/.

Additionally to the investigated materials, magnet builders have used a couple of other electrically (and thermally) insulating materials, e.g. Mylar, Kapton, Nomex etc.. Fig. 4.3-9 shows the irradiation behaviour of Kapton and Mylar. The breaking stress of Mylar is reduced substantially by irradiation at doses of about 5×10^6 Gy. Kapton, a polyimide film, holds up well and shows promise up to the highest applied doses /28/.

Table 4.3-4 shows the 75 %-dose for the materials irradiated at 4 K, warmed up to room temperature and tested at 77 K.

Fig. 4.3-10 shows a comparison between G-11 CR and Spaulrad. It is clearly shown the polyimide starts at lower flexure strength, but keeps its strength up to much higher doses than the epoxy-based G-11 CR. Therefore polyimide are better than epoxies with respect to radiation resistance.

Investigations of Weber et al. /62/ seem to confirm the above statement. Three kinds of samples used commercially for magnet insulation were irradiated at 77 K by neutrons and gammas in a reactor. Measurements were made at 77 K without warm up. The samples were:

A: 4 layers of glass woven tape, thermally desized and with special aminosilane finish, 20 x 0.14 mm^2 and magnet coil resin EP 305, BBC Brown Boveri & Cie, 0.93 mm thickness.

B: 4 layers of fiber silanized woven glass tape, 20 x 0.14 mm^2 and magnet coil resin EP 305, BBC Brown Boveri & Cie, 1.21 mm thickness.

C: A combination of the above glass tape with polyimide foil (Kapton), 20 x 0.05 mm^2, Du Pont de Nemours and Company, [glass/Kapton/glass/Kapton/glass] and same resin EP 305 as above.

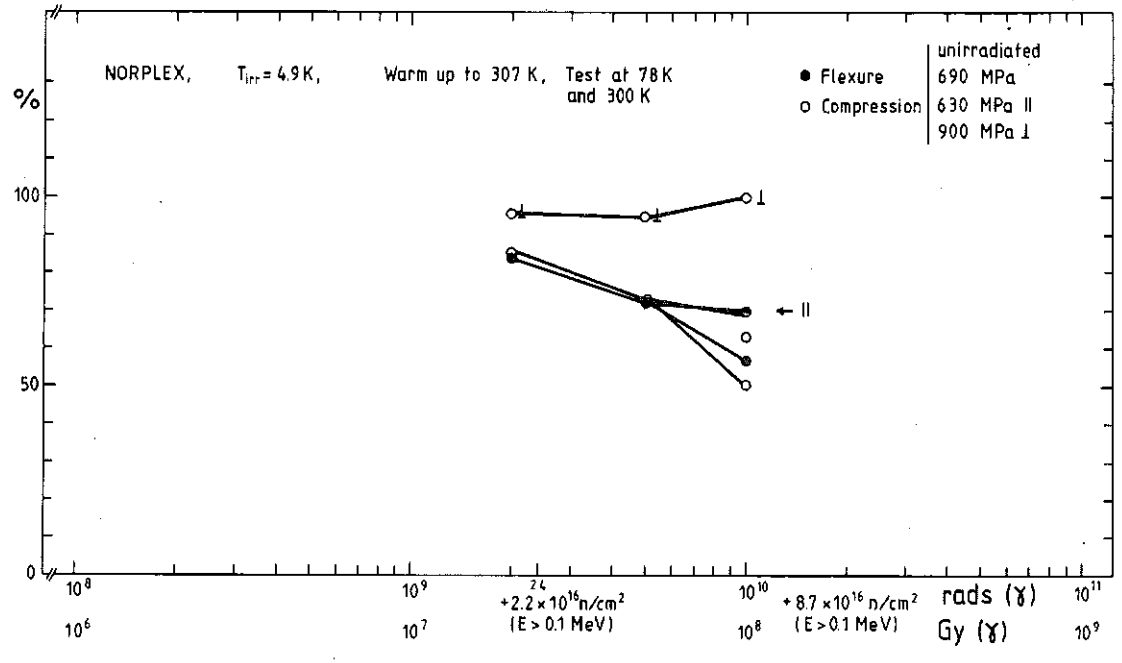

Fig. 4.3-7: Oak Ridge data for Norplex (polyimide).

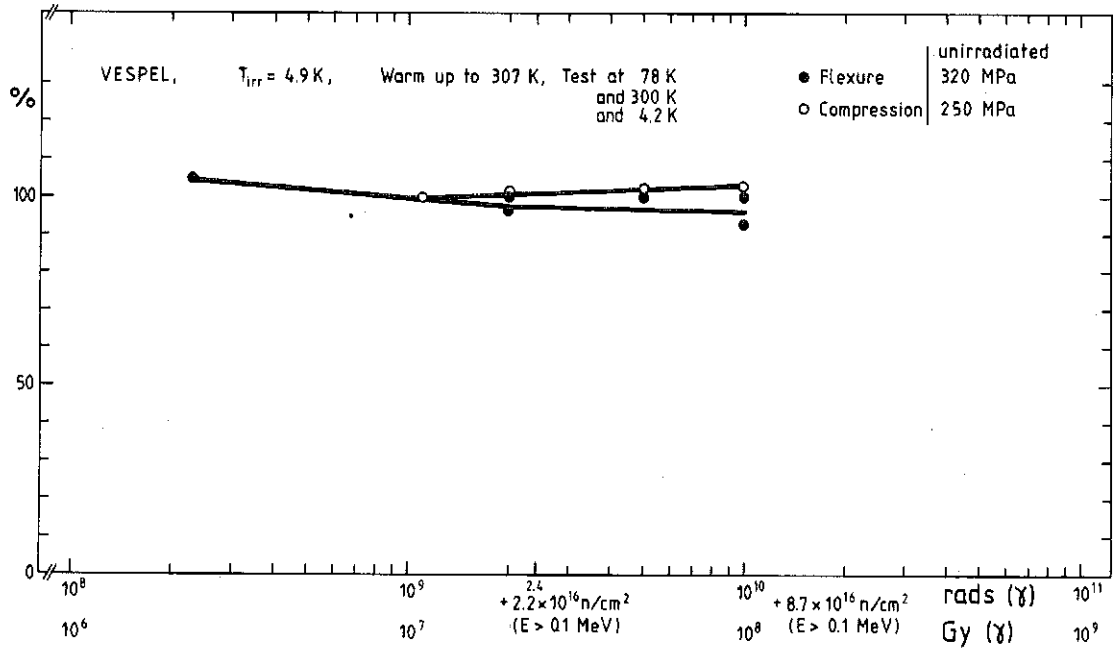

Fig. 4.3-8: Oak Ridge data for Vespel (polyimide).

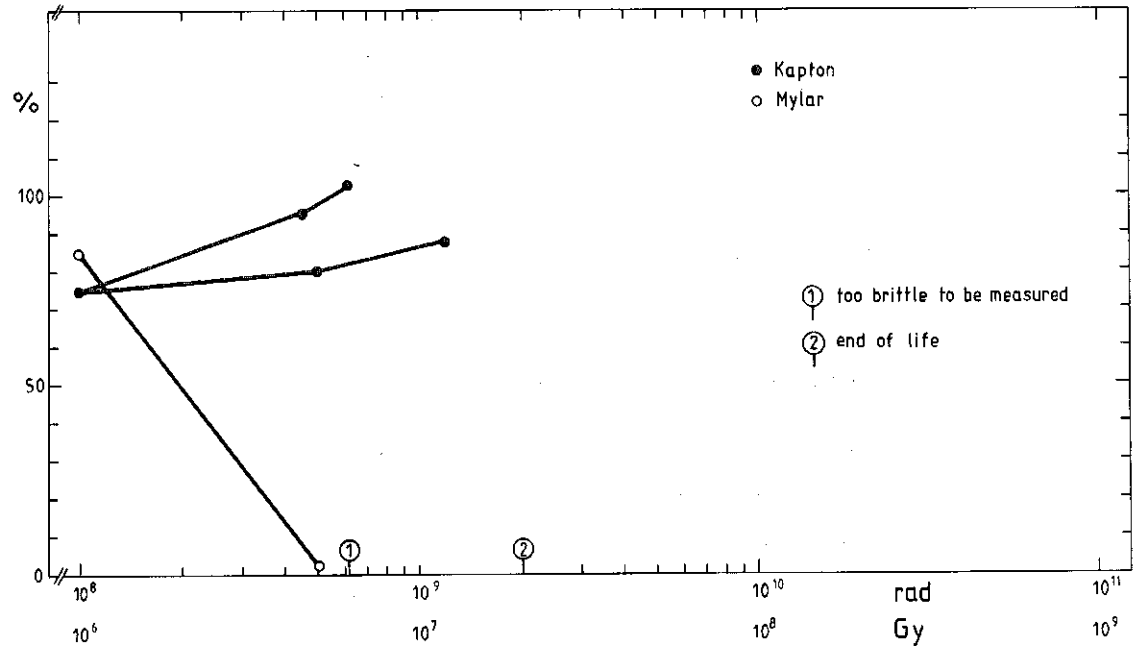

Fig. 4.3-9: Irradiation data for Mylar and Kapton.

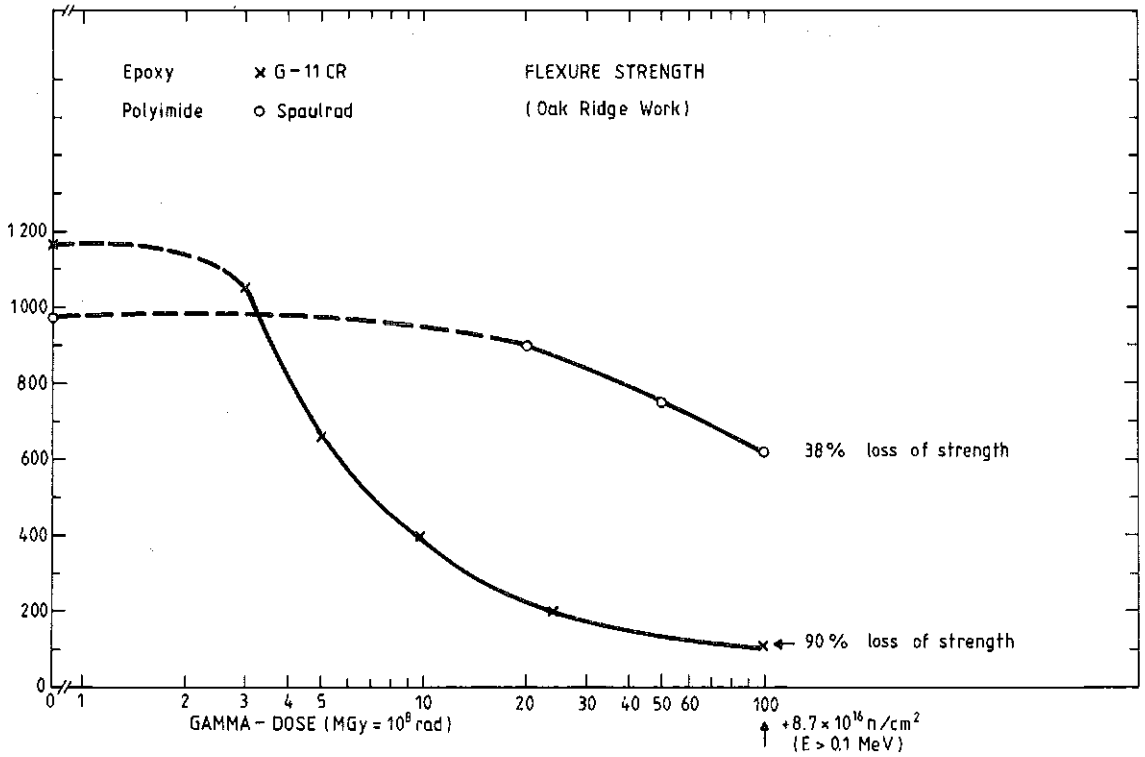

Fig. 4.3-10: Comparison of epoxy-based and polyimide based insulation materials.

Table 4.3-4: 75 %-dose for materials irradiated at 5 K

Material	75 %-dose (Gy)
G-10 CR	3×10^6
G-10 CR (BF)	3×10^6
G-11 CR	4×10^6
Epikote 828 (Epon 828)	7×10^6
Stycast 2850 FT	1×10^7
Spaulrad	5×10^7
Norplex	4×10^7
Vespel	$>10^8$
Mylar	1.2×10^6
Kapton	$>10^7$

The maximum applied dose was 1.5×10^8 Gy (2/3 gamma-, 1/3 neutron dose). The results are shown in Fig. 4.3-11. At a dose of 7.4×10^7 Gy, samples A and B lost about 30 % of their initial tensile strength, but C retained the initial value. At the dose of 1.5×10^8 Gy all composites were destroyed.

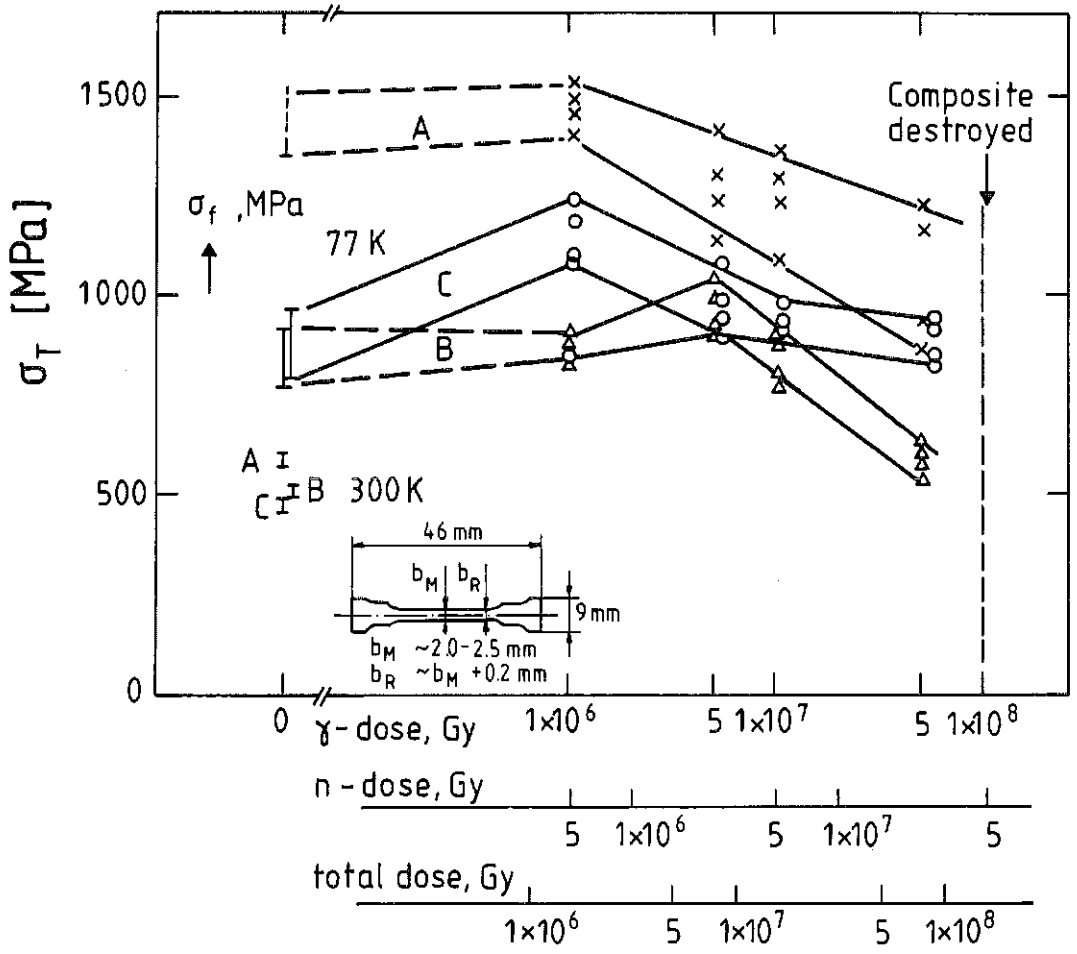

Fig. 4.3-11: Result of /62/ for various composites used commercially in magnet insulation.

4.4 Gas Evolution due to Radiation

The changes in the mechanical properties discussed in the previous subchapters 4.2 and 4.3 are accompanied by gas evolution. At room temperature these gas molecules can escape by normal diffusion processes from the specimen or agglomerate in the bulk materials. At cryogenic temperatures the escape mechanism is prevented. The generated gas is frozen out at 4 K and therefore trapped. During warm up the gas is released or agglomerates in gas bubbles in the specimen. With higher temperatures the gas pressure rises and the specimen swells and can break. In composite materials debonding occurs.

The atomic gas producing effects at low and room temperatures are being expected to differ significantly. Fig. 4.4-1 shows the evolution of hydrogen from the polymer polyethylene irradiated at various temeratures /51/. (Polyethylene is often used as power cable insulation.) The remarkable fact is seen at about 250 K where a discontinuity is seen at the glass transition temperature, the point at which movement of the polymer chain segments becomes severely restricted. This restriction of the movement of polymer chains is responsible for the differences produced by low temperature irradiation.

Reactions, which require chain segmet movements, are less likely to occur at low temperature, e.g. formation of cross-linkages and gas evolution. The mechanical properties are consequently different when tested with and without an intermediate warming up to ambient conditions.

The glass transition temperature for polymers are reported to be in the range of -150 °C to 150 °C. For highly-cross-linked polymers like the present epoxy and polyimide resins the glass transition temperature is well above 100 °C. Thus, sudden release of gas atoms during warming up from 4 K to 300 K is not expected, but a continuous release.

There are several measurements about gas evolution from different materials. Table 4.4-1 shows the gas evolution from diglycidyl ether of bisphenol A (DGEBA) epoxide resin cured with various hardeners /51/.

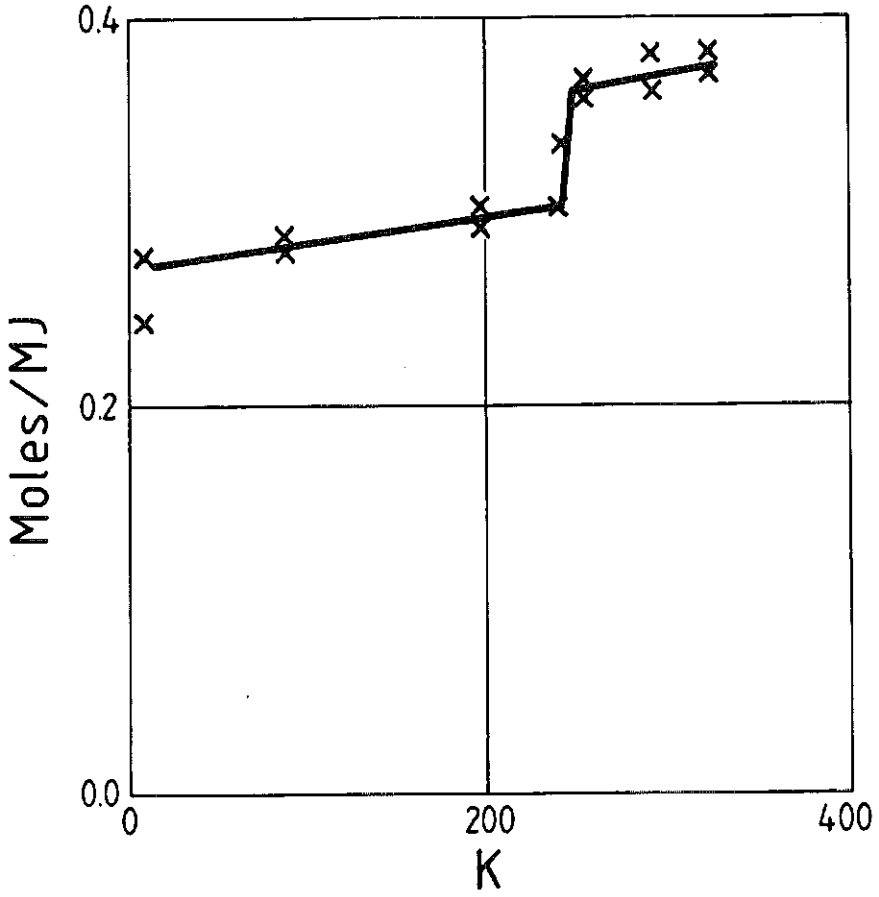

Fig. 4.4-1: Hydrogen evolution from polyethylene irradiated at various temperatures. (1 MJ = 1 MGy x kg) /51/

These data are for room temperature irradiation. Resins with aromatic amine hardeners are the most radiation resistive.

Gas evolution rates are given in /18/ for a variety of polymers irradiated at 293 K in air. Table 4.4-2 shows the values. The gas evolution for epoxy resins are by a factor of 50 to 2000 higher than those of polyimides.

The percentage weight composition of the gas mixture released from a variety of the gas mixture released from a variety of samples (Stycast 2850 FT blue, EPON 828, G-10 CR, G-10 CR (BF), G-11 CR, Nomex 410, Kapton H, Aluminized Kapton) during warm up is given in Table 4.4-3 for two different doses of 2.4 x 10^7 Gy and 10^8 Gy /27/. The gas composition changes with increasing dose, but the H_2 content remains fairly constant, while the methane, water and carbon-dioxide contents show significant differences.

Table 4.4-4 shows the gas evolved from a variety of samples (Stycast 2850 FT blue, Epon 828, G-10 CR, EF-527, Nomex 410, Kapton F, Al-coated Mylar film, and varnishes) during warm up after 5 K reactor irradiation.

Table 4.4-5 shows the gases evolved from polyimides at 307 K after irradiation at 5 K to 100 MGy /43, 38/.

The off-gas composition changes markedly with time after irradiation. The first gas sample shows a much larger proportion of hydrogen indicating its more rapid diffusion out of the material compared to the heavier species.

The gas evolution must be minimized to avoid fracturing of the insulators due to internal pressurization during magnet warm up. Magnet and refrigeration designer must consider methods to purge the helium and insulating vacuum systems of contaminants such as those quoted in tables 4.4-1 to 4.4-5.

Table 4.4-1: Gas Evolution from DGEBA Epoxide Resin Cured with Various Hardeners /51/

	Gas evolved	Composition (% volume)				
	(cm^3(NTP)/(gGy))	H_2	CO_2	N_2/CO	CH_4	C_2H_6
Aromatic amine	2.5×10^{-7}	77.1	0.8	20.9	0.9	0.3
Aliphatic amine	4.3×10^{-7}	88.4	-	10.0	1.3	0.3
Acid anhydride	7.3×10^{-7}	19.9	56.9	23.2	-	-

Table 4.4-2: Gas evolved from various polymers /18/

Polymer	cm^3 (NTP) / (g MGy)
Low density polyethylene	9
High density polyethylene	7
Polystyrene	0.06
Plasticised polyvinyl chloride	2.7
Unplasticised polyvinyl chloride	1.8
Polymethylmethacrylate	2.7
Polytetrafluoroethylene	0.22
Nylon 6	2.4
Polycarbonate	2
Polyethylene terephthalate	0.4
Epoxy resins	from 0.29 to 13
Polyimide	0.006

Table 4.4-3: Analysis of evolved gases (wt%) from all samples released during warm up after 5 K reactor irradiation /27/

Gas	Dose(Gy)			
	2.4×10^7		10^8	
Hydrogen	70.4		71.3	
Methane	2.1		1.2	
Water	5.8		0.6	
Nitrogen + Carbon Monoxide	17.1		20.5	
Carbon Dioxide	2.6		2.0	
Acetylene	2.0		4.0	1.5
Ethylene				1.3
Ethane				0.5
Propylene				0.4
Butane				0.3
Benzene				0.01

Table 4.4-4: Off-gas analyses (for all samples together)

Gas	Content (%) after each dose (MGy)		
	6	20	20 (35 days later)
H_2	58.9	93.7	35.8
CH_4	1.7	0.3	29.9
H_2O	21.7	3. 2	3.1
N_2 + CO	9.0	1.4	18.2
O_2	0.2	< 0.1	0.1
Ar	0.1	< 0.1	0.05
CO_2	6.9	0.7	8.9
Fluor carbons	0.2	~ 0.5	2.0
Organics*	1.4	~ 0.2	1.8

*) Heavier than methane up to molecular weight about 100.

Table 4.4-5: Gases evolved from polyimides /43/

Contaminants	Interval after irradiation			
10^{-4} g gas/g resin	(0 - 1) day	(weight %)	(1 - 6) days	(weight %)
H_2	14.3	(44.3 %)	0.2	(0.6 %)
CH_4	0.3	(0.9 %)	0.5	(1.4 %)
H_2O	0.6	(1.8 %)	7.4	(21.0 %)
N_2 and CO	16.0	(49.5 %)	23.4	(66.5 %)
O_2	0.02	(0.06 %)	0.1	(0.3 %)
CO_2	0.9	(0.28 %)	3.2	(9.1 %)
C_2H_4	0.2	(0.6 %)	0.4	(1.2 %)
Total	32.3		35.2	

4.5 Other Effects due to Radiation

Some other effects occur due to irradiation. They are weight changes, dimensional changes, color appearance and radioactive activation.

Weight loss occurs apparently as the result of the formation of gas atoms and molecules during irradiation. These are frozen out at low temperatures and freed during and after warm up they diffuse out through the surface of the specimen. Weight losses for Stycast 2850 FT, Epon 828, G-10 CR, G-10 CR (BF), and G-11 CR are reported /64/ to be less than 2 % for 10^8 Gy.

Dimensional changes for Epon 828, G-10 CR, G-10 CR (BF), and G-11 CR are less than 0.2 % at 10^8 Gy, but Stycast 2850 FT shows at the same dose up to 25 % dimensional changes /64/.

During irradiation the color changes. The general behaviour is: the higher the dose is, the darker the color is.

In the ATR-INEL (Advanced Test Reactor - Idaho National Engineering Laboratory) irradiated G-10 and G-11 CR specimens were found to be highly radioactive after months of radioactive cooldown. Tests were therefore conducted in a hot cell /36/.

Activation studies at MIT with 1.4×10^{18} n/cm^2 plus 5×10^9 rad by gamma rays showed that S-glass composites were less active than E-glass composites. The activation was measured after 258, 330, and 450 hours after irradiation /36/.

Activation studies at ORNL /38/ on specimens irradiated by 100 MGy (10^{10} rads) (gamma-ray dose) gave 48 mrad/h after one and 19 mrad/h after four weeks. It was found that almost all of the radioactivity originates in the glass fibres. Moreover glass-filled polyimides give almost the same results as G-10 CR (glass-filled epoxy). Both had nearly the same glass content.

4.6 Anneal

The warming up of organic insulators, irradiated at low temperature, to room temperature brings not much recovery as in the case of stabilization materials for superconducting magnets. On the contrary, it may even cause more damage, because the gases produced by irradiation and frozen out at low temperatures will be freed and escape from the insulators as discussed in chapter 4.4. There may be a building up of a considerable pressure in the magnet. Another effect could be the (partial) blocking of the coolant system.

Recent experiments /77/ investigated the annealing behaviour of specimens already described at the end of chapter 4.2. The samples were irradiated by ^{60}Co gamma-rays in air at ambient temperature up to 20 MGy. The irradiated and control specimens were put into a quartz capsule, and then annealed in vacuum at 180 °C for 2 h. The ultimate strength of the control specimens are practically the same before and after the annealing for all the composites (glass/-epoxy, glass/polyimide, carbon/epoxy, carbon/polyimide, alumina/epoxy). The ultimate strength of four irradiated samples (except the glass/polyimide sample) were decreased at a dose of 20 MGy by about 30 to 50 %. Only the glass/-polyimide sample was unchanged. The authors point out that the decrease in the ultimate strength after annealing is definitely attributable to latent radiation damage which can be activated by the annealing.

The Young's modulus of all composites remained practically unchanged up to 20 MGy after irradiation and annealing.

The shear modulus showed also a clear dose dependence after annealing for all the composites except the glass/polyimide sample, while without annealing after irradiation the shear modulus was not considerably changed. The dose dependence after annealing was quite similar to that described above for the ultimate strength. That indicates that the ultimate strength and the shear modulus are correlated with each other.

The annealing effects (at 180 °C) described here cannot be directly applied on low temperature behaviour. Generally, annealing of irradiated composites is considered to activate latent radiation damage near the fiber/matrix interface, thus causing a decrease in the load transfer capacity at the interface. Consequently, the shear modulus and the ultimate strength decrease.

5. Fatigue Effects on Polymers due to Irradiation

Fatigue effects are important for pulsed operation, e.g. in a Tokamak. In /36, Chapter 18/, compression fatigue test result are reported for a variety of test samples. All specimens were irradiated in the MIT reactor up to 2.3 x 10^{10} (γ)-rads and 1.06 x 10^{19} n/cm^2 (E > 1 MeV) or 2.16 x 10^{19} (total) n/cm^2. The thickness of the samples for the compression tests were 0.5 mm and the diameter was 12.7 mm. All specimens survived 30,000 cycles with a maximum applied stress of 310 MPa. At 77 K, G-10 samples survived also 30,000 cycles with a maximum applied stress of 345 MPa. The irradiation dose for this sample was 3.8 x 10^{11} rads (γ) and 1.6 x 10^{19} n/cm^2 (E > 0.1 MeV) or 10^{20} n/cm^2 (total).

Compressive fatigue tests on G-7, G-11 (single tests) and G-10 were performed for room and liquid nitrogen temperatures /42/. The materials are insulation materials investigated for the use in TFTR. Maximum doses are 5.2 x 10^5 Gy (γ) and 8.6 x 10^{16} n/cm^2 (E > 0.1 MeV). The result for G-10 is seen in Fig. 5-1.

The first fatigue data at liquid helium temperature after low temperature neutron irradiation is presented in /44/ for commercially available epoxy resins used in superconducting magnets. The irradiation was performed at KUR (Kyoto University Reactor) at 4.2 K; during irradiation the sample temperature was 27 K maximum. The doses are 2.8 x 10^6 Gy (γ) and 2.5 x 10^{16} n/cm^2 (E > 0.1 MeV) and 2.0 x 10^{17} n/cm^2 (thermal). The results are shown in Fig. 5-2. The load curves can be approximated by

$$S\ (kg) = 18.4 - 1.6 \log N \qquad \text{before irradiation}$$

and

$$S\ (kg) = 7.39 - 0.16 \log N \qquad \text{after irradiation.}$$

Tests of G-10 without irradiation /54, Chapt. 19/ were reported. For 10^6 to 10^7 cycles at least 20 % of the initial strength is retained.

The effect of cryogenic temperature and radiation on the fatigue resistance of G-11 CR glass-epoxy laminate is studied in /63/. Test temperatures are 295 K and 77 K for irradiated and unirradiated samples. The irradiation dose is relatively low, only 1 MGy (γ) from the 25,000 Curie ^{60}Co gamma facility of

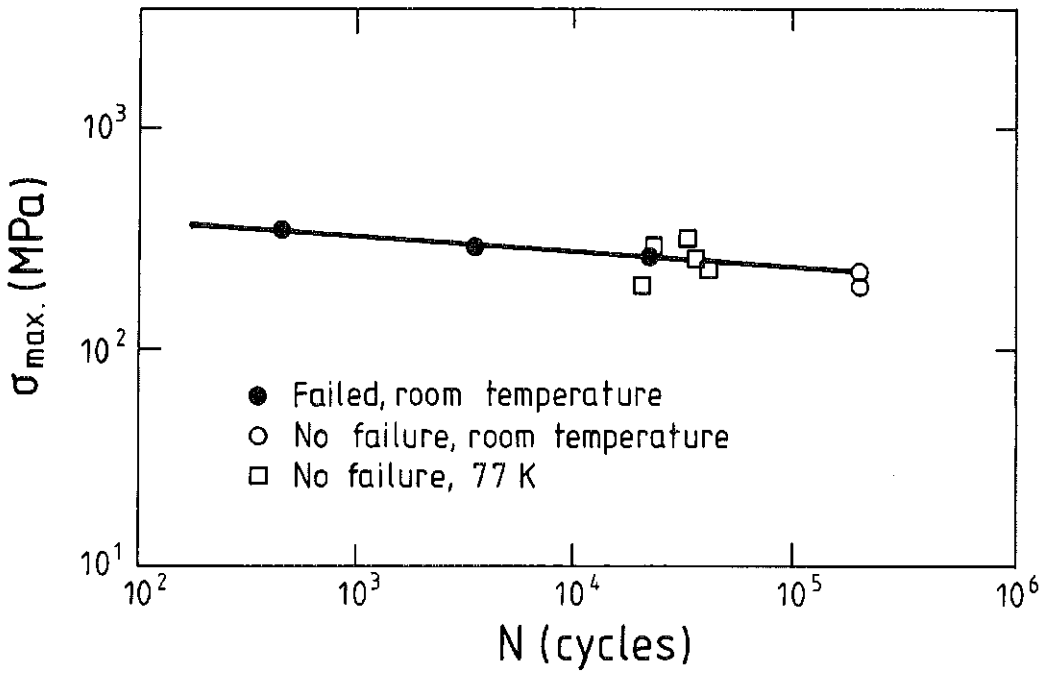

Fig. 5-1: Plot of compressive fatigue data for insulator G-10 plotted as maximum stress vs number of cycles in a test. [42]

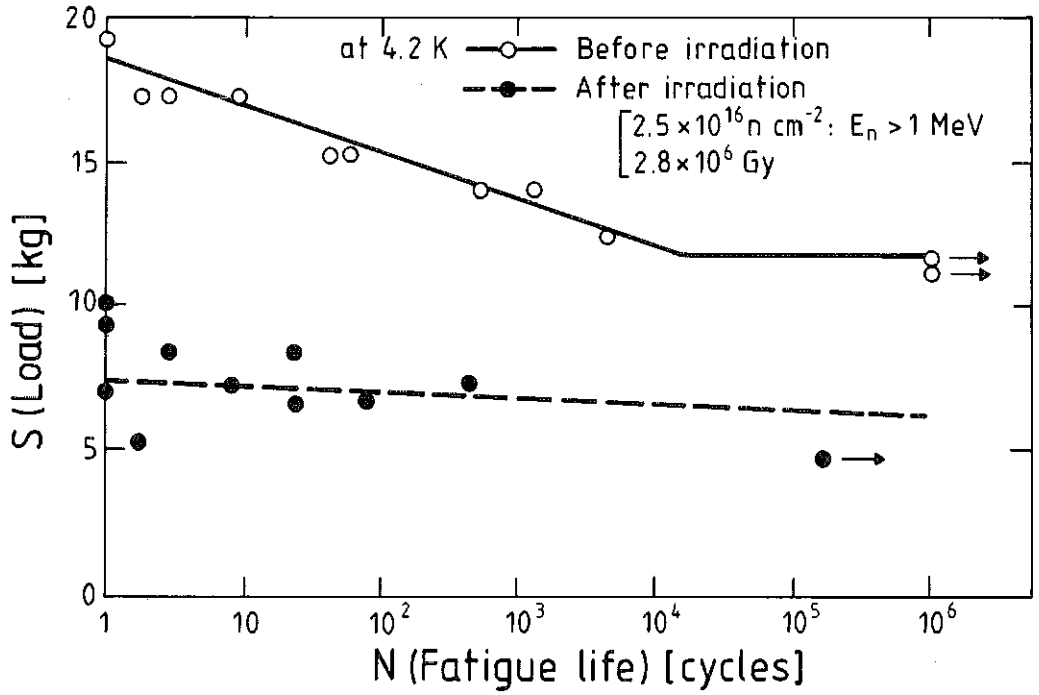

Fig. 5-2: Load-endurance diagrams before and after irradiation for an epoxy resin used in superconducting magnets.[44]

the Pennsylvania State University. The ultimate tensile strengths are σ_u = 463 MPa at 295 K and σ_u = 932 MPa at 77 K. The tests were performed with the maximum cyclic stress set at 90, 75, 60, 50 % of the measured tensile strength σ_u. The test results are shown in Fig. 5-3 and indicate a radiation effect at both test temperatures, an increase in fatigue resistance at cyogenic temperatures and substantially different failure modes at 295 K and 77 K.

At 295 K initial failure occurred as initiation of a through crack at the notch root. In this case, failure was defined as the number of cycles required to propagate the through crack 0.127 mm (0.005 in.) from the notch root. At 77 K, initial failure occurred as delamination early in the fatigue life. Delamination was followed by generation of surface cracks at points along the notch root and subsequent propagation of a dominant surface crack normal to the warp fibers. In this case, failure was defined as propagation of the dominant surface crack to a distance of 0.254 mm (0.01 in.) from the notch root.

The radiation effect for both temperatures and for a dose of 1 MGy decreases in terms of fatigue resistance above 3,000 cycles and increases below 3,000 cycles.

Recent irradiation data for epoxy and polyimide resins reinforced with E-glass and S-glass woven fabric reinforcements and irradiated at 325 K in the ATR (Advanced Test Reactor) in Idaho Falls up to doses of about 3×10^9 Gy (γ) and about 4×10^{19} n/cm^2 (E > 0.1 MeV) and a total neutron fluence of about 3.8×10^{20} n/cm^2 /72/. The total dose to the organic component of the laminates was about 4.4×10^9 Gy. Tests were performed with an applied maximum compression stress of 345 MPa up to 250,000 cycles. S-glass reinforced resins survived, but E-glass reinforced resins (G-10, G-11 CR) showed rapid failure after only a few hundred cycles. The reason is that the total dose to the materials containing E-glass is considerably higher due to the thermal neutron fission in ^{10}B (present as B_2O_3 in E-glass, 8 %, see Table 4.3-3).

Each fission event via $^{10}B\ (n,\alpha)\ ^7Li$ releases 2.8 MeV as kinetic energy to the reaction products resulting in an added dose to the E-glass of about 10^{10} Gy. These results recommend the use of S-glass, but S-glass is about six times more expensive than E-glass.

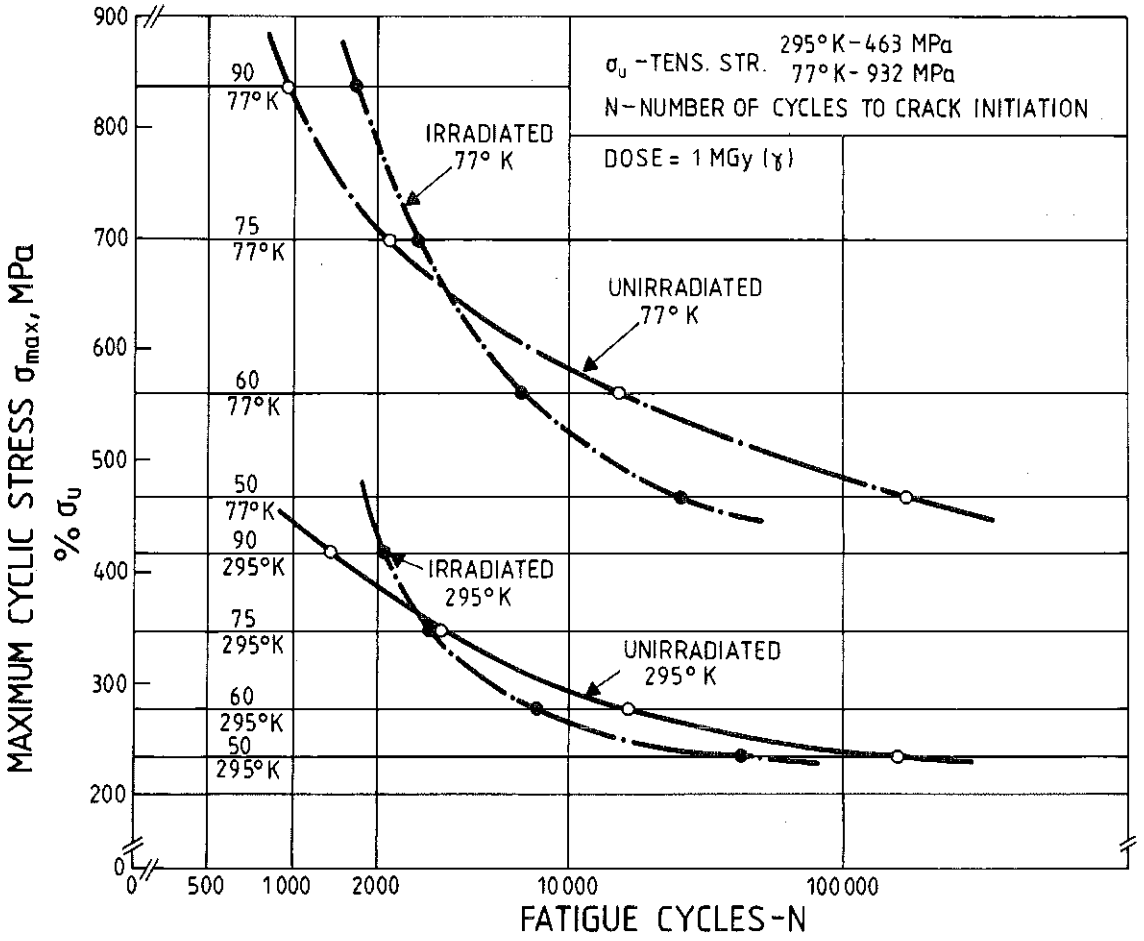

Fig. 5-3: Tensile Fatigue S-N Curves for G-11 CR.

6. Radiation Influence on Electrical Properties of Polymers

Radiation produces both transient and permanent changes of the electrical properties of polymers.

The transient effects are very sensitively affected by dose rate and are due to the ionisation of polymer molecules and the excitation of the freed electrons into conduction states. When irradiation is stopped, the number of conduction electrons decay as they are trapped and recombine with the ionised molecules.

The permanent effects are associated with permanet structural changes and begin to occur at similar total doses to whose which produce permanet changes in mechanical properties.

The transient changes of electrical conductivity are relatively small and do not have a significant effect on the insulating properties of polymers except at very high dose rates. It is not considered to be a problem for the operation of the magnets.

The permanent effects too are rather small until the polymer has absorbed a sufficiently high dose to degrade its mechanical properties.

In general the break down of electrical insulation at high doses is due primarily to the loss of mechanical properties, cracking, and gas evolution, rather than to intrinsic increases in conductivity of the irradiated material.

It is usually the case that failure of the electrical integrity occurs through degradation of the mechanical properties rather than the intrinsic electrical properties and that data about the former can be used to judge the probable suitability of a material.

Permanent changes in dielectric loss, dissipation factor and insulation from exposure to radiation have been recorded, but these are usually quite small. Measurements of electrical resistivity (volume and surface) and voltage breakdown strength are usually performed at room temperature. The most information can be found in /6/. In /36/ is stated that surface flashover, or tracking, is more likely to occur than is bulk breakdown (Tracking is running of an arc along one side of a surface).

Table 6-1 shows electrical measurements at room temperatur after irradiation at 5 K /29/. It is easily seen that the electrical resistivity of Stycast 2850 FT Nomex 410 (paper) and Kapton H is unaffected up to a dose of 10^8 Gy. The decrease of electrical breakdown strength begins in the range between 0.24 Gy and 1.0 Gy for Stycast, G-10 CR and G-11 CR.

Table 6-2 shows results of in-situ resistivity, postirradiation resistivity, breakdown strength, and dissipation factor measurements. The remarkable result is that the in-situ resistivity is about an order of magnitude lower than the postirradiation resistivity due to effects discussed before.

Table 6-3 shows recent results of electrical resistivity measurements for a very high dose irradiation at 325 K in the ATR reactor. Drastic changes were observed (up to nine orders of magnitude decrease in the electrical resistivity).

As a general conclusion it can be stated that electrical properties are generally degraded less than are mechanical properties, at a given dose.

Table 6-1: Electrical Measurements at Room Temperature after Irradiation at 5.0 K /29/.
(Each result is the average of three measurements.)

Dose 10^8 Gy	Stycast 2850 FT	EPON 828	G-10 CR	G-11 CR	Nomex 410	Kapton H
	Resistivity [1]			10^{15} Ωcm		
Control	0.24	20	8.2	4.4	3.3	20
0.24	0.25	5.5	8.5	2.8	2.2	29
1.0	0.27	8.1	0.64	0.14	2.0	22
	Electric Breakdown [2]			(kV/mm)		
Control	28^2	33°	23°	24°	36^2	66°
0.24	31°	34°	23°	23°	38^2	66°
1.0	8^3	31^1	8^3	10^3	36^2	68°

1) Electrical contact was established by use of a conducting silver-filled epoxy paint. To minimize handling difficulties from radioactivity, the paint was applied after irradiation. An EMF of 100 volts was used for the resistance measurements.

2) The digital superscripts indicate the number of specimens in a given group which showed a true voltage breakdown through the material in contrast to others for which a flashover around the edge of the specimen was observed. When all specimens in a given group exhibit flashover (superscript "0"), the true breakdown value may be significantly higher than the indicated minimum value shown.

Table 6-2: Results of Electrical Measurements /31/

Material	In-situ Resistivity (10^{15} cm)[1]		Postirradiation Resistivity (10^{15} cm)[2]		Breakdown (kV/mm)[3]		Dissipation Factor (tan δ) at 1 kHz[3]	
	C[4]	I[5]	C	I	C	I	C	I
Stycast 2850	1.0	0.35	14.0	5.0	32[6]	32[6]	0.024	0.023
Epon 828	1.5	0.75	10.0	6.0	24[6]	23[6]	0.026	0.028
G-10 CR	0.7	0.5	7.0	7.0	22[6]	22[6]	0.022	0.024
EF 527			12.0	0.005	31	11	0.025	
Nomex	3.5	1.5	13.0	14.0	38	39	0.008	0.008
Kapton F			15.0	19.0	70[6]	57[6]	0.008	0.008

1) Performed in Helium-environment from 5 - 300 K.

2) Dry nitrogen environment at 300 K.

3) Air at 300 K.

4) Control specimen.

5) Irradiated specimen, 2×10^{7} Gy dose.

6) Surface flashover, not breakdown.

Table 6-3: Results of Electrical Resistivity Measurements /72/

Material	Electrical Resistivity	
	Unirradiated (Ω cm)	Irradiated [1] (Ω cm)
G-10	1.1×10^{16}	3.8×10^{7}
G-11 CR	4.1×10^{15}	5.6×10^{9}
KERIMID-601	1.4×10^{15}	7.9×10^{11}
DGEBA[2]	8.9×10^{15}	1.6×10^{12}
TGPAP[3]	2.2×10^{15}	6.6×10^{11}

1) In the ATR (Advanced Test Reactor) in Idaho Falls at 325 K up to doses of about 3×10^{9} Gy (γ) and 4×10^{19} n/cm^2 (E > 0.1 MeV) and a total neutron fluence of about 3.8×10^{20} n/cm^2.

2) Diglycidyl ether of Bisphenol A.

3) Triglycidyl p-amino phenol.

7. Thermal Properties

Very few measurements exist for thermal properties after irradiation. In /17/ and /51/ the influence on the specific heat and the thermal conductivity is shortly discussed.

Chemical reactions due to irradiation can also be used to explain anomalies in specific heat measurements during warming up. The radiation effect on the thermal conductivity is small. Local heating may occur by energy deposition during the irradiation process; this is especially important for low thermal conductivity materials, because additional cooling power is required and differential thermal expansions can cause problems in very brittle materials at low temperatures.

8. General Conclusions and Recommendations

Some general conclusions can be drawn from the radiation data:

- Selections of insulator materials should be based on the effects of radiation on mechanical properties rather than electrical properties, because electrical properties are generally degraded less than are structural properties, at a given fluence.

- Commercially available polymers are constantly being modified by the manufacturers, and differences in their composition and in the dose rates and environments to be employed in a fusion reactor might significantly alter their behaviour. Existing information on the radiation effects should therefore be used as a guide to selecting promising commercial materials whose performance must then be evaluated in a radiation test or in a simulation of the proposed conditions if possible.

- The radiation response of organic insulation materials (and reinforcing glasses) depends on the mixture of neutron and gamma-ray intensities (and energies). These differ for various reactor designs.

- It is not evident that the damage production mechanism is the same for fast neutrons and gamma-rays, but the damage produced in organic materials appears to be proportional to the total energy absorbed.

- Typically 50 - 80 % of the deposited energy in the insulator material results from neutrons, the balance from gamma-rays. The deposited energy increases with increasing hydrogen content.

- The potential contribution of the thermal neutrons must be considered, in view of the neutron absorption not only in the organic materials but in all magnet components. Especially transmutation products are of interest. For example, insulators containing boron (in the E-glass) may not be suitable for use in fusion magnets because of the large thermal neutron absorption cross section leading to the nuclear reaction $^{10}B(n,\alpha)^{7}Li$.

- Irradiation (by neutron as well as by gamma-rays) induces crosslinking of polymeric chains and a depolymerization (chain fracturing) which results in

embrittlement. Aromatic hardeners (containing the benzene ring) generally have higher radiation resistance than aliphatic hardeners (which are more or less straight carbon chains).

- Consideration of individual effects in a material does not necessarily mean that the performance of a component subjected to irradiation at low temperatures can be reliably predicted. Changes may be caused or even enhanced by a combination of effects due to e.g. stress and magnetic field.

- A problem may be the formation of gas molecules at low temperatures and the release during warm up. This can cause structural and cooling problems.

- In fiber reinforced materials, the fiber-resin bond appears to be the radiation sensitive part, and the failure of the bond between fiber and resin seems to be the major mechanical breaking mode after irradiation.

- Candidate insulator materials based on epoxy resins typically begin to show serious reduction in mechanical properties for doses between 10^6 Gy and 10^7 Gy, while those based on polyimides show about an order of magnitude improvement. However, polyimides have inferior initial strength properties compared with epoxies. Therefore, the development of stronger polyimides laminates is desirable. (As a rule of thumb, 10^7 Gy corresponds to a fast neutron fluence of 10^{18} n/cm^2). It should be mentioned that vacuum impregnation with epoxy is easier than with polyimide resin.

- It appears that the use of aluminized Mylar as (thermal) superinsulation will be impossible, but aluminized Kapton may be a suitable superinsulation for the cryogenic systems of fusion magnets.

- Structural degradation of organic materials occurs at lower neutron doses than for other magnet materials, e.g. superconductors and stabilizer. The effect of radiation for these materials is better known than for the insulators.

- The graded use of insulating materials should be considered. Polyimide insulation should be used in interior magnet regions (plasma facing) where radiation doses are higher and stress levels are lower. Epoxy insulation should be used in outer magnet regions where radiation doses are lower and stress levels are higher.

- In this report the 75 %-dose (that means 25 % loss of strength) is used for comparison of materials. This dose seems to be acceptable for magnet designer.

- It must be noted, that e.g. the use of neutral beam injectors in fusion reactors and/or possibly some vacuum piping causes paths for intense radiation streaming. These "radiation hot spots" may seriously affect the local magnet performance and can drastically change the materials properties in certain winding regions.

- The low temperature irradiation data base for candidate magnet insulation materials is not sufficient. Detailed radiation performance data for magnet design are needed for epoxy- and polyimide-based insulation materials. The research effort on low temperature radiation effects in organic insulators should be expanded. A testing and development program should be installed. Suggestions are given in the appendix.

A. Appendix: Testing and Development

There is an inadequate data base for organic insulators for fusion magnets. The materials used in magnets are not standardized and the understanding of failure modes is incomplete.

Standard test procedures and adequate characterization of insulator properties are required.

The curves of mechanical properties vs dose or temperature or cycle number are usually bands, especially fatigue curves. The values are influenced by composition, age, and moisture of the polymer. There is also a lack of the reproducibility of the data.

Irradiation experiments at 4.2 K are difficult and expensive. Therefore experimental irradiation programs for 4.2 K irradiation of magnet component materials are very sparse. One of the programs is running at the Oak Ridge National Laboratory (ORNL) using the Low Temperature Irradiation Facility (LTIF). Another is running in Japan.

A.1 Development Goal

The development goal for the insulation should be specified as organic materials which can withstand a dose of about 10^9 Gy $\approx 10^{20}$ n/cm^2 (E > 0.1 MeV).

If organic insulators are found to be inadequate, inorganic (ceramic) materials should be investigated. The development of optimized materials with respect to radiation resistance and high initial strength is required. Methods of production control for consistent materials performance should be included.

The goal of the experimental investigations should be to interpret the experimental results in terms of the constitutive properties, i.e. to try to understand the effect at a fundamental level. That would lead to a better extrapolation of results and better predictability of the behaviour of the insulation. If the damage mechanism is understood, then it can be possibly predicted how new materials may be developed better to withstand the fusion environment.

A.2 Standardization of Materials

The molecular formula of a polymer is usually a secret of the manufacturer. Therefore standard sample specifications and compositions must be defined in order to perform various tests with and without irradiation on identical and well-characterized materials. Such a standardization program has been started in the U.S.A. by the National Bureau of Standards together with researchers and insulator manufacturers. A similar program should be initiated in Europe.

A.3 Identification of Failure Modes

The identification of failure modes due to irradiation is important for the development or improvement of fiber reinforced insulators. The question what the weakest point is, is very important for the design of the magnet insulation system. Is it the matrix, or the reinforcing fiber or the bond between both? Available experimental results suggest that radiation-induced debonding occurs between the fibers and the resin, which is responsible for the loss of strength /61, 65/. The inverse rule of mixture for the shear modulus G in a composite (subscript c) is given by

$$1/G_c = V_f/G_f + V_m/G_m$$

(V = volume, f = fiber, m = matrix) shows that the shear modulus is determined by the weakest part in the composite /76, 77/, i.e. the fiber/matrix interface. Separation or debonding between fiber and matrix is optically observed in irradiated composites even before the mechanical tests.

It is not quite clear, whether the debonding of the fiber/matrix interface is due to the migration and agglomeration of gas atoms at the interface or due to the higher dose due to the $^{10}B(n,\alpha)^{7}Li$ reaction in E-glass fiber reinforced materials.

The development of boron-free glass fiber reinforced composites should be envisaged. The use of R- or S-glass (both are boron-free) is strongly recommended. An advantage is the 50 % higher tensile strength, but the price is about 6 times higher than that of E-glass.

A.4 Role of Irradiation Temperature and Testing Temperature

Many of the authors point out that room temperature data should not be used as design data for fusion magnets, because composites irradiated at 4 K decreases much more significantly when tested at 77 K than at room temperature /e.g. 74, 64/. Results of Takamura and Kato /37, 45, 66/ suggest that results of testing at 4.2 K may be different from those at 77 K, and that intervening room temperature warming up may also affect the results, because latent radiation damage can be activated by warm up.

It appears that the sensitivity to radiation damage is greater at lower temperature /38/. May be, different damage modes may operate at low temperature irradiation or during postirradiation warming up compared with room temperature irradiation /31/. Materials that are more resistant to irradiation at room temperature are generally found to be more resistant to irradiation at low temperatures /39/. Another conclusion seems to be the more heat resistant a material is the more radiation it can withstand.

The optional irradiation test procedure might be:

Step 1: Irradiation of a standardized material at 5 K.
Step 2: Testing of all relevant properties at 5 K to determine property changes by irradiation.

Step 3: Warming up to 77 K.

Step 4: Testing of all relevant properties at 77 K to determine property changes by warming up. (Step 3 and 4 are helpful in the case of a partial warm up of a fusion magnet.)

Step 5: Warming up to 300 K (RT).

Step 6: Testing of all relevant properties at 300 K. Freed gases should be collected and analyzed.

Step 7: Cooling down to 4 K.

Step 8: Testing of all relevant properties at 4 K to compare with properties at 4 K irradiation before warm up.

It is clear that such a procedure is very expensive, but it would give a fundamental understanding of the role of irradiation and testing temperatures.

A.5 Test Matrix

The properties to be measured, the testing conditions and standard testing procedures have to be defined.

Properties to me measured should be:

Mechanical properties:	tensile strength
	compression strength
	flexure strength
	shear strength
	fatigue strength
	creep properties
Electrical properties:	voltage breakdown strength
	resistivity
	dielectric constant
	dissipation factor
Other properties:	brittleness/hardness
	dimensional changes
	weight changes
	gas evolution
	radioactivity
	color and appearance changes
	surface tracking (current along surface)
	electrolysis
	friction (resistance to fretting in pulsed magnets)
	thermal conductivity

Stress measurements should be performed also for multiaxial stresses (tension-torsion), not only for uniaxial stress.

Testing conditions should be:

- Well-known material compositions.

- Variation of sample dimensions.

- The thermal history (treatment) for all specimens should be equal.

- The influence of environment should be taken into account (especially before irradiation: air, nitrogen, water, cooling medium).

- Irradiation at 5 K in the right fusion spectrum, i.e. the right mixture of neutrons and gammas. This is a requirement which cannot ideally be fulfilled in a fission reactor.

- Dose and dose rate effects should be investigated.

- Applied stress during irradiation.

- Applied magnetic field during irradiation.

- Measurements during irradiation.

- Temperature and testing cycling as described above.

- Cyclic warming up and irradiation.

These test conditions cannot be reached simultaneously, e.g. the application of stress and magnetic field is difficult. The measurement without applied stress and/or magnetic field cannot give the full information, because synergic effects cannot be realized.

A.6 Properties of a Low Temperature Neutron Irradiation Facility

A low temperature neutron irradiation facility should allow 4.2 K sample temperature during irradiation. For measurements after irradiation, a temperature control up to 300 K should be possible.

A fast neutron flux of about 10^{13} n/cm^{2}s (E > 0.1 MeV) is required to attain the required fluences of about 10^{20} n/cm^2. In addition, the operating schedule should allow to accumulate the required fluence in an acceptable time interval (10^7 s correspond to about four months at steady state operation).

It should be possible to change the spectrum from the pure fission spectrum to a thermal spectrum. A control of the flux level is highly desirable.
The irradiation volume should be in the order of 1 liter. Enough refrigeration capacity should be available (several hundred W at 4.2 K).

Measurements should be possible either in-situ or a sample transfer capability at 4.2 K from the irradiation volume to the measurements devices should be available. The latter is preferred due to the higher measurement flexibility.

Neutron dosimetry is essential and supporting neutronics calculation capability should be available.

A.7 Extrapolation from Specimens to Magnets

The applicability of irradiation data from very small laboratory specimens to large scale devices has to be proven. Sample dimensions of e.g. 1.6 x 3.2 x 25.4 mm^3 or 6.4 mm Ø x 12.7 mm are very small compared with insulation materials dimensions in magnets. Therefore, there is a risk of incomplete simulation of the real case. May be edge effects in small samples predominate or surface-to-volume effects are possible.

It is not clear for the magnet designer which of the stress modes is the most important to have radiation data.

Also, the radiation behaviour of potting materials is not very wellknown. Irradiation investigations are strongly recommended.

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