

EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH



CERN ISR-MA/71-12

RADIATION RESISTANCE OF ORGANIC MATERIALS AT
CRYO-TEMPERATURES

by

M.H. Van de Voorde



CM-P00064762

Paper presented at the 1971 U.S. Particle Accelerator Conference

Chicago, 1 - 3 March 1971

Geneva, Switzerland

February 1971

RADIATION RESISTANCE OF ORGANIC MATERIALS AT CRYO-TEMPERATURES

M. H. Van de Voorde - CERN / ISR
Geneva Switzerland

Summary

The experimental procedures, the irradiation conditions and the radiation behaviour of a number of organic materials at liquid nitrogen temperature are reported.

1. INTRODUCTION

The progress made in superconductivity and the opportunities for reducing electric losses provided by the use of extremely pure metal conductors at very low temperatures has encouraged the development of superconducting- and cryogenic magnets. A new problem has therefore arisen, that of finding suitable insulators and structural organic components.

Organic materials used in the construction of such magnets which have to be operated in high energy accelerators must retain good mechanical and electrical properties at low temperatures in a radiation environment. As these materials are the least radiation resistant components used in magnet construction, they determine the life-time of the magnet. In order to obtain an acceptable life for a superconducting accelerator, special care must therefore be given to the selection of these materials. The paper describes experiments in order to determine the radiation behaviour of a number of plastics and elastomers at low temperature. The experimental procedures, the irradiation conditions and the first results are reported.

2. RADIATION AND CRYOGENIC TESTING FACILITY

The radiation source used in these tests was the "Melusine" reactor (Centre d'Etudes Nucléaires de Grenoble, CENG - France), a heterogeneous, highly enriched, thermal reactor utilizing water as neutron moderator and reflector, as radiation shielding and coolant. Maximum power generation is 4 MW.¹

The cryogenic facility, located at about 10 cm from the reactor core, is permanent, being continuously filled with liquid nitrogen (LN₂) of high purity (77° K) during the irradiations.² It contains a sample container of aluminium which measures 36 mm diameter and 300 mm length and in which about 16 dumb-bell type specimens of 113 x 22 x 2 mm can be irradiated at the same time. All irradiations have been performed under static conditions.

The neutron flux was monitored during the irradiations at several material locations along the sample container. Measurements were made with nickel for fast neutron flux, and cobalt for thermal neutron flux. Standard foil techniques were used in specifying the neutron field. The results of the neutron flux measurements along the container are given in Figure 1. The gamma dose rate inside the Dewar was measured with a TM type calorimeter (Figure 1) using graphite as absorber material.¹ From this data and knowing the energy neutron spectrum in the irradiation position³, the chemical composition of the irradiated material and their corresponding atomic absorption coefficients,

the total absorbed dose rate in the organic material in question has been calculated ($\sim 5 \cdot 10^7$ rad/hr).

After irradiation, tensile tests are carried out using an Instron tensile testing machine on which a LN₂ cryostat has been fitted. Load versus extension graphs for test specimens are plotted directly by an X-Y recorder. The load signal is taken from an Instron load cell and the strain recorded from the output of a displacement transducer. The sensitivity of the system allows strain in the specimen to be determined to better than 10⁻³ cm. The load may be accurately recorded up to 5,000 kg. The rate of crosshead travel is defined as 2 mm/min.

The materials studied are plastics and elastomers in sheet form and glass mica laminates. Only those which might be of interest in cryogenic magnet technology have been chosen. Before immersing in LN₂ and subsequent irradiation the specimens have been stored for several days in a desiccator to prevent water absorption. Four samples have been tested at each radiation level.

3. MATERIAL TEST RESULTS AND CONCLUSIONS

The effects of radiation on the mechanical properties of some plastics at LN₂ temperatures are given in Figures 2 - 7. In order to facilitate the selection of organic materials for cryogenic purposes in a radiation field, the relative radiation stability at LN₂ temperature of a number of organics is shown in Figure 8.

From these data and the experience gained during this studyprogram, the following conclusions may be derived :

1. The mechanical properties of organic materials irradiated at LN₂ temperature degrade. The radiation damage threshold value seems to differ from that measured at air-ambient temperature⁴, e.g. teflon and PVC resist better radiation at LN₂ compared to air, while the inverse is valid for the polypropylene group.
2. The first organic materials that come to mind for very low temperatures in a radiation field are : aromatic based epoxy resins, polyimides and polystyrenes. Polyacetals, polypropylene and many elastomers are discouraged in these applications although they are already widely used in cryogenic engineering.
3. At 77° K, these solids brittle. The elongation at break is often only 1 % of the value at room temperature. This brittleness still increases with radiation. One of the first considerations in deciding whether a material is or is not useful for cryogenic magnet technology in a radiation environment is the elongation measurement at break.
4. If one compares these data with the few data reported elsewhere⁵ on their electrical behaviour at LN₂ in a radiation field, it can be concluded that as long as the material withstands mechanically, the electrical properties practically do not alter.

5. A number of materials showed cracks or considerable shrinkage effects when immersed in LN₂, e.g. some polyethylenes, cyclic based epoxies, fluorinated silicones, etc. These materials have not been considered further.

6. Irradiations at LN₂ temperature adversely effect laminates, since such materials become "delaminated" under stress.

7. A general trend was noted in all test specimen colour changes but less pronounced at the same doses as in the air irradiations at 25°C (Fig. 9).

8. Humidity and impurities absorbed by organic materials show cracks when irradiated at LN₂ as a result of the formation of ice due to water present in the free state. Silicone resin used as demoulding agent gives the same effect as ice.

The irradiation results discussed have been obtained at LN₂ temperatures. The experiments are being expanded to lower temperatures, already now to liquid neon (27° K) and hopefully later to liquid helium (4° K) environments. As a preparatory part of this we have performed mechanical tests at LHe temperatures without radiation. These tests also serve to distinguish the effects of cooling and radiation. The results indicate that the choice of organic materials for magnets to be operated in a radiation field is much more limited in cryogenic and superconducting temperatures than at room temperature.

REFERENCES

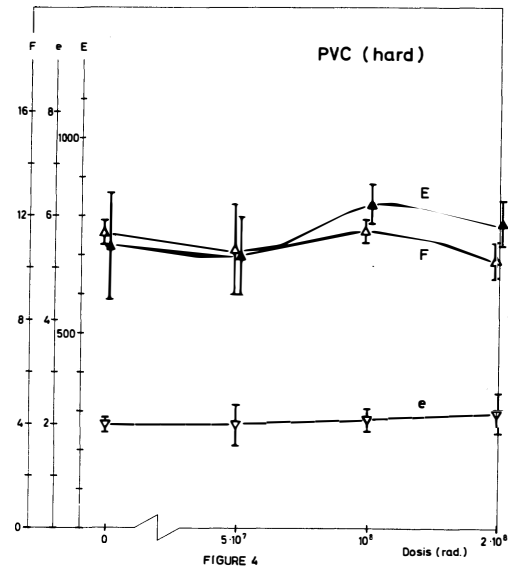
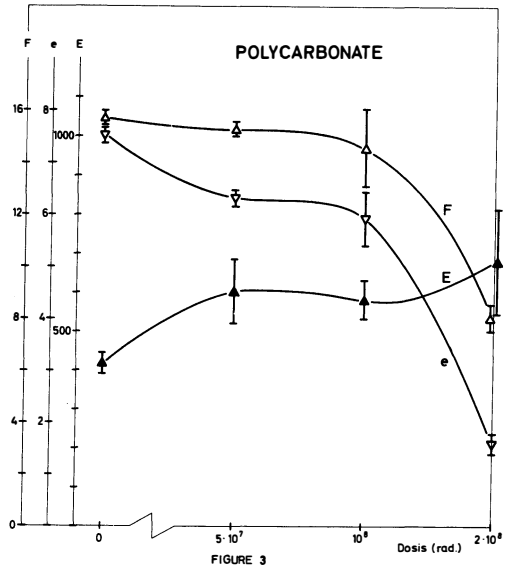
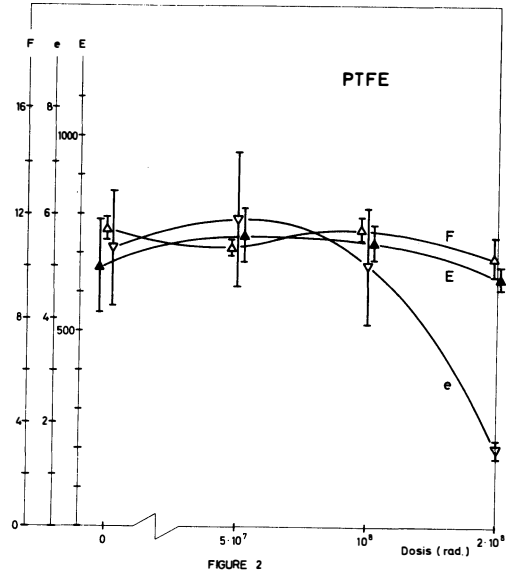
1. A.W. Boyd, IAEA - 128, Vienna (1970)
2. L. Bochirol, Industries Atomiques 7/8, 29(1968)
3. T. Mas, R.Lloret and J.Comera, CEA R3180 (1967)
4. M.H. Van de Voorde, CERN 70-05 (1970)
5. H. Brechna, BNL 50.155, 1011 (1969).

ACKNOWLEDGEMENTS

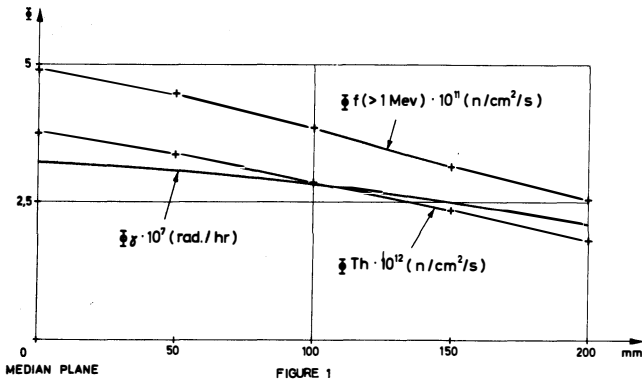
I should like to thank Dr. L. Resegotti for his encouragement and useful suggestions in this study paper, and Messrs. M. Blin and P. Bouriot for their devotion in the delicate technical work. The results have been obtained due to a close co-operation with CENG to whom I express my gratitude in particular to Dr.L.Bochirol, L.Brauns and P.Mas.

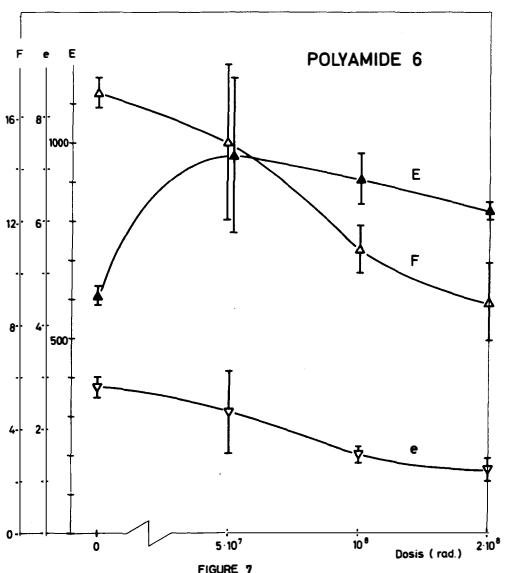
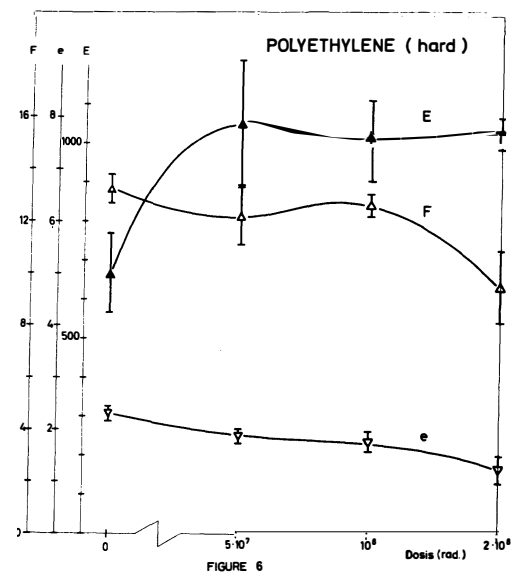
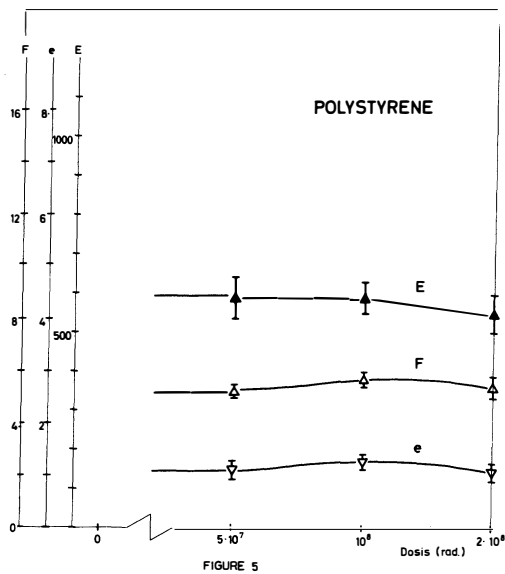
RADIATION RESISTANCE OF PLASTICS AT 77° K

△ E = MODULUS OF ELASTICITY IN kp/mm²
 ▽ F = TENSILE STRENGTH IN kp/mm²
 ▽ e = ELONGATION IN %



VERTICAL REPARTITION OF FLUX





COLOUR CHANGE AFTER IRRADIATION AT 77°K

Temperature (°K)	77				293 after irradiation at 2,10 ⁸
	0 Before irradi.	5,10 ⁷	1,10 ⁸	2,10 ⁸	
PRODUCT:					
Polyethylene (hard)	black	black	black	black	black
PVC (hard)	grey	brown	brown	brown	grey-brown
Polyamide	white	pale yellow	yellow	dark yellow	dark yellow
Polystyrene	white	white	white	white	white
Polycarbonate	transparent	black	black	black	dark green
Polypropylene	grey	grey	grey	grey	grey
Polyacetal	white	light brown	light brown	light brown	light brown
Polytetrafluoroethylene	white	pink	pink	dark pink	white

FIGURE 9

RELATIVE RADIATION RESISTANCE OF ORGANIC MATERIALS AT LIQUID NITROGEN TEMPERATURE

