LOW-TEMPERATURE IRRADIATION EFFECTS ON MATERIALS AND COMPONENTS FOR SUPERCONDUCTING MAGNETS FOR HIGH-ENERGY PHYSICS APPLICATIONS

Edited by M. Van de Voorde
Propriété littéraire et scientifique réservée pour tous les pays du monde. Ce document ne peut être reproduit ou traduit en tout ou en partie sans l’autorisation écrite du Directeur général du CERN, titulaire du droit d’auteur. Dans les cas appropriés, et s’il s’agit d’utiliser le document à des fins non commerciales, cette autorisation sera volontiers accordée.

Le CERN ne revendique pas la propriété des inventions brevetables et dessins ou modèles susceptibles de dépôt qui pourraient être décrits dans le présent document; ceux-ci peuvent être librement utilisés par les instituts de recherche, les industriels et autres intéressés. Cependant, le CERN se réserve le droit de s’opposer à toute revendication qu’un usager pourrait faire de la propriété scientifique ou industrielle de toute invention et tout dessin ou modèle décrits dans le présent document.

© Copyright CERN, Genève, 1977

Literary and scientific copyrights reserved in all countries of the world. This report, or any part of it, may not be reprinted or translated without written permission of the copyright holder, the Director-General of CERN. However, permission will be freely granted for appropriate non-commercial use. If any patentable invention or registrable design is described in the report, CERN makes no claim to property rights in it but offers it for the free use of research institutions, manufacturers and others. CERN, however, may oppose any attempt by a user to claim any proprietary or patent rights in such inventions or designs as may be described in the present document.
ABSTRACT

Experimental details and results are presented of irradiations carried out at the Centre d'Études Nucléaires, Grenoble, from 1972 to 1975. The pool-type nuclear reactor Melusine was used as a source of mixed neutron and gamma radiation. The irradiation facilities and dosimetry methods are described, followed by a chapter on radiation-damage effects on temperature sensors and liquid-level indicators. Irradiation baths of boiling liquid nitrogen (77 K) and liquid neon (27 K) permitted simultaneous exposure of several samples; the results are considered adequate for the assessment of irradiation effects down to liquid-helium temperature. The radiation effects on various types of copper and the low-temperature behaviour of a great number of organic materials in radiation fields are discussed. For the latter samples, test procedures and experimental results concerning the thermal conductivity, specific heat and mechanical properties (flexural tests) are presented.
CONTENTS

INTRODUCTION  L. Bochiroi, M. Van de Voorde  

CHAPTER I  IRRADIATION CONDITIONS  
L. Bochiroi, P. Mas, H. Petitoolas, M. Van de Voorde, J. Verdier  

I.1 IRRADIATION FACILITIES IN THE MELUSINE REACTOR  
I.2 DOSIMETRY METHODS  5  
I.2.1 Measuring the fluxes  5  
I.2.2 Calculation of radiation dose in material  6  
I.2.3 Neutron damage analysis  8  
I.3 CRYOGENIC IRRADIATION FACILITIES USED IN THE MELUSINE REACTOR  9  
I.4 INDUCED RADIOACTIVITY  10  
FIGURES  14  

CHAPTER II  RADIATION DAMAGE EFFECTS ON TEMPERATURE SENSORS AND LIQUID-LEVEL INDICATORS  
A. de Combarieu, M. Van de Voorde  

II.1 INTRODUCTION  19  
II.2 EXPERIMENTAL PROCEDURES  19  
II.2.1 Irradiation conditions and components tested  19  
II.2.2 Measurement techniques  19  
II.2.3 Calibration method  20  
II.3 RESULTS  20  
II.3.1 Resistors  20  
II.3.2 Diodes  21  
II.3.3 Thermocouples  22  
II.4 DISCUSSION OF RESULTS  22  
II.4.1 Germanium resistors  22  
II.4.2 GaAs diodes  22  
II.5 CONCLUSIONS  24  
FIGURES  26
CHAPTER III EFFECTS OF RADIATION ON COPPER
M. Couaou, M. Van de Voorde

III.1 INTRODUCTION 31

III.2 MEASURING TECHNIQUES AND IRRADIATION CONDITIONS 33
   III.2.1 Copper types studied 33
   III.2.2 Sample preparation 33
   III.2.3 Measuring technique and experimental errors 33
   III.2.4 Irradiation conditions - Dosimetry 34

III.3 RESULTS 34

III.4 ANALYSIS AND COMMENTS 35

FIGURES 42

CHAPTER IV LOW-TEMPERATURE BEHAVIOUR OF ORGANIC MATERIALS IN A RADIATION FIELD
E. Bonjour, P. Brauns, R. Lagnier, M. Van de Voorde

IV.1 INTRODUCTION 53

IV.2 THERMAL CONDUCTIVITY 53
   IV.2.1 Experimental procedure 53
   IV.2.2 Results 53

IV.3 SPECIFIC HEAT 54
   IV.3.1 Experimental procedure 55
   IV.3.2 Results 55

IV.4 Mechanical properties: Flexural tests 55
   IV.4.1 Test method 55
   IV.4.2 Results 56

IV.5 ANALYSIS OF DATA 58

FIGURES
INTRODUCTION

L. Bochirol (CENG) and M. Van de Voorde (CERN)

This report presents the results of the low-temperature irradiation experiments carried out in the Grenoble Nuclear Research Centre from 1972 to 1975 according to the program of Dr. M. Van de Voorde, ISR Division, CERN.

For the development of superconducting magnets and their use near high-energy particle beams, a thorough investigation of the radiation behaviour of the materials and components used in the superconducting (SC) magnets was necessary.

To irradiate materials and components under actual accelerator conditions at helium temperature is virtually impossible, hence evaluation of irradiation effects must be made by simulation. The source used for the experiments, the results of which are given in this report, was the mixed neutron and gamma radiation from a pool-type nuclear reactor.

Irradiations in a reactor at helium temperature need very sophisticated equipment and hence only a limited number of samples (generally a single sample) can be studied simultaneously. Helium-temperature irradiations are more adapted to fundamental studies than to technological ones. However, simultaneous irradiation of a number of samples is possible using boiling liquid nitrogen (77 K) and liquid neon (27 K) as irradiation baths. The cost of the irradiations is consequently reduced and, in most cases, 27 K is sufficient (and often 77 K) to "freeze" the created defects especially in technical materials. In fact, there would be very little difference, if any, in the irradiation effects at lower temperatures.

We think that the results presented in this report give a good indication of the behaviour of some technological materials and components when submitted to irradiation at low temperature. The results should also be useful to those involved in SC magnet conceptions and buildings for high-energy applications.

*) CENG: Centre d'Etudes Nucléaires de Grenoble.
CHAPTER I

IRRADIATION CONDITIONS

L. Bochirol, P. Mas, H. Petitcolas and J. Verdier
Centre d’Etudes Nucléaires de Grenoble, France

and

M. Van de Voorde
CERN, Geneva, Switzerland
I.1 IRRADIATION FACILITIES IN THE MELUSINE REACTOR

The materials and components were irradiated in the Melusine reactor of the Nuclear Research Centre, Grenoble. Melusine is a heterogeneous, highly enriched thermal reactor utilizing water as a) neutron moderator and reflector and b) radiation shielding and coolant, and has a maximum power generation of 8 MWe. The fuel elements are of the plate type, and the fuel plates are made of uranium-aluminium alloy containing 93% $^{235}$U.

Many irradiation devices are put inside the core or in its vicinity, and the irradiation temperature range is from -246°C (liquid-neon loop) to +2000°C (high-frequency furnace). Several in-pile continuous instrumentations are optional (incident fast neutron and thermal fluxes, temperature, dimensional variations, etc.).

Figure I.1 shows a simplified cross-section of the core at the centre of the fuel elements, and the neutron fluxes and gamma heating in each irradiation position.

The CERN materials and components were mainly irradiated in the following positions:

- in the special "low-temperature notch" at the centre of the four positions 28-29-38-39 and behind the lead shield, shown cross-hatched on the diagram (the aim of the lead shield is to decrease the gamma heating in the cold loops without appreciably degrading the fast flux intensity) (Fig. I.2);
- in positions 49-2 and 49-4, opposite the hole 49 but away from the core (Fig. I.3);
- in position 91, the cryogenic facility which contains an aluminium sample container (36 mm Ø and 300 mm long) in which about 16 dumb-bell-type specimens (113 x 22 x 2 mm) can be irradiated simultaneously (Fig. I.4).

I.2 DOSIMETRY METHODS

During each irradiation, the radiation doses received by the samples were measured.

I.2.1 Measuring the fluxes

a) Gamma flux

The gamma dose-rate inside the dewar is measured with a calorimeter (Refs. I.1 and I.2), which is shown in Fig. I.5. This is an isothermal calorimeter made of graphite absorber suspended by thermocouples in a narrow cylindrical stainless-steel container. The hot weld is in the centre of the graphite core and the cold one on the external calorimeter wall, which is at the temperature of the "swimming-pool" water. Calibration of the calorimeter is made by electrical heating coaxial cable. The radiation heating power released in the graphite core can be obtained from the temperature difference measured between the core and the external wall. The measuring range of this calorimeter type is from 0.01 to 6 W/g deposited power in graphite material. Comparisons made between numerous different laboratories gave consistent results (Refs. I.3 and I.4).

b) Fast neutron flux

Fast neutrons contribute to part of the energy released within irradiated plastic materials and produce some permanent damage in metallic materials.
In order to evaluate these two irradiation effects, the flux is measured by flux detectors in the vicinity of the samples during irradiation. For the CERN irradiations, the flux was measured from the threshold reaction of nickel:

\[ ^{58}\text{Ni}(n,p)^{58}\text{Co} \quad (72 \text{ d}) , \]

which has an effective threshold of 3 MeV, and by the indium reaction

\[ ^{115}\text{In}(n,n')^{115}\text{SmIn} \quad (4.5 \text{ h}) , \]

which has a threshold energy of 1 MeV. The selected fission spectrum mean cross-sections are 101 mb for nickel and 168 mb for indium. Hence from this indium-nickel spectrum the fast neutron flux with energy higher than 1 MeV can be measured, and by addition of the reaction

\[ ^{27}\text{Al}(n,\alpha)^{24}\text{Na} \quad (15 \text{ h - 6 MeV threshold}) , \]

the spectrum shape can be determined in the irradiation position (Ref. I.5).

The absolute disintegration rate of \(^{59}\text{Co}\) is obtained by comparison with a calibrated \(^{58}\text{Co}\) source for which the absolute activity was measured by the triple coincidence method (Ref. I.6).

c) Thermal neutron flux

The effect of thermal neutrons is important especially in highly absorbing materials, e.g. boron-glass laminates. The thermal neutron flux is measured with cobalt detectors from the reaction

\[ ^{59}\text{Co}(n,\gamma)^{60}\text{Co} \quad (5.27 \text{ y}) . \]

The selected cross-section is 38 b at 2200 m/sec.

\[ \text{I.}\underline{2} \quad \text{Calculation of radiation dose in material} \]

The radiations producing damage in plastic materials are gamma photons and fast neutrons. Radiation doses are expressed in rads (1 rad = 100 ergs/g).

a) Gamma dose

Knowing the energy spectrum in the irradiation position, the composition of the material, and the energy absorption coefficient of each constituent in the material, the total absorbed dose in the materials can be calculated.

The gamma photons are measured in graphite, and the mean energy of the gamma radiation in Melusine is taken as 0.8 MeV.

b) Calculation of gamma dose in polymeric materials

Taking \(D_\gamma\) as the dose in rads due to gamma-rays:

\[ D_\gamma = 10^5 \times \left( \frac{N_E}{N_M} \right) \times \rho M \times \frac{(\mu/\rho)_E}{(\mu/\rho)_C} \times t , \]
where
\[ \phi_{\gamma} E_0 = \text{gamma flux in W/g of graphite, in the dummy container of the low-temperature facility at the level of the sample during the calibration;} \]
\[ \phi_{\gamma} M_0 = \text{gamma flux in W/g of graphite, measured by the monitor during calibration;} \]
\[ \phi M = \text{gamma flux in W/g of graphite, measured by the calorimeter during irradiation;} \]
\[ (\rho/\mu)_C = \text{energy absorption coefficient of graphite, taken as the mean energy (E = 0.8 MeV) of gamma-rays;} \]
\[ (\rho/\mu)_E = \text{energy absorption coefficient of sample;} \]
\[ t = \text{irradiation time in seconds.} \]

The results can be given in rads using the coefficient $10^5$.

Table I.1 gives the conversion factor between graphite and some plastic materials.

c) Fast neutron dose

Taking $D_R$ as the fast neutron dose in rads
\[ D_R = 10^{-15} \Gamma E \times \phi_R \times 10^5, \]

where $\Gamma_E$ = energy absorption coefficient of the fast neutrons in the sample. For example, for a sample of formula $C_n H_m$ we have:
\[ \Gamma_E = \frac{12 n \Gamma_C + 1 m \Gamma_H}{12 n + m}. \]

The coefficients $\Gamma_C$, $\Gamma_H$, etc., are defined and calculated elsewhere (Ref. I.7).

$\phi_R$ = fast neutron fluence in n/cm$^2$, measured during irradiation.

d) Example of gamma and fast neutron dose calculations

Sample of polyethylene $CH_2$

i) Gamma dose

Flux measurements $\phi_{\gamma} E_0 = 0.090$ W/g
$\phi_{\gamma} M_0 = 0.400$ W/g
$\phi M = 0.370$ W/g
\[ \frac{(\rho/\mu)_E}{(\rho/\mu)_C} = 1.14, \]

where
\[ D_\gamma = 10^8 \times \left[ \begin{array}{c} 0.090 \\ 0.400 \end{array} \right] \times 0.370 \times 1.14 \times 5400 \]
\[ = 5.12 \times 10^7 \text{ rads}. \]
ii) Fast neutron dose

First determine \( \Gamma_E \):

\[
\Gamma_E = \frac{12 \times 6.8 + 2 \times 390}{12 + 2} = 61.54.
\]

We have measured \( \Phi_R = 2.19 \times 10^{15} \), where

\[
D_R = 10^{-15} \times 61.54 \times 2.19 \times 10^{15} \times 10^5
\]

\[= 1.35 \times 10^7 \text{ rads}.\]

I.2.3 Neutron damage analysis

Fast neutron energy is mainly released during the slowing down by elastic and inelastic collisions on the irradiated material nuclei (Ref. I.8). The "power" released by a flux having a spectral distribution \( \Phi(E) \) is:

\[
p = K \int \Phi(E) \sigma_p(E) \, dE;
\]

\( K \) is a constant which takes into account the energy transfer from the neutron to the nucleus and the units. If \( \Phi(E) \) is known, coefficients such as \( \delta = p/\Phi(>1) \) can be determined, where \( \Phi(>1) \) is the fast neutron flux with energy higher than 1 MeV.

After measuring the fast neutron fluence (\( E > 1 \) MeV), the corresponding deposited energy can easily be deduced.

In the copper and thermometer irradiations the fast neutron fluences, which produce the damage, had energy higher than 1 MeV. The damage in irradiated metals is actually produced by neutrons colliding with lattice atoms which can be ejected from their lattice positions with a certain energy and in turn collide with new atoms producing Frenkel pairs, i.e. vacancies and interstitial atoms. Therefore, the number of created defects depends on the energy of the incident neutrons, the probability of colliding with lattice atoms, and the energy transmitted to the individual atoms.

It was therefore decided to take the fluences producing damage as displacements per atom (d.p.a.), and hence comparisons can easily be made between irradiations carried out in various reactors with different neutron energy spectra and irradiations with particles of different natures and energies.

The number of d.p.a. can be written

\[
d = \frac{E_d}{E_0},
\]

with \( E_0 = 2E_d/0.8 \),

where the displacement energy \( E_d \) can be taken equal to 40 eV;

\[
E_d = W(E) \Phi(E) \, dE,
\]

where \( \Phi(E) \) is the neutron energy spectrum;

\( W(E) \) is the product of the collision probability times the number of displacements. The function is represented by the Lindhard model.
For a given material, the number of d.p.a. can be written:

\[ d = \alpha \phi (\geq 1) \]

where \( \phi (\geq 1) \) is the neutron flux with neutron energy higher than 1 MeV. In the case of the copper and with the Melusine irradiation position spectrum, calculations give

\[ \alpha = 1.3 \times 10^{-21} \text{ d.p.a./n/cm}^2. \]

I.3 CRYOGENIC IRRADIATION FACILITIES USED IN THE "MELUSINE" REACTOR

Melusine is equipped with four cryogenic irradiation facilities. Two operate at liquid nitrogen (LN\(_2\)) temperature level (77 K) and two others at liquid neon (LNe) level (27 K). These rigs can be put at different positions near the core of the reactor, allowing exposition of the samples to very different neutron and gamma flux levels, as has been indicated in Section I.1 (see Fig. I.1).

All the facilities used are of the same general concept, a schematic view of which is given in Fig. I.6.

The samples, with attached neutron dosimeters, are put in a container, immersed in a cryogenic bath (either boiling LN\(_2\) or LNe). This container is put in place for irradiation through a bent access tube; in the same way, the container can be removed very quickly at the end of irradiation and decay period to be transferred in a transport dewar. In all irradiation positions, vertical neutron flux gradients can be considered negligible for each sample.

Irradiation temperature is well defined (it is that of the boiling cryogenic fluid), and it is possible to transfer the irradiated samples to the laboratory for measurements, without any re-heating. Many of them have thus been regularly transferred from CENG, Grenoble to CERN, Geneva.

The vapours of a boiling cryogenic bath are condensed on the surface of a heat exchanger-condenser located in an immersed part of the lateral branch of the device. This is an original feature of cryogenic rigs developed at the Grenoble Nuclear Research Centre.

It is now well known (Ref. I.9) that direct use of commercial LN\(_2\) (containing oxygen as an impurity) is not possible in high gamma flux: the formation of ozone and nitrous oxides under irradiation may cause explosions. The solution is to use very pure LN\(_2\) (impurities at ppm level) in limited quantity as cryogenic bath, and condense its vapours on a condenser located at a distance from the core of several metres, where the gamma irradiation level is low. It is thus possible to feed the condenser of the rig with commercial LN\(_2\). One of the two LN\(_2\) cryogenic devices of Melusine is still of this type, and has been running for years without any trouble.

Later, since 1967, when we gradually equipped our devices with cryogenerators to keep our rigs at low temperature, we have kept up this concept (Refs. I.10 and I.11).
As is shown in Fig. 1.6, a flexible bellows connection makes it possible to approach the lower part of the loop (where the samples are located) quite near to the reactor core, or to keep it at a distance (max. 1.20 m between irradiation and de-activation positions). Of course, these movements do not affect feeding of the condenser with cryogenic fluid nor the vacuum insulation.

If necessary, it is possible to do in situ electrical measurements on the samples during or just after irradiation. This has been the case, for instance, when testing the behaviour of temperature sensors under irradiation at low temperature. For this type of measurement, electrical connections to the sample are provided through the access tube.

1.4 INDUCED RADIOACTIVITY

Induced radioactivity in an irradiated sample is always a potential problem in nuclear reactor irradiations. The samples irradiated were mainly polymeric materials.

The induced radioactivity in polymers depends largely on the nature and mass of plasticizers, mineral fillers, antioxidants, etc., which are eventually introduced, e.g. sulfur in elastomers and some epoxies, titanium dioxide in thermosetting resins, etc. Since the chemical composition of commercial polymers is often unknown, it is only at the end of reactor irradiation that the presence of these substances becomes obvious. Also, some types of polymer structure in themselves induce radioactivity, e.g. polyvinylchloride, chlorotrifluoroethylene.

Although the radiation hazards are not critical in polymers, safety procedures for handling, storage, and disposal must be followed. Some materials are very radioactive for a short time; for example, chlorine-38 and titanium-51 both emit energetic beta particles and gamma-rays with half-life of approximately 0.6 hours and 6 minutes. Hence such samples would either have to be handled remotely or be allowed to cool for approximately six hours and one hour, respectively; they can then be handled with safety by ordinary means. Certainly, after a period of 24 hours, sufficient decay would have occurred to render the chlorine or titanium samples completely innocuous.

Some elastomers and resins may be radioactive for long periods; for example, phosphorus-32 constitutes a serious hazard. It has just the right combination of rate of formation energy of beta particles (1.7 MeV) and half-life (14 days) to yield a high specific activity of penetrating rays of long duration. However, the shielding problem for phosphorus-32 is not too difficult because the betas can be absorbed in as little as one centimetre of plexiglas. But the high level of activity and long half-life means that the samples cannot be handled directly and, after measurement, must be stored for a rather long period of time (1 year) before the induced activity has decayed to a negligible level.

The majority of irradiated plastics are fairly weak beta-radiation emitters and do not constitute radiation hazards, e.g. carbon-14 (half-life 5700 years), sulfur-35 (half-life 87 d), calcium-45 (half-life 165 d), etc. However, owing to the very long half-life of these isotopes, an accumulation of samples is equivalent to an accumulation of activity over any period of time, and hence adequate storage and disposal procedures are required.
It is recommended that small representative samples of all new formulations be first tested in order to define the actual problems that may arise in the case of the full-scale test samples. In this way, problems can be anticipated and measures taken beforehand to control any difficulties either from radiation hazards during handling and testing, storage during and after testing, or contamination of equipment, area, etc.

Also, the aluminium containers themselves (each weighing about 26 g) are generally the most important source of activity (formation of sodium-24) and need about 10 to 15 hours for de-activation in cold conditions.

In order to evaluate the "danger parameter" of radioactivity induced by the interaction of reactor thermal neutrons with copper, a simulation irradiation experiment was carried out at room temperature and about $1.6 \times 10^{18}$ n/cm².

The radiation dose of the samples at 1 m was measured as a function of post-irradiation time. A dose-level of 200 mrad/h was measured 62 h 30 min after exposure. In order to reduce the dose-level to a level acceptable for manipulation, a cool-down of 15 days was necessary, after which time the dose measured at the surface of the sample was around 30 mrad/h per sample.
<table>
<thead>
<tr>
<th>Material</th>
<th>Empirical formula</th>
<th>Ratio of absorbed dose in material to that in graphite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyethylene</td>
<td>((\text{CH}_2)_n)</td>
<td>1.14</td>
</tr>
<tr>
<td>Polyamide (Nylon)</td>
<td>((\text{C}<em>6\text{H}</em>{11}\text{ON})_n)</td>
<td>1.09</td>
</tr>
<tr>
<td>Polydimethyl siloxane (Silicone)</td>
<td>((\text{C}_3\text{H}_6\text{OSi})_n)</td>
<td>1.08</td>
</tr>
<tr>
<td>Ethylene polysulfide (Thiokol)</td>
<td>((\text{C}_6\text{H}_6\text{S}_x)_n)</td>
<td>1.02</td>
</tr>
<tr>
<td>Vinlylidene chloride copolymer (Saran)</td>
<td>((\text{C}_6\text{H}_6\text{Cl}_x)_n)</td>
<td>1.03</td>
</tr>
<tr>
<td>Polytetrafluorethylene (Teflon)</td>
<td>((\text{CF}_2)_n)</td>
<td>(\sim 1)</td>
</tr>
<tr>
<td>Polychlorotrifluorethylene (Kel-F)</td>
<td>((\text{C}_2\text{F}_3\text{Cl})_n)</td>
<td>(\sim 1)</td>
</tr>
<tr>
<td>Polyvinylchloride (PVC)</td>
<td>((\text{C}_2\text{H}_3\text{Cl})_n)</td>
<td>1.05</td>
</tr>
<tr>
<td>Polyvinyl pyrrolidone</td>
<td>((\text{C}_5\text{H}_4\text{NO})_n)</td>
<td>1.08</td>
</tr>
<tr>
<td>Polyvinylcarbazole</td>
<td>((\text{C}<em>{14}\text{H}</em>{11}\text{N})_n)</td>
<td>1.06</td>
</tr>
<tr>
<td>Polyvinylacetate</td>
<td>((\text{C}_6\text{H}_6\text{O}_2)_n)</td>
<td>1.07</td>
</tr>
<tr>
<td>Polymethylmethacrylate</td>
<td>((\text{C}_5\text{H}_8\text{O}_2)_n)</td>
<td>1.06</td>
</tr>
<tr>
<td>Tributyl phosphate</td>
<td>((\text{C}_3\text{H}_9)_3\text{PO}_4)</td>
<td>1.10</td>
</tr>
<tr>
<td>Polystyrene</td>
<td>((\text{CH})_n)</td>
<td>1.08</td>
</tr>
<tr>
<td>Epoxy resin</td>
<td>((\text{C}_3\text{SiO}_3\text{H}_2)_n)</td>
<td>1.08</td>
</tr>
<tr>
<td>Fricke dosimeter</td>
<td>((\text{C}_4\text{H}_8)_3\text{PO}_4)</td>
<td>1.10</td>
</tr>
</tbody>
</table>

* Incident photon energy
REFERENCES TO CHAPTER I


I.2 H. Petitcolas et al., Mesures par calorimétrie de la dose absorbée dans le graphite, dans les réacteurs de recherche Mélusine (8 MW), Siloe (35 MW), et dans le réacteur de Puissance Bugey 1 (1900 MW), paper presented at the Premier Symposium ASTM-EURATOM sur la dosimétrie en réacteur, Petten, 22-26 September 1975.


I.5 P. Mas, R. Lloret and J. Comera, Spectre des neutrons rapides dans le réflecteur d'une pile à eau légère, CEA R-3180 (1967).


I.10 L. Bochirol, Récents développements de techniques cryogéniques au CENG, Industries Atomiques 7-8, 29 (1968).

Fig. I.1 Flux map of Melusine (8 MW)

Fig. I.2 Vertical distribution of the flux in irradiation positions 28, 29, 38, and 39 of Melusine (low temperature; behind lead shield)

Fig. I.3 Vertical distribution of the flux in irradiation position 49.2 of Melusine (low temperature; liquid nitrogen)
Fig. I.4 Vertical distribution of the flux in irradiation position 91 of Melusine (low temperature; liquid neon)

Fig. I.5 The calorimeter

Fig. I.6 Schematic view of the Melusine reactor cryogenic loops
CHAPTER II

RADIATION DAMAGE EFFECTS ON
TEMPERATURE SENSORS AND LIQUID-LEVEL INDICATORS

A. de Combarieu
Centre d'Etudes Nucléaires de Grenoble, France
and

M. Van de Voorde
CERN, Geneva, Switzerland
11.1 **Introduction**

The use of cryogenic equipment such as superconducting magnets in particle accelerators and storage rings necessitates data on the radiation damage effects on temperature sensors in the temperature range 4.2 K to 200 K and on cryogenic liquid-level indicators.

The investigation was limited to devices commonly in use, e.g. thermocouples, metal, carbon and semiconductor resistors, and semiconductor diodes.

The irradiations were performed in liquid neon (27 K), liquid nitrogen (77 K), and in air at ambient temperature. In all cases the irradiations were performed in mixed fast-neutron/gamma fields.

11.2 **Experimental Procedures**

11.2.1 **Irradiation conditions and components tested**

This data is contained in the following tables: Table II.1: Characteristics of the radiation sources used and Table II.2: Components tested.

11.2.2 **Measurement techniques**

- **Thermocouples**: Reference temperature, 4.2 K. Thermoelectric voltage is measured with a potentiometric method and a null detector, or directly with a digital voltmeter of input resistance > $10^{10}$ Ω. Accuracy of measurement, ±2 μV.

- **Resistors**: The resistance is measured with a four-wire direct current method. The voltages developed by a constant current across the component and a standard resistor are compared. A current source of 10 or 100 μA is used, depending on the sample temperature and resistance value. Voltmeter input resistance > $10^{10}$ Ω. Accuracy of measurement, $1 \times 10^{-4}$.

- **Diodes**: The forward voltage is measured with a current source, of a similar form to that of the resistors: 100 μA for the GaAs diodes; 1 μA for the Si diodes. Accuracy of measurement, $1 \times 10^{-4}$.

In situ measurements are done in the cold loop (liquid neon or liquid nitrogen) of the Melusine reactor. During the experiments the sensors are immersed directly in the cryogenic liquid near the core of the reactor. For resistors and diodes, the voltage and current are recorded at equal intervals of time. Measurements are done with the same four-wire technique as in the gas thermometer, and the precision is the same. Slight oscillations of some tenths of a degree Kelvin in the cryogenic bath temperature must be taken into account, resulting in pressure variations introduced by the regulation system of the test rig.

Measurement in a high magnetic field (5 tesla) are done using a superconducting coil of the SHE Company:

- **Bore**: 87.1 mm
- **Winding length**: 154 mm
- **Over-all diameter**: 174 mm
- **Over-all height**: 236 mm
Gauss/ampere: 969
Maximum field at 4.2 K: about 8 tesla
Field homogeneity: no compensating coil
Field stability versus time: $10^{-3}$.

In the case of the thermocouple measurements, the dimensions of the sensor being several centimetres it is difficult to extrapolate the results to another geometry because the field homogeneity is not so good. For the field measurement the SHE specifications are used without other measurements (969 G/A).

II.2.3 Calibration method

All sensors are calibrated before and after irradiation, using a classical, constant volume, helium gas thermometer, of which a detailed description is given by Bedin et al. (see Bibliography). A known quantity of helium gas is contained in a bulb of copper (volume $12\ell \text{ cm}^3$). When the temperature of the bulb varies, the pressure and the temperature of the gas are related by

$$PV = RT.$$  

If $V$ is constant and $T_0$, a known temperature, for another temperature $T_1$ we have

$$T_1 = \frac{P_1}{P_0} T_0,$$

and the measurement of temperature is converted in a pressure measurement.

It is obvious that this formula is true in an ideal case and for a perfect gas. In the real case, numerous correction terms must be applied. For our gas thermometer we have about 3 mb for 1 K. An important feature of this equipment is a special differential pressure-measuring system. The pressure measurement is done with an error of ±0.1 mb, and consequently the error in temperature determination is at least ±1/30 K. When we take into account all the errors, the relative accuracy of the measurements varies from $8 \times 10^{-3}$ at 4 K to $8 \times 10^{-4}$ at 300 K, but the reproducibility is quite better.

II.3 RESULTS

II.3.1 Resistors

a) Carbon and platinum

Carbon and platinum resistors were irradiated in SNIF (Standard Neutron Irradiation Facility of the ASTRA reactor, Seibersdorf, Austria) to a fast neutron fluence of $3.0 \times 10^{14}$ n/cm² and in Ebene 1 to a gamma dose of $5.0 \times 10^8$ rad at an irradiation temperature of ~ 300 K, and then measured in the temperature range 4.2 K to 20 K (C) and 77 K to 300 K (Pt). They did not show any change in characteristics outside the manufacturer's tolerance limits. Figures II.1 and II.2 give the $R = f$ (temp.) curves for C and Pt resistors, respectively, after irradiation to $5 \times 10^8$ rad ($\gamma$) and $3.6 \times 10^{15}$ n/cm².

A carbon resistor was irradiated in Mélusine at 27 K for 30 hours to a fast neutron fluence of $8.4 \times 10^{15}$, during which time the resistance was measured as a function of the neutron fluence, Fig. II.3.
The resistance of the irradiated component has also been measured as a function of temperature from 4.2 K to 30 K before and after irradiation at 27 K (Fig. II.4). The measurements show no change in resistance up to an integrated fluence of $8.8 \times 10^{15}$ n/cm$^2$; they are independent of the studied neutron fluxes between $6.8 \times 10^{13}$ and $2.8 \times 10^{14}$ n/cm$^2$ and of the post-irradiation room temperature heat treatment.

Also, the carbon resistors irradiated at 27 K have been measured in a magnetic field at 5 tesla, from 4.2 K to 30 K, before and after irradiation (Fig. II.4). Carbon resistors seem rather insensitive to these combined conditions of magnetic field and radiation.

b) Germanium

Sample A: Resistance was measured as a function of temperature from 4.2 K to 80 K. The component was then irradiated at 27 K for 6 hours to a fast neutron fluence of $4.1 \times 10^{14}$ n/cm$^2$, during which time the resistance was measured as a function of fast neutron fluence as shown in Fig. II.5. After raising the temperature to ambient, a further measurement of resistance as a function of temperature from 4.2 K to 80 K was made. The pre-irradiation and post-irradiation resistance/temperature curves are given in Fig. II.6.

Sample B: The same experimental procedure as that for sample A, except that the irradiation temperature was 77 K. Figures II.7 and II.8 give resistance as a function of fast neutron fluence and pre- and post-irradiation resistance as a function of temperature, respectively.

Samples C, D and E: These samples were measured for resistance as a function of temperature from 4.2 K to 80 K, after which they were irradiated at 77 K for 2 hours (sample C, $1.4 \times 10^{14}$ n/cm$^2$), 10 hours (sample D, $6.8 \times 10^{14}$ n/cm$^2$), and 20 hours (sample E, $1.4 \times 10^{15}$ n/cm$^2$). The sample temperature was then raised to ambient, and subsequently lowered to re-measure the resistance as a function of temperature from 4.2 K to 80 K. The experimental curves are given in Figs. II.9, 10, and 11, respectively. Sample E has again been measured after being kept for 105 days at room temperature. It showed no variation during this time (Fig. II.11).

II.3.2 Diodes

a) GaAs

Sample A: The forward voltage was measured as a function of temperature from 4.2 K to 300 K, followed by irradiation at 27 K for 6 hours to a fast neutron fluence of $4.1 \times 10^{14}$ n/cm$^2$. During the irradiation the forward voltage was measured as a function of neutron fluence, as shown in Fig. II.12. The sample temperature was then raised to ambient and re-cooled for a post-irradiation measurement of forward voltage as a function of temperature from 4.2 K to 300 K. The pre- and post-irradiation voltage/temperature curves are shown in Fig. II.13.

Sample B: The same experimental procedure was followed as for sample A, with the exception that the irradiation temperature was 77 K. Figures II.14 and II.15 give the forward voltage as a function of neutron fluence, and pre- and post-irradiation voltage as a function of temperature, respectively.
Sample C: The forward voltage was measured as a function of temperature from 4.2 K to 300 K followed by irradiation for 10 hours ($6.8 \times 10^{14}$ n/cm$^2$). The sample was then heated to ambient and re-cooled for the post-irradiation forward voltage measurements from 4.2 K to 300 K. The two curves are shown in Fig. II.16.

This sample was measured again after 180 days storage at room temperature. It showed a slight variation, especially at higher temperature (see Fig. II.16).

b) Si (liquid-He level indicator)

The effect of room temperature irradiation of Si diodes, both in SNIF and Ebene 1, is shown in Table II.3.

II.3.3 Thermocouples

A thermocouple of AuFe (0.03% At)-Cr was irradiated at 27 K to a fast neutron fluence of $4.8 \times 10^{14}$ n/cm$^2$. After irradiation, the thermocouple was raised to ambient temperature, then re-cooled for measurement. The calibration from 4.2 K to 30 K before and after irradiation in a zero- and 5-tesla magnetic field is given in Fig. II.17. The effect of irradiation was negligible at a zero field and at 5 teslas, but the thermoelectric voltage for a given temperature increases slightly as a function of magnetic field.

II.4 DISCUSSION OF RESULTS

II.4.1 Germanium resistors

i) Resistance increases approximately as a linear function of absorbed dose. The effect of this increase in resistance on the accuracy of the temperature measurement is shown in Fig. II.18. This gives the error in the temperature measurement $\Delta T(K)$ as a function of fast neutron fluence for four different temperature values. For example, after 12 hours irradiation, i.e. a fluence of $8.2 \times 10^{14}$ n/cm$^2$, a true temperature of 20 K would be indicated as $(20 + 8)$ K, i.e. 28 K.

ii) Radiation damage is not a function of irradiation at temperatures of 27 K and 77 K as shown in Fig. II.18.

iii) After irradiation in Melusine at 77 K to a fast neutron fluence of $6.8 \times 10^{14}$ n/cm$^2$, the radiation-induced increase in resistance was found to fade.

iv) From Figs. II.5 and 7, it can be seen that the immediate pre- and post-irradiation resistance values are greater than those at the start and end of the irradiation. This results from the heating effect of the gamma component of the reactor radiation field, as the resistance of a semiconductor is inversely proportional to temperature.

v) Stepwise fluctuations appear in the resistance/neutron fluence curves, and are due to injection of liquid to maintain a given liquid level in the cryostat.

II.4.2 GaAs diodes

i) The diode forward voltage decreases as a function of absorbed dose.

ii) From Figs. II.13, 15, and 16 it can be seen that, after irradiation, the temperature indicated by the diode is lower than the true value by $\Delta T(K)$. Values of $\Delta T(K)$ are
plotted as a function of fast neutron fluence for a series of true temperatures ranging from 27 K to 250 K, as shown in Fig. II.19. For example, a true temperature of 200 K after irradiation with a fast neutron fluence of $6.8 \times 10^{15}$ n/cm$^2$ would be $(200 - 52)$ K, i.e. 148 K.

iii) Radiation damage is not a function of irradiation at 27 K and 77 K, and the radiation-induced measurement error $\Delta T$(K) tends to saturate after fast neutron fluences in excess of $10^{15}$ n/cm$^2$. Both these phenomena are shown in Fig. II.19.

iv) Figures II.12 and II.14 indicate that the diode forward voltage shows a tendency to be lower in value immediately before and after irradiation than the corresponding values at the start and finish of irradiation. This is again attributed to heating from the gamma component of the reactor irradiation field.

v) As discussed in Section II.2.1, stepwise fluctuations appear in the forward voltage over neutron fluence characteristic.

vi) The effects of a 300 K post-irradiation annealing in the low-temperature range are very small (Fig. II.16).

II.5 CONCLUSIONS

i) Carbon resistors and silicon diodes irradiated at ambient temperature to a fast neutron fluence of $3.6 \times 10^{15}$ n/cm$^2$ and a gamma dose of $5 \times 10^8$ rad or irradiated at 27 K to $8.8 \times 10^{15}$ n/cm$^2$ are suitable devices for measuring liquid-helium levels in cryostats. The radiation-induced changes in the characteristics of these two types of device are small when compared with the differences in resistance and forward voltage when immersed in liquid helium or 10 mm above the liquid surface.

Also, carbon resistors have the additional advantage of being weakly insensitive in a magnetic field up to 5 tesla even in the above-mentioned radiation fields.

ii) Platinum resistors show no change in characteristics, outside that of the manufacturer's specification, when irradiated to the same fluences and doses. They can therefore be used for temperature measurement for cryogenic applications in a radiation area without the need for recalibration after irradiation.

iii) Ge resistors and GaAs diodes used as temperature measuring devices are sensitive to radiation in that their respective calibration curves are functions of absorbed radiation dose. It is therefore necessary to have a dosimetry system in parallel with the temperature measuring system in order to utilize the appropriate calibration curve.

iv) AuFe/chromel thermometers are radiation-stable independent of the applied magnetic field. Their thermoelectric voltage is field-dependent over the temperature range studied (4.2 K to 30 K).
### Table II.1
Characteristics of the radiation sources

<table>
<thead>
<tr>
<th></th>
<th>ASTRA reactor, Vienna</th>
<th>CENG, Grenoble</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>SNIF</strong></td>
<td><strong>Detectors</strong></td>
<td><strong>Detectors</strong></td>
</tr>
<tr>
<td><strong>Irradiation temperature (K)</strong></td>
<td>300</td>
<td>300</td>
</tr>
<tr>
<td><strong>Irradiation medium</strong></td>
<td>Air</td>
<td>Air</td>
</tr>
<tr>
<td><strong>Fast neutron flux</strong> a) (n/cm²/h)</td>
<td>1.5 × 10¹³ (E &gt; 1 MeV)</td>
<td>7.2 × 10¹³</td>
</tr>
<tr>
<td><strong>Gamma dose rate (h)</strong></td>
<td>1.1 × 10⁶</td>
<td>1.0 × 10⁷</td>
</tr>
<tr>
<td></td>
<td>Ni Activation detectors</td>
<td>Ni Activation detectors</td>
</tr>
<tr>
<td></td>
<td>Ionization chamber</td>
<td>Ionization chamber</td>
</tr>
<tr>
<td></td>
<td>Ni Activation detectors</td>
<td>Ni Activation detectors</td>
</tr>
<tr>
<td></td>
<td>Calorimeter graphite absorber</td>
<td></td>
</tr>
</tbody>
</table>

a) Conversion factor: 10¹³ n/cm² (E > 1 MeV) = 3 × 10⁶ CH-rad.

### Table II.2
Components tested

<table>
<thead>
<tr>
<th>Component</th>
<th>Use a)</th>
<th>Supplier</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 Ω, 1/4 W carbon resistor</td>
<td>L, T</td>
<td>Allen Bradley</td>
</tr>
<tr>
<td>Pt resistor, type P3</td>
<td>T</td>
<td>Scientific Instruments, Inc.</td>
</tr>
<tr>
<td>Ge resistor, type N2</td>
<td>T</td>
<td>Scientific Instruments, Inc.</td>
</tr>
<tr>
<td>GaAs diode, type GA-300</td>
<td>T</td>
<td>Scientific Instruments, Inc.</td>
</tr>
<tr>
<td>Si diode Class 1</td>
<td>L</td>
<td>Silec</td>
</tr>
<tr>
<td>AuFe/chromel thermocouple</td>
<td>T</td>
<td>Johnson-Matthey</td>
</tr>
</tbody>
</table>

a) L = liquid-He level indicator  
   T = temperature indicator

### Table II.3
Radiation damage suffered by Si diodes

<table>
<thead>
<tr>
<th>Condition</th>
<th>Forward voltage at 4.2°K</th>
<th>Forward voltage 10 mm above liquid-He surface</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zero radiation</td>
<td>≈ 1.6 V</td>
<td>≈ 0.7 V</td>
</tr>
<tr>
<td>3 × 10¹⁴ n/cm² (SNIF)</td>
<td>7.0% decrease</td>
<td>15% decrease</td>
</tr>
<tr>
<td>10⁷ rad (Ebene 1)</td>
<td>0.5% decrease</td>
<td>5% decrease</td>
</tr>
</tbody>
</table>
BIBLIOGRAPHY TO CHAPTER II


F. Pavese and S. Limbarini, Accuracy of gallium arsenide diode thermometer in the range 4-300 K, in Temperature, its measurement and control in science and industry (Instrument Society of America, Pittsburgh, 1972), Vol. 4, p. 1103.
Figs. II.1 to II.11  Change in the resistance of different resistor samples

Fig. II.1 As a function of temperature: carbon resistor in Ebene l irradiated to $5.0 \times 10^8$ rad and $3.6 \times 10^{15}$ n/cm$^2$, at ambient temperature

Fig. II.2 As a function of temperature: platinum resistor in Ebene l irradiated to $5.0 \times 10^8$ rad and $3.6 \times 10^{15}$ n/cm$^2$, at ambient temperature

Fig. II.3 As a function of irradiation time: carbon resistor irradiated by $4.5 \times 10^7$ rad/h and $2.8 \times 10^{14}$ n/h, at 27 K

Fig. II.4 As a function of temperature: carbon resistor irradiated to $8.8 \times 10^{15}$ n/cm$^2$, at 27 K
Fig. II.5 As a function of fast neutron fluence: 
germanium sample A, with an integrated fast neutron 
fluence of $4.1 \times 10^{14}$ n/cm$^2$, at 27 K

Fig. II.6 As a function of temperature: 
germanium sample A, same conditions as Fig. II.5

Fig. II.7 As a function of fast neutron fluence: 
germanium sample B, with an integrated fast neutron 
fluence of $4.1 \times 10^{14}$ n/cm$^2$, at 77 K

Fig. II.8 As a function of temperature: 
germanium sample B, same conditions as Fig. II.7
Fig. II.9 As a function of temperature: germanium sample C, with an integrated fast neutron fluence of $1.4 \times 10^{16}$ n/cm$^2$, at 77 K

Fig. II.10 As a function of temperature: germanium sample D, with an integrated fast neutron fluence of $6.8 \times 10^{15}$ n/cm$^2$, at 77 K

Fig. II.11 As a function of temperature: germanium sample E, with an integrated fast neutron fluence of $1.4 \times 10^{16}$ n/cm$^2$, at 77 K
Figs. II.12 to II.15 Decrease in the forward voltage of GaAs diode samples A (at 27 K) and B (at 77 K). The integrated fast neutron fluence is $4.1 \times 10^{15}$ n/cm$^2$.

**Fig. II.12** As a function of fast neutron fluence: sample A

**Fig. II.13** As a function of temperature: sample A

**Fig. II.14** As a function of fast neutron fluence: sample B

**Fig. II.15** As a function of temperature: sample B
Fig. II.16 Decrease in the forward voltage of the GaAs diode sample C (at 77 K) as a function of temperature. The integrated fast neutron fluence is $6.8 \times 10^{14}$ n/cm$^2$.

Fig. II.17 Change in the thermoelectric voltage as a function of temperature for a AuFe/chromel thermocouple. The reference temperature is 4.2 K.

Figs. II.18 and II.19 Radiation-induced error in the temperature measurement of samples as a function of fast neutron fluence. • At an irradiation temperature of 77 K; × At an irradiation temperature of 27 K.

Fig. II.18 Ge resistor sample

Fig. II.19 GaAs diode sample
CHAPTER III

EFFECTS OF RADIATION ON COPPER

M. Couach
Centre d'Etudes Nucléaires de Grenoble, France

and

M. Van de Voorde
CERN, Geneva, Switzerland
III.1 INTRODUCTION

The effects of nuclear radiation on some coppers of interest for the electrotechnical industry have been studied.

The irradiations were performed either in liquid nitrogen or liquid neon, and the electrical resistance was measured at 4.2 K as a function of the magnetic field.

Section III.2 gives a description of the measuring techniques and irradiation conditions, and an analysis of the results is given in Section III.3.

III.2 MEASURING TECHNIQUES AND IRRADIATION CONDITIONS

III.2.1 Copper types studied

Table III.1 shows the characteristics of the copper types studied.

III.2.2 Sample preparation

In order to minimize induced-radioactivity problems, samples of about 0.4 mm diameter and 1 m length, weighing 1.5 to 2.0 g, were selected. This allowed five or six samples to be irradiated simultaneously in one container.

The dimensions and layout of the sample holder are shown in Fig. III.1. One metre of wire is wound on the spirally grooved hollow holder, which is made from the aluminium alloy A9G3, and the tension wires are soldered to the sample with indium.

The fast neutron detector, in the form of a ring, is placed between the sample holder and the control arm.

The complete preparation and mounting of the irradiated samples has been carried out in liquid nitrogen or liquid neon followed by transfer to the liquid-helium cryostat without reheating (< 1 K). In the case of mounting the sample in liquid neon, it was necessary to use a glove box to avoid condensation of solid air and also to recuperate the neon gas which is an expensive product. Therefore special instrumentation was necessary to transfer the sample from the glove box to the helium cryostat without re-heating above 27 K. (Fig. III.2 and Ref. III.1.)

The initial resistivity values at 4.2 K and the ratio $\rho_{290K}/\rho_{4.2K}$ without magnetic field for the various coppers measured are shown in Table III.2.

The chemical composition is given in Table III.1.

III.2.3 Measuring technique and experimental errors

The resistance of the wire in coil form and in a transverse magnetic field was measured by the four-wire method. This method overcomes the problems of resistance in the leads and contacts because the unknown resistor is set up as a four-terminal resistor, supplied with separate current and potential leads.

All resistance measurements were made at 4.2 K. The sample placed in the middle of the superconducting coil, which has a field homogeneity of 0.5% over a 10 mm diameter sphere, is fed by a current of 1 A stabilized at $10^{-9}$. The tension is read directly from a digital voltmeter, which has a precision of 1 $\mu$V.
To ensure that the remanent field caused by the induction coil had negligible effect on the resistivity measurements, each sample (non-irradiated and irradiated) was first measured at H = 0 up to H = 8 and then re-measured at zero field. The absolute resistivity measurements were made with a "Δρ/ρ" precision better than 1%. This required a wire of 1 m length and a diameter of 400 ± 0.1 μ.

III.2.4 Irradiation conditions - Dosimetry

The copper wires have been irradiated in the Melusine reactor hole BT28, the layout of which is shown in Fig. I.1. The irradiations were performed in either liquid nitrogen or liquid neon.

Nickel (σ = 156 mb) and cobalt (σ = 38 b) detectors were used for measuring the fast (E > 1 MeV) and thermal neutrons, respectively.

The neutron flux for the fast neutrons was about $1.2 \times 10^{13}$ n/cm² and for the thermal neutrons about $4.1 \times 10^{13}$ n_th/cm². The integrated fast neutron fluences for the exposed coppers are shown on the corresponding curves. In principle, two dose-levels were aimed at, namely $1 \times 10^{17}$ n/cm² and $5 \times 10^{17}$ n/cm².

The gamma dose, measured with a calorimeter containing a graphite probe, was $3.1 \times 10^8$ rad and $1.6 \times 10^9$ rad in the two experiments, respectively.

II.3.3 RESULTS

The results are summarized in the figures, which are self-explanatory.

i) Figures III.3 and 4 show the relative changes in resistivity at 4.2 K as a function of the magnetic field and for two doses ($10^{17}$ and $5 \times 10^{17}$ n/cm²)

$$\frac{\Delta \rho}{\rho_0} = f(H)$$

\[\Delta \rho = \rho_{irr}(H) - \rho_0(H) \quad \text{irr: irradiated} \]
\[0: \text{before irradiation} \]

where Δρ is a direct measurement at 4.2 K of the damage created by the fast neutrons at the irradiation temperature, and ρ₀ the initial value of the copper considered.

Figure III.5 gives the radiation-induced changes in resistivity as a function of the magnetic field for coppers irradiated in liquid nitrogen and liquid neon.

It can be seen that:
- Δρ/ρ₀ decreases with the magnetic field for all irradiated coppers;
- the decrease is linear for the cold-worked coppers;
- at 8 tesla the changes in all coppers irradiated at 77 K are only between 10 and 20;
- Δρ/ρ₀ increases with the exposure dose for all measured fields;
- thermally annealed coppers are much more sensitive to radiation damage than cold-worked ones;
the induced changes are much more pronounced in the liquid-neon irradiations, particularly at low field levels, and we have $\left[ (\Delta \rho / \rho_0)_{27K} / (\Delta \rho / \rho_0)_{77K} \right] \sim 2$ for a given type of copper and at constant field and dose.

ii: The variation of the magnetoresistivity

$$M(H) = \frac{\rho(H) - \rho(O)}{\rho(O)}$$

is shown in Figs. III.6 to 11 as a function of the magnetic field, for coppers irradiated at 77 K. Figures III.12 and 13 show the effect of irradiation temperature (LNe against LN$_2$) on the magnetoresistivity of two copper types. For a given type of copper, for a given field, and for the same dose, we also have

$$\frac{\left[ \rho(H) - \rho(O) \right]}{\rho(O)}_{77K} \sim 2.$$  

At a fixed field, a log log plot of the magnetoresistivity versus absolute resistivity shows a linear dependence in the resistivity range between $10^{-8}$ and $10^{-7}$ $\Omega \cdot$cm for the coppers studied (irradiated or not) (Fig. III.14). This leads to $\left[ \rho(H) - \rho(O) \right] / \rho(O) \sim \rho(O)^{-n}$, where the exponent $n$ is given by the slope of the log log plot; $n$ appears to be a decreasing function of $H$. The magnetoresistivity decreases with radiation exposure dose, while absolute resistivity increases. So the representative point of a given copper tends to shift to the lower right on the universal curve $\left[ \rho(H) - \rho(O) \right] / \rho(O)$ as a function of $\rho(O)$ for a given field (Fig. III.15).

Another point we can mention is related to the magnetoresistance of some thermally annealed non-irradiated coppers; a linear plot versus applied field gives a straight line over the range of field 1-8 teslas. This has been mentioned by Fickett (Ref. III.2) for the purity range 7000 < RRR < 200 *), and so our results are in agreement; the bending of the curve at low fields seems to be opposite.

iii: The relative changes in magnetoresistivity $\Delta M/M_0$ with irradiation and with post-irradiation annealing at 300 K as a function of the magnetic field are given in Figs. III.16 to 21 for coppers irradiated at 77 K and 27 K. $\Delta M = M_{irr}(H) - M_0(H)$, where the index $irr$ means irradiated, and the index 0 means before irradiation.

These figures show that:

- $\Delta M/M_0$ diminishes with irradiation but re-increases with annealing;
- $\Delta M/M_0$ for irradiated and post-irradiated annealed coppers is hardly affected by magnetic field.

III.4 ANALYSIS AND COMMENTS

All the technological information about the impact of low-temperature neutron irradiation of the different coppers studied is contained in the figures where $\Delta \rho / \rho_0$,

$$M = \left[ \rho(H) - \rho(O) \right] / \rho(O),$$

and $\Delta M/M_0$ are plotted versus magnetic field after irradiation and annealing at 300 K.

*) RRR = Residual Resistance Ratio = $R(273 \, K)/R(4 \, K)$. 

*
It is interesting to point out the effect of irradiation temperature (77 or 27 K) upon \( \Delta \rho/\rho \) and \( [\rho(H) - \rho(O)]/\rho(O) \). Figure III.5 shows that \( (\Delta \rho/\rho)_{27K} \) is higher by a factor of 2 than \( (\Delta \rho/\rho)_{77K} \) whatever the copper (OHHC or 99.999) and the value of the magnetic field. Figures III.12 and III.13 show that \( [\rho(H) - \rho(O)]/\rho(O) \) \( \rho_{27K} \) is lower by a factor of 2 than \( [\rho(H) - \rho(O)]/\rho(O) \) \( \rho_{77K} \) in the same conditions as mentioned previously.

At this stage we can ask, What will happen if the irradiation is made at 4.2 K? We can give a partial answer by pointing out that in the stage I of the recovery spectrum of \( \Omega \) after 4.2 K irradiation, the stage ID (recombination of non-interacting interstitials and vacancies) is the most important and occurs in the temperature range 30-40 K whatever the particle used (fast neutron or electron) (Ref. III.3); moreover, in the particular case of fast neutrons, the damage produced at 27 K (deduced from annealing experiments of copper irradiated at 4.2 K) differs only by around 6% (Ref. III.3), that is to say

\[
\frac{\Delta \rho_{27K}}{\Delta \rho_{4.2K}} = 0.94,
\]

for an initial damage \( \Delta \rho_0 = 1.86 \, \text{m\Omega}\cdot\text{cm}; \) in our case the damage produced at 27 K and measured at 4.2 K is much more important (\( \Delta \rho_0 = 63 \, \text{m\Omega}\cdot\text{cm} \) for OHHC and 99.999 copper after \( \approx 5 \times 10^{17} \, \text{n cm}^{-2} \)).

Nevertheless, in the case of copper, we would have an idea of the damage produced by neutron irradiation at 4.2 K by making the same experiment at 27 K.

From another point of view, it is possible from this study to have some insight into the fundamental behaviour of the irradiated copper by comparing our results with those of other workers. It is useful to analyse and discuss the experimental results obtained with these commercial coppers after several treatments (type of irradiation, annealing) in terms of Kohler's rule. This rule states that the magnetoresistance \( [\rho(H) - \rho(O)]/\rho(O) \) is a function of \( H/\rho(O) \), i.e.

\[
\frac{\rho(H) - \rho(O)}{\rho(O)} = f \left( \frac{H}{\rho(O)} \right),
\]

where the function \( f \) is independent of the concentration of the scattering centres and thus of the residual resistance ratio as long as one type of scattering is significant.

Firstly, a Kohler plot for all the samples of the six types of copper studied, before and after irradiation and after annealing at 300 K (an amount of 300 experimental points), shows that the Kohler rule is well obeyed for copper and for the purity range \( \text{RRR} < 300 \). Fickett (Ref. III.4) has also shown that copper obeys Kohler's rule over the range of field (0-10 teslas) and over the purity range \( 200 \leq \text{RRR} \leq 7000 \); evidently a deviation exists, as shown by Fig. III.22; the maximum deviation from the mean line is about 10%.

So the Kohler rule in this case appears to be a very useful law for knowing the magnetoresistance of a given copper.

But the Kohler plot is also very useful for investigating more precisely the behaviour of these coppers after low-temperature irradiation. For this purpose and to avoid the deviation observed on several experimental points of various samples, it is better to make the Kohler plot for one sample and to see how it is modified by irradiation.
All the results are summarized in Figs. III.23-31. After examination it appears that
a) all the coppers studied obey very well the Kohler rule for the three states:
   before irradiation;
   after $10^{17}$ n/cm$^2$ irradiation at 77 K;
   after annealing at 300 K (Figs. III.23 and 24).

b) After 77 K irradiation at a dose near $5 \times 10^{17}$ n/cm$^2$ some deviations appear:
   after such an irradiation only the copper 99.999 obeys the Kohler rule (Fig. III.25).
   For all the annealed copper, we observed a positive deviation, i.e. for a given
   $H/\rho(0)$ the magnetoresistance is higher after irradiation than before (Figs. III.26
   and 27); but for cold-drawn samples we observed an opposite behaviour; the
   deviation is negative (Figs. III.28 and 29).

c) After 77 K irradiation at $5 \times 10^{17}$ n/cm$^2$ and annealing at 300 K, the general tend-
   dency for all the coppers studied is an increase of magnetoresistance for a given
   $H/\rho(0)$:
   - for cold-drawn samples this means that the curves before irradiation and after
     annealing are the same (Figs. III.28 and 29);
   - for annealed copper the plots after irradiation and annealing are nearly the
     same except for copper 99.999 (Fig. III.25).

d) After 27 K irradiation at a dose of $5 \times 10^{17}$ n/cm$^2$,
   - the behaviour of copper 99.999 is the same as previously mentioned. The plots
     before and after irradiation are the same. A positive deviation appears with
     annealing at 300 K (Fig. III.30).
   - For annealed OFHC (Fig. III.31), the plots of irradiated and annealed are nearly
     the same but tend to separate with a higher value of magnetoresistance for the
     annealed sample.

It is interesting to compare the behaviour observed for copper after irradiation and
annealing, with previous work done on this subject by several workers (Refs. III.5 and II.6)
by means of longitudinal magnetoresistance. Their experiments were conducted on very pure
polycrystalline copper (99.999 annealed two hours at 600°C) (Ref. III.5). Neutron irradi-
a tion made at 4 K with a dose of $3 \times 10^{18}$ n/cm$^2$ shows an increase of the magnetoresistance
for a given $H/\rho(0)$.

It increases very slightly between irradiation and annealing at $T < 200$ K, but the in-
crease is very strong for an annealing at 300 K. This strong increase appears during
stage III (150-300 K) and is related by Winkler to the formation of dislocation loops re-
sulting from defect reactions.

We observe the same tendency with our annealed coppers, especially for copper 99.999; furt hernore, an irradiation made at 27 K with a dose of $6 \times 10^{18}$ n/cm$^2$ (Ref. III.7) has
shown that the behaviour previously outlined is quite general; we have to note that in our
case (Fig. III.32) the plots before and after irradiation are nearly the same (the irradia-
ted curve is slightly higher than the non-irradiated one). Thus this stabilizer behaves
much more like an annealed OFHC copper than a cold-drawn one; this fact is also clearly demonstrated by its absolute resistivity, \( \rho_0 = 2.6 \times 10^{-8} \ \Omega \cdot \text{cm} \), close to that of an annealed copper (\( \rho_0 = 1.9 \times 10^{-8} \ \Omega \cdot \text{cm} \)). The previous workers have found (Ref. III.6) that a very pure copper RRR = 714, monocristalline or not, obeys perfectly the Kohler rule after irradiation at 4.2 K; that is to say, the irradiated and non-irradiated curves are the same. So we can interpret the small gap lying between our two curves as an effect of interaction between irradiation defects and pre-existing ones. In fact, these workers have shown that the magnetoresistance is sensitive to the scattering anisotropy of the scattering centres.

Thus the results obtained at 27 K at low and high doses (\( 5 \times 10^{17} \) and \( 6 \times 10^{18} \) n/cm\(^2\)) are in agreement and lead to the same conclusions. We have also shown that cold-drawn copper shows an irradiated behaviour quite different from that of annealed copper; the effect of annealing at 300 K is the same for both.
Table III.1
Copper types studied

<table>
<thead>
<tr>
<th>Type of copper</th>
<th>Condition of wire</th>
<th>Composition</th>
<th>Preparation treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>OHC *)</td>
<td>Annealed</td>
<td>99.99% Cu, no oxygen</td>
<td>Cold-worked from 7 mm to 0.85 mm. Annealed. Cold-drawn from 0.85 mm to 0.4 mm. Annealed at 300°C.</td>
</tr>
<tr>
<td>Cu 99.999 **)</td>
<td>Annealed</td>
<td>99.999% Cu</td>
<td>Annealed (Ø 1.5 mm) 1 h at 300°C. Drawn from 1.5 mm to 0.4 mm. Annealed 1 h at 300°C.</td>
</tr>
<tr>
<td>Cu-OF **)</td>
<td>Annealed</td>
<td>99.99% Cu, O₂: 3 ppm, Sb: 5, Fe: 7, Pb: 3, Se: 3, S: 11, Ag: 14, Na: 4, As: 2</td>
<td>&quot;</td>
</tr>
<tr>
<td>OHC ***</td>
<td>Hard</td>
<td>Cu 99.99% Cu, P &lt; 0.00003%, S &lt; 0.0015, Zn &lt; 0.0001, Cd &lt; 0.0001, Hg &lt; 0.0001, [Bi + Pb + Se + Te + O] &lt; 0.0010% min., [Bi + Sn + Mn + Sb + As + Se + Te] &lt; 0.0040%.</td>
<td>Extruded to 8 mm Ø. Drawn through several stone diameters to 0.4 mm.</td>
</tr>
<tr>
<td>ETP **)</td>
<td>Annealed</td>
<td>99.96% Cu, O₂: 230 ppm, Fe: 19, Pb: 0.5, Se: 0.3, S: 7, Ag: 7, Bi: 0.2</td>
<td>Annealed (Ø 1.5 mm) 1 h at 300°C. Drawn from 1.5 mm to 0.4 mm. Annealed 1 h at 300°C.</td>
</tr>
<tr>
<td>ETP **)</td>
<td>Hard</td>
<td>99.96% Cu, O₂: 230 ppm, Fe: 19, Pb: 0.5, Se: 0.3, S: 7, Ag: 7, Bi: 0.2</td>
<td>Annealed (Ø 1.5 mm) 1 h at 300°C. Drawn from 1.5 mm to 0.4 mm.</td>
</tr>
</tbody>
</table>

OHC (oxygen-free, high-conductivity copper) and ETP (electrolytic tough-pitch copper) produced by:
*) Treffmetaux, France.
**) Schweizerische Metallwerke, Selve, Switzerland.
***) Outonmyon, Oy, Finland.
Table III.2

Initial resistivity of copper samples

<table>
<thead>
<tr>
<th>Copper type</th>
<th>Initial resistivity (Ω cm) 4.2°K</th>
<th>$\rho_{290K}/\rho_{4.2K}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>OFHC (annealed)</td>
<td>$1.926 \times 10^{-8}$</td>
<td>80.6</td>
</tr>
<tr>
<td>Cu 99.999 (annealed)</td>
<td>$0.608 \times 10^{-8}$</td>
<td>262.1</td>
</tr>
<tr>
<td>Cu-OF (annealed)</td>
<td>$1.953 \times 10^{-8}$</td>
<td>93.3</td>
</tr>
<tr>
<td>OFHC (hard)</td>
<td>$7.787 \times 10^{-8}$</td>
<td>22.2</td>
</tr>
<tr>
<td>ETP (annealed)</td>
<td>$1.083 \times 10^{-8}$</td>
<td>182.1</td>
</tr>
<tr>
<td>ETP (hard)</td>
<td>$4.067 \times 10^{-8}$</td>
<td>37.6</td>
</tr>
</tbody>
</table>
REFERENCES TO CHAPTER III


III.7 M. Couach, private communication (see Fig. III.32).
Figs. III.3 and III.4 Radiation-induced changes in electrical resistivity versus magnetic field for various coppers irradiated in liquid nitrogen
Fig. III.5 Radiation-induced changes in electrical resistivity versus magnetic field for various coppers irradiated in liquid neon and nitrogen

Figs. III.6 to III.13 Changes in magnetoresistivity versus magnetic field for different types of copper

Fig. III.6 OFHC (annealed)

Fig. III.7 Cu 99.999 (annealed)
Fig. III.8 Cu-OF (annealed)

Fig. III.9 OFHC (hard)

Fig. III.10 ETP copper (annealed)

Fig. III.11 ETP copper (hard)
Fig. III.12  Cu 99.999 (in LN₂ and LNE)

Fig. III.13  OFHC (annealed; in LN₂ and LNE)

Fig. III.14  Variation of magnetoresistivity versus resistivity for different fields and copper types

Fig. III.15  Variation of magnetoresistivity versus radiation dose and resistivity for a field of 8 teslas and six copper types
Figs. III.16 to III.19: Relative changes in magnetoresistivity versus radiation, annealing and magnetic field for different types of copper.

Fig. III.16: OPHC (annealed)

Fig. III.17: Cu 99.999 (annealed)

Fig. III.18: Cu-OF (annealed)

Fig. III.19: OPHC (hard)
Fig. III.20 ETP copper (annealed)

Fig. III.21 ETP copper (cold worked)

Fig. III.22 Kohler plot for all samples of the six types of copper studied
Figs. III.23 to III.31 Kohler plot for the different samples of copper

Fig. III.23 Sample 1A of OFHC (annealed)

Fig. III.24 Sample 2A of Cu 99.999 (annealed)

Fig. III.25 Sample 2B of Cu 99.999 (annealed)

Fig. III.26 Sample 1B of OFHC (annealed)
Fig. III.27 Sample 3B of Cu-OF (annealed)

Fig. III.28 Sample 6B of ETP copper (cold drawn)

Fig. III.29 Sample 4B of OFHC (cold drawn)

Fig. III.30 Cu 99.999 (annealed)
Fig. III.31 OFHC (annealed)

Fig. III.32 Kohler plot for the stabilizer (copper $\rho = 2.68 \times 10^{-8} \, \Omega \, \text{cm}$) of the superconducting wire Vacryflux 6501
CHAPTER IV

LOW-TEMPERATURE BEHAVIOUR OF
ORGANIC MATERIALS IN A RADIATION FIELD

E. Bonjour, P. Brauns, R. Lagnier
Centre d'Etudes Nucléaires de Grenoble, France

and

M. Van de Voorde
CERN, Geneva, Switzerland
IV.1 INTRODUCTION

Polymers and composites are used either as electrical insulators or as constructional materials, e.g. clamps, spacers, etc., in superconducting magnet technology.

Every possible aspect of their physical properties during irradiation at low temperature must be well known before they can be used for magnets in high-energy physics.

In fact, polymer materials are much more sensitive to irradiation than are metals and alloys. Hence, it has mainly been these materials which have been studied in a program concentrating on the behaviour of their mechanical and thermal properties.

IV.2 THERMAL CONDUCTIVITY

IV.2.1 Experimental procedure

The thermal conductivity measurements have been done by a classical constant gradient method in the measurement cell which is schematized in Fig. IV.1. The samples are in the form of cylinders, 10 mm in diameter and 30 mm long.

A longitudinal gradient is created by a heater at the bottom of the sample (see Fig. IV.2 and measured between two thermal copper plugs which are fixed in 0.6 mm holes, bored perpendicular to the axis of the samples and 5 mm from the middle plane. Also, junctions of gold-iron/chromel thermocouples and little copper supporting tubes for carbon or platinum thermal resistance sensors are soldered on these plugs.

The upper end of the sample is fixed on a holder maintained at constant temperature by a control system. A heater (strain-gauge) is fixed to the bottom end of the sample applying thermal power (W) and establishing thermal flow in the sample. Around this is a shield which is also heated at the bottom so that it has a temperature profile similar to that along the sample, hence minimizing radial thermal radiation losses. Other thermal losses are suppressed by maintaining a good vacuum (10^{-5} Torr) in the measurement cell. This cell can be immersed in cryogenic liquids (either liquid nitrogen or liquid helium) or refrigerated by their vapours. Details of the experimental technique is given elsewhere (Ref. IV.1).

IV.2.2 Results

Taking:

\[ L \] = distance between the thermal plugs \( F_1 \) and \( F_2 \) on sample
\[ D \] = diameter of sample
\[ S \] = sample cross-sectional area
\[ W \] = longitudinal thermal flux
\[ T_1 \] = \( F_1 \) temperature
\[ T_2 \] = \( F_2 \) temperature
\[ T \] = mean sample temperature \( [ (T_1 + T_2)/2 ] \)
\[ V \] = thermocouple e.m.f.
\[ V_0 \] = value of \( V \) when \( W = 0 \)
\[ P(T) = \text{thermoelectric power of thermocouple at temperature } T \]
\[ K(T) = \text{thermal conductivity of sample at temperature } T \]

one can write:

\[ T_2 - T_1 = \frac{V - V_0}{P} \]

\[ T = \frac{T_1 + T_2}{2} = T_1 + \frac{T_2 - T_1}{2} = T_1 + \frac{V - V_0}{2P} \]

and

\[ K(T) = \frac{L}{S} \frac{W}{T_2 - T_1} = \frac{L}{S} \frac{W \times P}{(V - V_0)} . \]

Since \( P \) varies with \( T \), \( T = T_1 + [(V - V_0)/2P] \) is calculated by successive approximations either from tables or with the aid of a computer.

A detailed discussion of the incertitude of the measurements is also given in Ref. IV.1. Finally, it appears that experimental precision is better than 3%.

The results of the thermal conductivity versus temperature measurements are given in Figs. IV.3 to IV.14.

**IV.3 SPECIFIC HEAT**

**IV.3.1 Experimental procedure (Refs. IV.2 and IV.3)**

Specific heat was measured using a differential adiabatic calorimeter (power compensated by using linear-rise temperature control) over the range 4.2 K to 300 K. The principle, schematized in Fig. IV.15, is to balance exactly the electrical heating power necessary to raise, at the same speed, the temperature of a reference cell and one containing the sample. The difference is then directly recorded and corresponds to the heat capacity of the sample.

The calorimeter cell (Fig. IV.16) is maintained under high vacuum (less than \( 10^{-7} \) Torr) by means of a miniature cryopump placed inside. There are two thermal radiation shields, the second one being linearly temperature-controlled.

The two sample holders are inside this second shield, i.e. the reference holder (usually empty) and the sample holder. Thermal coupling between the sample and the walls of the sample holder is provided by means of a partial pressure of helium gas.

A gold-iron/chromel thermocouple placed on the temperature-controlled shielding allows a linear temperature rise from liquid-helium temperature to be recorded on a functional potentiometer. The differential thermocouples fixed between this shielding and the sample holders allow the temperature to be maintained accurately between shielding and sample holders throughout the programmed temperature rise.

Hence the difference \( (\Delta P) \) between the recordings of the electrical power heating supplied \( (P_1 + P_2) \) to each of the sample holders is the direct heat capacity of the sample.
(i.e. \( \Delta P = P_2 - P_1 \)), and if one of the two holders is empty, then the specific heat

\[
C_p = \frac{\Delta P}{mV},
\]

where \( m \) = specific gravity of the sample and \( V \) = its volume.

The precision of the measurement depends on:

- the inaccuracy of the temperature value, which is estimated to be \( \pm 0.2 \) K between 4 K and 50 K, and \( \pm 0.4 \) K from 50 K to 300 K;

- the inaccuracy in \( \Delta P \), which is the difference between two power measurements and is estimated to be about \( \pm 2\% \) in the present case.

Therefore, the total error in specific heat measurements could be about \( \pm 3\% \).

IV.3.2 Results

The specific heat versus temperature has been studied for various materials, and in order to check a possible effect of thermal diffusivity at very low temperature each sample was heated at 0.5 K/min and 1 K/min but no systematic difference was found between the two rates of heating.

The results of some of the measurements are shown in Figs. IV.17 to IV.29.

IV.4 MECHANICAL PROPERTIES: FLEXURAL TESTS

IV.4.1 Test method

The samples are in the form of plates \((70 \times 10 \times 2 \text{ to } 5 \text{ mm})\) and their flexural strength is measured by the four-point loading system (Figs. IV.30 and 31). The distance between the external and internal supporting points is 60 mm and 30 mm, respectively. Bending of the sample is detected by a rod transmitting its displacement to a differential transformer.

This support is fixed in a special cryostat which is used for tensile, compression, or flexion tests at liquid nitrogen, neon, or helium temperatures according to the type of sample holder and cryogenic liquid used. It is schematically represented in Fig. IV.32. The cryostat is fitted on a 5-ton Instron TTOML tensile testing machine. The load can be accurately recorded to 5000 kg and the rate of cross-bar displacement is 2 mm/min (Ref. IV.4).

IV.4.2 Results

Stress \((\sigma)\) and strain \((\epsilon)\) at the surface of the samples, where they have maximum values, are calculated from the applied force \((F)\) and sag \((f)\) of the sample (Fig. IV.33), using the formula:

\[
\sigma = \frac{3}{2} \frac{F \, (b - a)}{Ce^2}
\]

\[
E = \frac{\sigma}{\epsilon} = \frac{3L^2 \, (b - a)}{2Ce^3 \, f}.
\]
where \( a \) = distance between internal supporting points
\( b \) = " " external " "
\( C \) = sample width
\( e \) = sample thickness
\( E \) = modulus of elasticity,

The results of the experiments are summarized in Figs. IV.34-IV.45.

IV.5 **ANALYSIS OF DATA**

1. Irradiation at low temperature seems to reduce the flexural strain of polymers. Many polymers have a radiation threshold of \( 10^8 \) rad. The Young's modulus is much less affected by radiation.

2. Glass-reinforced epoxy resins withstand \( 10^3 \) rad.

3. The thermal properties of polymers are not influenced by radiation at low temperatures as long as the mechanical strength of the material is good.

4. The thermal properties of all polymers studied fall with decreasing temperature. The specific heat is particularly temperature-sensitive; it falls by more than two orders of magnitude between 300 and 4.2 K.

5. Polymers commonly used for high-temperature applications (\( > 200^\circ \text{C} \)) also seem to have the most suitable physical properties at very low temperatures.
REFERENCES TO CHAPTER IV

IV.1  P. Brauns, Appareil de mesure de conductibilité thermique entre 4 et 300 K, CENG-Service des Basses Températures, Internal note SBT 320/73, October 1973.

IV.2  R. Lagnier, Etude, réalisation et essais d'un dispositif de détermination de chaleur spécifique en mesure dynamique de 4 à 300 K, CENG-Service des Basses Températures, Report CEA R-4419 (1973).


Fig. IV.1 Thermal conductivity measurement cell

Fig. IV.2 Sample wiring diagram

Figs. IV.3 to IV.14 Thermal conductivity versus temperature for different organic materials

Fig. IV.3 Polyurethane (Adiprene)

Fig. IV.4 Polyvinylchloride (PVC)
Fig. IV.5 Araldite D + HY 951 (100-11 phr)

Fig. IV.6 Cast epoxy resin (Araldite B + HY 901) (100-30 phr)

Fig. IV.7 Araldite B + HY 901 (100-90 phr)

Fig. IV.8 Polyimide (Vespel)
Fig. IV.9 Polyphenylene oxide (PPO)

Fig. IV.10 Vetonite

Fig. IV.11 Vetonite

Fig. IV.12 Aluminium oxide (Al$_2$O$_3$)
Fig. IV.13 Brass

Fig. IV.14 ETP copper, OPHC (Grade II), phosphorus deoxidized copper (low residual phosphorus)
Figs. IV.15 to IV.29  Specific heat versus temperature for different organic materials
Fig. IV.19 Polytetrafluoroethylene (teflon) (irradiated to $10^9$ rad)

Fig. IV.20 Polyethylene (RCH-1000) (non-irradiated)

Fig. IV.21 Polyethylene (RCH-1000) (irradiated to $10^8$ rad)

Fig. IV.22 Epoxy resin (Araldite D + Hf 951) (non-irradiated)
Fig. IV.23 Epoxy resin (Araldite D + HY 951) (irradiated to $10^8$ rad)

Fig. IV.24 Epoxy (F + HY 905 + DY 040)

Fig. IV.25 Epoxy resin (Araldite F + D 230)

Fig. IV.26 Filled epoxy (Araldite F + HY 906)
Fig. IV.27 Glass-reinforced epoxies (Vetronite EPG-II)

Fig. IV.28 Aluminium oxide ($\text{Al}_2\text{O}_3$)

Fig. IV.29 Copper 5N
Fig. IV.30 Schematic diagram of the flexural test system (4 supporting points)

Fig. IV.31 Diagram of a sample

Fig. IV.32 Cryostat for mechanical tests

Fig. IV.33 Schematic diagram of forces
Figs. IV.34 to IV.45  Effect of radiation, at 77 K, on the flexural properties of different organic materials

Fig. IV.34  Polyurethane (Adiprene L 100)

Fig. IV.35  Unfilled epoxy resin (MY 745 + EPN 1139 + HY 905 + DY 063)

Fig. IV.36  Filled epoxy resin (Stycast 2850)

Fig. IV.37  Epoxy resin (Epikote 171 NMA BDMA)
Fig. IV.38 Epoxy resin (Epikote 828 DDM Epikote 154)

Fig. IV.39 Polyethylene terephthalate (Ertalyme)

Fig. IV.40 Polyphenylene oxide (Ertaphenyl)

Fig. IV.41 Polyethylene (RCH 1000)
Fig. IV.42 Polytetrafluoroethylene (PTFE)

Fig. IV.43 Polymethyl methacrylate (Flexiglas)

Fig. IV.44 Acrylonitrile-butadiene-styrene (Novodur BGV)

Fig. IV.45 Polypropylene (Ertalene)