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Within the framework of the Large Hadron Collider (LHC) R&D programme, CERN and the Department of Physics E21 of the Technical University Munich have established a collaboration to carry out irradiation experiments at liquid helium and liquid nitrogen temperatures on epitaxial diodes for the superconducting magnet protection.

Small diode samples of 10 mm wafer diameter from two different manufacturers were submitted to doses of up to 50 kGy and neutron fluences up to 10^{15} n/cm² and the degradation of the electrical characteristics was measured versus dose. During irradiation the diodes were submitted to current pulse annealing and after irradiation to thermal annealing. After exposure some diodes show a degradation in forward voltage drop of up to 600 % which, however, can be reduced to about 15 % - 20 % by thermal annealing. The degradation at liquid helium temperature is very similar to the degradation at liquid nitrogen temperature.

These degradations of electrical characteristics during the short term irradiation in a nuclear reactor are compared with degradations during long term irradiation in an accelerator environment at liquid nitrogen temperature.

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IRRADIATION OF QUENCH PROTECTION DIODES AT CRYOGENIC TEMPERATURES IN A NUCLEAR RESEARCH REACTOR

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ABSTRACT

Within the framework of the Large Hadron Collider (LHC) R&D programme, CERN and the Department of Physics E21 of the Technical University Munich have established a collaboration to carry out irradiation experiments at liquid helium and liquid nitrogen temperatures on epitaxial diodes for the superconducting magnet protection.

Small diode samples of 10 mm wafer diameter from two different manufacturers were submitted to doses of up to 50 kGy and neutron fluences up to 10^{15} n/cm² and the degradation of the electrical characteristics was measured versus dose. During irradiation the diodes were submitted to current pulse annealing and after irradiation to thermal annealing. After exposure some diodes show a degradation in forward voltage drop of up to 600 % which, however, can be reduced to about 15 % - 20 % by thermal annealing. The degradation at liquid helium temperature is very similar to the degradation at liquid nitrogen temperature.

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INTRODUCTION

Irradiation tests at liquid helium temperatures are essential for a decision as to whether these diodes can be used for the current by-pass at super fluid helium temperatures for the LHC-superconducting magnets ¹.

Our original intention to irradiate full size (75 mm wafer) diodes of the epitaxial type in liquid helium in a nuclear reactor has been given up because of the very high costs for adequate cryogenic installations. Instead, small diode samples of 10 mm wafer diameter have been irradiated in the nuclear research reactor at Munich in liquid helium and also at liquid nitrogen temperature for comparison, up to gamma doses of 50 kGy and neutron fluences up to 10^{15} n/cm² ($E > 0.1$ MeV) ².

IRRADIATION FACILITY AND DOSIMETRY

The low temperature irradiation facility (TTB) at the 4 MW swimming pool type Munich Research Reactor (FRM) offers the possibility to perform neutron and gamma

irradiation experiments in the temperature range between 4.6 K and 450 K. The irradiation facility has been described in detail elsewhere^{2,3}.

The layout of the irradiation cryostat restricts the sample size to a maximum length of $l_{\max} = 120$ mm and a maximum diameter of $\varnothing_{\max} = 16$ mm. The maximum weight of the TTB-specimens is limited by the gamma-heating in the irradiation position to be about 35-40 g. For the diode irradiations the core position TTB was used. In order to perform irradiations during reasonably long time periods the nominal power of the reactor had to be reduced from 4 MW to 40 kW. During irradiation at 4.6 K the temperature was controlled to within about ± 0.1 K and during 77 K irradiation to within about + 5 K and -15 K.

Dosimetry measurements were carried out prior to the irradiations at reduced reactor power of 50 kW. The neutron flux was determined with standard activation detector methods. The value adjusted to the reactor power was $3.3 \cdot 10^{11}$ n/cm²s ($E > 0.1$ MeV). The gamma dose was measured by means of Alanine and RPL glass dosimeters which were calibrated in a Cobalt-60 source at 4.2 K and at 77 K⁴. The value found was 16.7 Gy/s. The accumulated gamma dose and neutron fluences are presented together with the various irradiation steps in Table 1. It shows, that during all irradiations the gamma dose is a rather important part of the radiation spectrum. This is, however, not in disagreement with the situation to be expected in the LHC-dipoles where also the integrated dose will be a more important factor compared to the neutron fluence⁵.

DIODE TYPES AND SAMPLE HOLDERS

The irradiation tests described here were performed on 10 mm diameter samples of the same wafer material used for the 75 mm diameter wafers. The small diameter of only 10 mm was chosen in order to meet the dimensional and cryogenic requirements of the low temperature irradiation facility. All diodes are of the epitaxial type with an epitaxial width of about 22 μ m.

For the diodes from EUPEC (Siemens) - diode number starting with the letter E - rectangular pieces were sawn out of large diameter wafers and cylindrically ground to disk-like samples of 10 mm diameter. Since the rims were not passivated the reverse blocking voltages are very poor (in the range of a few volts) and neither reverse voltage nor junction capacity measurements could be carried out. The diodes from GEC-PLESSEY - diode number starting with the letter M - were fabricated in the normal way with passivated rims. Diode samples No. E1, E2, E5, E6, and M4N - M7N were irradiated at 4.6 K and diode samples No. E3, E4, E7, E12, M1, M3, M4, and M14 at about 77 K in the nuclear reactor. The diodes E8 - E11 and M8 - M11 were exposed to target irradiation at liquid nitrogen temperature only in the SPS Accelerator at CERN. Each of the six sample holders contained four diode samples. The diodes were stacked - together with copper disk electrodes between each of the diodes - and clamped under a force of 1 kN as shown in Fig. 1. Two Carbon (Allan Bradley) resistors were mounted in the 4.6 K-sample holder and two Copper-Constantan thermocouples in the 77 K-sample holder to control the temperature during irradiation.

TEST PROCEDURES

Table 1 shows the irradiation procedure which was applied to the six sets of diodes. For the reactor irradiation at 4.6 K the procedure consisted of 5 exposure periods of increasing duration. At 77 K the irradiation was carried out in three steps up to the maximum dose of about 50 kGy for the EUPEC diodes and up to 30 kGy for the GEC diodes. After each exposure step the samples were removed from the reactor core and the

Figure 1. Sample holder with diodes and temperature sensor.

electrical characteristics measured. The electrical tests have been described in detail already elsewhere³. On the diode samples marked with an asterisk (*) current pulses up to about 110 A were applied after each exposure step and the forward voltage at peak current level ($dI/dt = 0$) monitored. On the other diodes high current pulse measurements were carried out before and at the end of the total irradiation procedure only, to minimise the self-annealing effects by carrier injection and temperature rise (induced by the tests themselves).

One of the four diodes of each of the six sets was submitted to intermediate current pulse annealing during irradiation. After the last irradiation step at 4.6 K all four diodes were warmed up to room temperature for about 15 min and then measured again at 4.6 K to

Table 1. Irradiation procedures in the nuclear reactor at 4.6 K and at 77 K and in the accelerator target area at 77 K.

Irrad. Step No. /Temperature		Diode No.				Acc.Dose [kGy]	Acc.Neutrons 10 ¹³ [n/cm ²]	Acc.Irrad. Time
Reactor Irrad.								
1	4.6 K	E1*,	E2,	E5*,	E6	1.5	3	90 s
2	4.6 K	E1*,	E2,	E5*,	E6	5	10	300 s
3	4.6 K	E1*,	E2,	E5*,	E6	15	30	900 s
4	4.6 K	E1*,	E2,	E5*,	E6	30	60	1800 s
5	4.6 K	E1*,	E2,	E5*,	E6	50	100	3000 s
1	77 K	E3*,	E4,	E7*,	E12	5	10	300 s
2	77 K	E3*,	E4,	E7*,	E12	15	30	900 s
3	77 K	E3*,	E4,	E7*,	E12	50	100	3000 s
1	4.6 K	M4N,	M5N,	M6N*,	M7N*	1.5	3	90 s
2	4.6 K	M4N,	M5N,	M6N*,	M7N*	5	10	300 s
3	4.6 K	M4N,	M5N,	M6N*,	M7N*	15	30	900 s
4	4.6 K	M4N,	M5N,	M6N*,	M7N*	30	60	1800 s
5	4.6 K	M4N,	M5N,	M6N*,	M7N*	50	100	3000 s
1	77 K	M1*,	M3,	M4,	M14*	5	10	300 s
2	77 K	M1*,	M3,	M4,	M14*	15	30	900 s
3	77 K	M1*,	M3,	M4,	M14*	30	60	1800 s
Target Irrad.								
1	77 K	E8, E9, E10*,	E11*,	M8, M9, M10*,	M11*	6.80	10.0	505 h
2	77 K	E8, E9, E10*,	E11*,	M8, M9, M10*,	M11*	13.90	16.2	750 h
3	77 K	E8, E9, E10*,	E11*,	M8, M9, M10*,	M11*	20.48	19.0	879 h
4	77 K	E8, E9, E10*,	E11*,	M8, M9, M10*,	M11*	36.98	34.6	1641 h
5	77 K	E8, E9, E10*,	E11*,	M8, M9, M10*,	M11*	51.60	55.2	2370 h

* diodes measured versus dose

verify the effect of temperature annealing. Finally all diodes were re-measured again at 4.6 K and 77 K after staying for about two months at room temperature.

MEASUREMENT RESULTS

The forward voltage drop U_f of these diodes, which governs the thermal transient in the diodes during de-excitation of the magnets and the dimensioning of the heat sinks, is the most important characteristic affected by irradiation. If during irradiation this voltage exceeds a certain value then by excessive joule heating the diode will be damaged because the generated heat cannot be transferred sufficiently fast to the heat sink⁶.

Reactor Measurements at 4.6 K

Figure 2 shows as typical examples the change of the current-voltage characteristic $I_f = f(U_f)$ of the diodes E5 and M6N before irradiation and after irradiation up to 50 kGy and thermal annealing at 300K. The deformation of the curve of diode M6N at higher doses is due to heating effects at higher current and voltage levels causing a decrease in forward voltage. The degradation - increase in forward voltage U_f - of the diode M6N is significant.

In Fig. 3 the relative increase of forward voltage $\Delta U_f/U_{f0}$ at the peak current level of 100 A is shown versus dose and fluence for the 8 diodes irradiated at 4.6 K. The maximum degradation of almost 600 % was measured on the diode M7N whereas the diodes E2 and E6 show an increase in U_f of about 180 %. The degradation in forward voltage versus dose is smaller for the diodes E1 and E5 which had intermediate annealing caused by the measurement itself. After 50 kGy and one thermal cycle of 15 minutes to room temperature the degradation in forward voltage drops to about 22 % for all of the EUPEC-diodes and to about 50 % for all of the GEC diodes.

The effect of annealing during 2 months at 300 K amounts to a further reduction of the degradation in forward voltage down to about 20% for the EUPEC diodes and to about 25 % for the GEC diodes. Intermediate current pulse annealing gave a final reduction in U_f of a few per cent only.

Reactor Measurements at about 77 K

In Fig. 4 the relative increase in forward voltage $\Delta U_f/U_{f0}$ for the EUPEC diodes E3, E4, E7, E12 and for the GEC diodes M1, M14 at about 77 K is shown. Similar to the

Figure 2. I_f - U_f -characteristic of diode E5 and M6N at 4.6 K before and after irradiation.

Figure 3. Relative increase of the forward voltage versus dose and fluence at 4.6 K.

results for the irradiation at 4.6 K the increase in forward voltage amounts to about 150 % for the EUPEC diodes E3 and E7 which were measured versus dose and up to about 175 % for the diodes E4 and E12 which were measured only before and after irradiation. The relative increase in U_f for the GEC diodes amounts to about 400 % after a dose of 30 kGy. After about 2 months at 300K the degradation is reduced to about 10 to 20 % for all of the diodes.

Comparison of Results at 4.6 K and 77 K

The comparison of results obtained for EUPEC diodes at 4.6 K and 77 K is shown in Fig 5. The degradation versus dose is surprisingly similar, although a larger degradation was expected at 4.6 K due to the reduced electrical conductivity. The corresponding results for the GEC diodes are given in Fig. 6 up to a dose of 30 kGy for the irradiation at 77 K. The GEC diodes irradiated at 4.6 K and 77 K gave similar results up to a dose of 30 kGy. If

Figure 4. Relative increase of the forward voltage versus dose and fluence at 77 K.

Figure 5. Relative increase of forward voltage versus dose and fluence at 4.6 K and 77 K for EUPEC diodes.

further tests on similar diodes should confirm this results then irradiation tests could be limited to tests at 77 K only.

The full reverse characteristic $I_r = f(U_r)$ was monitored on the GEC diodes after each exposure period in the I_r -range from 1 μ A to about 1 mA. The reverse voltage U_r versus dose at $I_r = 1$ mA is shown in Fig. 7 during irradiation at 4.6 K up to a dose of 50 kGy and during irradiation at 77 K up to a dose of 30 kGy. During 77 K irradiation the reverse voltage increased for most of the diodes (up to a maximum of about 50 % for diode M4) without any significant decrease after annealing at 300 K. During 4.6 K irradiation the reverse voltages also increased for all diodes (up to a maximum of about 100 % for diode M4N). After annealing at 300 K however the reverse voltage decreased for most of the diodes to a value less than before irradiation. On diode M5N even a short circuit was measured.

Accelerator Target Area Irradiation Measurements

Four GEC diodes and four EUPEC diodes of 10 mm diameter were exposed to irradiations at 77 K in the SPS target area at CERN up to a dose of about 52 kGy and a

Figure 6. Relative increase of forward voltage versus dose and fluence at 4.6 K and 77 K for GEC diodes.

Figure 7. Reverse voltage (at $I_r=1$ mA) versus dose at 4.6 K and 77 K irradiation.

neutron fluence of up to about $50 \cdot 10^{13} \text{ n/cm}^2$ which is about half of the fluence compared to the reactor irradiations. The relative increase in forward voltage $\Delta U_f/U_{f0}$ at $I_f = 100$ A for these diodes is shown in Fig. 8. Intermediate current pulses annealing was applied to the GEC diode M11 and to the EUPEC diode E11 at about 37 kGy. The reduction in forward voltage especially for diode M11 is significant and mainly due to partial thermal annealing caused by the measurement itself.

Comparison of Reactor Irradiation and Accelerator Target Area Irradiation

For the comparison between reactor irradiation and accelerator target irradiation typical results are presented in Fig. 9 for the EUPEC diodes E7 and E10 and for the GEC diodes M10 and M14 where the relative increase in forward voltage is given versus dose. For the EUPEC diodes the increase in forward voltage during reactor irradiation and target irradiation are almost identical up to a dose of about 15 kGy and more than a factor of two higher during reactor irradiation at a dose of about 50 kGy. For the GEC diodes the

Figure 8. Relative increase of forward voltage versus dose and fluence for EUPEC and GEC diodes during target irradiation at 77 K.

Figure 9. Forward voltage increase versus dose during reactor- and target irradiation at 77 K.

increase in forward voltage is similar up to a dose of about 5 kGy only and also more than double during reactor irradiation at a dose of 30 kGy.

CONCLUSIONS

Epitaxial diodes of the same epitaxial width (22μm) of two different manufacturers show substantial differences when irradiated, which can be attributed to different fabrication processes. Results from irradiations at 4.6 K and 77 K are similar, at least for the EUPEC diodes, and it should be possible in future to restrict irradiation tests to 77K. For the EUPEC diodes, reactor and target irradiations show almost identical degradation in forward voltage up to about 15 kGy, so that long term irradiations (over 6 month) in the target area could be replaced by short term irradiations in a nuclear reactor (a few days).

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