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PUBLICATION

CERTIFICATION OF THE RADIATION RESISTANCE OF COIL INSULATION MATERIAL

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DELIVERABLE REPORT

CERTIFICATION OF THE RADIATION RESISTANCE OF COIL INSULATION MATERIAL

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Abstract:

The goal of the WP 7.2.1 sub-task of the EuCARD program has been to determine the Nb_3Sn based accelerator magnet coil electrical insulation resistance against irradiation, which will occur in future accelerators. The scope of the certification covers determination of mechanical, electrical and thermal properties changes due to irradiation. The report presents a selection of the insulation material candidates for future accelerator magnets as well as the definition of the radiation certification methodology with respect of radiation type, energy, doses and irradiation conditions. The test methods and results of the electrical and mechanical insulation materials properties degradation due to irradiation are presented. Thermal conductivity and Kapitza resistance at temperature range from 1.5 K to 2.0 K (superfluid helium conditions) are given.

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Delivery Slip

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1. EXECUTIVE SUMMARY

Within the WP 7.2.1 sub-task of the EuCARD program the radiation resistance of the potential materials for future Nb_3Sn accelerator magnets electrical insulation has been accomplished. S-glass fibre reinforced DGEBA epoxy (RAL Mix71), TGPAP epoxy (RAL Mix 237), cyanate ester-epoxy mixes (CE-Epoxy) and CTD101 ceramic epoxy (LARP) laminates have been certified with respect to the requirements resulting from the radiation conditions estimated for future accelerators. The materials have been irradiated with 50 MGy, 4 MeV electron beam at 77 K conditions. Electrical, mechanical and thermal properties tests of the materials before and after irradiation have been performed in cryogenic conditions (LN2 environment).

The electrical strength of 0.5 mm thick insulation samples at 77 K tests shows strong degradation of the insulation properties due to the irradiation. Nevertheless, electrical strength of each irradiated material is a few times higher than the required 5 kV/mm.

Mechanical ultimate tensile strength tests at 77 K also show strong degradation of the materials due to the irradiation. The strength of RAL Mix 71 after irradiation has decreased almost to 0 MPa, which disqualifies this material for use in the accelerator magnets.

Thermal properties of the materials have, due to program time limit, been determined for unirradiated samples only. Nevertheless, the obtained results can be very useful in Nb_3Sn magnets thermal design. The thermal measurements of the irradiated samples are in progress.

2. INTRODUCTION

Magnets in accelerators like HL-LHC and neutrino factories will be subjected to very high radiation doses. The electrical insulation employed in the coils needs to be resistant to this radiation. A certification program for the radiation resistance of the insulation material has been launched within the WP7.2.1 of EuCARD program sub-task.

Within the program the list of potential materials for electrical insulation has been established as well as a set of certification criteria: certification irradiation type and doses, irradiation conditions, insulation materials properties to be certified as well as certification test methods and conditions.

3. SELECTION OF THE MATERIAL CANDIDATES FOR MAGNET COIL ELECTRICAL INSULATION

Wind-and-react techniques for Nb_3Sn magnet coils require coil heat treatment at a temperature of 650^{o}C or higher. Therefore, the process of the coil electrical insulation production is divided into two steps. Before coil winding, the coil cables are wound with a high temperature resistant glass fiber tape to separate adjacent cables in the formed coil. After the heat treatment, the spaces created by the fibers and the space in the cable structure are vacuum impregnated with polymer matrix material, which, after polymerization, bonds the insulation fibers and cable into a monolith.

3.1. FIBER GLASSES

The most market available glass fibers tapes are made of the E-glass. This material contains boron dioxides, which reduce the glass melting temperature but activate the material after

irradiation. Therefore, E-glass fibers are not suitable for Nb_3Sn accelerator magnet applications.

Boron free glass fibers are those with S-glass and quartz-glass. Since the quartz-glass is proven to be difficult to weave and it is not easily available, the S-glass is chosen as electrical insulation structure material.

3.2. MATRIX MATERIALS

The matrix material should be characterized by low initial and long time viscosity to allow sufficient time for the magnet mould to fill, good toughness to avoid matrix material cracking as well as good electric strength and good thermal conductivity. Besides the viscosity, all listed properties should be highly resistant for the irradiation.

Based on the information available in the literature and on program participants self experience, the following matrix materials, which could fulfill the above listed requirements, were selected for the certification program:

3.2.1. RAL MIX 71

RAL Mix71 is a flexible epoxy formulated from DGEBA (standard epoxy resin) and POPDA (an amine hardener). This material has been developed during the Next European Dipole (NED) program by Rutherford Appleton Laboratory (UK) for Nb_3Sn magnet applications. It has a low glass transition temperature and is not expected to be very radiation tolerant because the hardener is a long chain molecule. This material is a benchmark material in that it has a long history of successful use in superconducting magnets. It has a relatively short pot life (around 3h at 40^oC).

3.2.2. RAL MIX 237

RAL Mix 237 is a trifunctional resin (TGPAP) and DETD(2002) aromatic hardener. The resulting cured material has a very high crosslink density and so a high radiation resistance. It has been exposed to 100 MGy in a fission reactor for ITER purposes, and retains 75% of its compressive strength. The epoxy is more expensive than RAL Mix71.

3.2.3. CYANATE ESTER – EPOXY RESIN

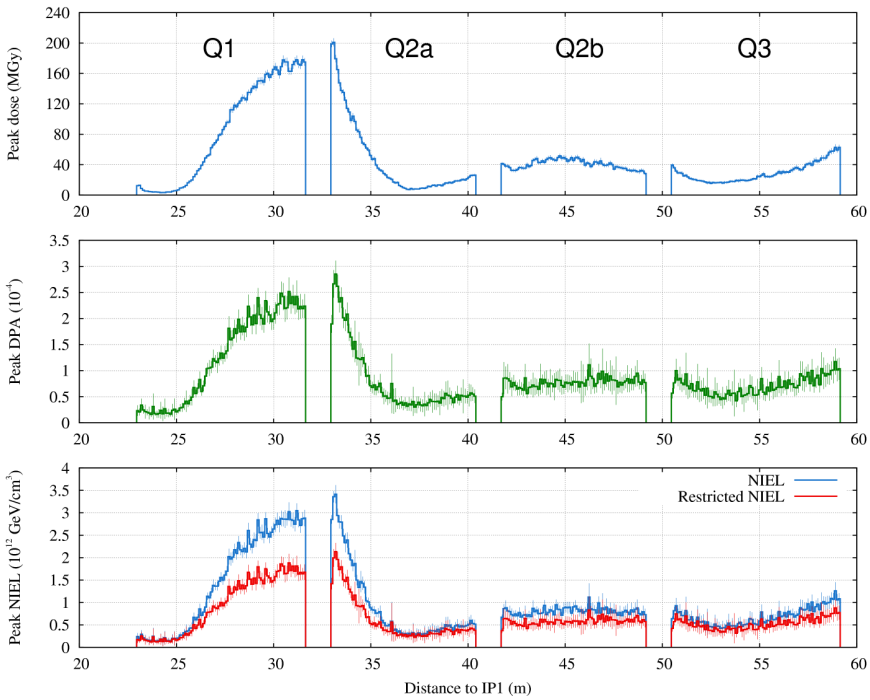
Literature data from ITER work shows that CE-Epoxy mixes are highly radiation stable. Pure cyanate esters are expected to be very radiation resistant, because of trifunctional bonds and high crosslink density. A liquid cyanate ester was used for its suitability for vacuum impregnation. For the program purpose the mixture consists 40% of CE and 60% of epoxy.

3.2.4. LARP

The radiation sensitive material in the so-called “LARP” ceramic-epoxy composite is the epoxy itself. It has been shown that the ceramic is reacting with the glass fibers. Therefore, for the program purpose it has been decided to make materials using S-glass, as for the other materials, and an epoxy equivalent to the CTD101. This is an epoxy with an anhydride hardener, widely used for magnets (both resistive and superconducting) because of its low viscosity and very long pot life. This epoxy is brittle, but potentially radiation resistant.

4. RADIATION SPECTRUM IN FUTURE ACCELERATORS

To define the certification irradiation procedures the conditions that can occur in the future Nb_3Sn superconducting magnets have to be recognized and estimated. The following parameters have to be defined: radiation type, energy spectrum and doses. It was decided to perform radiation certification for conditions corresponding to the maximum radiation doses expected in the HL-LHC accelerator. Figure 4.1 presents the radiation map for the Interaction Region (IR) Quadrupoles (Q) for HL-LHC calculated with FLUKA model. It can be seen that the peak of irradiation will occur in the Q2a, 35m from Collision Point.



4.1 Radiation map for the Interaction Region (IR) Quadrupoles [1] for the innermost 3 mm for the unshielded case with an integrated luminosity of 3000 nb^{-1}

FLUKA simulations allow defining the contents of given radiation type in the Q2a radiation peak point. The simulation information and influence of given radiation type on the SC magnets coil material are collected in Table 4.1

Table 4.1 Radiation spectrum at Q2a: 35m from Collision Point [1,2] and the estimated effect on the coil.

Radiation type	**Contents, %**	**Influence on magnet coil materials**
Neutrons	4.82	SC and Cu
Protons	0.14	SC and Cu
Photons (γ)	88.93	Insulation
Electrons	4.31	small effect
Positrons	2.23	small effect
Pions +	0.19	probably small effect
Pions -	0.26	probably small effect

It results from Table 4.1 that photon (γ) radiation has the strongest influence on the SC magnet coil electrical insulation. It is expected to constitute almost 90% of the total radiation on the Q2a magnet.

Simulations done with the FLUKA code enable to estimate the energy spectrum for all radiation types listed in table 4.1. Figure 4.2 presents the photons spectrum in the inner coil of Q2a at the peak location.

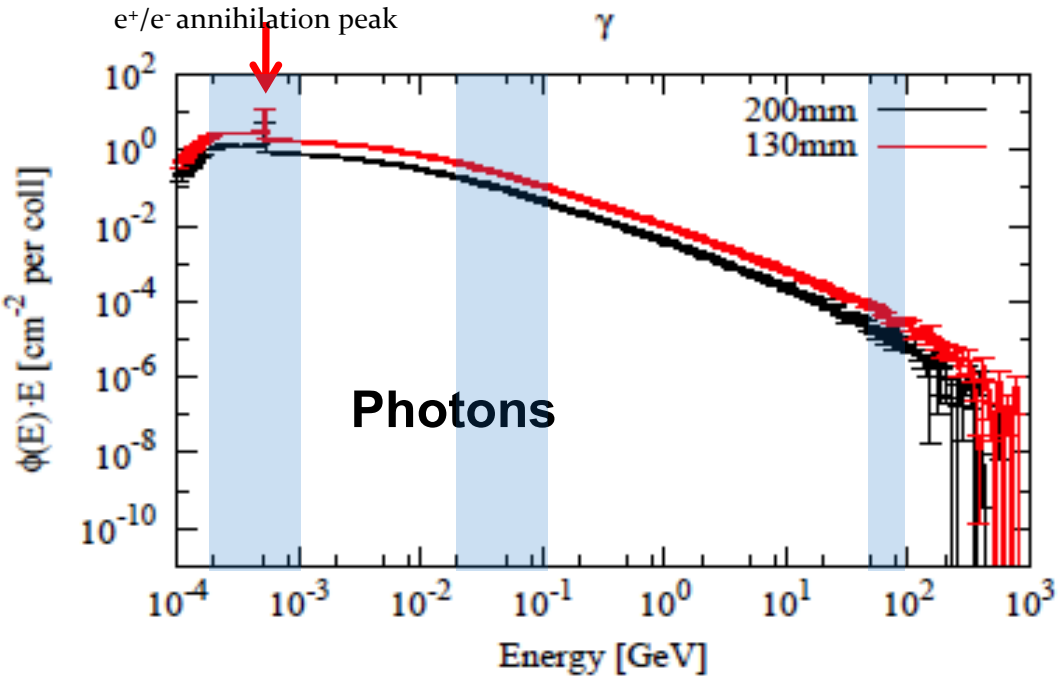


Figure 4.2 Photon spectrum on the inner coil of Q2a at the peak location for 2 different magnet aperture diameters [2]

It can be seen from Figure 4.2 that the peak number of photons colliding with Q2a has energy from range 0.1 MeV to 1.0 MeV. Also the influence of higher energy (up to 100 MeV) photons on the insulation properties should not be neglected. It would be also profitable for a better understanding of the organic materials irradiation processes to investigate the influence of the protons with energies in the GeV range. Taking this into account, the irradiation photon energy bands have been preliminary defined as: 0.2 MeV – 1.0 MeV, 20 MeV – 100 MeV, 50 GeV – 100 GeV (marked with light-blue area in Figure 4.2).

The certification maximum radiation dose has been targeted at the value of 50 MGy.

5. CERTIFICATION RADIATION TYPE, IRRADIATION CONDITIONS AND FACILITY

5.1. SELECTION OF THE CERTIFICATION RADIATION TYPE

After reviewing the ability and accessibility of the irradiation sources installed over Europe, the National Center for Nuclear Research (NCBJ) in Poland (former Andrzej Soltan Institute

for Nuclear Problems) has been selected as irradiation Institute. NCBJ operates an industrial accelerator for irradiation with 4 MeV to 15 MeV photons. Photons are produced by bombarding electrons on a 1 mm thick tungsten and a 0.2 mm thick gold target [3]. Available photon spectra from the electron linac for different energies are presented in Figure 5.1.

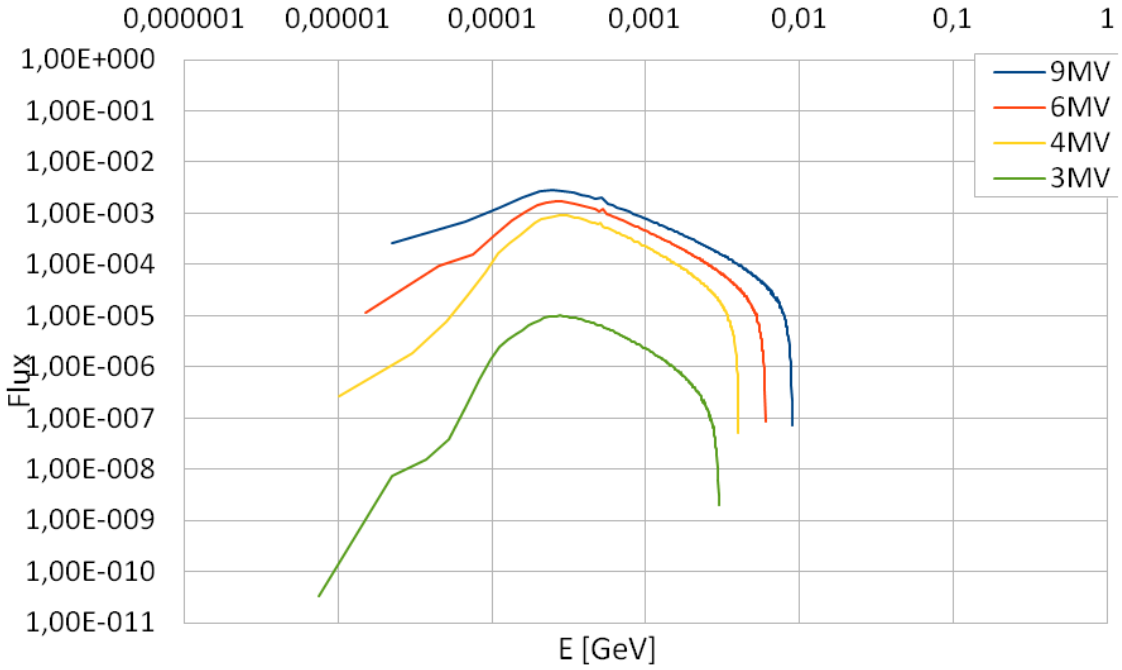


Figure 5.1 Typical spectrum of brehmstrahlung radiation induced by: 9 MeV electrons (blue), 6 MeV electrons (red), 4MeV electrons (yellow) and 3 MeV electrons (green).[3]

It can be concluded from Figure 5.1 that the flux of photons is between 3 to 7 orders of magnitude lower than the flux of electrons colliding with the target. It results that to obtain the proposed 50 MGy dose, a relatively long time would be needed.

It is know that the character of the damage to the insulation material from electrons with E > 1 MeV is similar to that of photons with E > 1 MeV due to the formation of a shower with photons and pair produced electrons and positrons. Therefore, an electron beam has been chosen as the source of the certification radiation.

5.2. SELECTION OF THE ELECTRON BEAM ACCELERATOR STRUCTURE

To determine the best irradiation parameters, three different accelerator structures, namely 6 MeV, 12 MeV and 15 MeV were tested at NCBJ between Oct 2010 and Jan 2011. Figures 5.2 and 5.3 present the experimental results of the electron beam penetration depths in PMMA (Plexiglas) for the 12 MeV and 15 MeV structures. The results are obtained for optimized accelerator gun current, gun voltage, solenoid voltage and with a pulse repetition rate of PRR = 5 Hz.

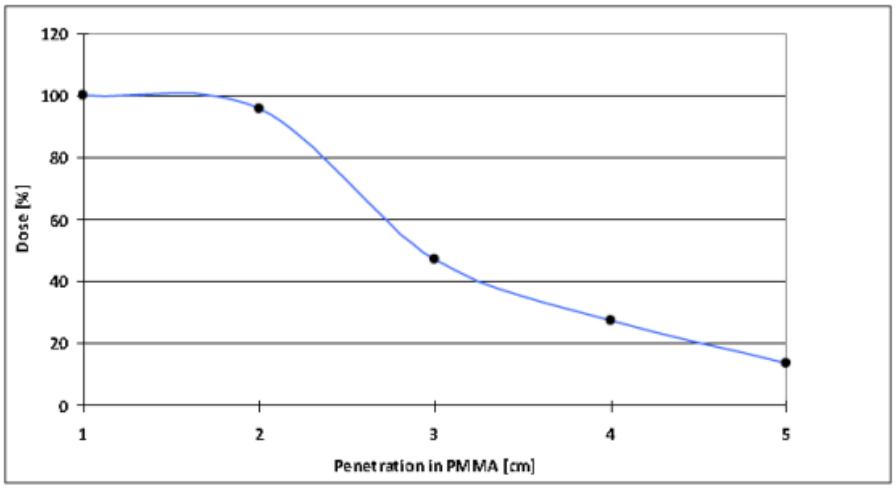


Figure 5.2 Measured depth-dose curve of "12 MeV" structure - confirmed energy: 7 MeV - 8 MeV.

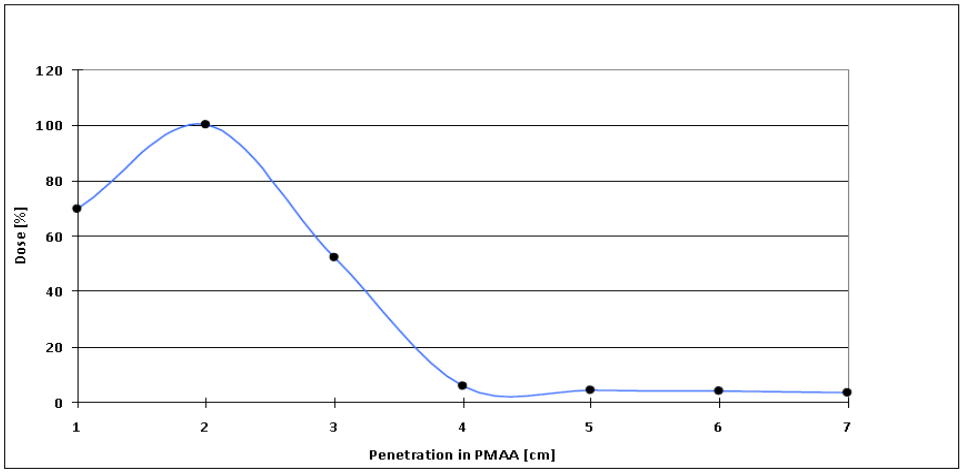


Figure 5.3 Measured depth-dose curve of "15 MeV" structure - confirmed energy: 10 MeV - 11 MeV.

Based on the PMMA penetration results, it has been concluded that for the 12 MeV structure the confirmed energy is 7 MeV - 8 MeV, while for the 15 MeV structure the confirmed energy is 10 MeV - 11 MeV. The energy of the 6 MeV structure has been confirmed in another experiment as 4 MeV.

Figures 5.4 and 5.5 present experimental results of the radiation dose as a function of the distance from the radiation source for 6 MeV and 15 MeV structures. The PRR was 76,4 Hz and 5 Hz respectively.

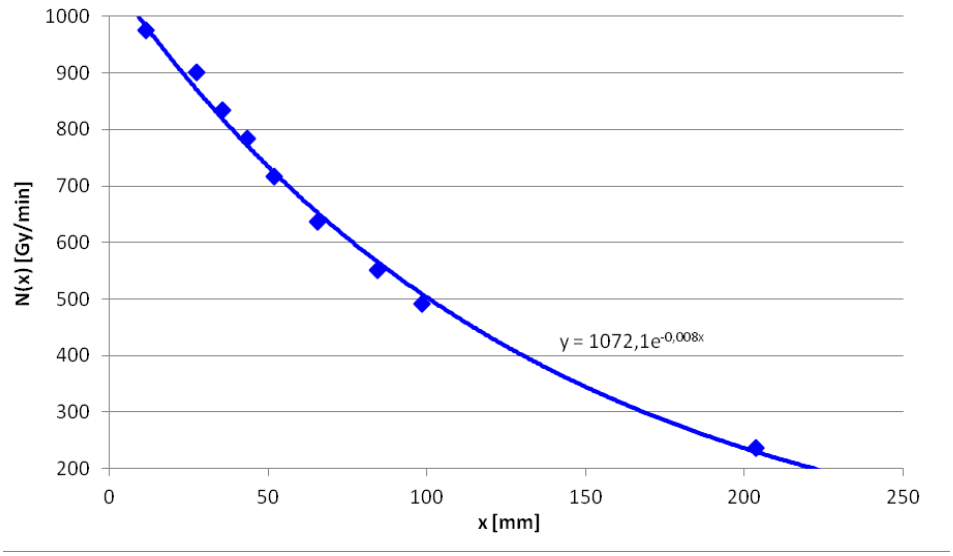


Figure 5.4 Dose rate variation as function of the distance from the radiation source in air for the 6 MeV structure, PRR = 76.4 Hz. Accelerator parameters not optimized. [4]

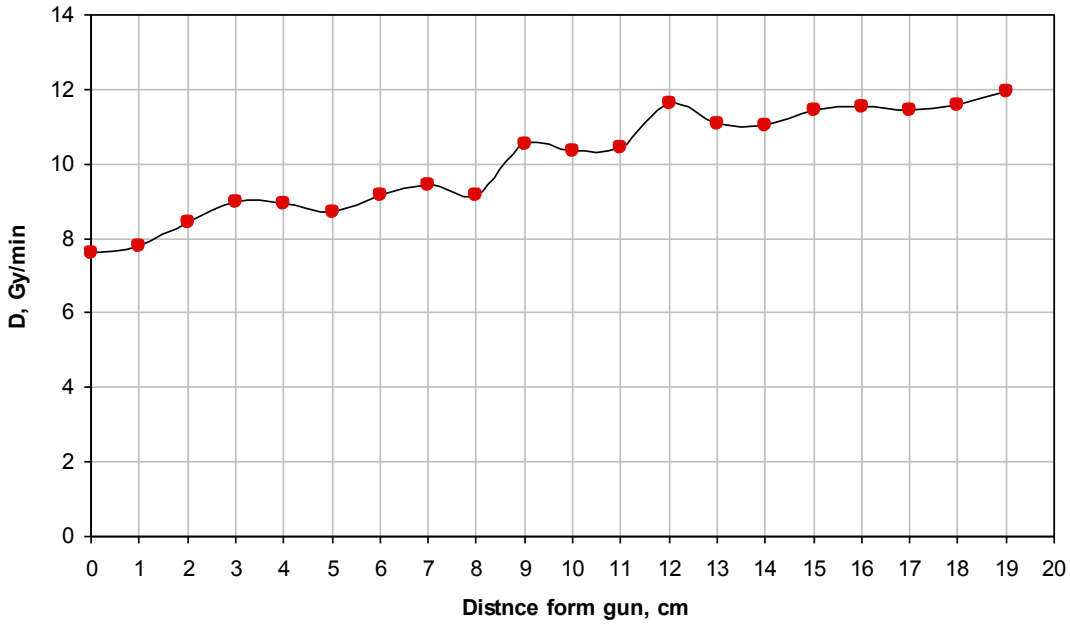


Figure 5.5 Dose rate variation as function of the distance form the radiation source in air for the 15 MeV structure, PRR = 5 Hz. Accelerator parameters not optimized.[5]

It should be pointed out that the dose rate variation results presented for the 6 MeV structure in Figure 5.4 have been obtained for not optimized accelerator parameters. The maximum dose for optimized parameters for PRR = 76.4 Hz was measured as 2200 Gy/min [4]. The results for the 12 MeV structure are very similar as for the 15 MeV structure and therefore not presented in the report.

To compare and select the best acceleration structure, the calculation of the necessary irradiation time for one irradiated insulation material sample has been done. For the calculation purpose the following assumptions have been made:

- the material samples can be assembled into a package,
- the average sample thickness is 0.5 mm,
- the sample density is about 1.8 g/cm^3 (density of G10 material, which is similar to insulation materials),
- irradiation dose 50 MGy,
- PRR 300 Hz (maximum available for the NCBJ electron linac).

The penetration depth in the insulation material for a given structure has been determined and this allows to decide the number of samples in the irradiation package. The dose rate scaled to PRR = 300 Hz allows to estimate the time necessary to accumulate 50 MGy in the package. Finally, the irradiation time of one sample can be found.

The calculation input parameters and the results for the considered structures are shown in Table 5.1

Table 5.1. Irradiation time for different accelerator structures

Structure		**6 MeV**	**12 MeV**	**15 MeV**
Confirmed electron energy	MeV	4	8	11
Depth of insulation penetration	mm	5,6	14,4	21,1
Nbrs of samples radiated at once	-	11	29	42
Max recorded dose rate	Gy/min	2200	12	12
Repetition frequency	Hz	76,4	5	5
Expected dose @f=300Hz	Gy/min	8638,7	720,0	720,0
Irradiation time for 50 MGy	Working days	12,1	144,7	144,7
Irradiated samples	Work. days/sample	1,1	5,0	3,4

It results from Table 5.1 that the 6 MeV structure allows irradiation of one material sample in a much shorter time than with the 15 MeV structure (1/3rd irradiation time) and 12 MeV structure (1/5th irradiation time). Additionally, the 6 MeV structure has the largest beam diameter (see paragraph 5.3), which allows irradiation of the larger surface with a single beam. Therefore, it has been decided to irradiate the insulation samples with 6 MeV electrons delivered by an electron linac.

5.3. DETERMINATION OF THE ELECTRON BEAM SIZE

As required for the post-irradiation testing, the irradiated area for insulation material samples is relatively large (see paragraph 5.4). Therefore, the beam size is an important parameter. The beam size has been determined by its projection on the PMMA plate, which allowed taking a picture of the beam shape – see Figure 5.6 a. Then the picture has been analyzed in a computer program, where the beam intensity distribution was determined - see Figure 5.6 b.

a)

b)

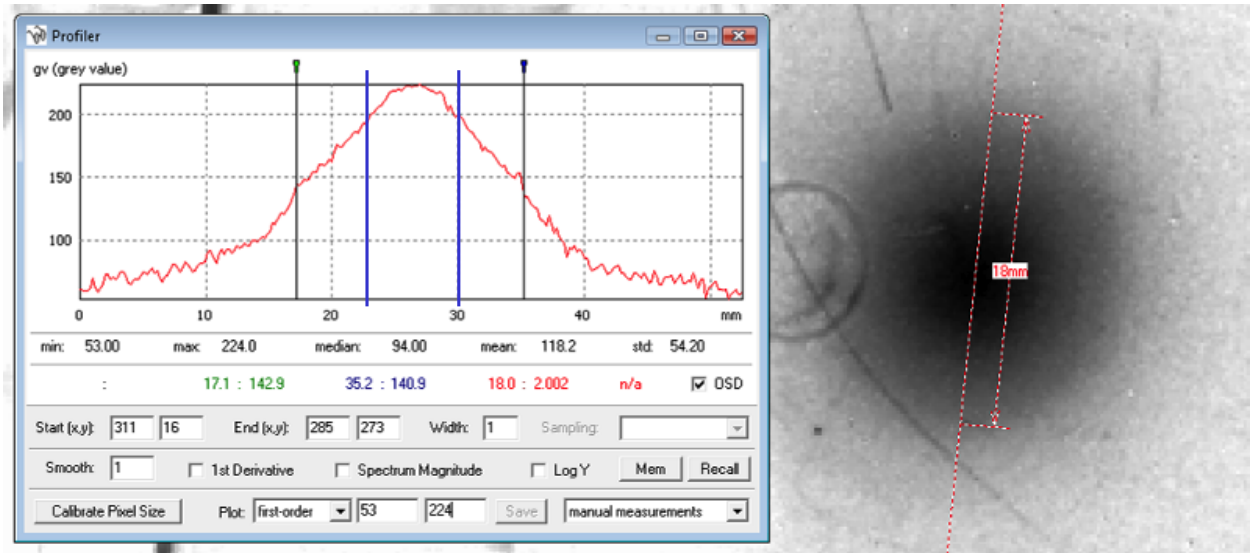


Figure 5.6. Measurement of the beam diameter for the 6 MeV structure, a) projection of the beam on a PMMA plate, b)determination of the beam intensity distribution with picture color analyses

From Figure 5.6.b it can be found that beam intensity distribution has a normal distribution shape, where the 6 mm and 18 mm diameter circles include respectively 90%-100% and 65%-100% of the maximum intensity. Similar measurements for 12 MeV and 15 MeV structures confirmed a beam diameter of 2 mm (90%–100% of the intensity) in both cases.

5.4. DETERMINATION OF THE IRRADIATION AND SAMPLE TRANSPORT CONDITIONS

Profound literature review concerning properties of low temperature irradiated epoxy/glass fiber composites [6] has indicated a lack of data, useful for future insulation materials development. The subject of radiation damage in reinforced polymers is complicated and poorly understood. Twenty articles concerning the irradiation effects have been identified and they cover the following materials: pure epoxy (2 articles) and composites (18 articles). Direct comparison of published data was impossible due to differences in radiation doses, materials, tested properties, radiation and testing conditions. Nevertheless the literature review has enabled to identify the main problems of the radiation influence on mechanical, thermal and electrical properties of the samples.

The effect of irradiation temperature (room/cryogenic temperature) is still not fully investigated and in case of irradiation at cryogenic temperature range, the influence of warm-up between the irradiation and the test process, on the change of insulators properties is not

verified. Literature review has shown that there is insufficient research data that takes into account the irradiation temperature.

Therefore it was decided to perform the irradiation followed by tests at cryogenic temperature without a meantime warm-up. Samples irradiation has been performed in liquid nitrogen (LN2) conditions as well as the electrical and mechanical certification tests. The tests followed the irradiation without warming-up the samples. Thermal certification tests have been performed in pressurized superfluid helium and the irradiated test samples have been warmed-up for installation in the test cryostat.

The irradiated electrical and mechanical samples have been transported in a dedicated, dry, liquid nitrogen cooled cryogenic transport vessel (Air Liquid Voyageur Plus). In case of the thermal samples transport, a nitrogen gas vented hermetic box has been used to avoid atmospheric oxygen diffusion into the irradiated parts of the samples.

5.5. DEVELOPMENT OF INSULATION IRRADIATION METHOD IN CRYOGENIC TEMPERATURE

A series of preliminary tests performed in NCBJ in Nov 2010 concerning the irradiation absorption ability of different solids and liquids have shown that a layer of 15 mm liquid nitrogen absorbs almost 100% of the electron beam [4]. Therefore, immersing of the insulation samples in LN2 during the irradiation process can not be applied. Nevertheless, the cooling of the samples with a thin film of liquid nitrogen has been compromised as a satisfactory solution.

a) *b)*

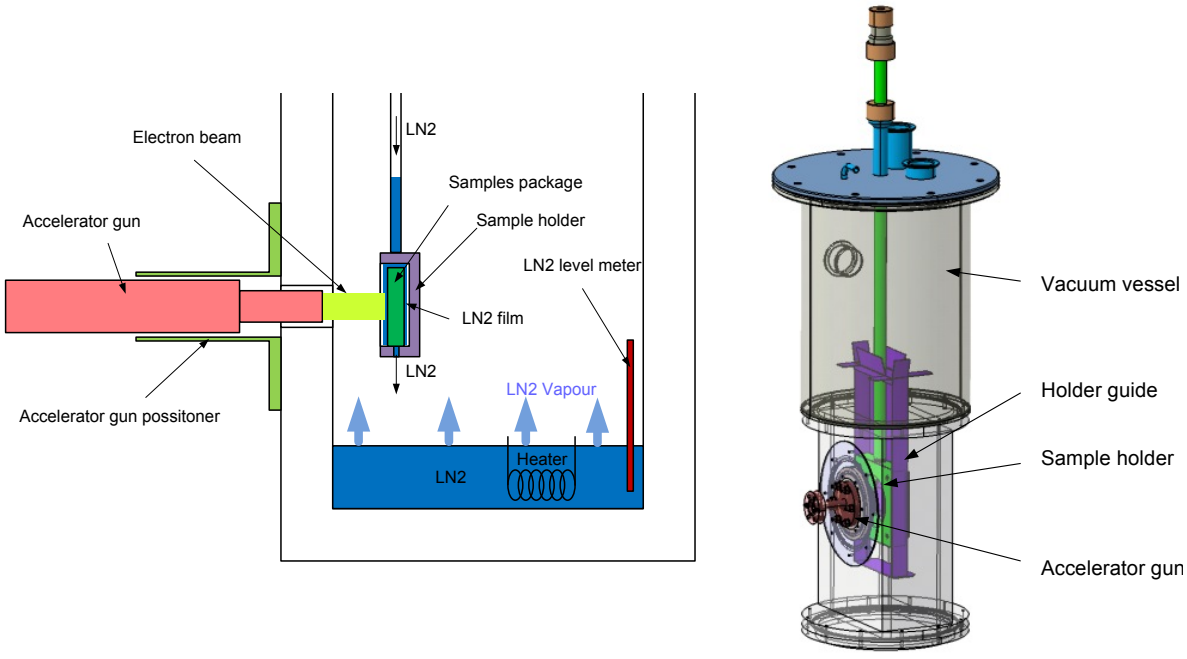


Figure 5.7. Irradiation cryostat, a) scheme, b) view of conceptual design.

The scheme and conceptual design of the cryostat for irradiation purpose, developed by PWR and manufactured by Kriosystem Sp z o.o. in Poland, is shown in Figure 5.7 while the photo of the irradiation set-up at NCBJ is presented in Figure 5.8. The distance between the accelerator window and the samples package in the holder is about 2 cm. The samples are

located in a sample holder (Figure 5.9) inserted in the open box made of aluminium and with a wall thickness of 0.2 mm. Liquid nitrogen (LN2) is supplied from an external dewar through a vacuum insulated transfer line and fills up the box from the top. Thanks to that the samples in the holder are always covered with thin film of LN2. The excess LN2 is over-flowing from the box and it is collected in the cryostat bottom vessel, where a 200 W electrical heater and a liquid nitrogen level sensor are installed. The purpose of the heater is to generate nitrogen vapour for the accelerator window cooling, while the level sensor protects the heater against its burning, in case of a lack of LN2. The sample holder can be moved inside the cryostat in vertical direction and can be fixed at different heights. Additionally, the holder design allows its rotation by 90 deg, so it is possible to irradiate different areas of the samples in vertical and horizontal directions.

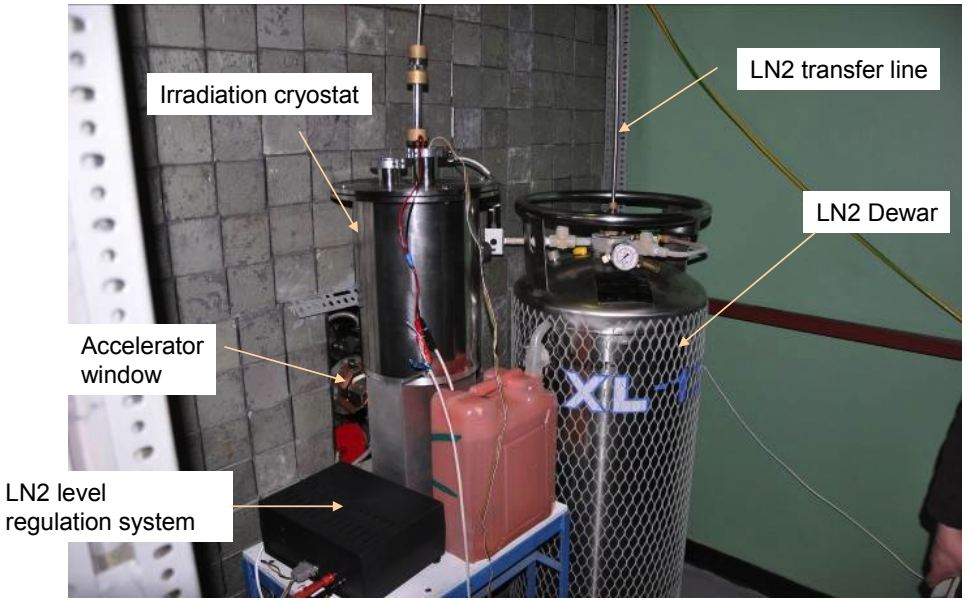


Figure 5.8. Irradiation set-up installed on the accelerator site in NCBJ, Poland .

5.6. IRRADIATION PROCESS

The selected test methods of electrical, mechanical and thermal properties requires a sample thickness in a range 0.1 mm – 0.5 mm (see paragraph 6). The materials to be tested were provided as square sheets 110 mm × 110 mm and of specified thickness. The available beam penetration depth allows simultaneous irradiation of 10 samples. Before the installation in the sample holder circular holes were cut in the corner of the sheet that allows griping the sheets in the holder with help of bolts. During installation each sheet was separated from the others with help of plastic washers, which creates slots for liquid nitrogen flow across the package and wetting both sides of each sheet. The view of the sheets package in the holder is shown in Figure 5.9.

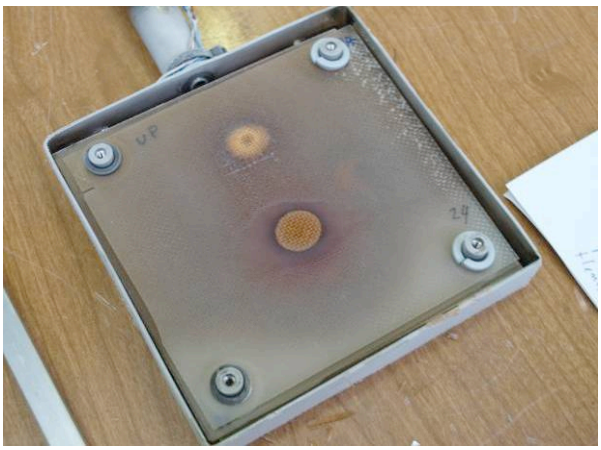


Figure 5.9. View of the sheets package after irradiation during irradiation set-up commissioning phase

The sample holder is equipped with a Pt100 temperature sensor located next to the liquid nitrogen inlet of the holder. Therefore, a presence of liquid nitrogen flow can be monitored. In case of lack of nitrogen flow, what can be observed by the temperature increase on the Pt100 sensor, the accelerator control system triggers the beam stop, which avoids the possibility of the beam burning the sheet.

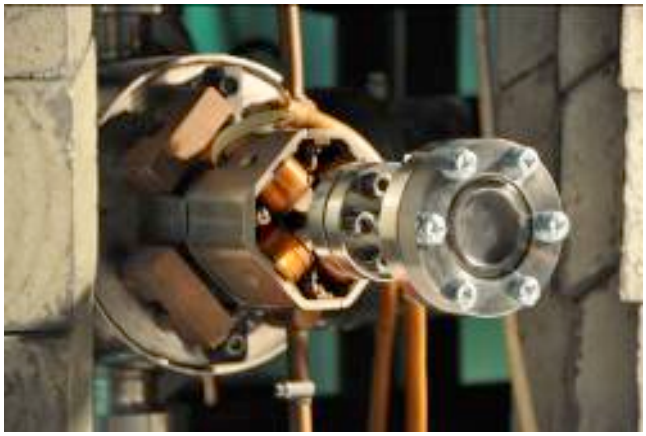


Figure 5.10. 0.2 mm thick Ti accelerator window.

During the irradiation, the beam dose rate is being monitored with use of a special designed solenoid coil, in which the electron beam generates a current. The generated current versus beam dose rate was scaled with help of a Farmer type ionisation chamber with an accuracy of 1%.

5.7. ANALISYS OF THE IRRADITION PATTERN FOR MECHANICAL AND THERMAL SAMPLES

As it was mentioned in paragraph 5.3, the beam size (intensity range 90%-100% of the maximum intensity) is 6 mm only. Such a beam size is sufficient to perform the electrical properties tests, where a 5 mm diameter electrode size can be used – see paragraph 6. In case of mechanical and thermal certification tests a single irradiation spot is not sufficient for uniform irradiation of the tested part of the specimens. Nevertheless, since 65% of the maximum irradiation intensity is still available at a distance of 9 mm from the beam center, a

proper overlapping of a few single spots can create a relatively large, uniformly irradiated area.

Since the cryostat design limits the possibility of the samples surface irradiation to vertical and horizontal directions, it is possible to irradiate the sample center as well as the area above, below, left and right from the centre. Figure 5.11 presents the irradiation dose distribution for specimen irradiated in 5 locations for a different distance *d* from the irradiated spots center. It can be seen that there is an optimum distance (*d*=15mm), where a regular, uniformly irradiated area is created.

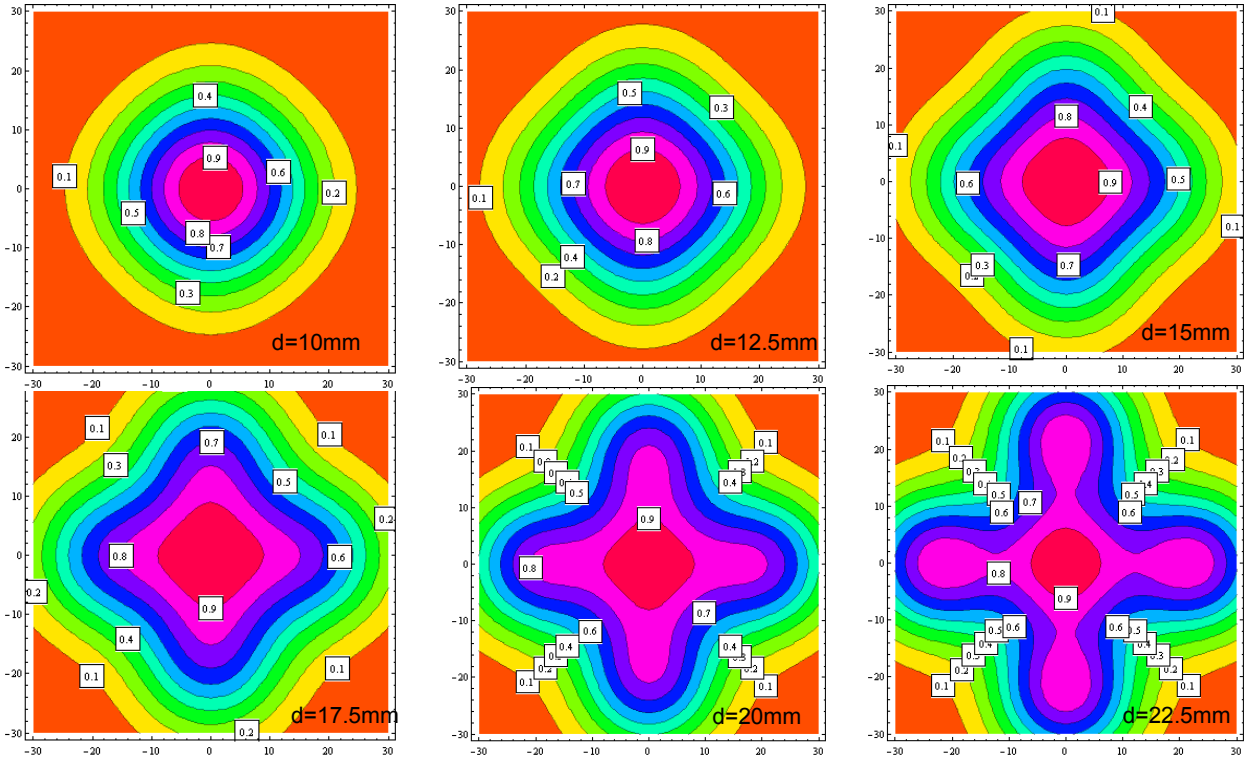


Figure 5.11. Irradiation dose distribution for specimen irradiated in 5 locations: central, top, bottom, left and right from the specimen centre, for different distance d from the spot's centre

Detailed analysis (Figure 5.12) of the picture obtained for *d*=15 mm shows that it is possible to extract a 24 mm circle irradiated with 80% to 100% of the total dose. Such a shape can be used for thermal tests of the irradiated insulation materials.

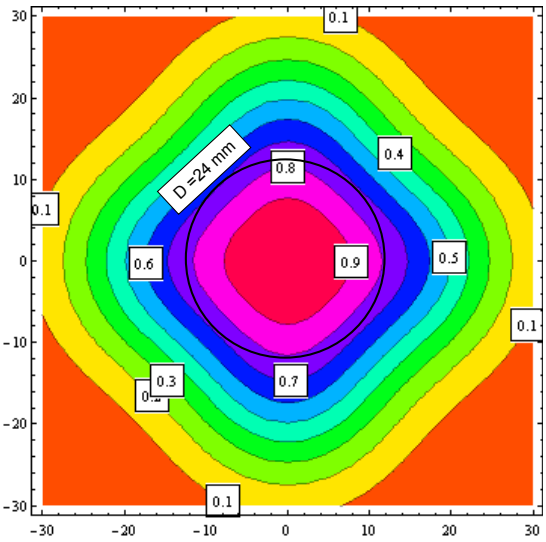


Figure 5.12. Irradiation dose distribution for specimen irradiated in 5 locations. Distance between irradiation spots d=15 mm.

A similar simulation has been performed for the mechanical specimen (standard shape Type 1, ISO 37:2005) which test part is 6 mm wide and 33 mm long – see Figure 5.13. For this case the 3 irradiation spots, placed in line 18mm from each other, are sufficient to irradiate the test part with 90-100% of the total dose.

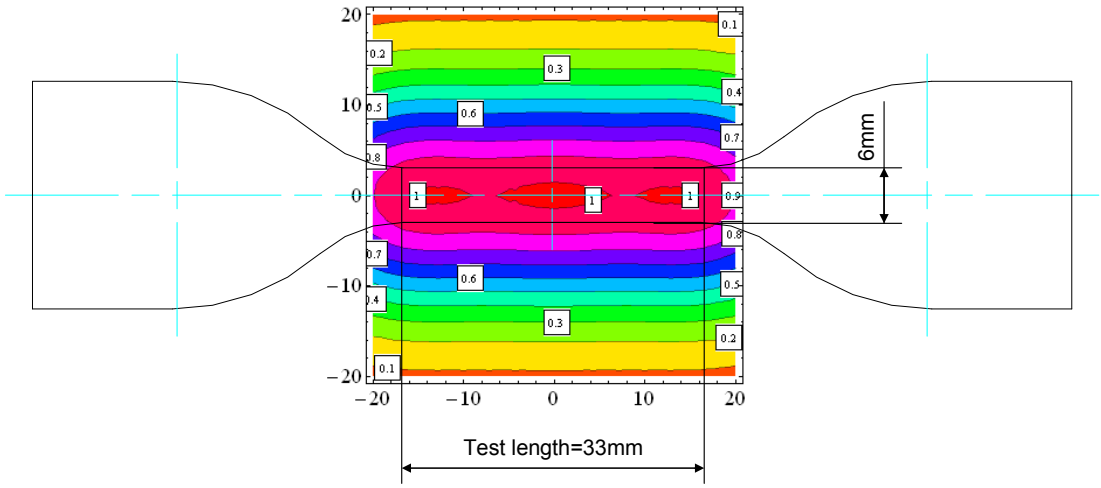


Figure 5.13. Irradiation dose distribution for mechanical specimen irradiated in 3 locations. Distance between irradiation spots d=18 mm.

6. ELECTRICAL CERTIFICATION OF IRRADIATED INSULATION SAMPLES

The main purpose of certification tests is the comparison of given material properties before and after irradiation. Therefore, each certification test method was repeated for unirradiated and for irradiated insulation candidates.

6.1. ELECTRICAL CERTIFICATION TEST METHOD

An EN 60243-1 standard: "Methods of test for electric strength of solid insulating materials. Tests at power frequencies" has been selected as the insulation materials electrical properties certification test. In this method, two electrodes are placed on the opposite side of the tested material sheet. An increasing voltage difference is applied on the electrodes until an electrical breakdown in the material occurs.

The EN 60243-1 standard recommends using a close to 0.5 mm thickness sheet and external dimensions of about 100 mm × 100 mm. Such size perfectly matches the developed irradiation method requirements.

6.2. ELECTRICAL CERTIFICATION TEST STAND

For the certification program purpose it was required to implement the electric strength test method in liquid nitrogen conditions. Therefore, a dedicated electric test cryostat was designed by PWR and manufactured by Kriosystem Sp z o.o. in Poland. The conceptual design as well as the cryostat view is presented in Figure 6.1.

The cryostat consists a double wall vacuum insulated dewar, where the insert with a test part is placed. The test part is composed of an upper and a lower plate. Both plates are made of high electric strength and low temperature resistance fibre glass – epoxy resin laminate G10. The lower plate is suspended on the G10 rods connected to the insert top plate. The upper plate is movable in the vertical direction – the horizontal movements of the plate are limited by the suspension rods. Each plates caries one, 5 mm diameter electrode. The lower electrode is fixed in the centre of the lower plate while the upper electrode is moveable in vertical direction in the centre line of the upper plate.

The 110 mm x 110 mm dimension test sample is placing on the lower plate in a specially shaped grove, which doesn't allow dislocation of the sample in the horizontal direction. Due to that, the centre of the sample lies exactly on the bottom electrode. After sample installation the upper plate is sliding down and the upper electrode loosely lies on the top surface of the sample. The mass of upper electrode is 50 g, which fulfils the EN 60243-1 standard requirements and assures the reproducible contact pressure between the electrodes and sample in each test.

The electrodes are connected to an external SL600 type Spellman High DC Voltage source, which can generate a voltage of up to 60 kV. Voltage supplying copper wires are Kapton polyimide insulated to withstand the liquid nitrogen temperature conditions without mechanical damages. The nominal electric strength of the wire insulation is 30 kV. Further tests show that the insulation can withstand up to 54 kV. For the tests where higher voltage values were required the wires were wrapped with an additional layer of the adhesive Kapton tape, that increases their insulation strength to over 60 kV.

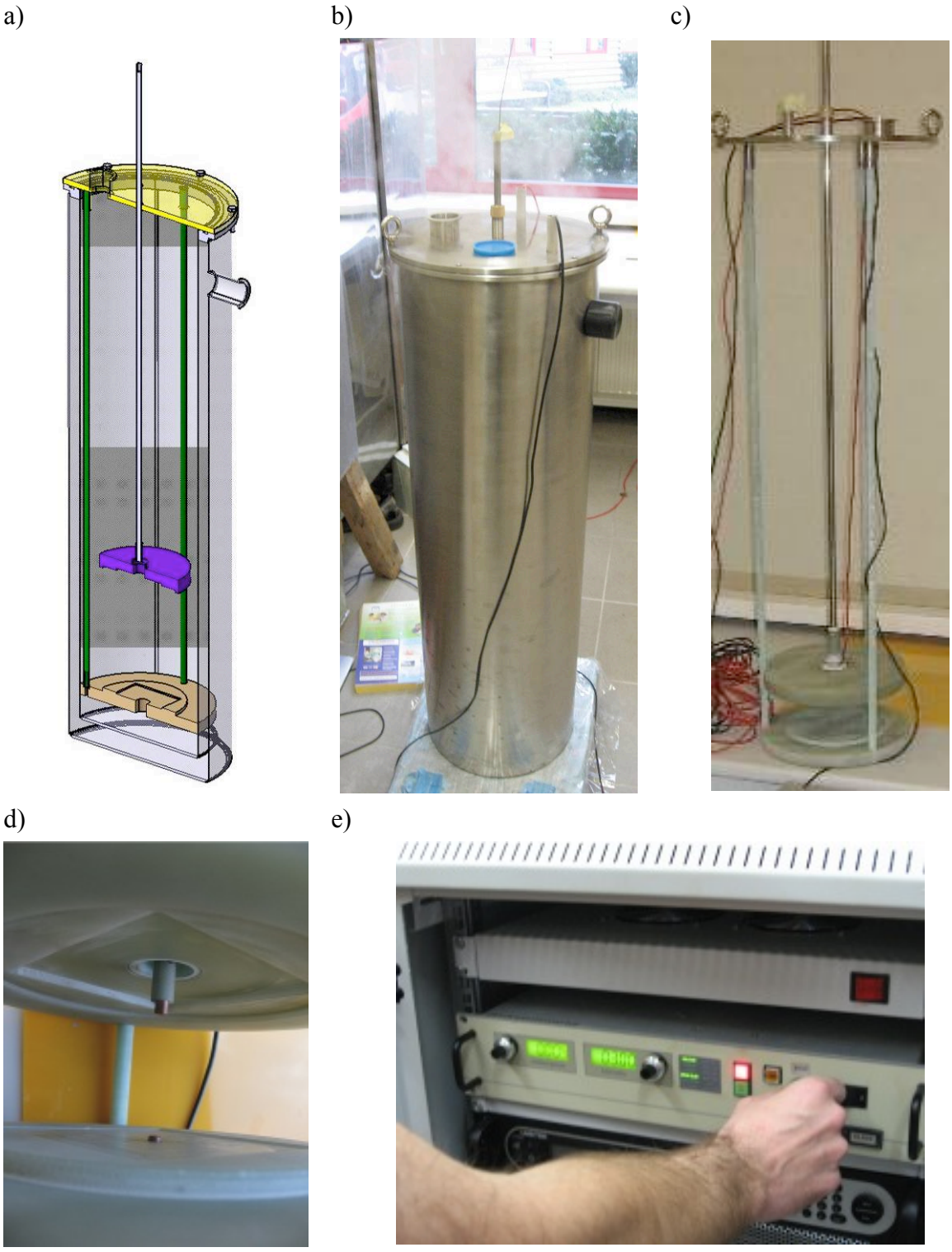


Figure 6.1. Electrical test stand view, a) electrical cryostat conceptual design, b) the cryostat view, c) the cryostat insert view, d) the test part of the insert view, e) SL600 type Spellman High DC Voltage source

6.3. ELECTRICAL TEST METHODOLOGY

For the calibration (see paragraph 6.4) and unirradiated insulation tests the samples were cleaned with methanol. Before the test the sample thickness was measured in the test point (in the sample centre) with a micrometer. Then the insert was installed in the cryostat dewar. Since the calibration tests were preformed at room temperature, the cryostat dewar wasn't filled with liquid nitrogen, while in case of the certification tests the test part of the insert was completely immersed in liquid nitrogen.

Special care was taken to avoid warm-up of the irradiated sample during installation in the test set-up. Therefore, the samples, after being removed from the transport vessel, were very quickly installed in the liquid nitrogen pre-cooled insert lower plate. The thickness of the irradiated samples in the test point (irradiated area) was measured after the test. The exemplary view of irradiated and tested insulation sample is presented in Figure 6.2.

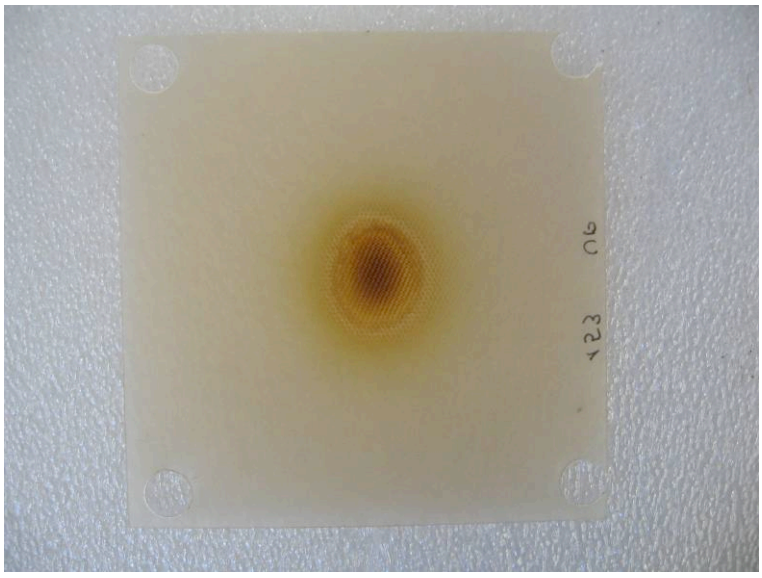


Figure 6.2. Irradiated and tested electrical sample

The high voltage source is computer controlled, which allows setting different voltage increase rates as well as monitoring an actual generated voltage value. In the electrical certification test program an uniform 1 kV/min voltage increase rate was used. Such value is recommended by the EN 60243-1 standard. The test lasts until the electric breakdown of the material, when the voltage source detects an electric short circuit and triggers off the voltage output. As breaking voltage, the last recorded value of the supplying voltage plus one ramp step value is considered. Finally, the electric strength of the single sample can be calculated as the breaking voltage value divided by the material thickness. The electrical strength test procedure recommends 5 times repetition of the test on a separate material samples. As the final electrical strength the average value from the 5 tests is calculated.

6.4. CALIBRATION TESTS OF ELECTRICAL TEST STAND

For electrical test stand commissioning a number of reference material tests at room temperature have been performed. The purpose of the tests was to determine the reproducibility of the results. As reference materials 50 μm and 150 μm thick Kapton sheets, and 190 μm and 550 μm thick G10 laminate sheets were used. The reference material electric

strength data have been obtained at room temperature in accordance to ASTM D-149: "*Standard Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies*". Since the certification test set-up uses DC voltage and in the ASTM D-149 standard AC voltage is used, the reference data should be scaled to the DC value by root-of-2 factor.

One of the key parameter of the Kapton polyimide electric strength, due its high hygroscopic properties, is the material humidity during the tests. It was found [12] that the dielectric strength can vary ±10% from the average value obtained for 50% humidity.

The Figure 6.3 and 6.4 present the calibration test results for 50 μm and 150 μm thick Kapton respectively where corresponding reference data [12] (scaled to the DC conditions) for 50% humid Kapton is marked.

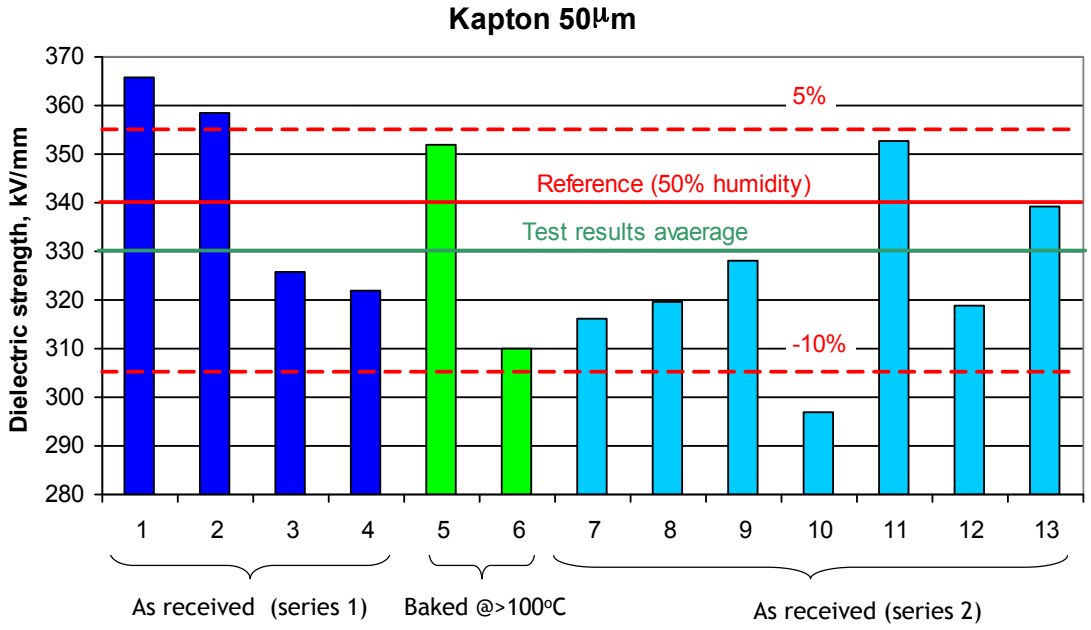


Figure 6.3. Reference electrical tests results for Kapton 50 μm, reference data [12]. AC measurements scaled to DC equivalence.

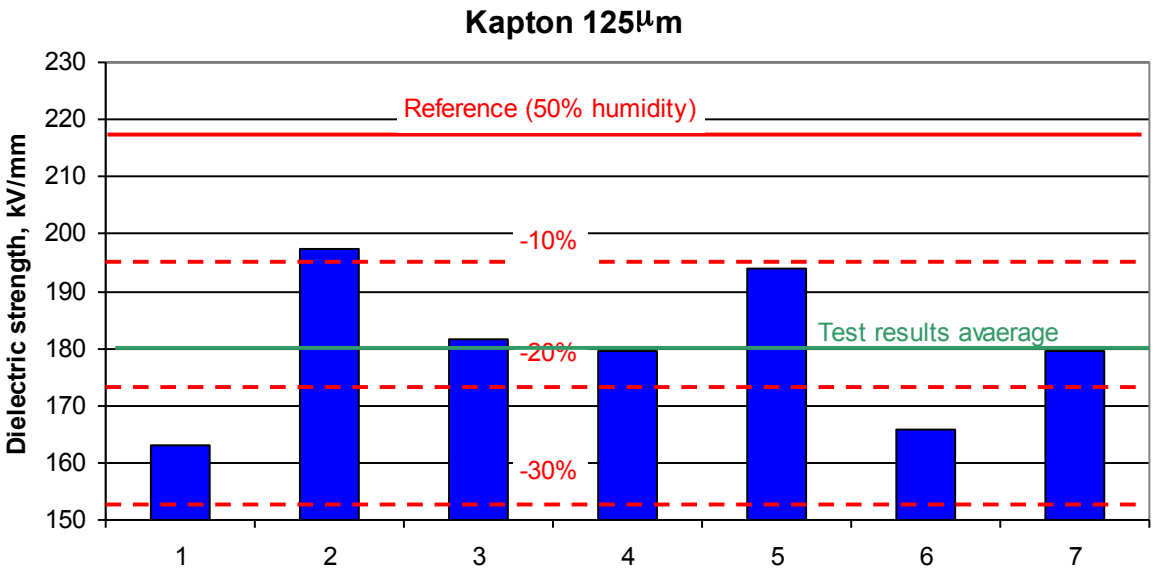


Figure 6.4. Reference electrical tests results for Kapton 125 μm, reference data [12] . AC measurements scaled to DC equivalence.

In the case of the 50 μm thick Kapton, 3 test series have been performed. The first and the last series have been done for samples without any additional treatment than the methanol cleaning. The 2nd series has been done for Kapton samples baked at 100°C to obtain close to 0% humid samples. As it can be seen, no specific differences between the particular test series can be observed. Almost all the results are in the ±10% range of nominal values. Moreover, the average test results value is only 3% lower than the reference value.

For the 150 μm thick Kapton the average test results value is significantly far (-17.5%) from the reference value. This disagreement can't be explained only by the tested and reference sample humidity difference. Nevertheless, it can be seen that the variation of the test results is relatively small – less than 10% of the average value.

Figure 6.5 presents the reference data [13] for G10 laminate tested with AC voltage. The electric strength for reference sheets thicknesses are marked with dotted blue lines. What is worth to underline is that for 550 μm thick G10 the reference data is interpolated from a number of measurement points. However, the 190 μm thick G10 reference data is extrapolated, moreover, since the values are presented on the logarithmic scale, an precision of reference data for small thicknesses can be expected to be poor.

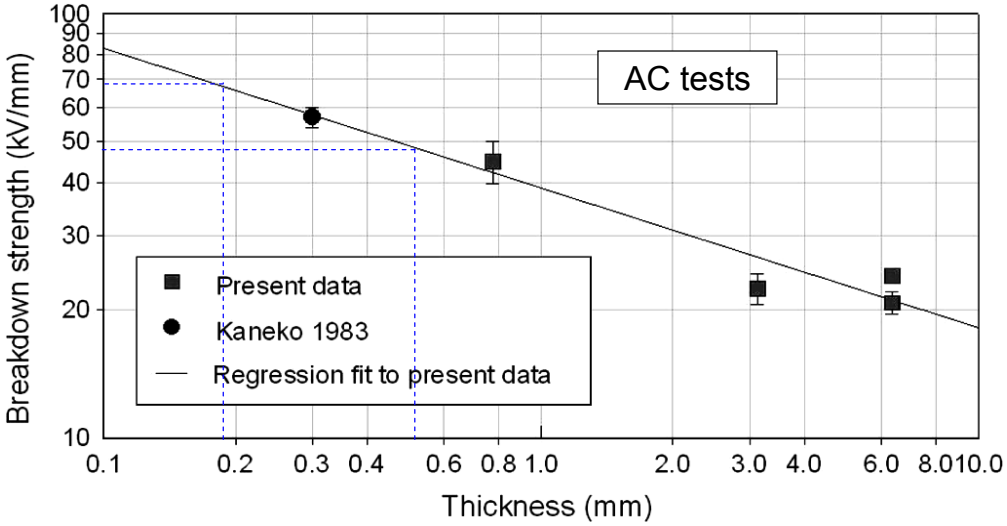


Figure 6.5. Rreference data for G10 laminate tested with AC voltage [13]

Figure 6.6 and 6.7 present the 190 μm and 550 μm thick G10 electric strength test results.

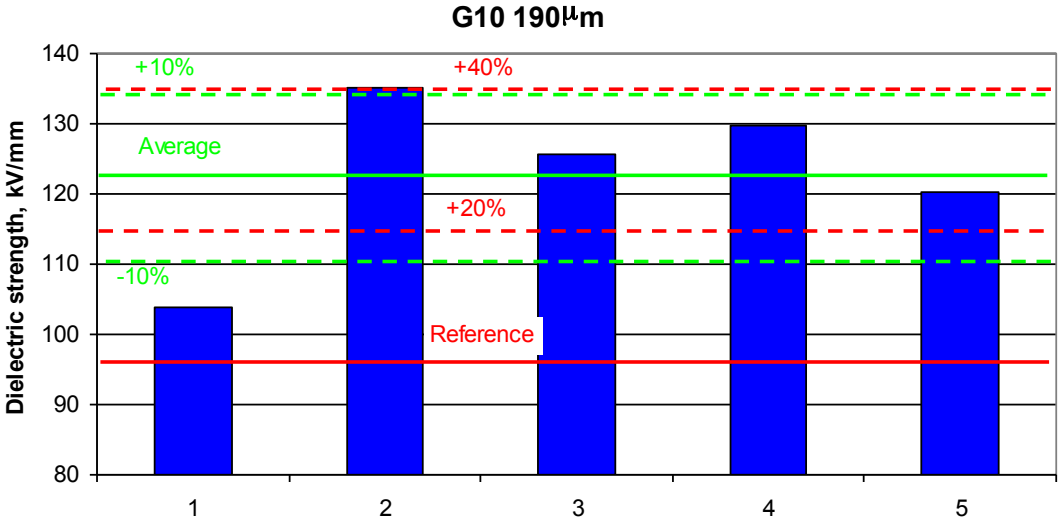


Figure 6.6. Electric strength test results of 190 μm thick G10 laminate, reference data [13]

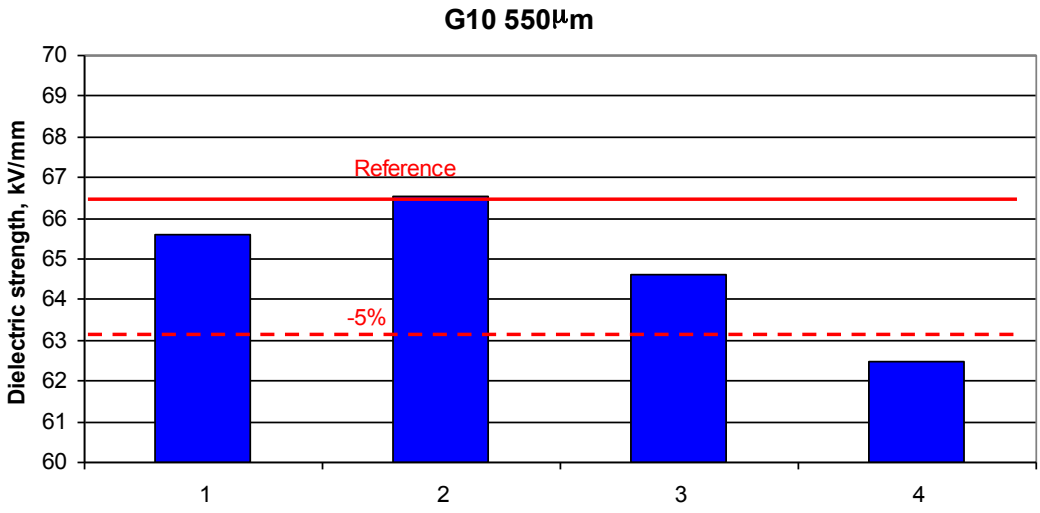


Figure 6.7. Electric strength test results of 550 µm thick G10 laminate, reference data [13]

It can be found that disagreement between the average and reference electric strength value for 190µm thick G10 is almost 30%. However, as the variation of the tests result around the average value is ±10%, the 30% of disagreement can be explained by the poor precision of the reference data estimation.

In case of the 550 µm thick G10, the tests results average is in a very good agreement with the interpolated reference data. Also test results variation around the average is small.

It can be concluded, that besides (in some cases) a large disagreement between the reference data and results obtained on the electric certification test stand-up, the variation of the test results around the average values is in ±10% range. Since the purpose of certification tests is to compare, not to determine a precise electric strength values, the ±10% results variation is acceptable for the tests purpose.

6.5. ELECTRICAL CERTIFICATION TESTS RESULTS

The Figure 6.8 to 6.14 present the test result for unirradiated and irradiated insulation materials considered in the program. The unirradiated samples could be measured in a few locations. Therefore, more the 5 results are presented on the corresponding graphs. In case of irradiated Mix 71, LARP and cyanate ester-epoxy resin (CE-Epoxy) one of the sample exhibited much lower electric strength value than the other samples. It is probably due to moisture condensation on the sample surface during the sample installation in the test set-up. Due of that, results for these samples were removed from a further consideration..

The Mix 237, LARP and CE-Epoxy samples thickness was about 0.5 mm, while the thickness of the Mix 71 samples was about 0.7mm.

In the case of unirradiated Mix 71, the breakage voltage wasn't achieved for supply voltages up to 60 kV. Therefore, separate results are not presented below and in Figure 6.15 are marked as *"better than 85 kV*".

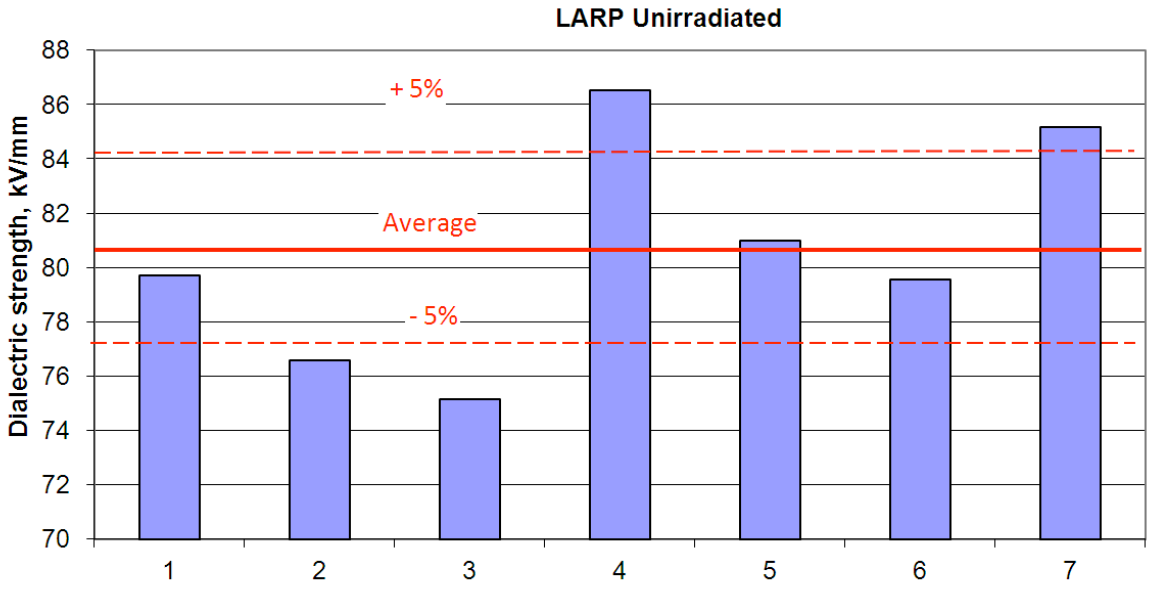


Figure 6.8 Unirradiated LARP electrical breakdown strength

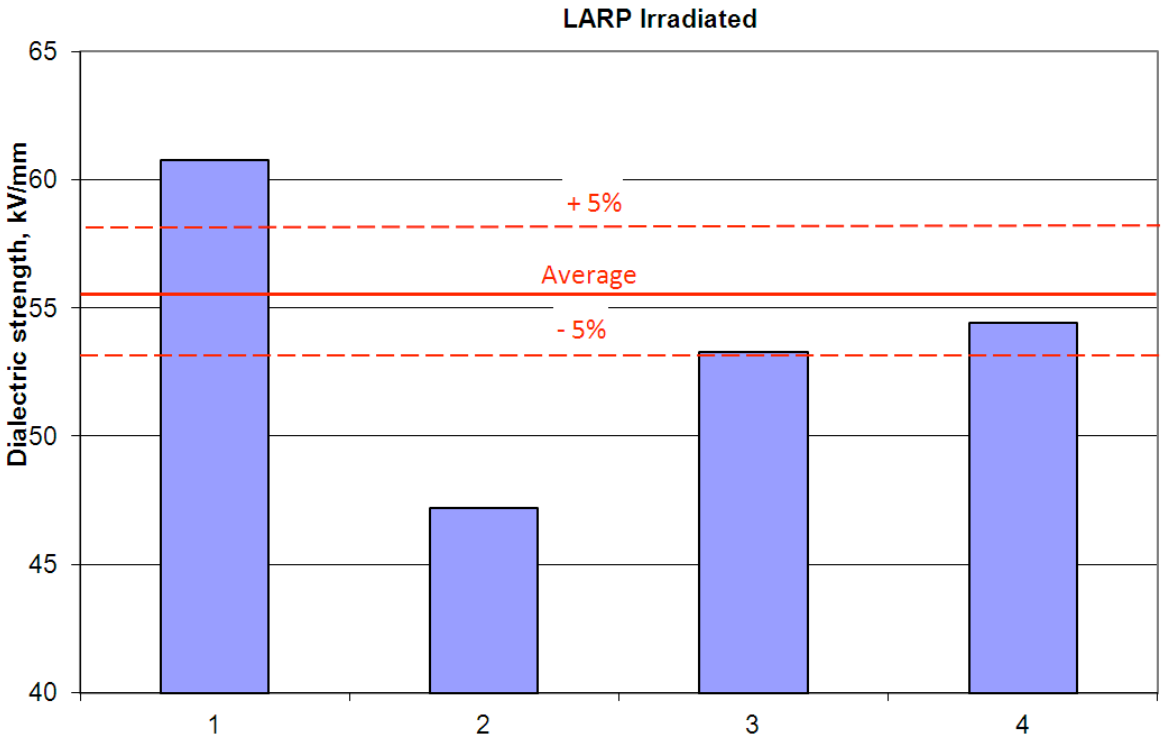


Figure 6.9. Irradiated LARP electrical breakdown strength

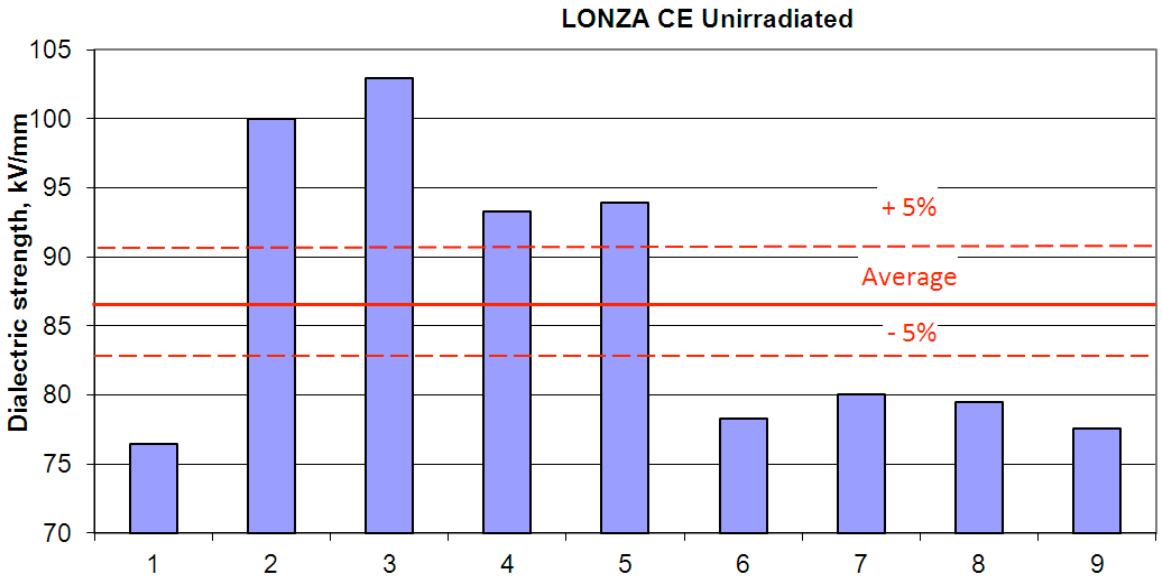


Figure 6.10. Unirradiated LONZA CE electrical breakdown strength

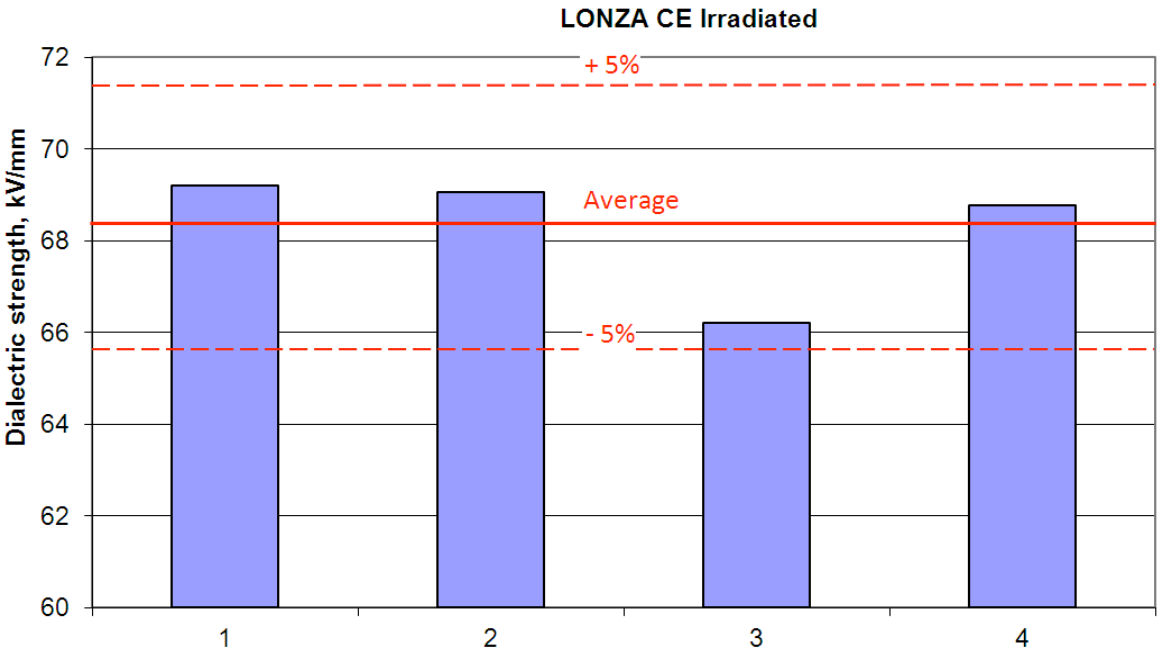


Figure 6.11. Irradiated LONZA CE electrical breakdown strength

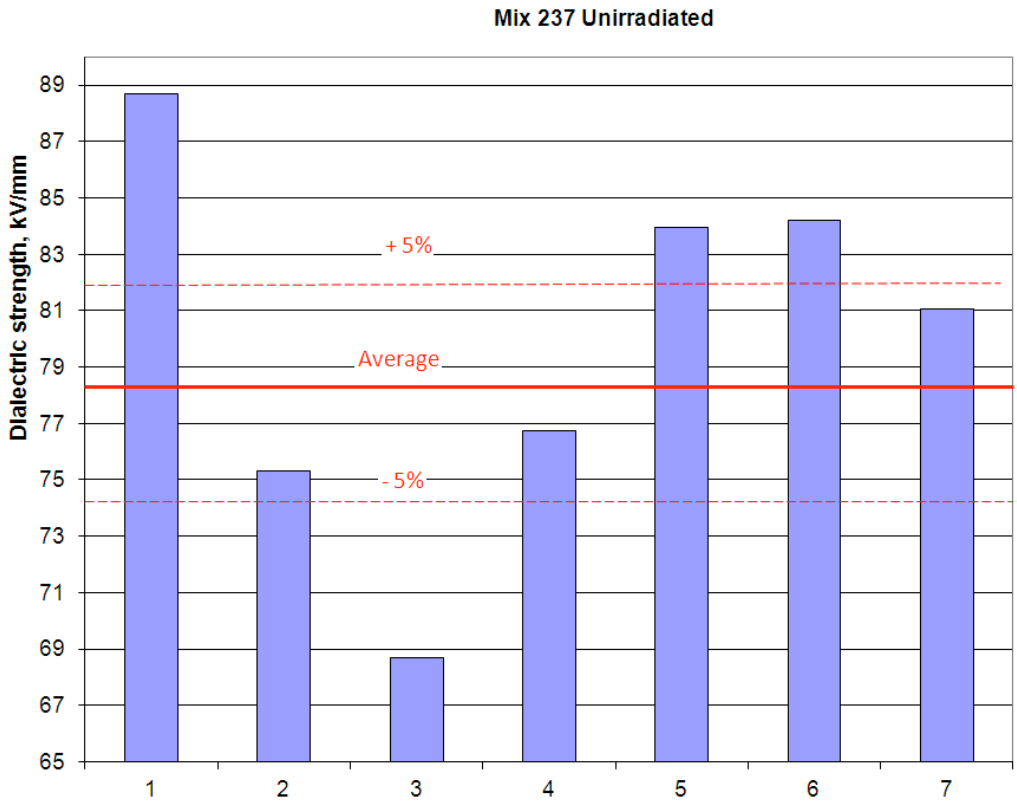


Figure 6.12. Unirradiated MIX 237electrical breakdown strength

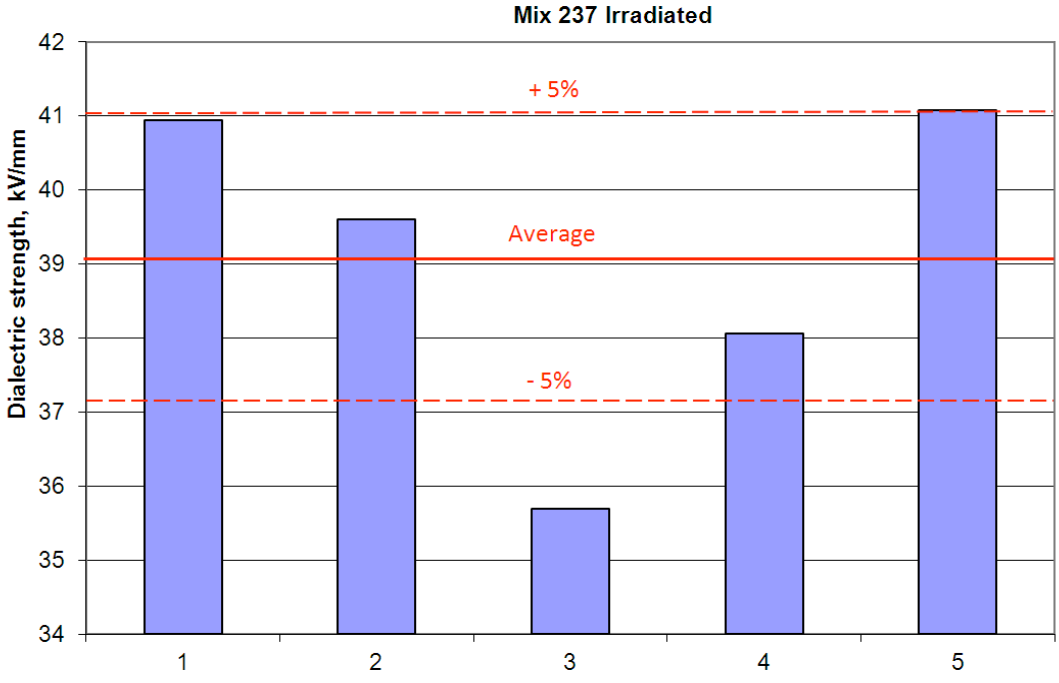


Figure 6.13. Irradiated Mix 237 electrical breakdown strength

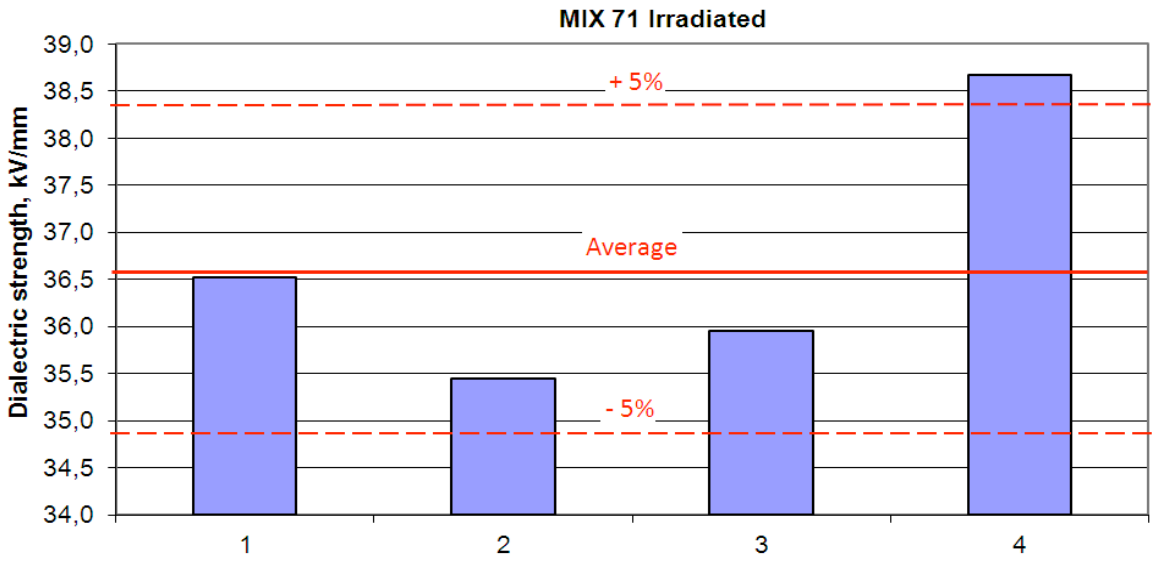


Figure 6.14. Irradiated Mix 71 electrical breakdown strength

Figure 6.15 present the summary of the electric strength certification test results. The bar height represents an average value of electric strength obtained for given material, while the error bars represent the standard variation of the test results around the average value. The 5 kV/mm criteria value, required for the SC magnet design, is also marked on the graph.

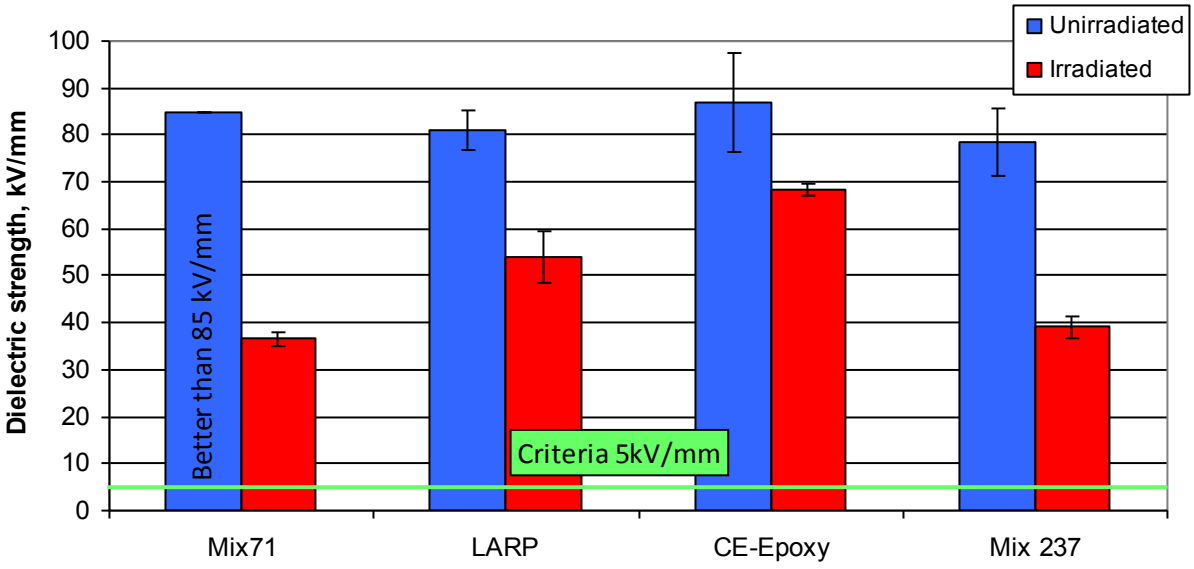


Figure 6.15. Synthesis of electrical tests

From Figure 6.15 it can be found that 50 MGy electron irradiation degrades the electric properties of the considered insulation materials. The highest degradation in the electric properties can be observed for Mix 71 and Mix 237 materials. Lower, but still significant degradation occurs for LARP and CE-Epoxy materials. Nevertheless, even for the most degraded insulation the electrical strength is a few times higher than required 5 kV/mm.

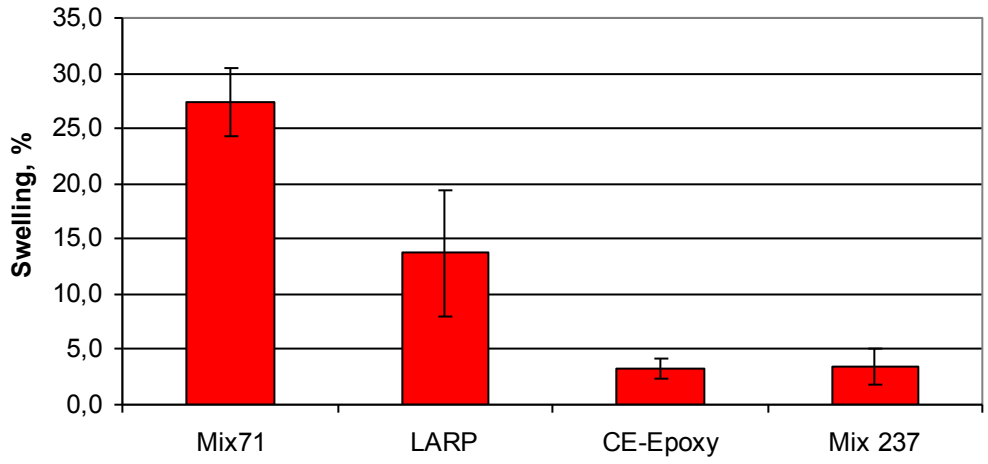
Due to program time constrain it was impossible to perform the test of materials irradiated with other doses, especially higher than 50 MGy. Therefore, an electric strength degradation

with irradiation doses was impossible to determine. Such characteristic would allow prediction of the material degradation at higher doses. It is strongly recommended to perform an additional test program with the irradiation doses as high as 100 MGy, if possible.

6.6 MATERIAL SWELLING EFFECTS

Organic materials can increase their volume due to irradiation. This effect, called the material swelling, is quite important to take into consideration in the SC magnets design, because swelled insulation provides additional mechanical pre-stress in a magnet coil structure.

With the irradiated electric samples it was in addition possible to determine the material swelling by measurement of the samples thickness on an unirradiated sample part and comparison with the thickness in the irradiated part. The swelling effect for all tested materials is presented in Figure 6.16. With the bars the average (from 5 samples) swelling values are represented, while the error bars represent the standard variation of the measurements results around the average value.



6.16. Insulation materials swelling due to 50MGy electron irradiation at 77K

Figure 6.16 shows that significant swelling occurs for Mix 71 and LARP materials – 27.5% and 13% respectively. For CE-Epoxy and Mix 237 the swelling effect is much smaller – below 5%.

7. MECHANICAL CERTIFICATION OF IRRADIATED INSULATION SAMPLES

7.1. MECHANICAL CERTIFCATION METHOD

For mechanical tests of laminate materials two methods are commonly used: a method based on EN ISO 14130 standard: "*Fibre-reinforced plastic composites – Determination of apparent interlaminar shear strength by short-beam method*" and a method based on ISO EN 15024" *Fibre-reinforced plastic composites — Determination of mode I interlaminar fracture toughness*"[8]. Nevertheless, in both methods the tested specimen volume needs to be uniformly irradiated. For this, the sample length should not exceed 5 mm in length (limitation due to beam penetration depth) and rectangular cross section shape dimensions should inscribe to the 5 mm - 7 mm (limitation due to beam diameter) circle. Since the

EN ISO 14130 standard specified the test specimen as rectangular bars of uniform thickness h, the length of the specimen should be l=10h and its width b=5h. It imposes the maximum specimen dimensions as: (thickness × width × length) 0.5×2.5×5.0 mm×mm×mm. It should be pointed out, that for the selected irradiation method only one such specimen can be irradiated at any time. Therefore, and due to fact that the above listed mechanical certification methods require the test of 5 specimens of a given material, irradiation time of mechanical samples can be relatively high.

A compromise between a proper certification test method and relatively low specimen's irradiation time brings the implementation of the ISO 37:2005 standard: "*Rubber, vulcanized or thermoplastic -- Determination of tensile stress - strain properties*" for laminates. ISO 37:2005 standard doesn't impose a specimen thickness so a sample thickness of 0.5 mm, suitable for simultaneous irradiation can be used.

7.2. MECHANICAL TEST SET-UP

For the mechanical properties certification tests a commercial available tensile test stand TIRAtest Table Unit 2705 has been used. The test stand allows a tensile test up to 5 kN with a speed up to 10 mm/min. The set-up consists of two sample holders: lower, fixed to the test table, and upper, fixed to the bar driving by the motor. Parameter such as force, elongation and test time can be computer recorded. A view of the test set-up is presented in Figure 7.1.

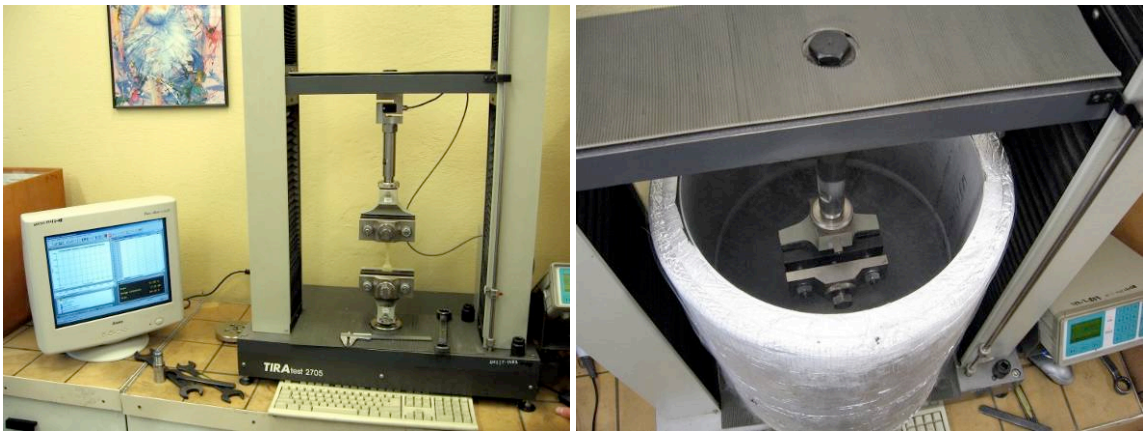


Figure 7.1. View of the mechanical test set-up (left) and its up-date layout with the cryogenic vessel.

For the WP7.2.1 certification test purpose, the mechanical test set-up has been updated with a liquid nitrogen foam insulated vessel – see Figure 7.1. The vessel design allows installation of the set-up lower sample holder inside the vessel, while the vessel can be installed on the set-up table instead of the lower holder.

7.3. MECHANICAL TEST METHODOLOGY

For the mechanical certification tests purpose a special shape of material sheet has been designed – see Figure 7.2. In the central part the dumb-bell Type 1 mechanical test sample shape (in accordance to the ISO 37:2005 standard) is created, while an external part, with 4 circular holes in the corners, aims for sheet installation in the irradiation set-up sample holder. The sheet shape allows fast extraction of the test sample by easy cut off of the sheet in the places marked with the dotted lines in Figure 7.2. Since the required sheet shape is difficult, a water-jet cutting technology has been used for mechanical sample sheets preparation.

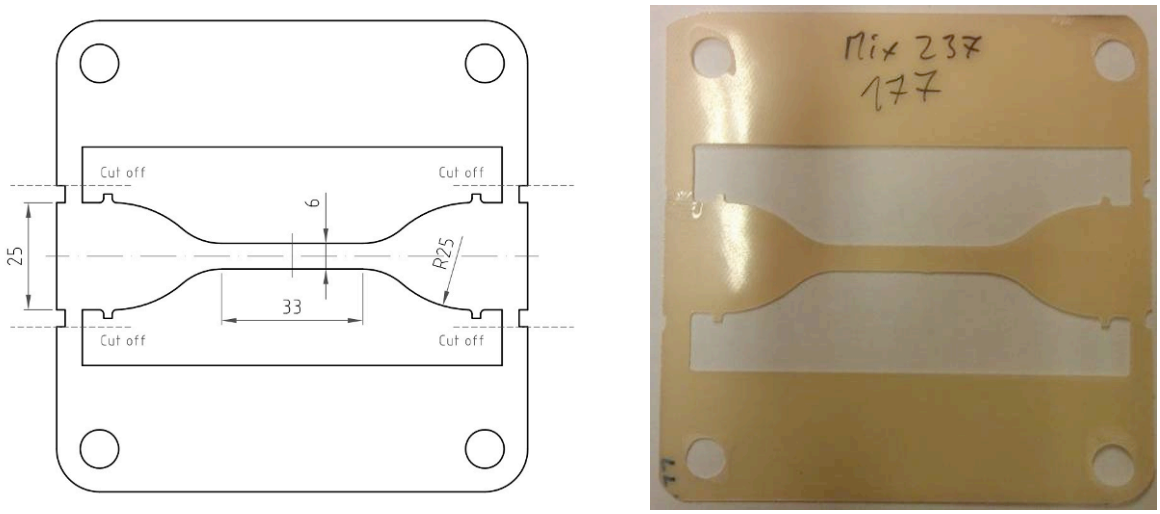


Figure 7.2. View of the mechanical test set-up (left) and its up-date with the cryogenic vessel.

As the tested materials are fiberglass – matrix material laminate, where the glass fibers are expected to be radiation resistant, the degradation of the mechanical properties due to irradiation is dominated by the matrix material degradation. Therefore, in order to determine the matrix material strength a test sample axis and glass fibers in the sample are at a 45-degree angle from each other. Thanks to that, the tensile force acts more on the matrix material than on the fibers. Actually, it can be expected, that the tensile test will determine the fibers – matrix material bond (adhesion) strength, which strongly depends on the matrix material properties.

Unirradiated samples were methanol cleaned and installed in a lower sample holder already placed in the liquid nitrogen vessel. Then the liquid nitrogen vessel was installed on the set-up table and the sample was attached to the upper sample holder. Next the vessel was slowly filled with liquid nitrogen. During this phase a thermal constriction of the test set-up elements and sample itself has been observed, what caused uncontrolled sample tension generation. To avoid this effect the upper test set-up bar was gradually moved down to keep the tension force around the 0 N value.

During the irradiated sample installation, special care was taken to avoid the sample warm-up. Therefore, the irradiated sheets were removed from the transport vessel and quickly moved to the nitrogen vessel. The vessel had been pre-cooled with liquid nitrogen and still was collecting a small portion of the liquid to keep it cold. There, the test samples were cut-off from the sheets and installed on the lower sample holder. Next, the installation procedure was identical as for the unirradiated samples.

Before each test the thickness and the width of the investigated part of each unirradiated sample were measured with a micrometer. In the case of an irradiated sample the dimensional measurement was skipped, because the avoidance of the samples warm-up was the priority, and the dimensional verification process could cause unavoidable sample warming. It was found that unirradiated sample dimensions did not vary in a significant way. Therefore, it was assumed that both unirradiated and irradiated samples had equal dimensions of the test part.

Each certification test has been performed with 1 mm/min ripper velocity. The tensile force has been recorded each 0.02 s. The material stress value was calculated as the tensile force value divided by a sample test part cross-section area.

7.4. MECHANICAL TEST RESULTS

Figure 7.3 present the tensile strength test results for the unirradiated and irradiated LARP insulation material, while the samples view after the tests is presented in Figure7.4.

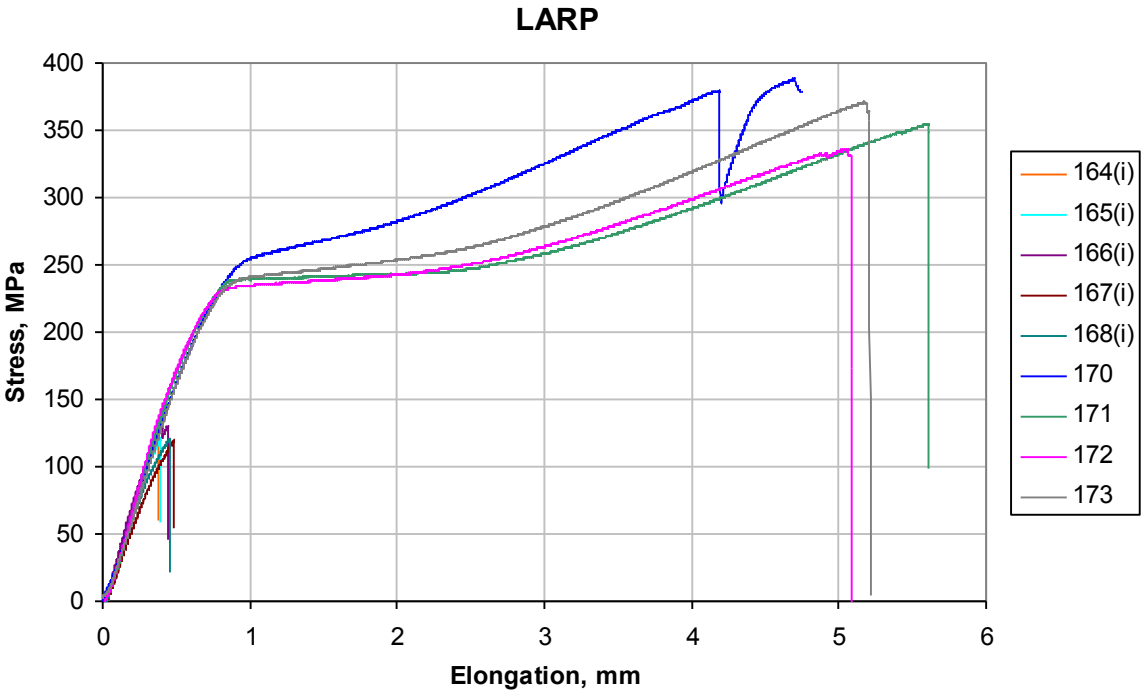


Figure 7.3. UTS test results for unirradiated and irradiated (i) LARP samples

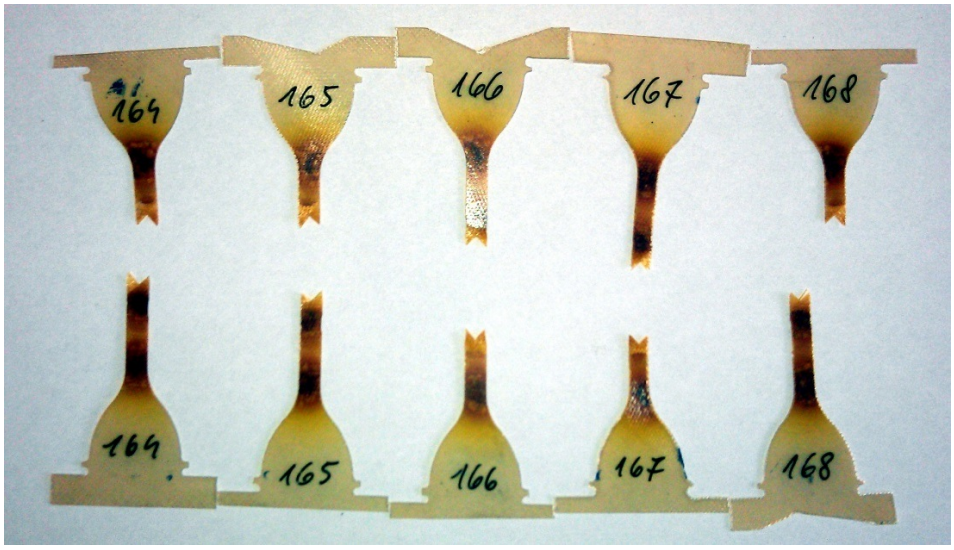


Figure 7.4. View of unirradiated (lower) and irradiated (upper) LARP samples after the UTS test

It can be found that the Ultimate Tensile Strength (UTS) for unirradiated samples is about 350 MPa , while the average elongation at the UTS is about 5.2 mm. For 50 MGy electron irradiated samples the UTS is reduced to 120 MPa and elongation at UTS is reduced to 0.5 mm.

From Figure 7.4 it can be seen that the both, unirradiated and irradiated samples have been broken in the middle of the test part, which proves the correctness of the sample preparation methodology as well as the sample irradiation uniformity obtained with the proper irradiation pattern - see description in the paragraph 5.7.

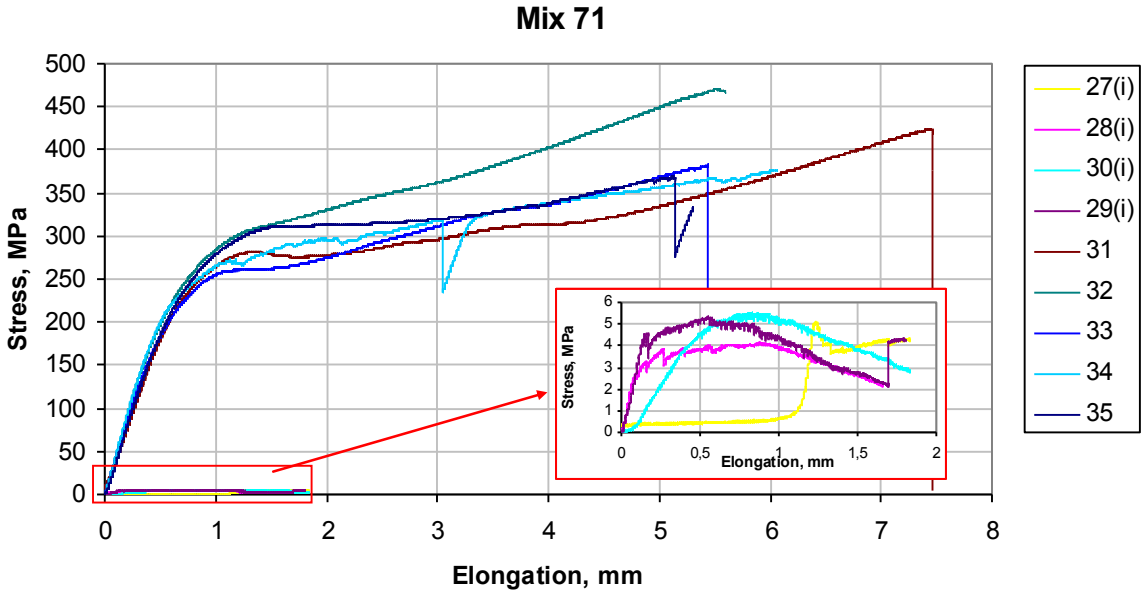


Figure 7.5. UTS test results for unirradiated and irradiated (i) Mix71 samples

Figure 7.6. View of unirradiated and irradiated LARP samples after the UTS test

The UTS test results for Mix 71 are present in Figure 7.5 while the views of the samples after the test are presented in Figure 7.6.

From Figure 7.5 it can be concluded that the UTS values for unirradiated Mix71 samples are not as regular as for LARP samples. Nevertheless the average UTS value remains in the range of 380 MPa – 420 MPa.

For Mix 71 irradiated samples the UTS is almost not measureable – the maximum UTS values are as low as 6 MPa only. Due to the UTS measurements and the view of the irradiated samples after the test (samples are broken in two locations) it can be concluded that the 50 MGy electron irradiation completely destroyed the Mix 71 material. Therefore, the Mix 71 insulation should be not longer be considered as potential electrical insulation material of the superconducting accelerator magnet coils.

At the time of the present report the Mix237 and the CE-Epoxy mechanical samples are under preparation for mechanical testing. The mechanical test results for these materials will be known in the near future and the present report will be updated.

8. THERMAL CERTIFICATION OF IRRADIATED INSULATION SAMPLES

8.1. THERMAL CERTIFICATION TEST

Thermal properties certification tests have been performed using a drum method in superfluid HeIIp (pressurized HeII) conditions. This method allows simultaneous determination of the thermal conductivity and the Kapitza resistance of the tested samples. Experimental data concerning those material properties are essential for HeIIp cooled SC magnet thermal modeling and for magnet design purposes, which is a goal of WP7.2.2 and WP7.3 of the EuCARD program.

In the drum method, minimum 2, typically 3 to 5 different thickness material samples need to be tested. In the certification program 4 different thickness of one tested material are specified to be 0.1 mm, 0.2 mm, 0.3 mm and 0.4 mm. The materials have been tested with use of dedicated sample holders placed inside the temperature-controlled bath of pressurized HeII.

8.2. THERMAL TEST SAMPLE HOLDER DESIGN

The sample holder consists of a central flange and two sets of sample and compression flanges screwed to each side of the central flange. All flanges are made of stainless steel. The schematic construction and views of the holder are presented in Figure 8.1. Inside the central flange an electrical heater (ceramic resistor) and a temperature sensor are located. The sample material is glued with help of 3M DP190 Scotch-Weld Epoxy Adhesive to the sample flange. The glued area is pressed by the compression flange being as required during the epoxy drying.

The sample has a circular shape with a diameter of 100 mm. The external 10 mm circumference of the sample is used for sample installation on the flange, so the active heat transfer area of the sample is limited by an 80 mm diameter circle. Two separate samples with this same thickness are installed on the central flange at once.

The central and the sample flanges contact surfaces are sealed with Apiezon N vacuum grease which helps to avoid uncontrolled superfluid helium leaks during the measurements. The assembled holder creates a confinement that is limited by the tested material and the central flange material.

The confinement is filled with superfluid helium from the cryostat test vessel bath with help of a long and small inner diameter capillary that ensures a minimization of heat losses due to heat conduction through the superfluid helium. The capillary caries also the temperature sensor and the heater instrumentation wires, which constitutes a further limitation of the area for the superfluid helium heat conduction.

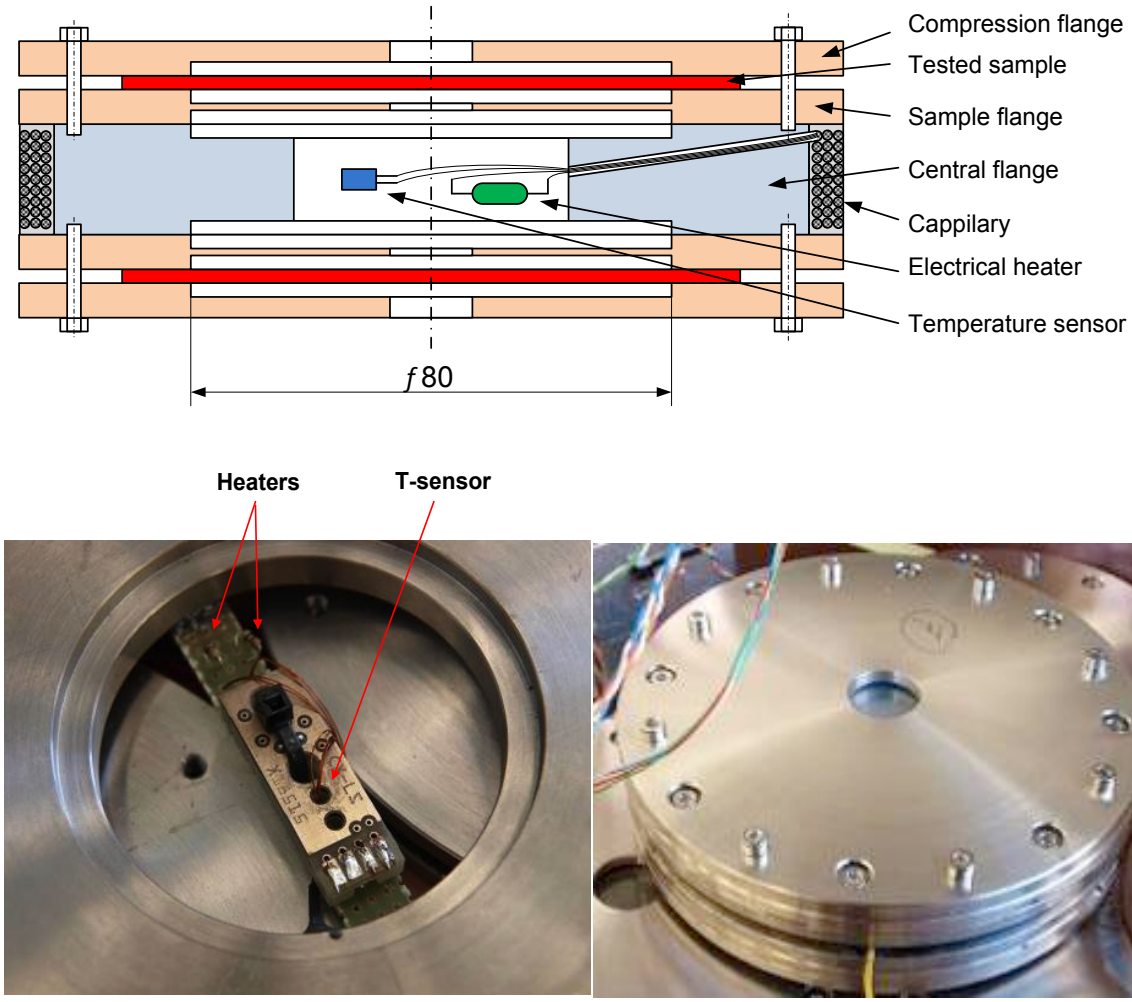


Figure 8.1. Sample holder for thermal tests of irradiated samples

8.3. THERMAL TEST METHODOLOGY

During the tests the superfluid helium inside the sample holder is heated up and its temperature is measured. As the tested material thermal resistance, due to its very small thickness, is much lower than the holder material, it can be considered that the heat generated inside the holder is transferred to the bath through the tested sample only. With such assumption, the total thermal resistance of the material can be calculated as the value of the heating power flux divided by the temperature difference between the holder inner space and external bath. The total thermal resistance of the sample is the sum of the sample heat conduction resistance and the double Kapitza resistance between the sample surfaces and the external and the internal superfluid baths. The temperature differences between the baths are kept in a range of 10 mK – 30 mK. It was shown with thermal modeling and experiments [7] that for temperature differences above 10 mK the regular measurement error provided by the capillary and holder material heat conduction is already negligible. On the other hand, the upper temperature difference is foreseen to be 30 mK to limit the error provided by the assumption that the Kapitza resistance on the both sample surfaces is equal. For different thicknesses of tested material the heat conduction resistance is proportional to the sample thickness while the Kapitza resistance remains constant, which allows determination of both resistance values.

8.4. THERMAL TEST SET-UP

The insulation materials thermal measurements have been split between two Institutes: CEA-Saclay, and PWR. The PWR HeII cryostat scheme is presented in Figure 8.2.

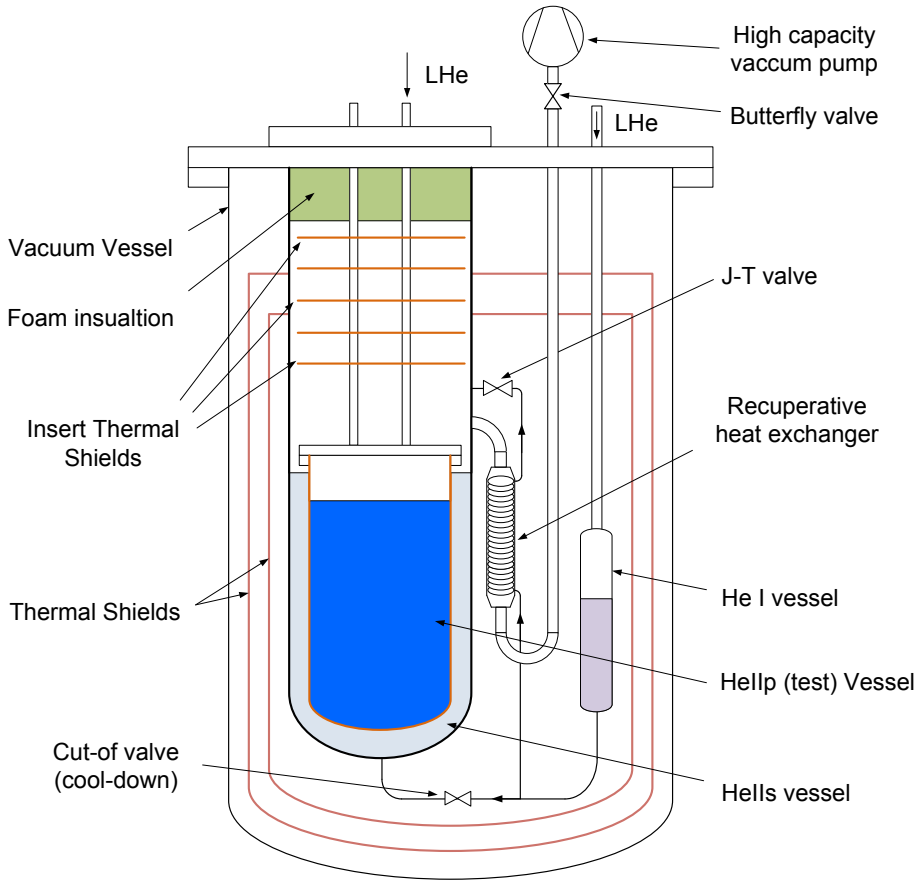


Figure 8.2. Superfluid helium test cryostat flow scheme

The PWR cryostat consists of the vacuum vessel that contains a saturated superfluid helium vessel (HeIIs), a normal helium vessel (HeI), a recuperative heat exchanger, two thermal shields and a cold helium circuit. In the HeIIs vessel the insert with the test, pressurized superfluid vessel (HeIIp), is placed. Inside the vacuum vessel a vacuum thermal insulation is created. Additional reduction of the heat transfer to the cold elements of the cryostat is provided by two thermal shields and multilayer insulation (MLI) covering the shields and all low temperature surfaces.

In the cryostat the temperature of the HeIIs bath can be controlled by means of regulation of the helium vapour pressure above the liquid. In order to create low pressure in the he HeIIs vessel, the vessel is connected with an external high capacity vacuum pump by a large diameter pumping line. The pumping line is equipped with a butterfly control valve allowing to steer the helium flow, to influence the vaporization pressure and finally to determine the helium temperature in the HeIIs vessel.

In the pumping line a recuperative heat exchanger is installed. The purpose of the exchanger is pre-cooling of the liquid helium with the helium vapour before its throttling on the J-T valve and flow to the HeIIs vessel. Thanks to that an increase of liquid fraction in the throttled two-phase helium is achieved, which increases the overall thermodynamic performance of the HeIIs production under nominal operation conditions of the cryostat.

During the cool down phase liquid helium is provided from an external dewar to the HeI vessel and, via the cut-off valve, to the HeIIs vessel. Simultaneously, liquid helium is supplied from second external dewar to the test vessel. After helium transfer termination, the helium transfer line is removed from the test vessel, the cut-off valve is closed, the external vacuum pump is started and the J-T valve is opened. In such operation mode the vacuum pump reduces the helium vapour pressure in the HeIIs vessel, which results in a HeIIs temperature decrease. Since the HeIIp vessel is made of high purity copper, a large heat transfer between the HeIIs and HeIIp baths occurs and the HeIIp helium temperature is lowered as well.

In the cryostat the HeIIp bath temperature and HeIIs level are controlled. The temperature of the HeIIp bath depends on the heat dissipated in the bath (by the test set-up for example) as well as on the temperature and the level of the HeIIs helium. As the HeIIs level is kept constant by the J-T valve, the HeIIp bath temperature is controlled by HeIIs bath temperature adjustment, basically, by the butterfly valve position adjustment. Such a control loop allows for stabilisation of the HeIIp temperature at a desired temperature level with a variation of ±0.25 mK, for whole measurement time period (usually 2-3 hours).

The dimension of the PWR cryostat test vessel (400 mm diameter and 400 mm high), allows simultaneous installation of the 4 sample holders. Thanks to that during one test session (one cool-down) it was possible to test all the samples of given a insulation material. It strongly reduced the test time and costs.

Figure 8.3. Superfluid helium test cryostat at Wroclaw University of Technology

For the thermal measurement purpose CEA-Saclay used the NED cryostat, which design is presented in [10,11] . The pressurized superfluid test vessel dimensions allow simultaneous

placing of two sample holders. A view of the CEA-Saclay cryostat as well as the sample holder installed on the cryostat insert is presented in Figure 8.4.

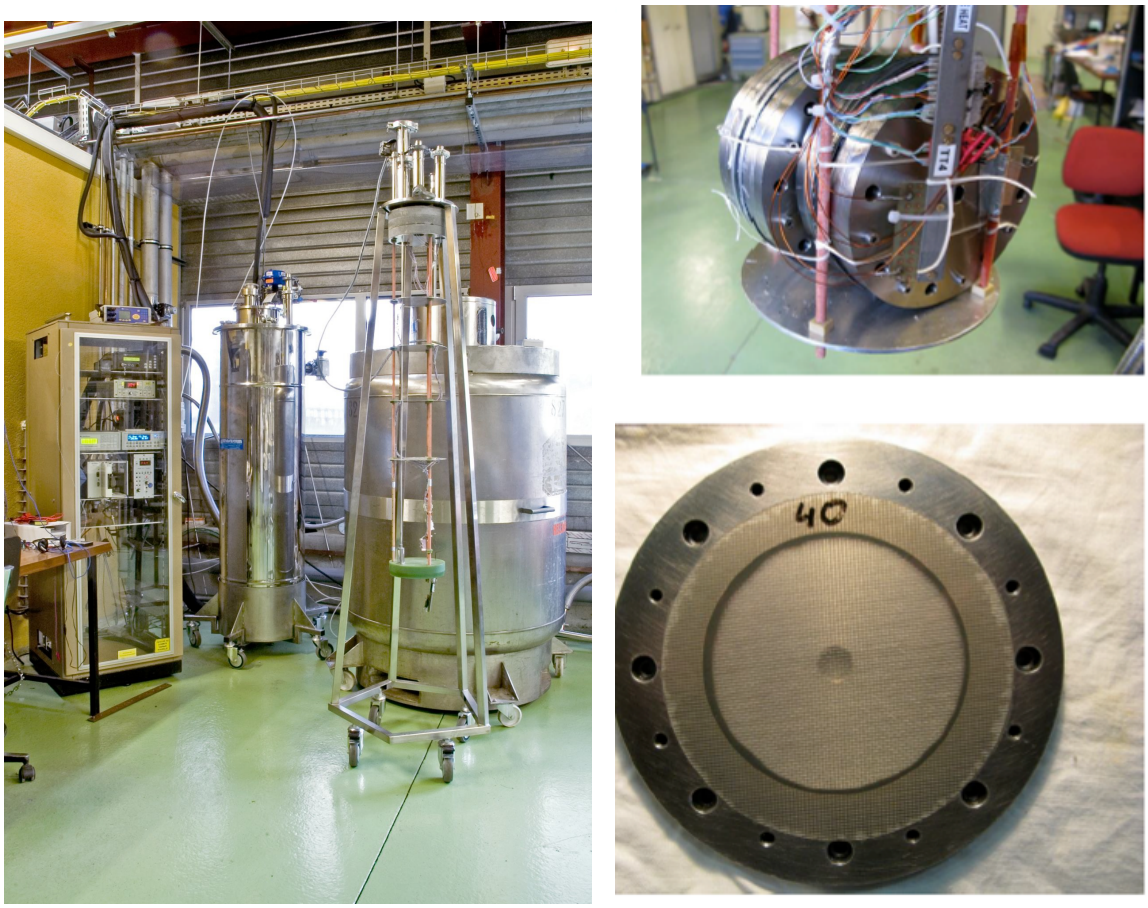


Figure 8.4. Superfluid helium HeII pressurized test cryostat at CEA Saclay

8.5. THERMAL TESTS RESULTS

Within the EuCARD program CEA-Saclay has tested the Mix 237 and CE – Epoxy in a temperature range 1.6 K – 2.0 K while the PWR has tested the LARP insulation in the temperature range 1.5 K – 2.0 K.

The Mix 71 material thermal properties have been previously measured at the CEA Saclay cryostat within the NED program [7]. Since the PWR HeII cryostat was used for such type of measurements for the first time it was decided to repeat the Mix 71 tests at the PWR set-up to check the RWR test results precision and accuracy.

Figure 8.5 presents the thermal conductivity, while Figure 8.6 depicts the Kapitza resistance values for all materials tested within the EuCARD program, and additionally the results for the Mix71 obtained during the NED program. With the points the calculation data are presented while with the solid lines the best data fitted curves are marked. The best data fitted curve equations are summarized in Table 8.1.

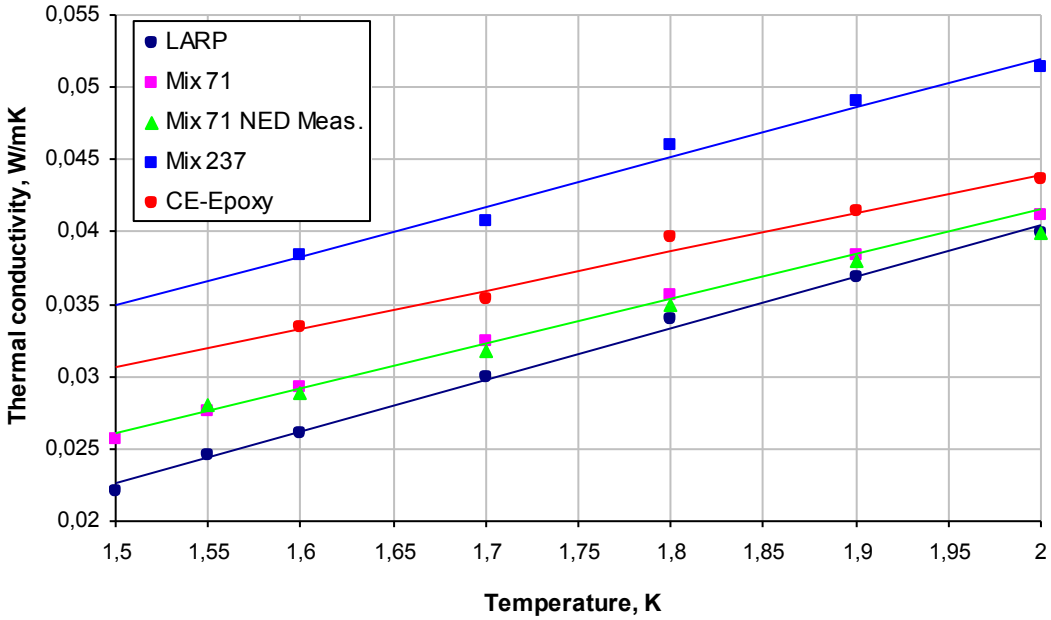


Figure 8.5. Thermal conductivity of the tested samples

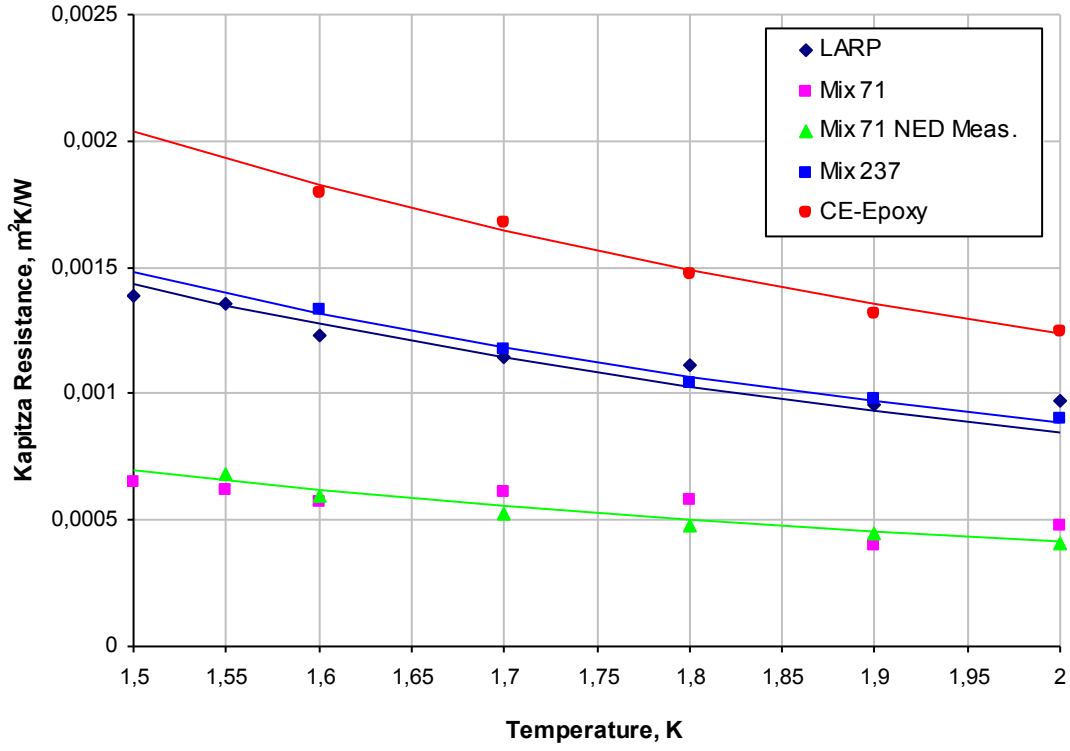


Figure 8.6. Kapitza resistance measurements results

Table 8.1 Best experimental data fitted curve equations

Insulation material	Thermal conductivity	Kapitza resistance
Mix 71 [7]	$25.8\times10^{-3}T-12.2\times10^{-3}$	$1462\times10^{-6}\cdot T^{1.86}$
Mix 237 [9]	$34.2\times10^{-3}T-16.4\times10^{-3}$	$3057\times10^{-6}\cdot T^{1.79}$
LARP	$35.8\times10^{-3}T-31.1\times10^{-3}$	$2997\times10^{-6}\cdot T^{1.79}$
CE-Epoxy [9]	$26.8\times10^{-3}T-9.6\times10^{-3}$	$4114\times10^{-6}\cdot T^{1.73}$

From the experimental results presented in Figures 8.5 and 8.6 it can by concluded that there is a very good agreement of the results for Mix 71 obtained with two different set-ups, what confirms correctness of the drum method as well as the precision of the measurements performed with the PWR set-up.

The thermal conductivity of tested materials in temperature range 1.5 K– 2.0 K can be approximated with a linear function. The highest thermal conductivity is exhibited by Mix 237, while the lowest by the LARP material. Nevertheless, the relative differences are not significant, and from thermal conduction point of view all tested material are suitable for Nb_3Sn coil electrical insulation.

Kapitza resistance values for tested materials differ more significant than their thermal conductivity. It can be seen that higher Kapitza resistance values have been found for the CE-Epoxy, while the lowest for the Mix 71 (from 2.5 to 3.0 times lower than the CE-Epoxy Kapitza resistance). The LARP and Mix 237 Kapitza resistance values are very similar and are of about 2 times higher than the Mix71 Kapitza resistance.

Unfortunately, due to necessity of a long time irradiation of the thermal samples, within the EuCARD program time frame it was impossible to perform the thermal tests for irradiated materials. It is strongly recommended to continue this work. It will allow a full and reliable qualification of electrical insulation materials for future accelerators.

9. CONCLUSIONS

The irradiation method reflecting the spectrum foreseen for future accelerators superconducting magnets has been proposed. The method makes use of industrial electron linac characterized by an energy of 4 MeV and allows the irradiation of the test samples with a 50 MGy dose in a reasonable time. The irradiation is occurring in cryogenic conditions, namely in LN2 environment.

The electrical, mechanical and thermal tests allowing the certification of the insulation materials with respect to their future applicability in SC Nb_3Sn magnets have been proposed and developed. Dedicated cryostats for the sample irradiation and several kinds of the tests have been designed, manufactured and commissioned. The synthesis of the certification program is presented in Table 9.1. It can be concluded that the Mix 71 material must be avoided due to its bad after-irradiation mechanical properties, while the LARP material can be considered as potential candidate for electrical insulation in HEP accelerators making use of Nb_3Sn superconductor. The usability verdict for Mix237 and CE-Epoxy will be given after the completion of the irradiation and test program.

Table 9.1. Summary of the electrical insulation materials certification program (properties after irradiation).

Material	Electrical properties	Mechanical properties	Thermal properties (non-irradiated / irradiated)		Applicability for accelerator magnets
Mix 71	Good	Very bad	Very good	TBD	Non applicable
Mix 237	Good	TBD	Good	TBD	TBD
LARP	Very good	Satisfactory	Good	TBD	Applicable
CE Epoxy	Very good	TBD	Satisfactory	TBD	TBD

TBD – to be defined

Due to the fact that the thermal performance of the irradiated samples has not been measured it is strongly recommended to continue the certification process allowing a full categorization of the proposed candidates.

10. REFERENCES

[1] L. Bottura, R. Flukiger, A. Ballarino, F. Liberati, L. Oberli, I. Pong, G. de Rijk, *An R&D program to Address Irradiation Effects in Superconductors for LHC* , EuCARD workshop on insulator irradiation, December 2, 2009 , CERN, Geneva, CH

[2] R. Flukiger, *Radiation spectra in LHC phase II - Effect on superconducting materials*, EuCARD workshop on insulator irradiation, December 2, 2009 , CERN, Geneva, CH

[3] S. Wronka, *The analysis of possible certification scenario based on research accelerators located at Świerk Nuclear Center*, NCBJ, June 2010

[4] J. Polinski, M. Chorowski, P. Bogdan, *Task 7.2.1 – Radiation certification status report*, EuCard-WP7-HFM Collaboration Meeting , CERN, Geneva 26.11.2010

[5] J. Polinski, M. Chorowski, P. Bogdan, S. Wronka, *Task 7.2.1 – Radiation certification status report*, EuCard-WP7-HFM Collaboration Meeting – CEA Grenoble 22.03.2011

[6] J. Polinski, M. Chorowski , E. Roszak, Review of literatures concern properties of low temperature irradiated epoxy glass fibre composites, 3th Radiation Working Group Meeting, 12.01.2010, phone conference

[7] B. Baudouy, J. Polinski, Thermal conductivity and Kapitza resistance of epoxy resin fiberglass tape at superfluid helium temperature, Cryogenics 49 (2009), p. 138–143

[8] F.Rondeaux: *Insulator radiation hardness: Definition of tests and samples*, EuCARD workshop on insulator irradiation, December 2, 2009 , CERN, Geneva, CH

[9] S. Pietrowicz, A. Four, S. Jones, S. Canfer, B. Baudouy ,*Thermal conductivity and Kapitza resistance of cyanate ester epoxy mix and tri-functional epoxy electrical insulations at superfluid helium temperature*, Cryogenics 52 (2012), p. 100–104

[10] M. Chorowski, J. Polinski, B.Baudouy, F.Michel, R. van Weelderen, *Optimization of the NED cryostat thermal shielding with entropy minimization method*, CryoPrague 2006. Proceedings of the Twenty First International Cryogenic Engineering Conference (ICEC 21). Icaris, 2006, p. 669-672

[11] B.Baudouy, J. Polinski, L. Vieillard, *Commissioning of the NED cryostat*,CARE-Report-2006-031-NED

[12] DuPont, *Summary of Properties for Kapton® Polyimide Films* , www2.dupont.com

[13] E. Tuncer, I. Sauers, , D.R. James , , A.R. Ellis, *Electrical Insulation Characteristics of Glass Fiber Reinforced Resins*, IEEE Transactions on Applied Superconductivity, Vol. 19, No. 3, June 2009