Annex to

Design Requirements and Guidelines Level 1 (DRG1)

Structural Material Database

Article 5. Qualification of Irradiated Insulation Materials (Document No. 4)

Association Euratom – ÖAW

TECHNOLOGY TASKS

Next step

Field: Magnets

Technology Task M16/03

ELECTRICAL PROPERTIES OF MAGNET INSULATION MATERIALS IN THE IRRADIATED STATE

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Final report

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1. Task objective and work program

Originally, a major part of the ITER Magnet Insulation Program had been assigned to the Russian Federation Home Team (RFHT), in particular irradiation and testing of various insulator properties in the Ekaterinburg Reactor Facility. However, after the cancellation of the RFHT test program, the Joint Central Team (JCT) devised a "Ekaterinburg Replacement Program" for further consideration by the other Home Teams (Task Proposal of January 28, 1997 from ITER JCT to EUHT). After extended discussions, the present task was agreed upon. It represents the core of the original program and refers to the assessment of the electrical properties (i.e. the dielectric strength and the breakdown voltage) of a composite, which is manufactured by a company of the EUHT, and to measurements of swelling and weight loss following low temperature irradiation in the FRM Garching.

2. Experimental

The irradiation experiments were not part of our project, but carried out in the context of the current research contract between the EUHT and the FRM Garching. In total, 9 Al-containers, filled with all of the samples needed for our research program, provided us with 4 irradiated samples of each type and specimen geometry, irradiated to a fluence of 1×10^{21} , 5×10^{21} and 1×10^{22} m⁻² (E>0.1 MeV), respectively. The responsibility of the Garching team ended with the storage of the samples for radioactive decay and a final warm-up cycle to room temperature for shipment to Vienna.

The Vienna group was responsible for the project coordination, for the transport of the radioactive materials to Vienna, and for the disposal of the radioactive materials after completion of the experiments. All of the experiments were carried out at the Atomic Institute in Vienna in cooperation with the Institut für Werkstoffe der Elektrotechnik, TU Wien. Firstly, the weight and the dimensions of all the samples were assessed prior to and following reactor irradiation, and secondly, the electrical measurements of the dielectric strength and of the breakdown voltage were made on all the samples at 77 K according to standard procedures.

In summary, important information on the electrical property changes of the magnet insulation after reactor irradiation according to ITER specifications and after a warm-up cycle to room temperature

as well as information on swelling and weight loss of this magnet component became available through this program in a form, which is suitable for ITER design purposes.

2.1 Preliminary tests on disk shaped samples

Prior to finalizing the sample geometries and the specifications for the manufacturing of the samples, it was decided to conduct tests of the electrical breakdown measurements on disk shaped materials and composites at Technical University of Vienna in the end of 1997. This decision was primarily motivated by the fact, that the sample geometries for our program were simply dictated by the space limitations in the FRM Garching, which required much smaller sample dimensions than originally envisaged for the Ekaterinburg reactor.

The results showed indeed, that the breakdown voltage could not be reliably assessed in this sample configuration. Based on the first tests and the experience gained at that time, further work was done in the beginning of 1998, in order to establish sample geometries, which were suitable for a reliable assessment of the electrical properties (i.e. the breakdown voltage and the dielectric strength). As a result, we suggested to reduce the sample thickness from 1.0 to 0.5 mm in the case of the pure laminate, and from 2 to 1 mm in the case of the sandwich (i.e. a thickness of 0.5 mm for the laminate and for the stainless steel each). In the case of the sandwich, we also suggested to reduce the diameter of the steel disk to 8 mm. It has to be pointed out, that, although these preliminary tests have certainly led to delays compared to our original schedule, we believe that they were absolutely worthwhile and beneficial to the program as a whole.

2.2 Final sample geometries

The final sample geometries and specifications, which were chosen in view of the existing space limitations in the Garching low temperature irradiation facility, can be seen in Figures 1 and 2. Three different types of specimen geometries were investigated. The first is a glass-fiber-resin laminate with a diameter of 12.5 mm and a thickness of 0.5 mm, the second a glass-fiber-resin laminate-stainless steel sandwich with a diameter of 12.5 mm and a total thickness of 1 mm, and the third a tubular arrangement of stainless steel and insulation, i.e. a conductor insulation prototype sample, with an outer diameter of 12 mm and a total length of 32 mm. Photographs of the specimen geometries are presented in Figures 3 and 4.

2.3 Distribution of sample specifications

The final sample specifications were then communicated to the EUHT. A detailed justification for the change of the design was presented at a meeting on radiation effects in insulators, which was presided by JCT and hosted by the EUHT in Garching, on March 13, 1998. The EUHT contacted several European companies and requested quotations for the sample fabrication. The final order was placed in July, the delivery - originally planned for October 1998 - was delayed to January 1999.

2.4 Sample manufacturing process

The investigated insulation system for the Toroidal Field Model Coil (TFMC) of ITER is a laminate, which consists of a combined Kapton/R-glass-fiber reinforcement tape, vacuum-impregnated with an epoxy DGEBA system. The above test specimens were manufactured by Ansaldo, Genova, Italy. The manufacturing processes were as follows.

Pure disk shaped laminate

The laminates from which the disks were cut, were obtained by wrapping the combined Kapton/glass-fabric tape (half-overlapped) of the same kind as the one used for the TFMC around a steel plate. The two longitudinal sides of the steel plate were well rounded, in order to enable safe wrapping. Before the application of the insulation, Mylar was wrapped around the plate for easy detachment of the laminate after the vacuum and pressure impregnation (VPI) process. Two extra steel plates and a set of bolted beams were used to press the insulation during the impregnation process. The whole arrangement was then vacuum impregnated. After the VPI process, two insulation plates were obtained by cutting the insulation along the two longitudinal sides of the steel plate. The disk shaped laminates were obtained by laser cutting the 0.5 mm thick insulation plates.

Disk shaped laminate-stainless steel sandwich

The manufacturing procedure of the insulation plates was the same as above. For the laminate-steel sandwich, the following fabrication steps were made. A 0.5 mm thick Teflon sheet was used to fix the steel disks on the insulation plate. The Teflon sheets had 35 holes with a diameter of 8 mm to accommodate the steel disks. As for the pure disk shaped laminates, an outer frame was used to press the steel disks against the insulation, and further on, the insulation against the core steel plate. After the VPI process, the disk shaped insulation plated with the steel disks (sandwiches) were cut from the mould by laser cutting.

Conductor insulation prototype sample

This specimen consists of two concentric stainless steel tubes and an insulation tube located between the steel tubes. In order to avoid excess voltages at the ends of the tubes, the sharp edges of the steel tubes were softened by flanging the inner and outer tube radially inwards and outwards, respectively, with suitably shaped tools. Four pieces of Teflon fillers were then manufactured and fitted inside the inner steel tube to keep this space free from resin during the impregnation process. The Kapton-glass insulation tape was then wrapped onto the inner steel tube, and the Teflon fillers were used to define the two boundaries of the whole tube. The outer steel tube was made by rolling and pressing a sheet of stainless steel onto the surface of the insulation tube. Finally, the whole arrangement was wrapped with a Tedlar (Teflon-type) tape on the outside to enable the extraction of the specimen from the cured resin bath. After the impregnation process, the Teflon filler and the Tedlar tape were removed.

2.5 Assessment of sample quality

When the complete set of samples was delivered, the specimens were subjected to an optical inspection. Firstly, all of the disk shaped laminates showed surfaces, which were not completely smooth. We noticed especially at the locations of the adhesives between the Kapton foils and the glass-fiber tapes the formation of small grooves, which led to a considerable decrease of the thickness of the laminate in these regions. Secondly, the Kapton-glass fabric tape was wrapped approximately half-overlapping within the laminate, and therefore, regions with one or two Kapton foils along the thickness were produced.

Consequently we noticed during the electrical tests, that the measured breakdown strength depended strongly on the position of the electrodes on the sample surface. Although we tried to find the optimal position of the electrodes for each sample (as far as this could be done considering the small sample dimensions), both effects lead to rather high standard deviations of the evaluated breakdown strengths (cf. Figures 10,12).

On the other hand, the insulation of the conductor insulation prototype samples were manufactured with a thickness of 1 mm instead of 0.5 mm (cf. section 2.1). The reason might have been the first draft of the "Ekaterinburg Replacement Program", in which a thickness of 1 mm had originally been foreseen. Because of this misunderstanding, the breakdown voltage could not be assessed on this type of specimen (cf. section 3.2).

2.6 Device for electrical measurements

For the electrical tests a specially designed device was manufactured at ATI to allow testing of both unirradiated and irradiated specimens under liquid nitrogen. The disk shaped laminates could be positioned easily and quickly between the two electrodes (cf. below). The device was placed into a polystyrene container filled with liquid nitrogen. As can be seen in Figure 5, the stainless steel electrodes were fixed onto brass rods, and further on, mounted into an insulating plastic frame. Two connecting leads were fixed with screws on both sides of the brass rods leading to the high voltage power supply.

2.7 Irradiation experiments

After initial measurements of the sample dimensions and weights, the samples were sent to Garching for irradiation in March 1999. All irradiations were performed in the FRM I reactor at 5 K to neutron fluences of 10^{21} , $5x10^{21}$ and 10^{22} m⁻² (E>0.1 MeV). This reactor is operating at a γ -dose rate of $2.8x10^6$ Gyh⁻¹, a fast neutron flux density of $2.9x10^{17}$ m⁻²s⁻¹ (E>0.1 MeV), and a total neutron flux density of $9.5x10^{17}$ m⁻²s⁻¹, respectively. The irradiation in Garching was completed in June 1999. After irradiation, all samples were warmed up to room temperature and stored in Garching until they were shipped to Vienna in August 1999.

2.8 Induced radioactivity and sample changes

After completion of the irradiations the samples were stored for radioactive decay. Whereas reactor irradiation of fiber-reinforced plastics leads to a very low activation of the material, stainless steel shows a rather high radioactivity. Consequently, calculations of the gamma dose rates of the stainless steel used for the samples were made to estimate the time for radioactive decay, the transportation and the handling of the samples during electrical testing. As can be seen from Figure 6, especially the nuclides Na-24, Al-28, Cr-51, V-52, Cr-55, Mn-56, Co-58C, Fe-59, Cu-64 and Ni-65 show higher gamma dose rates, but the low or moderate half-life times (from a few hours to a few months) allowed safe handling of the samples.

When the complete set of irradiated samples arrived in Vienna, the specimens were subjected to optical inspection. The outer layers of some specimens irradiated to a fluence of 10^{22} m⁻² (E>0.1 MeV) were found to have delaminated completely from the compound.

3. Results

3.1 Swelling and weight loss of the irradiated samples

The sample dimensions and weights were measured again in September and then analysed in order to evaluate the radiation induced swelling and weight loss of the specimens. The results are shown in Figures 7 and 8, where the swelling and the weight loss of the samples are plotted as a function of the fast neutron fluence. The swelling of the laminate (Figure 7) amounts to 1.5 % at a fast neutron fluence of 1×10^{21} m⁻² and to ~2 % at a fast neutron fluence of 5×10^{21} m⁻². A considerable increase of swelling (to ~5 %) is found at a fluence of 1×10^{22} m⁻². The swelling of the sandwich (fiber reinforced plastic laminate on stainless steel) amounts to ~1 % at 1×10^{21} m⁻² and increases continuously to ~9 % at a fluence of 1×10^{22} m⁻². We wish to point out that those parts of the sandwich consisting only of the pure laminate were also measured. Within experimental accuracy, the data on swelling agree with those obtained on the pure disk shaped laminates. Therefore, we assume that the difference in the swelling behavior between the pure disk shaped laminate and the sandwich may be related to the laminate-stainless stell interface which could not be measured seperately. An optical investigation of the samples did not show any obvious effects for both types of samples, and therefore, did not lead to further insights. On the other hand, the swelling of the

conductor insulation prototype sample (outer diameter) is almost negligible (~ 0 to 0.5 %, cf. Figure 7). It should be noted, that each data point in Figures 7 and 8 represents an average calculated from five to eight measurements (samples showing significant deviations were rejected in the statistical calculations), except those for the conductor insulation prototype sample, where the average was calculated from only two measurements.

As can be seen in Figure 8, the weight loss of the laminate increases continuously to 2 % at a fluence of 1×10^{22} m⁻², in good agreement with literature data on similar laminates.

The data on swelling (cf. Figure 7) will be addressed in more detail. At a fast neutron fluence of $1 \times 10^{21} \text{ m}^{-2}$ (E>0.1 MeV), no remarkable difference between the pure laminate and the sandwich was found. Special attention should be paid to the fluence of $5 \times 10^{21} \text{ m}^{-2}$, which is the designated ITER dose level. At this point we calculated the following data (mean value and standard deviation) on swelling. For the laminate 2.2 ± 1.2 % and for the sandwich 4.1 ± 1.8 %. Because of significant deviations, three measurements were rejected in each case. As can be seen in Figure 7, the error bars are almost half overlapping at this point. The overall effect is certainly ~ 4 %. A considerable scatter of the data can be observed at a fluence of $1 \times 10^{22} \text{ m}^{-2}$. Again, three measurements were rejected in each case, but no overlap of the error bars occurs anymore. The sandwich clearly shows higher swelling. On the other hand, the swelling of the conductor insulation prototype sample (outer diameter) is extremely small (~0 to 0.5 %). This may be attributed to the configuration and the insulation, and thus, effects of swelling are almost avoided. For an application of the insulation system in the configuration of a conductor insulation prototype, the effect of swelling can be neglected.

3.2 Electrical tests on the unirradiated and irradiated samples

From October to December 1999, the electrical tests (i.e. the dielectric strength and the breakdown voltage) were carried out at the Atomic Institute in Vienna in cooperation with the Institut für Werkstoffe der Elektrotechnik, TU Wien, on the unirradiated and irradiated disk shaped laminates, the stainless-steel-laminate sandwiches, and the conductor insulation prototype samples at 77 K.

Scaling experiments on unirradiated disk shaped samples

In the beginning, scaling experiments were made on approximately 0.5 mm thick pure laminates at room temperature (under oil environment) and at 77 K, in order to investigate the influence of the diameter of the disk shaped samples as well as of the size of the stainless-steel electrodes on the measured dielectric strength. The following electrode diameters were investigated.

Disk diameter:50 mmElectrode diameter:12.5 mm (two electrodes opposing)Disk diameter:25 mmElectrode diameter:6.4 mm (two electrodes opposing)Disk diameter:12.5 mmElectrode diameter:6.4 and1.6 mm (electrodes opposing)

Figure 9 shows a photograph of the set of disk shaped laminates used for the scaling experiments. Figure 10 summarizes the scaling results for both test temperatures. No influence of the disk diameter on the dielectric strength was found, which confirms the suitability of 12.5 mm diameter disk shaped laminates for a reliable assessment of the electrical properties (i.e. the breakdown voltage and the dielectric strength) under low temperature irradiation conditions. The dielectric strength is about 10 % lower at 77 K.

Electrical tests on the irradiated samples

Finally, the dielectric strength of the disk shaped laminate and of the sandwich was measured at 77 K prior to and after low temperature reactor irradiation. The breakdown voltage of the test samples was evaluated according to the "Standard Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies" (ASTM D149-97a); "Standard Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials under Direct-Voltage Stress" (ASTM D3755-97) with Appendix B: "Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Electrical Insulating Materials under DC Voltage at Cryogenic Temperatures". A photograph of the measuring device inserted in the liquid nitrogen container and connected with the high voltage power supply is presented in Figure 11. The dielectric strength of the laminate and of the sandwich as a function of the neutron fluence is plotted in Figure 12. As can be seen from this figure, no degradation of the dielectric strength was found for both the laminate and the sandwich over the whole dose range. Slightly higher values were found for the sandwich, but the overall effects are within the calculated standard deviations. The conductor insulation prototype sample (tube) was manufactured with a thickness of the insulations system, which is twice that of the disk shaped

laminates (cf. Figures 1 and 2). Unfortunately, the voltage needed for electrical breakdown through the laminate exceeded the maximum voltage generated by the high voltage power supply, and thus, breakdown could not be achieved on all of these tubes. Nevertheless, all conductor insulation prototype samples were found to withstand a voltage of at least 60 kV dc without electrical breakdown.

3.3 Fractography of irradiated samples after breakdown

After the electrical measurements had been carried out, fractographic investigations of the surface structure of the irradiated laminates were made in an SEM. Typical photographs are presented in Figures 13 to 15, where the surfaces of the laminates are shown following reactor irradiation at 5 K to a fast neutron fluence of 1×10^{21} , 5×10^{21} , and 1×10^{22} m⁻² (E>0.1 MeV), respectively, and following electrical breakdown at 77 K.

For the lower dose level, small epoxy plates of the outer layer of the laminate are found to be delaminated partially from the compound after breakdown. At higher doses, parts of the outer epoxy layers delaminate completely from the compound and are pulled out. In addition, some inner layers were also partly destroyed. In general, higher dose levels lead to larger areas of destroyed material.

4. Conclusions

The quality assessment of the Double Pancake Module of the TFMC of ITER involves the following electrical tests.

Insulation resistance for conductor to radial plate: 500 V dc; dielectric test for conductor to radial plate: 3000 V dc for 1 min followed by 3000 V ac peak-to-peak for 1 min; dielectric test radial plate to ground: 1500 V dc for 1 min; EDA parameter the ITER TF coil (standing 1995): maximum voltage between the layers of 1.33 kV.

Typical insulation thicknesses are in a range between 1 and 2 mm (e.g. TFMC ground insulation: thickness 1.3 mm). Considering the data on the electrical tests presented in this report, we conclude

that the *electrical performance of these materials exceeds* by far the design limits of the ITER EDA criteria.

At a fluence of $5x10^{21}$ m⁻² (E>0.1 MeV), i.e. the ITER dose level, the data on swelling show half overlapping error bars between the pure laminate and the sandwich, the overall effects are within ~ 4 %. In the configuration of the conductor insulation prototype sample, the effects of swelling are almost negligible.

On the other hand it should be pointed out that the *mechanical strength* of a similar insulation system (supplier Alstom, Belfort; also DGEBA epoxy resin, but another basic mixture and curing temperature) was drastically affected by reactor irradiation. Recent experiments (cf. Figure 16) on shear-compression insulation samples compared the Alstom insulation material with other systems prepared by the JAHT and the USHT. At a fast neutron fluence of $5x10^{21}$ m⁻² (E>0.1 MeV) all materials showed approximately the same strength (~ 120 MPa). Upon further irradiation to a fast neutron fluence of $1x10^{22}$ m⁻² (E>0.1 MeV), the JA material remained constant at 120 MPa, the US material degraded down to ~80 MPa, but the EU material failed more or less completely at a strength of only 6 MPa.

In summary, considering these drastic changes on the one hand and the data presented in this work on the other hand, we wish to point out again, that further investigations of the insulation system for the TFMC of ITER should concentrate on the mechanical properties (e.g. resin system, etched Kapton,...) and not on their electrical performance.

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INSULATION ONLY

SANDWICH



(WITH SAND BLASTED SURFACE NEXT TO INSULATION)

Figure 2

CONDUCTOR INSULATION PROTOTYPE



Figure 1: Insulator and insulator-metal sandwich sample.

Figure 2: Conductor insulation prototype sample.



Figure 3: Photograph of the disk shaped laminate and of the sandwich.



Figure 4: Photographs of the conductor insulation prototype sample (tube).





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Calculated gamma dose rates of the radionuclides for the stainless steel.



Figure 7: Swelling of the laminate, the sandwich, and the conductor insulation prototype sample (tube) after reactor irradiation.



Figure 8: Weight loss of the laminate after reactor irradiation.







Figure 10: Scaling result on unirradiated disk shaped laminates.



Figure 11: The measuring device in the liquid nitrogen container connected with the high voltage power supply.



Figure 12: Dielectric strength of the laminate and of the sandwich as a function of the neutron fluence.



Figure 13: SEM photographs of the surface of the laminate following reactor irradiation to a neutron fluence of 1×10^{21} m⁻² (E>0.1 MeV) and electrical breakdown at 77 K.



Figure 14: SEM photographs of the surface of the laminate following reactor irradiation to a neutron fluence of $5x10^{21}$ m⁻² (E>0.1 MeV) and electrical breakdown at 77 K.



Figure 15: SEM photographs of the surface of the laminate following reactor irradiation to a neutron fluence of 1×10^{22} m⁻² (E>0.1 MeV) and electrical breakdown at 77 K.





measured by TUM/H.Gerstenberg

Figure 16: Shear-compression strength of various insulation systems after reactor irradiation at 5 K.