Cryogenic electrical properties of irradiated cyanate ester/epoxy insulation for fusion magnets

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Abstract. The insulation materials used in high field fusion magnets require excellent mechanical properties, high electrical breakdown strength, good thermal conductivity and high radiation tolerance. Previous investigations showed that cyanate ester/epoxy (CE/EP) insulation material, a candidate insulation for fusion magnets, can maintain good mechanical performance at cryogenic temperature after 10 MGy irradiation and has a much longer pot life than traditional epoxy insulation material. In order to quantify the electrical properties of the CE/EP insulation material at low temperature, a cryogenic electrical property testing system cooled by a G-M cryocooler was developed for this study. An insulation material with 40% cyanate ester and 60% epoxy was subjected to ⁶⁰Co γ-ray irradiation in air at ambient temperature with a dose rate of 300 Gy/min, and total doses of 1 MGy, 5 MGy and 10 MGy. The electrical breakdown strength of this CE/EP insulation material was measured before and after irradiation. The results show that cryogenic temperature has a positive effect on the electrical breakdown strength of this composite, while the influence of ⁶⁰Co γ-ray irradiation is not obvious at 6.1 K.

1. Introduction

In order to maintain fusion for long periods of time, a high magnetic field is required in ITER to control the high temperature plasma, and it is crucial for superconducting magnets operating in cryogenic environment to maintain their superconductivity. Supercritical helium at 4 K is used to cool the ITER magnets and the superconducting magnet is surrounded by a large cryostat and an actively-cooled thermal shield with forced flow helium at 80 K [1]. At the same time, there are many high-energy neutrons generated by deuterium-tritium fusion reactions in the plasma, and the insulation materials covering magnetics are irradiated by these neutrons escaping from the plasma.

Insulation material is an important part of all superconducting magnets. It works as an insulator, supporter and protector, and needs to withstand a radiation environment in ITER. The insulation of the superconducting coils is affected by a combination of low temperature, irradiation and high voltage, as well as by mechanical stresses [2]. Thus, it is important to find insulation materials with excellent mechanical properties, high electrical breakdown strength, and high radiation resistance at low temperature [3, 4].

The electrical insulation system of the Toroidal Field (TF) magnet system requires an irradiation resistance of 10 MGy in ITER. Recent studies showed that cyanate ester/epoxy (CE/EP) insulation material has an excellent resistance against gamma ray and neutron irradiation, and thus it considered
as a candidate insulation material for fusion magnets [5-9]. However, previous works for this kind of material were focused on its mechanical properties, while electrical properties tests have been seldom conducted. Therefore, it is essential for us to test the electrical properties of cyanate ester/epoxy (CE/EP) insulation material after irradiation and compare the differences when irradiated by different doses of gamma rays.

This work reports on the fabrication of a device to measure the electrical properties of insulation materials under vacuum at cryogenic temperature, in order to assess the material performance under ITER relevant conditions, and then addresses the breakdown strength of cyanate ester/epoxy at 6.1 K before and after irradiation. Moreover, the insulation breakdown strength for different irradiation doses was measured in this study.

2. Experiment

2.1. Material

The insulation material used in this work consists of two parts, the cyanate ester, which is 1,1-Bis(4-cyanatophenyl) ethane (DCBE, Primaset LECy from Lonza), and the epoxy resin, which is bisphenol-F (DGEBF, GY285 from Huntsman). Figure 1 shows the chemical structures of DGEBF and DCBE. The CE content is 40%, and the EP content is 60%. The catalyst is acetyl acetone cobalt(II) bought from Alfa Aesar. The reinforcement material is boron free glass fiber cloth (RW220-90, Sinoma Science and Technology Co., China) treated by a silane coupling agent. The fabric was 0.2 ± 0.022 mm thick with a count of 18 ± 1 threads/cm in the warp and 14 ± 1 threads/cm in the fill.

This material was fabricated using the vacuum pressure impregnation (VPI) technique. Firstly, cut and seal the woven glass fiber into a metal mould coated with releasing agent. Then, dry the metal mould and glass fiber at 100°C for 10 h. The 60 wt% epoxy resin and 40 wt% cyanate ester with 0.01 wt% Co(acac)\textsubscript{2} were mixed in a 250 ml beaker. Impregnating the fluid resin into the preheated mould followed by curing at 100°C for 6 h, 120°C for 4 h, and then 150°C for 16.5 h. \textsuperscript{60}Co γ–ray was used to irradiate the specimens in air at ambient temperature with a dose rate of 300 Gy/min. The total doses were 1 MGy, 5 MGy and 10 MGy.

Previous studies showed that glass, resin and void content are important parameters that affect electrical properties of dielectric material [10-14]. The insulation tested in this study consists of resin, glass fiber and void, so the test methods of references [15], [16] and [17] are applicable to it. The fiber glass content was obtained using the method in reference [15], the resin content uses the method in reference [16], and the void content uses the method in reference [17]. The resin content was measured to be 45.2 volume %, the glass content was 53.0 volume %, and the void content was 0.8 volume %.

2.2 Apparatus

Figure 1 shows a sketch of the cryostat with a GM cryocooler, which was employed to measure the electrical breakdown strength and the breakdown characteristic of the insulation surface at low temperature. The GM cryocooler used in this equipment is an ESYCOOL KDE415SA with two stages. The GM cryocooler specifications are 35W@50K on the first stage, and 1.5W@4.2K on the second stage.
This testing system consists of three parts: A G-M cryocooler and a cryogenic liquid helium pot, which cools down the whole system, an electrode system that is used to measure the breakdown strength of samples, and finally a thermal insulation system to maintain a cryogenic environment. The electrode system can be changed so that the system can adapt to different kinds of electrical property tests.

Equipment designed in this study are able to reach 4.2 K, however, due to the low thermal conductivity of CE/EP insulation material, hard radiation heat transfer at low temperature and almost no convective heat transfer at high-vacuum, samples tested in this paper were unable to reach 4.2 K, but would stay at 6.1 K [18]. Therefore, the data obtained are at 6.1 K.

The main goal was to investigate the breakdown strength of cyanate ester/epoxy before and after irradiation at 6.1 K. Therefore, the electrodes used in this equipment were especially designed for electrical breakdown strength testing. The diameters of the top electrode and the bottom electrode were 6 mm and 25 mm. An insulation bushing is used to press the samples to avoid cold-contraction of the insulation materials.

Figure 1. Sketch of cryostat with a GM cryocooler

Figure 2. Structure of electrode system for electrical breakdown strength
A vacuum was used to reduce convective heat transfer and simulate the environment in ITER. This incorporated two thermal shields made of copper. The outer shield connects with the first stage cold head of the cryocooler, which can reach 50 K, and the inner shield connects with the second stage cold head, which can reach 4 K. The thermal shields were covered with ten layers of multi-layer insulation in order to reduce radiation from the vacuum chamber. A turbomolecular pump was used to create and maintain the vacuum inside the cryostat. The vacuum level was typically maintained below 10^{-5} Pa and monitored using a wide range vacuum gage mounted on the top flange of the cryostat.

The temperature monitoring and controlling system was based on a labview software platform, which can collect and then display the temperature of the samples and at other important places in the equipment. The electrical temperature controllers used in this system were Lakeshore Cryogenics model 340, which utilize a heater and thermometer coupled on an electrode. With this control system, the heating unit can be accurately controlled from 6.1 K to 300 K.

2.3 Test procedures
In this experiment, the sample was first sandwiched between the top electrode and the bottom electrode. The top electrode and bottom electrodes were connected to the high voltage and grounds, respectively. The samples were put on the bottom and the bolts by sides were adjusted until the electrode touch the sample. Second, air was exhausted from the vessel to create a vacuum environment. Next, the cryocooler was turned on until the temperature stayed constant, and then liquid helium was injected and a heater was turned on to keep the temperature. The high voltage DC power supply was then ramped up with a rate of 1 kV/s. Results were recorded when breakdown occurred. Each data represents an average, calculated from five measurements (samples showing significant deviation were rejected in the statistical calculations).

The breakdown voltage was divided by the thickness of the sample in order to obtain normalized values (in kV/mm) [19]. The thickness of the samples in this work is 0.5 mm. The electrical breakdown strength was calculated from the average voltage of five samples using the following formula:

$$E_b = \frac{U_b}{d}$$

E_b = electrical breakdown strength, 
U_b = average breakdown voltage of five samples, and 
d = thickness of the samples.

3. Results and discussion
In this paper, electric breakdown strength of cyanate ester/epoxy (CE/EP) insulation material at 6.1 K, 77 K (the temperature of liquid nitrogen) and 300 K (the room temperature) was studied. Table 1 shows that electrical breakdown strength of this composite improved obviously when the temperature decreases. Furthermore, the average breakdown strength at 6.1 K is 68.9 kV/mm, about 18% higher than that at 300 K. The average breakdown strength at 77 K is 63.8 kV/mm, about 9.4% higher than that at 300 K.

The increase of breakdown strength caused by low temperature may be explained as follows: the free volume of the CE/EP reduced due to the decrease of temperature. The molecular motion of the composite confined because less space of action if the free volume of the composite is reduced. There would not be enough molecular space for the charge carrier to accumulate easily when an external electrical field is applied, and much more polarized charge carriers in the composite would be released immediately, which helps the improved dielectric breakdown strength [20].
Table 1. Electrical breakdown strength of the CE/EP composite at different temperatures

| Temperature (K) | 6.1   | 77    | 300   |
|----------------|-------|-------|-------|
| Breakdown strength (kV/mm) | 68.6  | 65.2  | 57.9  |
|                  | 69.2  | 63.9  | 55.3  |
|                  | 69.9  | 64.6  | 61.6  |
|                  | 67.5  | 61.3  | 59.8  |
|                  | 69.3  | 64.0  | 56.9  |
| Average (kV/mm) (X±2 std. deviations) | 68.9±1.6 | 63.8±2.6 | 58.3±4.4 |

Table 2 shows the electric breakdown strength of cyanate ester/epoxy (CE/EP) before and after irradiation by a total dose of 1 MGy, 5 MGy or 10 MGy at 6.1 K. Slightly higher values were found with the increase of dose range, but the overall effects are within the calculated standard deviation. That evidently indicates irradiation by $^{60}$Co $\gamma$-ray effect slightly on electric breakdown strength of this composite. That is to say, the chemical structure of the CE/EP is scarcely changed by the irradiation up to 10 MGy. All results obtained by UV-Vis, FT-IR and mechanical properties tests in our previous study are consistent with this point [21].

Table 2. Electrical breakdown strength of the CE/EP composite at 6.1 K before and after irradiation

| Radiation doses (MGy) | 0     | 1     | 5     | 10    |
|-----------------------|-------|-------|-------|-------|
| Breakdown strength (kV/mm) | 68.6  | 75.5  | 72.1  | 73.8  |
|                       | 69.2  | 65.1  | 71.2  | 70.6  |
|                       | 69.9  | 69.5  | 76.1  | 71.2  |
|                       | 67.5  | 68.6  | 74.5  | 74.8  |
|                       | 69.3  | 72.3  | 72.6  | 73.6  |
| Average (kV/mm) (X±2 std. deviations) | 68.9±1.6 | 70.2±7 | 73.3±3.6 | 72.8±3.2 |

4. Conclusion
In this work, equipment was fabricated to measure electrical properties at a temperature range from 4.2 K to 300 K under vacuum. The electrical breakdown strength of cyanate ester/epoxy blend with 40% CE and 60% EP was measured at 6.1 K, 77 K and 300 K. At the same time, electrical breakdown strength of this composition at 6.1 K before and after irradiation was studied. In conclusion, cryogenic temperature has a positive effect on electrical breakdown strength of the composite, while the influence of $^{60}$Co $\gamma$-ray irradiation is not evidential at 6.1 K.

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