Properties of radiation stable insulation composites for fusion magnet

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Abstract. High field superconducting magnets made of Nb₃Al will be a suitable candidate for future fusion device which can provide magnetic field over 15T without critical current degradation caused by strain. The higher magnetic field and the larger current will produce a huge electromagnetic force. Therefore, it is necessary to develop high strength cryogenic structural materials and electrical insulation materials with excellent performance. On the other hand, superconducting magnets in fusion devices will experience significant nuclear radiation exposure during service. While typical structural materials like stainless steel and titanium have proven their ability to withstand these conditions, electrical insulation materials used in these coils have not fared as well. In fact, recent investigations have shown that electrical insulation breakdown is a limiting factor in the performance of high field magnets. The insulation materials used in the high field fusion magnets should be characterized by excellent mechanical properties, high radiation resistivity and good thermal conductivity. To meet these objectives, we designed various insulation materials based on epoxy resins and cyanate ester resins and investigated their processing characteristic and mechanical properties before and after irradiation at low temperature. In this paper, the recent progress of the radiation stable insulation composites for high field fusion magnet is presented. The materials have been irradiated by ⁶⁰Co γ-ray irradiation in air at ambient temperature with a dose rate of 300 Gy/min. The total doses of 1 MGy, 5 MGy and 10 MGy were selected to the test specimens.

1. Introduction

Compared to International Thermonuclear Experimental Reactor (ITER), the next fusion device like DEMO will require the higher magnetic field and larger plasma volume. It means that larger superconducting magnets with higher magnetic field would be designed and the conductor would endure the higher electro-magnetic force and higher levels of radiation. For example, the peak field for ITER TF is 11.8 T and 13.6 T for DEMO[1]. On the other hand, superconducting magnets in fusion devices will experience significant nuclear radiation exposure during service. Therefore, it should be noted that the irradiation effect of the superconducting magnet materials would be the main key issues for the future fusion device. Among the superconducting magnet materials, the insulation material is reported as the most radiation-sensitive, which consists of glass fiber reinforced polymer (GFRP) composites [2-4]. Generally speaking, radiation-resistant property of the composite is related to the chemical structures [5]. For example, aromatic structures are superior to aliphatic structures and radiation tolerance increases with resin functionality. Based on this, we designed various insulation
materials based on epoxy resins with different functionality and cyanate ester resins and investigated their processing characteristic and mechanical properties before and after irradiation at low temperature [6-8].

In this paper, the recent progress of the radiation stable insulation composites for fusion magnet is presented. All materials have been irradiated by $^{60}\text{Co}$ $\gamma$-ray irradiation in air at ambient temperature. The total doses of 1 MGy, 5 MGy and 10 MGy were selected to the test specimens.

2. Experiment
2.1. Materials
The epoxy resins used in this study were diglycidyl ether of bisphenol F epoxy resin (DGEBF, GY285, Huntsman Advanced Materials) and triglycidyl-p-aminophenol (TGPAP, MY0510, Huntsman Advanced Materials), respectively. The cyanate ester was 1,1-Bis(4-cyanatophenyl)ethane (DCBE, Primaset LECy from Lonza), The curing agent was diethyl toluene diamine (DETD, HY5200, Huntsman Advanced Materials). The modifier agent was isopropylidenebisphenol bis[(2-glycidyloxy-3-n-butoxy)-1-propylether] (IPBE, PY 4122, Huntsman Advanced Materials). The reinforcement was boron-free glass fiber cloth (RW220-90, Sinoma Science and Technology Co., China) treated with a silane coupling agent.

2.2. Specimen preparation
The composites were prepared by the VPI process. Details of the VPI process were described in reference [6] and reference [8]. The resin components are given in table 1. The prepared composites were cut into the specimens for the short beam shear test with the dimensions of 24 mm× 8 mm× 4 mm (length × width × thickness), as shown in figure 1.

| Sample code | DGEBF (g) | TGPAP (g) | IPBE (g) | CE (g) | DETD (g) |
|-------------|-----------|-----------|----------|--------|----------|
| 1           | 100       | 0         | 0        | 0      | 26.7     |
| 2           | 90        | 0         | 10       | 0      | 25.4     |
| 3           | 0         | 100       | 0        | 0      | 44.5     |
| 4           | 0         | 90        | 10       | 0      | 41.4     |
| 5           | 60        | 0         | 0        | 40     | 0        |

Figure 1. Schematic showing location of specimen removal from the composites.

2.3. Test procedures
The specimens were irradiated by $^{60}\text{Co}$ $\gamma$-ray irradiation in air at ambient temperature with a doses rate of 300 Gy/min. The maximum dose was 10 MGy.

The apparent interlaminar shear strength (ILSS) was assessed by the short-beam shear (SBS) test according to the ASTM D2344. The short beam shear (SBS) test at 77 K was carried out using a MTS-SANS 5000 universal testing with a cross-head speed of 1 mm/min and a span-to-thickness ratio of 5:1 was used. The ILSS can be calculated using the following formula:
where $P_B$ is the maximum load, $b$ is the specimen width, and $h$ is the specimen thickness.

3. Results and discussion

3.1. Viscosity

Generally, the insulation systems for superconducting magnets are fabricated by a vacuum press impregnation (VPI) process. The resin matrix with a rather low viscosity and long working life is essential to assure a complete impregnation of a large superconducting coil with a complicated shape [5, 9]. In this work, the resin viscosity was measured using a Brookfield viscometer. The viscosity of the resin systems at 45°C as a function of time is shown in figure 2. Clearly, compared with other resin systems, the CE/DGEBF system exhibits lower initial viscosity and longer pot life. This indicates that the CE/DGEBF system is more suitable for VPI process to impregnate large-scale superconducting magnets. Moreover, it can be seen that the addition of IPBE caused a decrease of the viscosity rise rate for both DGEBF and TGPAP. This implies that the IPBE reduced the rate of curing reactions and in turn lead to the increase of the effective working life.

![Figure 2. Viscosity versus time at 45 °C for various epoxy resin systems.](image)

3.2. Void content

The contents of glass fiber, epoxy resin and void for the prepared composites were measured according to ASTM D2734. The results are presented in table 2. The void contents of all prepared composites were less than 1 vol.%. Furthermore, the volume contents of the fiber and the matrix for all prepared composites were about 45 vol.% and 54 vol.%, respectively.

| Resin System # | Resin Content Vol. % | Glass Content Vol % | Void Content Vol % |
|----------------|----------------------|---------------------|-------------------|
| DGEBF          | 54.47                | 45.29               | 0.24              |
| TGPAP          | 53.72                | 45.83               | 0.45              |
| IPBE/DGEBF     | 54.03                | 45.38               | 0.59              |
| IPBE/TGPAP     | 53.84                | 45.84               | 0.32              |
| CE/DGEBF       | 53.98                | 45.21               | 0.81              |
3.3. Interlaminar shear strength
The ILSSs of the non-irradiated and irradiated composites were measured by the SBS test at 77 K. The ILSS as a function of the irradiation dose is shown in figure 3. It can be seen that among all formulations, the IPBE/DGEBF composite presented the highest shear strength value before irradiation, while TGPAP composite showed the lowest ILSS. TGPAP is known to result in cured materials with higher crosslink density than DGEBF resin, because of the presence of three reactive groups in the resin on a single aromatic nucleus. That is to say, TGPAP is more brittle than DGEBF and thus exhibited lower shear strength at 77 K. Moreover, the addition of IPBE improved the ILSS of DGEBF and TGPAP composite. The mechanism for the improvement of ILSS was discussed in reference [10].

![Graph showing ILSS results for composites with gamma-irradiation dose](image)

**Figure 3.** ILSS results for the composites, illustrating the variation with gamma-irradiation dose

Moreover, with the increasing of gamma-irradiation doses, ILSS of these composites show different trends. It is observed that ILSS of composites based on DGEBF, IPBE/TGPAP, TGPAP and IPBE/DGEBF slightly increases with the irradiation dose and then decreases. Especially for IPBE/DGEBF systems, the ILSS of composites are significantly decreased with the irradiation dose up to 10MGy. However, the DGEBF/CE systems show that the ILSS of composites increases with the increasing of irradiation doses. That is to say, IPBE/DGEBF composite could not withstand 10MGy gamma-irradiation. Moreover, DGEBF/CE systems show the best behavior under 10MGy gamma-irradiation.

SEM images of fracture surfaces of composites are shown in figure 4. Obviously, hackle pattern fracture surfaces are observed for irradiation and un-irradiation composites based on DGEBF and DGEBF/CE systems. These microstructure characteristics suggest that the interfacial adhesion of the composites after 10MGy irradiation is strong and the failure mechanisms for these composites are a combination of resin fracture and interfacial debonding. On the other hand, it is observed that fracture surface of IPBE/DGEBF composite after 10MGy irradiation shows smooth and few river lines, which indicates that the composite failure is caused by the brittle fracture of the polymer matrix. Therefore, less load was needed to destroy the composites.

The ILSS reduction mechanisms for IPBE/DGEBF composite can be related to the radiation-induced degradation in the mechanical properties of the matrix resin owing to molecular chain scission and crosslinking. In general, polymers after the high energy irradiation would be induced two competing processes: chain scission and crosslinking. IPBE has long aliphatic chain which is more easy to be destroyed by the high energy irradiation, and thus leading to a predominance of chain...
scission at high dose radiation, which results in the significant decrease in ILSS after 10MGy irradiation for the IPBE/DGEBF composite.

Therefore, based on the results we have discussed, DGEBF/CE composites have shown the best mechanical and irradiation resistant behaviors.

Figure 4. SEM images of fracture surfaces after SBS tests. (a) 0MGy, DGEBF; (b) 10MGy, DGEBF; (c) 0MGy, IPBE/DGEBF; (d) 10MGy, IPBE/DGEBF; (e) 0MGy, CE/DGEBF; (f) 10MGy, CE/DGEBF.

4. Conclusion
We designed various insulation materials based on epoxy resins and cyanate ester resins and investigated their processing characteristic and mechanical properties before and after irradiation at low temperature. The cyanate ester/epoxy system exhibits the lowest initial viscosity and the longest pot life which shows that the cyanate ester/epoxy system is suitable for VPI process to impregnate large-scale superconducting magnets. All insulation materials except for IPBE/DGEBF show good radiation resistance, especially CE/DGEBF. From these limited results, the CE/DGEBF system appears to be more suitable for the high field fusion magnets.

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