Mechanical performance of cyanate ester based insulation systems under ITER relevant conditions

R Prokopec, K Humer, R K Maix, H Fillunger and H W Weber

Atomic Institute of the Austrian Universities, 1020 Wien, Austria
rprokopec@ati.ac.at

Epoxy based glass fiber reinforced composites are candidate insulation systems for the superconducting magnet coils of fusion devices, e. g. of ITER. Because of their relatively low radiation hardness, the radiation-harder cyanate ester based systems have become of special interest for application in ITER and future fusion devices.

This paper presents recent results obtained on pure cyanate ester and cyanate ester/epoxy blends reinforced with R-glass fibers/Kapton H foil. The systems were investigated at 77 K prior to and after irradiation at ambient temperature (340 K) to a fast neutron fluence of $1 \times 10^{22}$ m$^{-2}$ (E>0.1 MeV) in the TRIGA reactor (Vienna). The mechanical material performance was characterized under static load conditions in tension and interlaminar shear. In addition, tension-tension fatigue measurements were carried out to simulate the pulsed operation of ITER and to assess the lifetime performance of these materials. The results show only slight mechanical degradations (by about 5 to 10%) after irradiation.

1. Introduction

Traditional epoxy resins were used for the impregnation of the toroidal field model coil (TFMC) of ITER [1]. Subsequent material tests on these fiber reinforced composites revealed their poor radiation hardness, especially after exposure to the ITER design fluence of $1 \times 10^{22}$ m$^{-2}$ (E>0.1 MeV). A severe degradation of the mechanical strength was observed due to the radiation induced damage of the epoxy resins [2, 3]. The mechanical performance does not fulfill the ITER design criteria. Therefore, an enhancement of the radiation hardness of the resin system is an important issue for applications in a radiation environment.

The application cyanate ester (CE) seems to be the most promising way for reducing the radiation damage in the composite. Recent results on pure CE and CE/epoxy blended insulation systems showed, that the mechanical material performance was hardly influenced by a fast neutron fluence of $1 \times 10^{22}$ m$^{-2}$ (E>0.1 MeV) [4-6].

This contribution addresses the material behavior of glass fiber reinforced laminates impregnated with pure and blended CE resins at 77 K before and after fast neutron irradiation to a fluence of $1 \times 10^{22}$ m$^{-2}$ (E>0.1 MeV). Static tests to determine the ultimate tensile strength (UTS) and the interlaminar shear strength (ILSS) as well as dynamic tests to simulate the pulsed ITER operating conditions were carried out.

2. Materials and Test Procedures

Various insulation systems were manufactured by Marti-Supratec Corporation, Switzerland using the vacuum pressure impregnation (VPI) technique. R-glass fiber / Kapton H tapes were wrapped half-
Table 1. Overview of cyanate ester based insulation systems

|      | T1a | T1b | T1o |
|------|-----|-----|-----|
| Type | Cyanate Ester | Cyanate Ester | Cyanate Ester |
| Resin | AroCy-L10 | AroCy-L10 | AroCy-L10 |
| Hardener | ---- | ---- | ---- |
| Additives | Mn-Acetylacetonate in Nonylphenol | Mn-Acetylacetonate in Nonylphenol | Mn-Acetylacetonate in Nonylphenol |
| Reinforcement | R-glass / Kapton | R-glass / Kapton | R-glass |
| Curing Temp. | 4 h @ 140°C / 5 h @ 210°C | 4 h @ 100°C / 5 h @ 160°C | 4 h @ 100°C / 5 h @ 160°C |

Table 2. Overview of cyanate ester / epoxy blend insulation systems

|      | T2 |
|------|----|
| Type | Cyanate Ester about 40 % | DGEBA about 60 % |
| Resin | AroCy-L10 | PY306 |
| Hardener | ---- | ---- |
| Additives | Mn-Acetylacetonate in Nonylphenol | ---- |
| Reinforcement | R-glass / Kapton | ---- |
| Curing Temp. | 4 h @ 100°C / 5 h @ 160°C | ---- |

overlapped around a steel plate, impregnated with the resin and pressed to obtain a sample thickness of about 4 mm.

In order to study the influence of the Kapton layers, which enhance the dielectric properties but reduce the mechanical strength of the laminate, the “T1o” system did not contain Kapton. The insulation systems investigated in this study are summarized in Tables 1 and 2.

The irradiation was done in the TRIGA reactor (Vienna) at ambient temperature (340 K) to a fast neutron fluence of $1 \times 10^{22}$ $m^{-2}$ ($E>0.1$ MeV), which corresponds approximately to a total absorbed dose of 50 MGy [7].

All static and dynamic tests were carried out at 77 K using a servo-hydraulic MTS 810 testing device, which was modified for measurements in a liquid nitrogen environment. The UTS was measured according to the DIN 53455 and ASTM D 638 standards. The ILSS was determined by the short-beam-shear (SBS) test according to the ASTM D 2344 standard. Span-to-thickness ratios of 4:1 and 5:1 were used to achieve interlaminar shear fracture. To simulate the pulsed operation mode of ITER, load controlled tension-tension fatigue measurements (ASTM D 3479) were done up to $10^6$ cycles at a frequency of 10 Hz and a minimum to maximum stress ratio of $R=0.1$. Each data point refers to at least 4 samples.

Because of their manufacturing process the materials have anisotropic properties. Therefore, tensile and SBS specimens were cut parallel (0°) and perpendicular (90°) to the winding direction of the reinforcing glass fiber tapes. All static material tests were carried out in both directions, whereas only the 90° direction was chosen for the dynamic tests.

3. Results

3.1. Ultimate tensile strength

The results of the static tensile tests are listed in Table 3. Because of the higher glass fiber content of the CE blend “T2”, the UTS is higher by about 250 MPa in 0° direction and by about 150 MPa in 90°
Table 3. Ultimate tensile strength (UTS) before and after reactor irradiation to a fast neutron fluence of $1 \times 10^{22} \text{m}^{-2} (E > 0.1 \text{ MeV})$ measured at 77 K.

| Insulation system | Unirradiated UTS (MPa) | Irradiated UTS (MPa) |
|------------------|------------------------|----------------------|
|                  | $0^\circ$ | $90^\circ$ | $0^\circ$ | $90^\circ$ |
| T1a              | 612 ± 32 | 206 ± 6  | 600 ± 59 | 222 ± 5  |
| T1b              | 757 ± 4  | 250 ± 19 | 769 ± 34 | 250 ± 22 |
| T1o              | 675 ± 19 | 386 ± 26 | 616 ± 44 | 335 ± 13 |
| T2               | 1027 ± 20| 414 ± 20 | 945 ± 18 | 397 ± 21 |

3.2. Interlaminar Shear Strength

The results of the SBS tests are summarized in Table 4. Both before and after irradiation the ILSS in $0^\circ$ direction is higher by about 10 MPa than in $90^\circ$ direction. The higher fiber content of “T2” results in an ILSS which is higher by about 30 MPa. In comparison to the UTS, no pronounced influence of the Kapton layers on the ILSS is observed in both directions.

In general, irradiation of the insulation systems to the ITER design fluence does not show any reduction of the ILSS, which confirms the radiation hardness of these materials.

Table 4. Interlaminar shear strength (ILSS) before and after reactor irradiation to a fast neutron fluence of $1 \times 10^{22} \text{m}^{-2} (E > 0.1 \text{ MeV})$ measured at 77 K.

| Insulation system | Unirradiated ILSS (MPa) | Irradiated ILSS (MPa) |
|------------------|-------------------------|----------------------|
|                  | $0^\circ$ | $90^\circ$ | $0^\circ$ | $90^\circ$ |
| T1a              | 58 ± 5  | 51 ± 6   | 58 ± 3   | 56 ± 2   |
| T1b              | 59 ± 8  | 42 ± 10  | 68 ± 4   | 48 ± 5   |
| T1o              | 71 ± 7  | 53 ± 6   | 65 ± 4   | 48 ± 6   |
| T2               | 92 ± 3  | 83 ± 4   | 91 ± 3   | 81 ± 4   |

direction than for the other systems. An effect of the Kapton layers in the reinforcement can be found especially in $90^\circ$ direction. The UTS of the CE system without Kapton (“T1o”) is higher by about 50% than that of the CE systems with Kapton (“T1a” and “T1b”). After irradiation to a fast neutron fluence of $1 \times 10^{22} \text{m}^{-2} (E > 0.1 \text{ MeV})$ only a small degradation of the UTS is observed in all systems.

3.3. Tension-Tension Fatigue Behavior

Tension-tension fatigue measurements were carried out to study the effects of irradiation and of the Kapton layers on the dynamic material performance of the insulation systems. The stress lifetime diagrams (Wöhler curves) are presented in Figure 1. In general, no significant radiation effect on the dynamic material behavior was found for all insulation systems, the Wöhler curves prior to and after irradiation are within the standard deviations. Only for “T1b”, a well defined life endurance limit was found at 0.5 UTS (125 MPa).

4. Summary

Based on the fact that conventional epoxy systems hardly withstand the high radiation levels expected for ITER, the application of the radiation harder cyanate esters (pure and blended with epoxy) as impregnation resins for fiber reinforced composites has become of great importance. Several pure and blended CE based insulation systems were investigated under static and dynamic load conditions before and after reactor irradiation to a neutron fluence of $1 \times 10^{22} \text{m}^{-2} (E > 0.1 \text{ MeV})$, i.e. the ITER design fluence level. The results may be summarized as follows:
No significant degradation of the UTS and ILSS was observed for the pure and the blended CE based insulation systems after exposure to the ITER design fluence.

The dynamic material performance was not affected by irradiation.

Reinforcement without Kapton leads to an enhancement of the UTS, especially in 90° direction.

With respect to the application to the ITER magnets, the CE laminates show higher radiation resistance compared to the commercial epoxy systems used for the TFMC. No differences in the radiation hardness were observed for the insulation systems impregnated with pure or blended CE. For economical reasons the cheaper 40:60 CE / epoxy blend is preferable for ITER.

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