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# Radiation effects on the mechanical integrity of novel organic insulators for the ITER magnet coils

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#### Abstract

High quality glass fiber reinforced composites will be used as electrical insulation systems for the windings of the superconducting magnet coils of the ITER fusion device. Recently, considerable interest in coil technology has focused on improving the radiation hardness of the organic impregnation resins, in order to avoid serious material damage, such as fiber-matrix debonding (delamination). This paper reports on first screening tests of novel cyanate-ester based glass fiber reinforced composites, which were developed especially for ITER-relevant applications. The material behavior was investigated at 77 K prior to and after reactor irradiation to a neutron fluence of  $1 \times 10^{22} \text{ m}^{-2}$  (E > 0.1 MeV) using the tensile and the short-beam-shear test, respectively. Furthermore, tension–tension fatigue measurements were carried out at low temperatures, in order to assess the mechanical performance under the pulsed operating conditions of ITER.

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## 1. Introduction

Because of their low viscosity, long pot-life and compatibility with the 'resin transfer moulding' process, standard di-functional epoxy resins were used for the large area impregnation of the toroidal field model coil (TFMC) of international thermonuclear experimental reactor (ITER) [1]. However, subsequent material studies on these R-glass fiber reinforced epoxy based laminates [2,3] showed an unacceptable degradation of their mechanical strength, especially after exposure to the ITER design fluence of  $1 \times 10^{22}$  m<sup>-2</sup> (E > 0.1 MeV). The material performance was seriously affected by radiation-induced damage of the impregnation resin resulting in pre-mature interfacial failure. These extended test series clearly demonstrated that the insulation material is still a critical component in fusion magnet design. Therefore, an enhancement of the impregnation's radiation hardness is highly desirable. Recent work [4–6] showed that the most promising improvement can be achieved by admixing a certain amount of cyanate-ester (CE) resin.

The present contribution addresses the material behavior at 77 K of novel glass fiber reinforced composites impregnated with pure and blended cyanate ester resins before and after irradiation to a neutron fluence of  $5 \times 10^{21}$  and  $1 \times 10^{22}$  m<sup>-2</sup> (E > 0.1 MeV). Screening tests of the ultimate tensile strength (UTS) and of the interlaminar shear strength (ILSS) under static and dynamic load conditions allow a first assessment of the material performance under ITER-relevant conditions.

#### 2. Materials and test procedure

In order to fulfill the objectives of high performance magnet insulation systems, standard epoxy resins were hybridized with special organic cyanate-ester thermosetting monomers. This innovative impregnation material became important recently due to its high ability to

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Table 1					
Material	specifications	of the	organic	insulation	systems

Code	Matrix	Fabrication process	Reinforcement	Fiber volume (nominal) (%)
CTD-404	Pure CE	VPI	S-2 glass fabric, 6781 style 8 h satin weave	50
CTD-422	CE/ epoxy blend	VPI	S-2 glass fabric, 6781 style 8 h satin weave	50
EU2	CE/ epoxy blend	VPI	R-glass/Kapton tapes wrapped half overlapped	50

 Table 2

 Results of the static and dynamic measurements at 77 K before and after irradiation

System	Fluence $(m^{-2})$ ( <i>E</i> > 0.1 MeV)	UTS (MPa)	ILSS <sup>SBS</sup> (MPa)	Fatigue limit @~10 <sup>6</sup> cycles <sup>a</sup> (%/MPa)
CTD-404	Unirradiated	$894 \pm 27$	$108 \pm 5$	_
CTD-422	Unirradiated	$1043 \pm 26$	$88 \pm 5$	0.25/261
EU2/0°	Unirradiated	$1027 \pm 20$	$92 \pm 3$	0.3/308
EU2/90°	Unirradiated	$414 \pm 20$	$83 \pm 4$	0.4/167
CTD-404	$5 \times 10^{21}$	_	_	_
CTD-422	$5 \times 10^{21}$	$1048 \pm 26$	_	0.2/210
EU2/0°	$5 \times 10^{21}$	_	_	_
EU2/90°	$5 \times 10^{21}$	_	_	_
CTD-404	$1 \times 10^{22}$	$868 \pm 12$	$97 \pm 3$	_
CTD-422	$1 \times 10^{22}$	$994 \pm 20$	$69 \pm 6$	0.22/219
EU2/0°	$1 \times 10^{22}$	$945 \pm 18$	$91 \pm 3$	0.3/284
EU2/90°	$1 \times 10^{22}$	$397 \pm 21$	$81 \pm 4$	0.4/160

<sup>a</sup> Normalized (%) and absolute (MPa) life endurance limits.

withstand demanding operating temperatures as well as working environments, moisture absorption and mechanical loads.

The present vacuum-pressure-impregnated (VPI) CEblended fiber composites (Table 1) were manufactured by Composite Technology Development (CTD) Inc., USA ('CTD-404' and 'CTD-422') and by MARTI-SU-PRATEC Corporation, Switzerland ('EU2'), respectively.

Because of the anisotropic material properties of EU2 (4 mm thickness), the tensile specimens <sup>1</sup> [7] and the short-beam shear (SBS)-specimens were loaded under two different directions, i.e., parallel (0°) and perpendicular (90°) to the winding direction of the reinforcing tapes. The 3 mm thick tensile <sup>1</sup> and SBS samples CTD-404 and CTD-422 were loaded only along their strongest direction, i.e., the warp-direction of the laminates' fiber reinforcement was chosen to be parallel to the longer axis of the specimens.

Both static and dynamic screening tests were done at 77 K using a servo-hydraulic MTS 810 testing device. The ultimate tensile strength (UTS) was measured according to DIN 53455 and ASTM D638. The interlaminar shear strength (ILSS) was determined from SBS tests according to the ASTM D2344 standard with a span-to-thickness ratio of 5:1 for EU2 and of 4:1 for the CTD-systems, respectively. To simulate the pulsed conditions of ITER, tension-tension fatigue experiments (ASTM D 3479) were done at 10 Hz and with R = 0.1 up to  $10^6$  cycles. Four or five samples were measured at each load level. A standard deviation of ~20% was observed.

All irradiations were done at ambient temperature (~340 K) in the TRIGA reactor (Vienna, Austria) to a neutron fluence of  $5 \times 10^{21}$  and  $1 \times 10^{22}$  m<sup>-2</sup> (E > 0.1 MeV), which corresponds approximately to a total absorbed dose of 20 and 40 MGy. The reactor is operating at a  $\gamma$ -dose rate of  $1 \times 10^{6}$  Gyh<sup>-1</sup>, a fast neuron flux density of  $7.6 \times 10^{16}$  m<sup>-2</sup> s<sup>-1</sup> (E > 0.1 MeV), and a total neutron flux density of  $2.1 \times 10^{17}$  m<sup>-2</sup> s<sup>-1</sup>, respectively.

<sup>&</sup>lt;sup>1</sup> Length×width:  $(70 \times 10)$  mm<sup>2</sup>.



Fig. 1. Normalized (a,c) and absolute (b,d) tension-tension stress-lifetime diagrams for CTD-422 and EU2<sup>0°+90°</sup> before and after reactor irradiation to a fast neutron fluence of  $1 \times 10^{22}$  m<sup>-2</sup> (E > 0.1 MeV) measured at 77 K. In addition, a subset of CTD-422 specimens was also tested after irradiation at  $5 \times 10^{21}$  m<sup>-2</sup> (E > 0.1 MeV). The measurements were stopped manually above  $10^6$  cycles, as indicated by the arrows.

### 3. Results

#### 3.1. Static tensile tests

As can be seen from Table 2, all laminates show an excellent high UTS in warp-direction, whereas the UTS of EU2/90° is  $\sim 60\%$  lower (a detailed description of the fracture behavior in the 90°-direction is reported in Refs. [2,3]). Irradiation to the highest fluence level does not show a significant influence on the UTS, neither for the pure nor for the CE-blended CTD laminates. The material strength of the R-glass/Kapton system EU2 remains also nearly constant in both directions after exposure to the ITER design fluence.

#### 3.2. Tension-tension fatigue behavior

Fig. 1(a) and (b) show the material performance of CTD-422 under tension–tension fatigue load before and after irradiation to a neutron fluence of  $5 \times 10^{21}$  and  $1 \times 10^{22}$  m<sup>-2</sup> (E > 0.1 MeV), respectively (Table 2). All Wöhler-curves of CTD-422 are characterized by a rapid decrease in the range from 80% to 30% UTS. The preirradiation life endurance limit  $\sigma_D$ , achieved at about 10<sup>6</sup> cycles, is 0.25 UTS (=261 MPa). These limits change slightly to 0.20 UTS (=210 MPa) after the first irradiation step and further to 0.22 (=219 MPa) at the ITER design fluence. These minor differences in  $\sigma_D$  after radiation of the experiment.

The tension-tension fatigue behavior in the 0°direction of EU2 (Table 2) is characterized by a rapid decrease between 0.8 and 0.4 UTS (Fig. 1(c) and (d)) prior to irradiation. The fatigue limit of this Kapton interleaved insulation system is 0.3 UTS (= 308 MPa) in the 0°-direction, whereas an endurance limit of 0.4 UTS (= 167 MPa) was found for the 90°-direction. After irradiation to the highest dose level, the fatigue values remain nearly constant at 0.3 UTS<sup>0°</sup> (= 284 MPa) and at 0.4 UTS<sup>90°</sup> (= 160 MPa).

#### 3.3. Interlaminar shear strength

The highest ILSS<sup>SBS</sup> is found for the unirradiated pure CE-system CTD-404 followed by the CE/epoxy blend EU2<sup>0°</sup> (Table 2). Both CTD-422 and EU2<sup>90°</sup> show a shear strength of about 88 MPa before irradiation. Irradiation to a fast neutron fluence of  $1 \times 10^{22}$ m<sup>-2</sup> (E > 0.1 MeV) does not lead to any significant change in the shear behavior of the CE/epoxy-blend EU2. The ILSS of the laminate degrades only by 2.5% under both loading directions, which is within the standard deviation. On the other hand, CTD-404 degrades to 97 MPa, whereas the CTD-422 laminate looses  $\sim 28\%$  of its initial ILSS<sup>SBS</sup>, presumably due to a higher epoxy-content.

#### 4. Summary

Based on the fact that the traditionally employed epoxy resins show serious problems to withstand high radiation dose levels (>20 MGy), the impregnation materials of novel glass fiber reinforced ITER-relevant coil insulation systems were blended with special cyanate-ester thermosets, in order to enhance the radiation hardness. Two CE/epoxy blends (CTD-422 and EU2) and a pure CE-composite (CTD-404) were mechanically screened before and after irradiation to a neutron fluence of  $1 \times 10^{22}$  m<sup>-2</sup> (E > 0.1 MeV). These initial results may be summarized as follows:

- In 0°-direction, all laminates show a high ultimate tensile strength (UTS) of up to 1043 MPa, which degrades slightly (by 5%) after irradiation at the highest dose level. A significantly lower (~60%) UTS was measured perpendicular to the tape winding direction of the EU2 blend, which also does not change after irradiation to  $1 \times 10^{22}$  m<sup>-2</sup> (E > 0.1 MeV).
- In general, the tension-tension fatigue behavior of all laminates remains almost the same after irradiation to a fluence of  $1 \times 10^{22} \text{ m}^{-2}$  (E > 0.1 MeV). The endurance limits are 0.25 UTS for CTD-422, 0.3 UTS for EU2<sup>0°</sup> and 0.4 UTS for EU2<sup>90°</sup> before irradiation.
- The highest interlaminar shear strength (ILSS) of 108 MPa was found for the pure CE-system CTD-404. A ~10% lower ILSS was observed for both CE/epoxy blends. At the final dose, irradiation leads to a small decrease (up to 10%) of the ILSS for CTD-404 and EU2, whereas the ILSS of CTD-422 degrades to 69 MPa due to a higher epoxy content.

As a first result of these screening experiments, we wish to point out, that all newly developed insulation systems show improved mechanical integrity under ITER-relevant conditions, and thus, meet the actual ITER requirements. In particular, the cost-effective blending of epoxy with cyanate-ester clearly demonstrates the improvement potential of commercially available resins. Consequently, these blends are of special interest for fusion coil technology. Detailed mechanical studies are under way at present.

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