

Insulation systems for superconducting fusion magnets based on cyanate ester blends

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ARTICLE INFO

Article history:

Available online 18 December 2008

Keywords:

Fiber reinforced composites
Cyanate ester
Mechanical properties
Reactor irradiation
Cryogenic temperatures

ABSTRACT

Advanced epoxy-based glass fiber reinforced plastics are commercially used as insulating materials in fusion magnet technology. However, their mechanical strength drops dramatically upon irradiation to a fast neutron fluence of $1 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$), i.e. the ITER design fluence. The recent results demonstrated that cyanate ester (CE)/epoxy blends were not affected at this fluence level. In this work, various magnet insulation systems containing boron-free R-glass fiber reinforcements embedded in CE/epoxy blends are investigated. The mechanical properties were assessed at 77 K in tension as well as in the interlaminar shear mode prior to and after reactor irradiation at ambient temperature ($\sim 340 \text{ K}$) to neutron fluences of up to $4 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$) under static and dynamic load conditions. The results show only a small reduction of the mechanical properties at twice the ITER design fluence. At the highest irradiation level the interlaminar shear strength of blends with at least 40% CE is only reduced by 20–30%.

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1. Introduction

During the past few years extensive research was carried out to find a suitable radiation hard insulation system for the toroidal field coils of ITER [1–6]. Especially the admixture of cyanate ester (CE) lead to innovative blends showing excellent mechanical material performance even after exposure to the ITER design fluence of $1 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$). Recent investigations have shown that the percentage of the expensive CE in the CE/epoxy blend can be reduced to only 20% without any significant degradation of the mechanical properties [7,8]. However, the industrial handling experience is less developed and has to be thoroughly checked for large-scale applications. Based on the improved radiation resistance, a CE/epoxy blend containing 40% CE is foreseen for the ITER TF coil insulation.

With respect to the application of these innovative blends in upcoming fusion devices, e.g. for DEMO, with possible higher demands on radiation hardness it is important to investigate the mechanical properties of these blends at higher radiation levels.

In this study the mechanical behavior of several magnet insulation systems containing a glass fiber reinforcement impregnated with various CE/epoxy blends is investigated at 77 K before and after reactor irradiation to fast neutron fluences of up to $4 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$). Tests in tension and interlaminar shear were carried out under static and dynamic load conditions, in order to assess the material performance under ITER relevant conditions and beyond.

2. Materials, irradiations and test procedures

The blends were fabricated by Marti-Supratec Corp., Switzerland. The reinforcement of the laminates, i.e. seven layers of R-glass fiber ($0.24 \times 40 \text{ mm}$)/Kapton H tapes and one pure glass fiber layer, were wrapped half-overlapped around a steel plate, pressed to a thickness of 4 mm, and vacuum pressure impregnated either with pure CE or with different mixtures of CE and epoxy, supplied by Huntsman, Switzerland. A short summary of the investigated laminates is presented in Table 1. The curing cycle was chosen according to the recommendations of the supplier and the needs of ITER.

All irradiations were performed at ambient temperature (340 K) in the TRIGA reactor (Vienna) to fast neutron fluences of up to $4 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$), i.e. to a total absorbed dose of $\sim 200 \text{ MGy}$ [9].

The mechanical tests were carried out 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) according to the ASTM D2344 standard. To ensure interlaminar shear failure, span-to-thickness ratios of 4:1 and 5:1 were used, respectively. In addition, tension–tension fatigue measurements (ASTM D 3479) were carried out under load control at 10 Hz for $R=0.1$. Each data point refers to four or more samples. After 10^6 load cycles the tests were stopped manually.

Because of the wrapped glass fiber tapes the materials have anisotropic properties. For this reason, specimens were cut parallel (0°) and perpendicular (90°) to the winding direction of the tapes. For the 90° samples the mechanical strength is mainly determined

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Table 1
Overview of cyanate ester-based insulation systems.

		Insulation system		CE/Epoxy ratio
Type	Cyanate ester	DGEBF	T1 (100)	100:0
Resin	AroCy-L10	PY306	T2 (40)	40:60
Hardener	–	–	T8 (30)	30:70
Additives	Mn acetylacetonate in nonylphenol	–	T10 (20)	20:80
Reinforcement		R-glass/Kapton		
Curing Temp.		4h @ 100 °C 5h @ 160 °C		

by the interfaces between the reinforcing tapes and the resin, and thus, the influence of radiation damage is more pronounced than in the 0° samples.

3. Results

3.1. Ultimate tensile strength

The results of the static tensile tests are summarized in Table 2. In the unirradiated state no significant influence of the CE content on the UTS is found for all investigated materials. The results are in the range from 250 to 270 MPa. Only for the system with a CE content of 40% (T2) the UTS is slightly higher, i.e. 313 MPa. This is in good agreement with expectations by the resin supplier, suggesting that the best mechanical properties should be reached with 40% CE in the blend. After irradiation to the ITER design fluence no reduction of the UTS was observed, whereas at twice the ITER

level slight degradations (5–15%) were found for all investigated systems.

3.2. Fatigue behavior

For the characterization of the mechanical material performance under dynamic load, tension–tension fatigue measurements were carried out before and after irradiation to neutron fluences of up to $2 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$). Fig. 1 shows the absolute and normalized stress–lifetime diagrams (S–N curves, Wöhler curves) for all investigated systems before irradiation. In general, no significant influence of the CE content on the lifetime of the materials was observed. The S–N curves of all systems show a continuous decrease up to 10^6 load cycles, except for the pure CE system (T1), where a life endurance limit was found at 0.5 UTS (=125 MPa). The slight variations at higher residual strengths are mainly related to differences in the UTS, as can be seen from the normalized curves.

Table 2
Ultimate tensile strength (UTS) measured at 77 K before and after irradiation to fast neutron fluences of up to $2 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$).

Insulation system	T1 (100), UTS 90° (MPa)	T2 (40), UTS 90° (MPa)	T8 (30), UTS 90° (MPa)	T10 (20), UTS 90° (MPa)
Unirr.	250 ± 19	313 ± 18	269 ± 19	265 ± 16
$1 \times 10^{22} \text{ m}^{-2}$	250 ± 22	296 ± 10	274 ± 6	243 ± 12
$2 \times 10^{22} \text{ m}^{-2}$	228 ± 13	253 ± 16	260 ± 7	218 ± 8

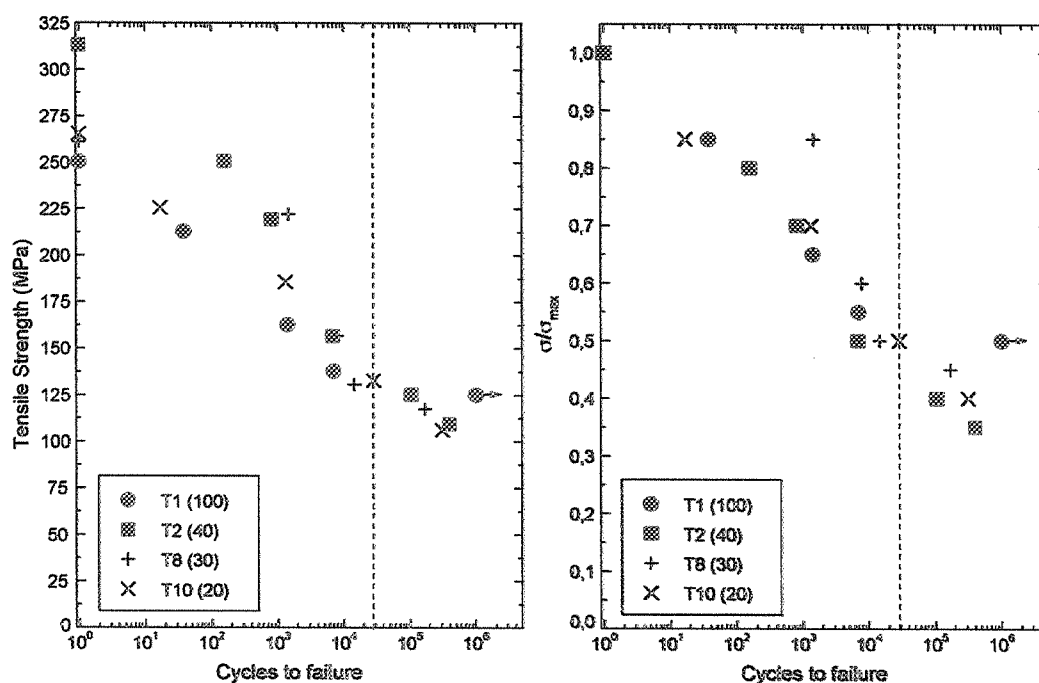


Fig. 1. Absolute (left) and normalized (right) tension–tension stress–lifetime diagrams before irradiation measured at 77 K. All measurements were stopped manually above 10^6 cycles, as indicated by the arrows.

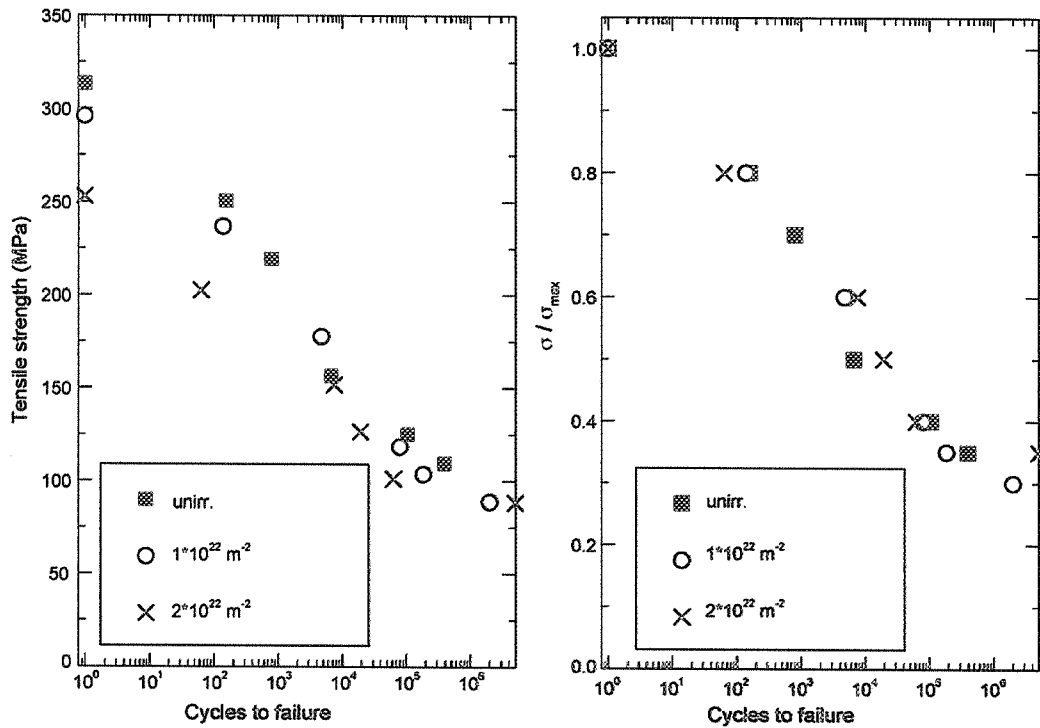


Fig. 2. Absolute (left) and normalized (right) tension–tension stress–lifetime diagrams of T2 (40) measured at 77 K before and after irradiation to fast neutron fluences of up to $2 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$).

Table 3

Interlaminar shear strength (ILSS) for both directions measured at 77 K before and after irradiation to fast neutron fluences of up to $4 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$).

Insulation system	T1 (100)		T2 (40)		T8 (30)		T10 (20)	
	0°	90°	0°	90°	0°	90°	0°	90°
Unirr.	59 ± 8	42 ± 10	77 ± 4	57 ± 3	74 ± 4	63 ± 9	62 ± 6	48 ± 9
$1 \times 10^{22} \text{ m}^{-2}$	68 ± 4	48 ± 5	66 ± 5	58 ± 3	69 ± 8	58 ± 7	65 ± 4	48 ± 5
$2 \times 10^{22} \text{ m}^{-2}$	50 ± 2	46 ± 1	61 ± 2	52 ± 3	62 ± 3	51 ± 3	53 ± 6	48 ± 3
$4 \times 10^{22} \text{ m}^{-2}$	46 ± 2	37 ± 2	52 ± 2	45 ± 6	43 ± 3	37 ± 5	36 ± 4	26 ± 4

Regarding ITER, where 30,000 load cycles are expected, the residual strengths of all four materials is about 130 MPa, which is more than adequate.

After neutron irradiation up to twice the ITER level, no distinct changes of the dynamic material performance were observed. As an

example, Fig. 2 shows the absolute and normalized S–N diagrams for the 40% CE system (T2). As can be seen there, irradiation reduces the residual strength especially at lower cycle number to failure. At the ITER point (3×10^4 cycles) and above the residual strength is less affected by irradiation. The Wöhler curves of all other systems

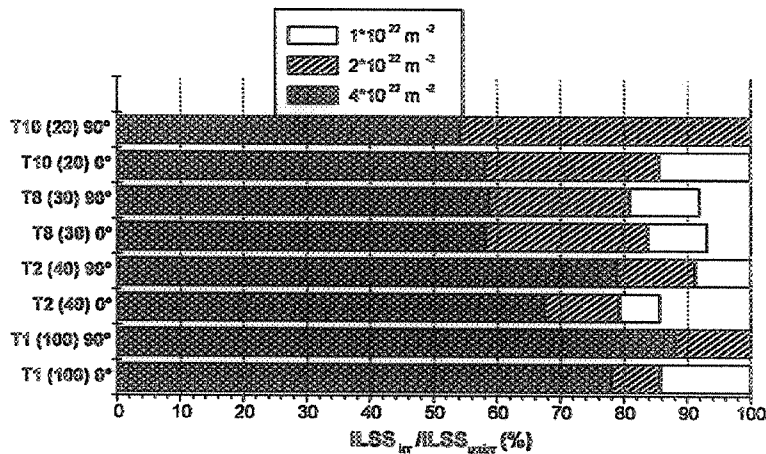


Fig. 3. Normalized interlaminar shear strength after irradiation to fast neutron fluences of up to $4 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$).

show the same behavior, except for the system with the least CE content of 20% (T10), where the shape and the absolute values of the Wöhler curves are hardly affected and an increase in the scatter of the data points is found, which indicates an enhancement of material inhomogeneities.

3.3. Interlaminar shear strength

A detailed summary of the ILSS determined in both load directions is presented in Table 3. In general, the results are in good agreement with those from the UTS. For all systems, the ILSS varies from 60 to 80 MPa for 0°, whereas in 90° the ILSS is lower by about 10–15 MPa. As expected from the previous section, slightly higher values were found for T2 (40). However, the CE content does not have a significant influence on the mechanical properties.

To make the irradiation effects more obvious, Fig. 3 shows the relative ILSS compared to the unirradiated state. Up to a neutron fluence of $2 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$) the reduction of the ILSS is not pronounced. At $1 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$) material inhomogeneities lead to small deviations, whereas at $2 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$) radiation damage starts to degrade the mechanical properties by up to 20%, which confirms the results of the tensile tests. No distinct correlation between the radiation resistance and the CE/epoxy ratio was found.

Depending on the insulation system, the irradiation effects are more or less pronounced at $4 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$). For all materials the ILSS lies between 36 and 52 MPa for 0° and between 26 and 45 MPa for 90°. Taking the variations of the initial shear strength (in the unirradiated state) into account, the normalized values demonstrate the radiation hardness of these materials. The ILSS of the insulation systems impregnated with pure CE is only reduced by about 20%. The 40% system (T2) shows similar results. On the other hand, severe radiation damage was found for the 30% (T8) and the 20% CE systems (T10), where the ILSS drops by 45%. Compared to the results on conventional DGEBA epoxy resins, such as the systems used for the toroidal field model coil of ITER, the least radiation resistant blend (T10) has still better mechanical properties, even after exposure to a four times higher neutron fluence [1,2,6].

This extensive database demonstrates that composites containing more than 40% CE have the potential to withstand neutron fluences even beyond $4 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$).

4. Summary

Fiber reinforced superconducting magnet insulation systems based on a mixture of cyanate ester (CE) and epoxy resin proved their radiation resistance up to the ITER design fluence of $1 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$). In view of applications at higher radiation levels, i.e. DEMO, the limits of these new insulation systems and the influence of the CE concentration on the radiation hardness will have to be investigated.

In the present study, several CE/epoxy blends with varying CE content were investigated under static and dynamic load conditions before and after reactor irradiation to the ITER design fluence of $1 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$) and far beyond (up to $4 \times 10^{22} \text{ m}^{-2}$). The results may be summarized as follows:

- In general, the influence of the CE content on the mechanical material properties is low. The best static material performance was found for a CE content of 40%.
- At twice the ITER fluence, i.e. at $2 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$), slight reductions of the ultimate tensile strength and of the interlaminar shear strength were observed, especially for a CE content of 20%.
- At a neutron fluence of $4 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$) the interlaminar shear strength is slightly reduced by 20–30%. Materials with a CE content below 40% are less radiation resistant.
- Under dynamic load in tension, the CE content does not have a significant influence on the material performance. Especially at the ITER point, i.e. at 30,000 cycles, and above, all investigated systems behave nearly in the same way.
- After irradiation the residual strength of all materials is not reduced at load levels around the ITER point and below. For the system with the lowest CE content (20%) an increase of material inhomogeneity was found, which indicates radiation damage.

With respect to applications at high radiation levels, the CE/epoxy blends with a CE content down to 20% showed an improved material performance even up to neutron fluences of $4 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$), when compared to conventional epoxy insulation systems exposed to $1 \times 10^{22} \text{ m}^{-2}$ ($E > 0.1 \text{ MeV}$). Especially for a CE content of 40% and beyond, the materials withstand even higher neutron fluences and retain acceptable mechanical properties.

Acknowledgements

Technical assistance by Mr. E. Tischler and Mr. H. Hartmann is acknowledged. This work, supported by the European Communities under the contract of Association between EURATOM-OEAW, was carried out within the framework of the European Fusion Development Agreement. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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