Qualification of the Resin System for the ITER Toroidal Field Coils

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***Abstract*—In view of the radiation environment prevailing at the location of the ITER toroidal field coils, the replacement of “traditional” magnet insulation systems, i.e. glass-fiber reinforced epoxies, by less radiation sensitive materials was found to be necessary. After detailed studies of all kinds of resin systems (various types of epoxy, bismaleimides, etc.), composites consisting of boron-free glass fabrics and polyimide layers impregnated by cyanate ester (CE) or cyanate ester – epoxy blends turned out to fulfill all the requirements set for the ITER TF coils. These are in particular (i) radiation tolerance of the electrical and mechanical properties up to twice the ITER lifetime fluence of fast neutrons, (ii) suitability for vacuum pressure impregnation, (iii) sufficiently long pot life for this purpose, (iv) reasonably low curing temperature, (v) suitability for multiple impregnations, and (vi) suitability for large scale industrial application. Among all the blends investigated, the resin consisting of 40 wt% CE and 60 wt% epoxy was found to perform best. As a consequence, a qualification program was implemented by IO with the aim of fabricating sample plates under identical conditions and testing the products provided by industrial suppliers with regard to their radiation hardness and their mechanical performance under ITER-like conditions. All four resins provided so far by companies in Europe (Huntsman), the US (CTD) and Japan (IST) passed the qualification procedures and are now fully qualified as insulation materials for the ITER TF coils.**

***Index Terms*—Bonded glass fiber / polyimide tapes, Qualification, Radiation resistant resins, ITER**

I. INTRODUCTION

EXTENSIVE work has been done to find alternative insulation systems, which can withstand the challenging environment of the ITER toroidal field coils. Conventional resins (various types of epoxy, bismaleimides, etc.) show severe degradation at the ITER design fluence of $1\mathrm{x}10^{22}$ m^{-2} (E>0.1 MeV). Compared to epoxy resins, cyanate esters (CE) offer enhanced temperature and radiation resistance as well as high mechanical strength (cf. Fig 1) [1]. Also the gas evolution due to irradiation is significantly lower [2, 3]. In addition, no technological changes in the coil fabrication are needed, since CE resins can be treated like epoxies and are compatible with the VPI process. Blending cyanate ester with epoxy resins is the best way to reduce the costs as well as the reactivity. A mixing ratio of 40 % CE and 60 % epoxy turned out to be the solution for the TF coil insulation [4]. Various companies offer pure CE as well as CE blends, which were mechanically characterized before and after irradiation [5, 6]. However, these results were not directly comparable due to differences in the fabrication process as well as the reinforcement. Tests have also shown that the total absorbed dose is a better scaling quantity than the neutron fluence, just when materials were irradiated in different sources [7].

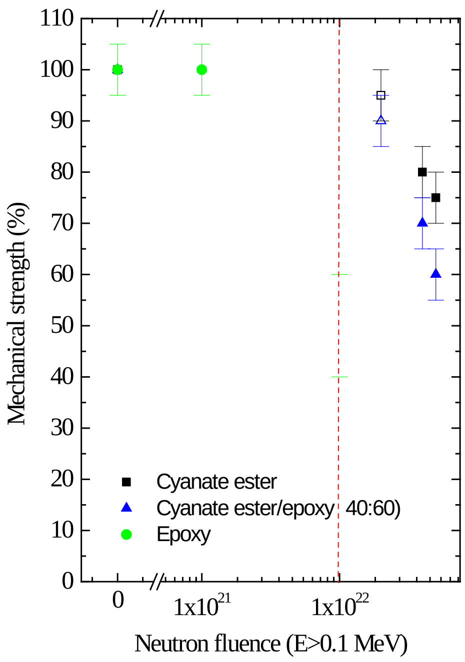

Fig. 1. Mechanical strength of various types of resins

In order to find the best resin, a qualification program [8, 9] on the insulation system selected for the ITER TF coils was launched by the ITER Organization (IO). Various industrial resin systems, supplied by companies from Europe, US and Japan were investigated and test samples produced under exactly the same conditions. In addition, pre-bonded glass fiber / polyimide tapes, which would simplify and also enhance the speed of wrapping the insulation, were tested.

Finally, the mechanical properties of a low temperature curable cyanate ester / epoxy blend suitable for local repairs were investigated, which is essential, especially for a large and complex structure like the TF coils.

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TABLE I
PROPERTIES OF FOUR RESIN SYSTEMS COMPARED TO THE ITER RESIN SPECIFICATION

	ITER spec.	CTD-425	Huntsman	IST	CTD-435
Processing temperature	< 60 °C	50 °C	50 °C	55 °C	60 °C
Jellification	24 h @ 100 ± 10 °C	22 h @ 100 °C	14 h @ 100 °C	12 h @ 100 °C	24 h @ 100 °C
Curing	24 h @ 160 ± 10 °C	24 h @ 170 °C	18 h @ 155 °C	18 h @ 150 °C	24 h @ 155 °C
Pot-life	> 100 h	> 100 h	> 100 h	> 100 h	> 100 h

All materials were characterized at 77 K before and after irradiation to a fast neutron fluence of $2x10^{22}$ m^{-2} (E>0.1 MeV).

The present paper summarizes these extensive investigations.

II. SAMPLE FABRICATION AND TEST PROCEDURES

A. Sample fabrication

All materials were fabricated by Marti-Supratec Corporation, Switzerland, using the vacuum pressure impregnation (VPI) technique. For the qualification of the different resins, test plates were fabricated using an identical reinforcement in order to make the results comparable. The reinforcement of these materials consists of one R-glass fiber layer (0.25 x 40 mm) and seven layers of glass fiber/ polyimide (Kapton H tape (0.05 x 36 mm)) sandwich tapes, which were wrapped half-overlapped around an aluminium plate. The wrapped plates were dried under vacuum at ~100 °C to remove moisture which may be absorbed during storing and wrapping. Afterwards they were impregnated with CE/ epoxy mixtures from Huntsman, Switzerland, (LMB6653 / LMB6622-4), Composite Technology Development, U.S.A, (CTD-425 and CTD-435) and Industrial Summit Technology, Japan, (IST) and cured according to the recommendations of the resin supplier within the ITER resin specification (cf. TABLE 1).

For the qualification of bonded glass fiber / polyimide tapes, test materials were fabricated according to the lay-up of the ITER turn insulation, i.e. one layer of S2 glass fibers (0.15 mm thickness), 3 layers of bonded tapes and 1 layer of S2 glass fibers (0.25 mm thickness), where each layer is wrapped half overlapped. The tapes were supplied by Arisawa, Japan, and Advanced Composite Group (ACG), U.K. The Arisawa tape consists of a 40 mm x 0.13 mm S2 glass fiber tape and a 40 mm x 0.025 mm Kapton HN tape. The tapes were bonded with a cyanate ester (CE) based resin. In case of the ACG tape, S2 glass fiber tapes (40 mm x 0.15 mm) from Streiffband, Switzerland, were used for bonding. An Upilex polyimide film (36 mm wide) was glued onto the glass fiber tape. The actual thickness of the two tapes was between 0.18 and 0.19 mm, measured with a micrometer caliper (cf. Fig. 2). The wrapped plates were impregnated with the CE / epoxy blend from Huntsman (LMB6653/LMB6622-4).

Fig. 2. Bonded glass fiber / polyimide tapes from Arisawa (left) and ACG (right).

In order to simulate a realistic repair scenario, material plates from the EFDA project 07-1709/1605 (Task TW7-TMSM-BLEND) were used as an existing insulation, where the mechanical properties before and after irradiation are known [10]. These plates consist of a R-glass fiber / polyimide reinforcement impregnated with a CE / epoxy blend (LMB6653 / LMB6622 from Huntsman [11]). Half of the layers were removed by grinding such that the boundary between the original insulation and the repair insulation is located at the center of the composite. Afterwards, the surfaces of the plates were sand blasted, cleaned and degreased in order to achieve a higher bonding strength. The new layers of glass fiber / polyimide sandwich tapes were wet wrapped half overlapped to obtain again the original thickness of 4 mm.

B. Test procedures

All mechanical tests were carried out at 77 K using a servo-hydraulic MTS 810 test device, which was modified for measurements in a liquid nitrogen environment. The ultimate tensile strength (UTS) was measured according to the DIN 53455 and the ASTM D638 standards. However, the ASTM D638 Type M-I sample geometry is not suitable for irradiation experiments due to the limited space inside the irradiation facility. Therefore, the irradiation experiments were carried out on a downscaled sample geometry, i.e. dog-bone shaped samples for the qualification of the resins [12] and downscaled

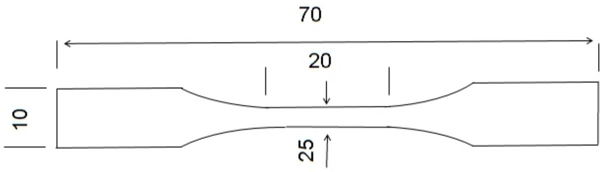

Fig. 3. Downscaled ASTM (dASTM) sample geometry used for irradiation tests.

ASTM samples (cf. Fig. 3) for testing the bonded glass fiber / polyimide tapes.

The tensile stresses are relatively low, but the stress distribution at the TF coil insulation is of mixed mode. Therefore, it is important to determine the intrinsic material properties e.g. under pure tension as well for use as an input parameter for FEM modeling of the material, i.e. to simulate appropriate loading conditions for specified applications.

For the simulation of the pulsed ITER operation, tension-tension fatigue measurements (ASTM D3479) were done at a frequency of 10 Hz applying a sinusoidal load function up to 10^6 cycles at minimum-to-peak stress ratios (R-ratio) of 0.1. Each data point refers to the average of 4 or more samples.

The apparent interlaminar shear strength (ILSS) was assessed by the short-beam-shear (SBS) test according to the ASTM D2344 standard using cuboid specimens of 23 x 6.4 x 4 mm^3 size. To ensure interlaminar shear failure, span-to-thickness ratios of 4:1 and 5:1 were chosen.

Because of the wrapped glass fiber tapes the materials have anisotropic properties. Therefore, specimens were cut parallel (0°) and perpendicular (90°) to the winding direction of the tapes.

All irradiations were performed at ambient temperature (340 K) in the TRIGA reactor (Vienna) to a fast neutron fluence of $2x10^{22}$ m^{-2} (E>0.1 MeV), i.e. to a total absorbed dose of ~100 MGy [13].

The glass transition temperature was determined by evaluating the DMA tan-delta peak. Samples with dimensions of 6 mm x 20 mm and a maximum thickness of 2 mm were prepared. The repaired area was separated from the old insulation and T_g was only determined for the repaired section.

III. RESULTS

A. *Qualification of resins*

1) *Tensile properties*

The UTS of the materials in 90° direction was measured on the dog-bone shaped specimen geometry at 77 K prior to and after reactor irradiation to twice the ITER design fluence (cf. TABLE 2).

Before irradiation, the UTS of all four materials is nearly identical. Only the UTS of the Huntsman material (309 MPa) is slightly lower compared to the ~360 MPa of the other materials. After irradiation, the UTS is slightly reduced. In general, it degrades by ~5 % (Huntsman) to ~15 % (CTD-425 and IST). A higher reduction was only observed for the CTD-435 material (~ 30 %).

TABLE 2
ULTIMATE TENSILE STRENGTH (MPA) OF THE HUNTSMAN, CTD-425, IST AND CTD-435 MATERIALS IN 90 ° DIRECTION BEFORE AND AFTER IRRADIATION TO A FAST NEUTRON FLUENCE OF $2x10^{22}$ M^{-2} (E>0.1 MEV)

	unirradiated (MPa)	irradiated (MPa)
Huntsman	309 ± 5	292 ± 13
CTD-425	360 ± 5	294 ± 13
IST	359 ± 19	310 ± 11
CTD-435	349 ± 9	247 ± 11

Also under dynamic tensile load, all four materials show excellent mechanical properties as well as the expected radiation hardness after irradiation to $2x10^{22}$ m^{-2} (E>0.1 MeV). Fig. 4 shows the absolute stress-lifetime diagrams (S-N curves, Wöhler curves). As can be seen, all the Wöhler curves show a continuous decrease up to 10^6 load cycles and the shape of the curves is nearly identical apart from minor deviations at high load levels. Regarding ITER, where 30000 load cycles are expected, the residual strength of all materials is between 130 MPa and 140 MPa, which is more than adequate for ITER. This situation is hardly changed after neutron irradiation. The materials are not affected by irradiation, especially in the ITER relevant area.

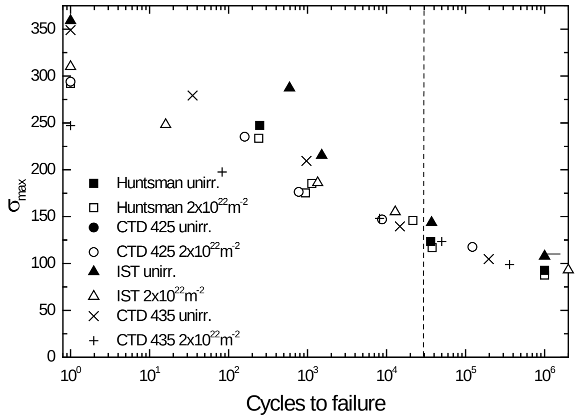

Fig. 4. Absolute stress-lifetime diagrams of the Huntsman, CTD-425, IST and CTD-435 materials in 90° direction measured at 77 K prior to and after reactor irradiation to a fast neutron fluence of $2x10^{22}$ m^{-2} (E>0.1 MeV).

TABLE 3
APPARENT INTERLAMINAR SHEAR STRENGTH (MPA) OF THE HUNTSMAN, CTD-425, IST AND CTD-435 MATERIALS IN 0° AND 90° DIRECTION MEASURED AT 77 K PRIOR TO AND AFTER REACTOR IRRADIATION TO A FAST NEUTRON FLUENCE OF 2X10^{22} M^{-2} (E>0.1 MEV)

Material	ILSS 0° (MPa)		ILSS 90° (MPa)	
	unirradiated	irradiated	unirradiated	irradiated
Huntsman	76 ± 6	72 ± 4	56 ± 4	51 ± 4
CTD-425	71 ± 3	70 ± 4	58 ± 6	53 ± 5
IST	78 ± 2	74 ± 3	64 ± 7	61 ± 4
CTD-435	75 ± 4	62 ± 5	65 ± 2	52 ± 6

2) *Apparent interlaminar shear strength*

TABLE 3 presents the static test results (77 K) on the apparent ILSS of all materials in 0° and 90° direction prior to and after reactor irradiation to twice the ITER design fluence. For the unirradiated materials the ILSS is on average about 75 MPa for 0° and about 60 MPa for 90°. This is in good agreement with the tensile properties, where also no significant differences between the materials were found. After irradiation, the ILSS remains nearly unchanged for all materials in 0°, and is slightly reduced by 10 % for 90°. Only the CTD-435 material shows a higher reduction by ~20 %.

This also confirms the excellent radiation resistance of the materials up to this fluence level.

B. Bonded glass fiber / polyimide tapes

The UTS values, summarized in TABLE 4, were obtained on the downscaled ASTM sample geometry and measured at 77 K in their weakest direction (90°). Surprisingly, there is a significant difference between the two materials. The UTS of the Arisawa is higher by about 30 % compared to the ACG material. The reason for this seems to be the bonding strength between resin and polyimide. As mentioned in the previous section, two types of polyimide tapes were used for the fabrication of the bonded tapes. Looking at the fractured specimens (Fig. 4), one can clearly see their different behavior. For the Arisawa material only the top layers, which consist of the thicker glass fiber tapes, delaminate. The part consisting of bonded tapes is still a single composite, where no obvious delamination is observed, indicating excellent adhesion between the fiber/ polyimide/ matrix interfaces. In case of the ACG material, nearly each layer is separated; however, the polyimide tape is still partly attached to the glass fiber tape.

TABLE 4
ULTIMATE TENSILE STRENGTH (MPA) OF THE ARISAWA AND THE ACG MATERIAL IN 90 ° DIRECTION BEFORE AND AFTER IRRADIATION TO A FAST NEUTRON FLUENCE OF 2X10^{22} M^{-2} (E>0.1 MEV) MEASURED AT 77 K

	Arisawa	ACG
unirr.	445 ± 13	332 ± 21
irr.	407 ± 15	278 ± 6

Neutron irradiation has only a small effect on the mechanical properties. The UTS of the Arisawa material is reduced by ~ 10 %, i.e. the same as observed for the material consisting of un-bonded tapes and the same resin [9]. The degradation of the ACG material is slightly higher (~ 15 %), but not a big issue. The UTS value is still well above the ITER specification. This indicates that the bonding agent of both materials is extremely radiation resistant and does not affect the mechanical properties in a negative way, even at this high fluence level. However, the bonding strength between resin and polyimide, which seems to be much higher in combination with Kapton than with Upilex, needs further investigations.

Fig. 4. Fractured tensile specimen of the Arisawa (left) and ACG (right) material.

C. Repair solution

Two types of a “repaired” insulation were fabricated using different curing cycles. One material (Rep A) was cured at 120 °C according to the recommendation of the resin supplier. For the second (Rep B), the curing temperature was reduced to only 80 °C and the curing time was doubled for compensation. The fabrication parameters are summarized in TABLE 5.

TABLE 5
OVERVIEW OF THE REPAIRED MATERIALS

Material	Rep A (recommended)	Rep B (test)
Impregnation temperature	RT	RT
Jellification	6 h @ 60 °C	6 h @ 60 °C
Curing	48 h @ 120 °C	96 h @ 80 °C

TABLE 6
APPARENT INTERLAMINAR SHEAR STRENGHT (MPA) OF THE REPAIRED MATERIALS IN 0° AND 90° DIRECTION MEASURED AT 77 K PRIOR TO AND AFTER REACTOR IRRADIATION TO A FAST NEUTRON FLUENCE OF 2×10^{22} m^{-2} (E>0.1 MEV)

Material	ILSS 0° (MPa)		ILSS 90° (MPa)	
	Unirr.	irr.	unirr.	irr.
Rep A	59 ± 5	46 ± 5	49 ± 4	34 ± 4
Rep B	50 ± 6	36 ± 2	47 ± 4	31 ± 2
Original	74 ± 5	47 ± 3	58 ± 6	39 ± 4

1) *Apparent interlaminar shear strength*

Both repaired materials were characterized under static interlaminar shear load before and after irradiation. The results of these tests are listed in TABLE 6. In 0° direction, the Rep A material cured at 120 °C shows a higher ILSS value by about 20 % compared to the Rep B material cured at 80 °C in the unirradiated state. In 90° direction, the ILSS is about the same for both the Rep A and the Rep B material. In general, the ILSS of both repaired insulations is lower by at least 20 % than that of the original sample. Looking at the test samples shows that the reduced ILSS is not caused by the properties of the repair resin. The main reason for this reduction seems to be the quality of the repaired section. As can be seen in Fig. 5 the uniformity of the reinforcement in the repaired section is not as good as in the un-repaired area. The reinforcing tapes are more undulated. In addition, investigations under an optical microscope show a significant number of voids or bubbles causing an earlier failure of the composite (cf. Fig. 5, right). This problem can be overcome by improving the quality of the repair leading to an increase of the mechanical strength.

After irradiation the situation is similar to the unirradiated state. The material cured at 120 °C has a higher ILSS value by up to 30 % in 0° direction and by up to 10 % in 90° direction, respectively. Compared to the original material, the ILSS of Rep A is only slightly lower. Both materials show an excellent radiation resistance demonstrating their suitability as a possible repair solution for the ITER TF coil insulation.

2) *Glass transition temperature*

In addition, the glass transition temperature was measured in order to assess the influence of the modified curing schedule far below the recommendation. The results on the glass transition temperature are summarized in TABLE 7. The material cured at 120 °C has the higher T_g, which is in the range of the four CE/epoxy blends for the ITER TF coil insulation, which have already been qualified [8]. T_g values of around 170 °C were obtained on these materials. The T_g of the material cured at 80 °C is significantly lower and marginally below the ITER specification. The lower T_g indicates a lower degree of the cross linked chemical structure, which explains the differences of the ILSS between these two materials.

TABLE 7
GLASS TRANSITION TEMPERATURE OF THE INVESTIGATED MATERIALS

Material	T_g
Rep A	175 °C
Rep B	146 °C
TF coil spec.	> 150 °C

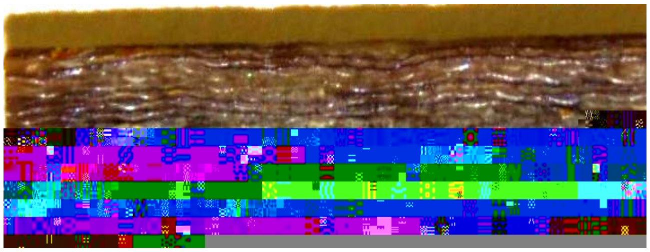

Fig. 5. Short beam shear specimen of the “repaired” insulation. The top half of the sample is the repaired section and the bottom the original insulation (left) and magnified view of the repaired area with a significant number of bubbles or voids.

IV. SUMMARY

Research on advanced insulating materials for fusion magnets led to a qualification program for the ITER magnet insulation system from various industrial suppliers. Various resin systems, pre-bonded glass fiber / polyimide tapes and a repair resin were characterized prior to and after neutron irradiation to a fast neutron fluence of 2×10^{22} m^{-2} (E>0.1 MeV) and tested at 77 K with regard to their suitability for the ITER TF coils according to the ITER specifications.

The results may be summarized as follows

- Each of the four resins shows excellent mechanical properties prior to and after irradiation to twice the ITER design fluence. The ultimate tensile strength in their weakest direction is between 309 MPa and 360 MPa. After irradiation, the degradation is 5 % (Huntsman), 15 % (CTD-425 and IST) and 30 % (CTD-435), which indicates the high radiation hardness of all materials.
- Under dynamic tensile load, the material performance of the Huntsman, CTD-425, IST, and CTD-435 materials is very similar especially in the ITER relevant range of 30000 load cycles. The residual strength is in the range from 130 MPa (Huntsman, CTD-425, CTD-435) to 140 MPa (IST). Neutron irradiation does not show any influence.
- The interlaminar shear strength of the unirradiated Huntsman, CTD-425, IST, and CTD-435 materials is on average about 75 MPa for 0° and about 60 MPa for 90°. Neutron irradiation does not reduce the ILSS in 0° direction. In 90° direction, the reduction amounts only to ~10 %, except for the CTD-435 material (~20 %).
- The ACG material shows an unexpectedly low ultimate tensile strength compared to the Arisawa material. Severe delamination can be seen, indicating weak bonding between the cyanate ester / epoxy blend and the Upilex polyimide. However, the bonding agent itself has proved its radiation resistance.
- The Arisawa material shows excellent mechanical properties before and after irradiation. No influence of the bonding agent on the mechanical properties is observed.
- Both types of bonded glass fiber polyimide tapes clearly fulfill the ITER specification.
- The interlaminar shear strength of the “repaired” material cured at 120 °C is higher by up to 20 % compared to that cured at 80 °C depending on the loading direction. In general, the interlaminar shear strength of both “repaired” insulations is lower than that of the original material, which is caused by the lower quality of the repair. However, the radiation resistance is sufficiently high.
- The glass transition temperatures of both materials are well above or close to the specification. Especially the material cured at 120 °C shows the same glass transition temperature as the resins qualified for the TF coils.
- The repair resin can be applied with minor modifications of the curing cycle at temperatures down to ~80 °C.

All tested materials have demonstrated their excellent mechanical material performance both under static and dynamic load in tension as well as under static interlaminar shear load before and after reactor irradiation up to a radiation level, which will be accumulated over the ITER lifetime and beyond, i.e. at twice the ITER design fluence. All materials are successfully qualified.

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