

# MECHANICAL CHARACTERIZATION OF THE ITER MOCK-UP INSULATION AFTER REACTOR IRRADIATION

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## ABSTRACT

The ITER mock-up project was launched in order to demonstrate the feasibility of an industrial impregnation process using the new cyanate ester / epoxy blend. The mock-up simulates the TF winding pack cross section by a stainless steel structure with the same dimensions as the TF winding pack at a length of 1 m. It consists of 7 plates simulating the double pancakes, each of them is wrapped with glass fiber / Kapton sandwich tapes. After stacking the 7 plates, additional insulation layers are wrapped to simulate the ground insulation. This paper presents the results of the mechanical quality tests on the mock-up pancake insulation. Tensile and short beam shear specimens were cut from the plates extracted from the mock-up and tested at 77 K using a servo-hydraulic material testing device. All tests were repeated after reactor irradiation to a fast neutron fluence of  $1 \times 10^{22} \text{ m}^{-2}$  ( $E > 0.1 \text{ MeV}$ ). In order to simulate the pulsed operation of ITER, tension-tension fatigue measurements were performed in the load controlled mode. Initial results show a high mechanical strength as expected from the high number of thin glass fiber layers, and an excellent homogeneity of the material.

**KEYWORDS:** Fiber reinforced composites, ITER mock-up, Cyanate ester blend, Mechanical behavior, Neutron irradiation

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## INTRODUCTION

In view of the need for new radiation resistant resins for the ITER toroidal field (TF) coil insulation system, mixtures of cyanate ester (CE) and epoxy resins were found to show the necessary radiation resistance [1-4].

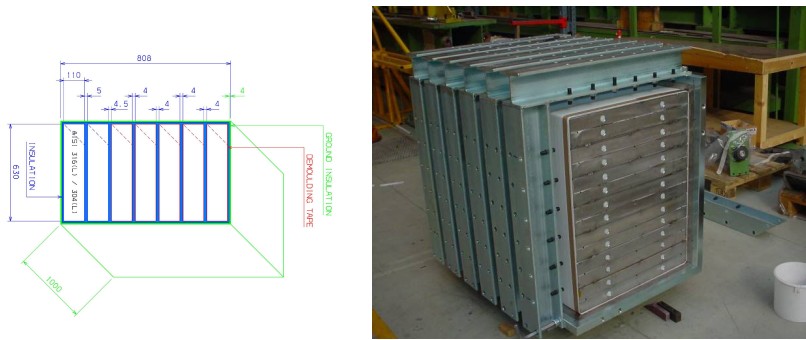


FIGURE 1. Schematic view of the mock-up (left) and photo of the assembled mock-up before installation inside the autoclave (right).

However, the test materials were fabricated under laboratory conditions and industrial experience was lacking. Therefore, a mock-up of the ITER TF coil winding pack with a length of 1 m was designed by CEA, Saclay, France, and built by ASG Superconductors, Genoa, Italy. It consisted of seven steel plates wrapped with glass fiber/Kapton tapes, which were stacked in order to simulate the pancake structure of the coil (cf. FIGURE 1 left). The objective of this work was to demonstrate the industrial feasibility of employing the new resin system with respect to vacuum pressure impregnation (VPI), large volume impregnation and safety aspects. The dimensions of the whole assembly including the mixing device, pipes and mould were chosen for handling an amount of  $\sim 50$  kg of the CE/epoxy resin supplied by Huntsman, Switzerland.

The insulation consists of R-glass fiber tapes (0.12 x 40 mm) and Kapton HN tapes (0.05 x 36 mm), which were wrapped half overlapped (ho). An insulation thickness of 2 mm was applied on each steel plate. By stacking the plates the final interlayer insulation had a thickness of 4 mm. Afterwards, additional insulation layers were wrapped around the stacked plates, simulating the ground insulation.

This paper presents the results on samples cut from these plates. The insulation system was characterized under static and dynamic tensile load as well as under static interlaminar shear load at 77 K. In order to check the influence of radiation, mechanical tests were carried out after irradiation to the ITER design fluence of  $1 \times 10^{22} \text{ m}^{-2}$  ( $E > 0.1$  MeV). In addition, the results will be compared with a similar insulation system fabricated under laboratory conditions [5].

## EXPERIMENTAL DETAILS

### Materials

The investigated insulation system was fabricated by ASG Superconductors, Genoa, Italy, using the vacuum pressure impregnation (VPI) technique. The assembled mock-up (cf. FIGURE 1 right) was placed into an autoclave. Before the impregnation the autoclave was externally heated and evacuated to dry the glass fiber tapes, in case of moisture absorption during wrapping. The VPI process was carried out at 60 °C, where  $\sim 20$  l of resin were introduced into the mock-up. Then the curing cycle described in TABLE 1 was

TABLE 1. Overview of cyanate ester based insulation systems

|               | Mock-up   |                                | T13   |                                |
|---------------|---|--------------------------------|---|--------------------------------|
| Type          | Cyanate Ester<br>about 40 %                                       | DGEBF + catalyst<br>about 60 % | Cyanate Ester<br>about 40 %   | DGEBF + catalyst<br>about 60 % |
| Resin         | AroCy-L10<br>(LMB 6653)   | PY306 + catalyst<br>(LMB 6622) | AroCy-L10<br>(LMB 6653)   | PY306 + catalyst<br>(LMB 6622) |
| Hardener      | ----  |                                | ----  |                                |
| Catalyst      | Co Acetylacetonate<br>in Pentylphenol                             |                                | Co Acetylacetonate<br>in Pentylphenol   |                                |
| Reinforcement | R-glass / Kapton<br>2G (ho) + 5 GK (ho) + 5 GK (ho) + 2 G<br>(ho) |                                | R-glass / Kapton<br>1 G (no) + 3 GK (ho) + 1 G (ho) + 3 GK<br>(ho) + 1 G (no) |                                |
| Comments      | R-glass: 0.12 x 40 mm<br>symmetric lay-up                         |                                | R-glass: 0.25 x 40 mm<br>symmetric lay-up                                     |                                |
| Curing Temp.  | 12 h @ 100 °C<br>12 h @ 155 °C                                    |                                | 6 h @ 100 °C<br>4 h @ 120 °C<br>16.5 h @ 150 °C                               |                                |

applied with a heating rate of 2 °C/h. After the impregnation two interlayer insulation plates were delivered to ATI and the results of the mechanical characterization are presented in this work.

The second material designated as T13 was fabricated by MartiSupratec, Switzerland. The reinforcement consists of multiple half overlapped (ho) and non overlapped (no) glass / Kapton tapes to simulate the symmetric mock-up material. Four wrapped aluminium plates were enclosed by stainless steel plates, which were welded to form the mould. Press and heat plates were attached to the assembly to compress the wrapped plates and to achieve a more homogeneous temperature distribution. Before the vacuum impregnation the mould was heated and evacuated to dry the glass fiber tapes. Afterwards the vacuum impregnation was carried out at 55 °C, followed by the curing cycle described in TABLE 1.

There are certain differences between these two materials, mainly concerning the build-up of the reinforcement, since the mock-up insulation consists of thinner glass fiber tapes than those used in our previous test program. However, the final thickness of both insulation systems was 4 mm and the.

An overview of the material parameters is given in TABLE 1.

## Irradiation and Test Procedures

The reactor irradiation was done in the TRIGA reactor (Vienna) at ambient temperature (340 K) to fast neutron fluences of up to  $1 \times 10^{22} \text{ m}^{-2}$  ( $E > 0.1 \text{ MeV}$ ), which corresponds approximately to a total absorbed dose of 50 MGy [6]. Calculations of the absorbed dose show that the neutron contribution is in the range of 30 %.

In order to characterize the material, 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 ultimate tensile strength (UTS) was measured according to DIN 53455 and ASTM D638. Because of space limitations inside the irradiation facility, dog- bone shaped samples of 70 mm length were cut from the sample plates. Scaling experiments showed that the smaller size did not significantly influence the results compared to the geometry of the standards [7, 8].

The interlaminar shear strength (ILSS) was determined by the short-beam-shear (SBS) test according to the ASTM D2344 standard (23 x 6.4 x 4 mm<sup>3</sup> sample size) using 10 samples each. Span-to-thickness ratios of 4:1 and 5:1 were used to obtain interlaminar fracture. Only samples which have shown interlaminar failure were taken for evaluation. To simulate the pulsed TOKAMAK operation, tension-tension fatigue measurements (ASTM D 3479) were carried out in the load controlled mode at a frequency of 10 Hz and a minimum to peak stress ratio of R=0.1. Each data point refers to 4 or more samples. After 10<sup>6</sup> load cycles the tests were stopped manually.

Because of the manufacturing process the materials have anisotropic properties. Therefore, short-beam-shear specimens were cut parallel (0°) and perpendicular (90°) to the winding direction of the reinforcing glass fiber tapes. For the tensile tests, the samples were only cut in 90° direction. In this direction the influence of radiation damage is more pronounced, because the mechanical strength is determined mainly by the fiber/resin adhesion.

## RESULTS

### Ultimate Tensile Strength

The UTS of the unirradiated material in 90 ° direction is 465 MPa. This value is much higher than for T13 (324 MPa). However, this increase by ~ 50 % compared to the material fabricated under small scale conditions is not unexpected and related to the reinforcement. As mentioned above, the glass fiber tapes used for the mock-up are thinner by a factor of two, but the final thickness is the same. Therefore, the mock-up material has more layers (28 compared to 16), and consequently, more fiber/resin interfaces, which results in the higher UTS. Assuming an ideal stress distribution, one interface has to carry individually lower loads, when more interfaces exist.

Similar results were found in previous tests [9]. An UTS increase by 50 % was observed, when the glass fiber thickness was reduced by a factor of two, while the sample thickness was kept the same.

Neutron irradiation to the ITER design fluence of 1x10<sup>22</sup> m<sup>-2</sup> (E > 0.1 MeV) does not significantly affect the mechanical properties of the composite material. The UTS is slightly reduced to 390 MPa and 282 MPa, respectively. Apart from the differences of the UTS in the unirradiated state the relative drop after irradiation is nearly the same for both materials. The results of the static tensile tests are summarized in TABLE 2.

### Tension Tension Fatigue Behavior

In addition, tension tension fatigue measurements were carried out on both materials. FIGURE 1 shows the absolute and normalized stress-lifetime diagrams (Wöhler curves). Over the entire cycle range the residual strengths of the mock-up material are higher than for T13. Especially at the ITER point, i.e. 30000 cycles, the residual strength is higher by 100 MPa with absolute values of 225 MPa and 125 MPa, respectively. However, this is mainly due to the fact that the UTS is significantly different, as demonstrated by the normalized Wöhler curves, which give a better indication of the material quality. As can be seen in FIGURE 2 (right), the material properties are quite similar. However, at load levels below  $\sigma = 0.8 \sigma_{\max}$ , the mock-up material withstands slightly more cycles. Also the scatter

TABLE 2. Ultimate tensile strength (UTS) in 90° direction measured at 77 K before and after irradiation to a fast neutron fluence of  $1 \times 10^{22} \text{ m}^{-2}$  ( $E > 0.1 \text{ MeV}$ ).

| Insulation system                 | Mock-up       | T13           |
|-----------------------------------|---------------|---------------|
|                                   | UTS 90° (MPa) | UTS 90° (MPa) |
| unirr.                            | $465 \pm 8$   | $324 \pm 15$  |
| $1 \times 10^{22} \text{ m}^{-2}$ | $390 \pm 9$   | $282 \pm 23$  |

of the results at a given load (not shown) is lower for the mock-up material, which indicates the excellent material properties as well as the impregnation quality achieved even on a large scale.

### Interlaminar Shear Strength

As expected from the tensile tests, the ILSS of the mock-up material is significantly higher than that of T13. A summary of the results of the short-beam-shear tests can be found in TABLE 3. For the mock-up material, the ILSS in 0° direction is higher by ~ 25 MPa than in T13, whereas in 90° direction the difference is ~ 20 MPa.

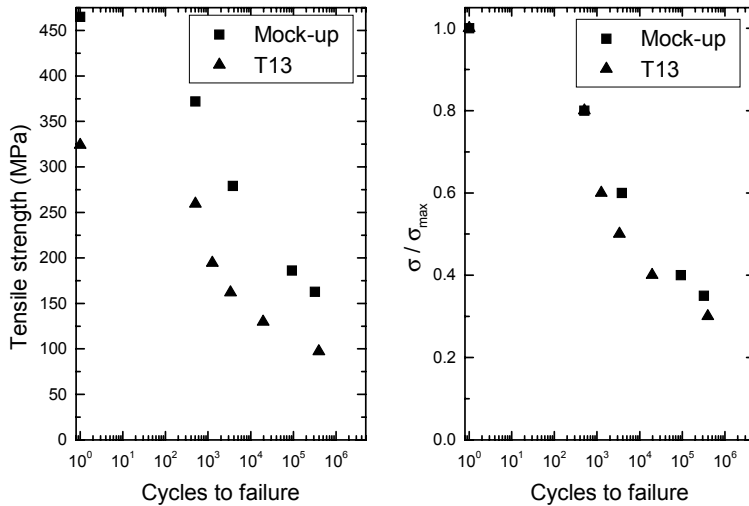


FIGURE 2. Absolute (left) and normalized (right) tension-tension stress-lifetime diagrams of the unirradiated mock-up and the T13 insulation system measured at 77 K.

TABLE 3. Interlaminar shear strength (ILSS) in 0° and 90° direction measured at 77 K before and after irradiation to a fast neutron fluence of  $1 \times 10^{22} \text{ m}^{-2}$  ( $E > 0.1 \text{ MeV}$ ).

| Insulation system                 | Mock-up          |                   | T13              |                   |
|-----------------------------------|------------------|-------------------|------------------|-------------------|
|                                   | ILSS 0°<br>(MPa) | ILSS 90°<br>(MPa) | ILSS 0°<br>(MPa) | ILSS 90°<br>(MPa) |
| unirr.                            | 102 ± 4          | 79 ± 6            | 74 ± 5           | 58 ± 6            |
| $1 \times 10^{22} \text{ m}^{-2}$ | 85 ± 5           | 67 ± 8            | 68 ± 3           | 53 ± 2            |

After irradiation to the ITER design fluence of  $1 \times 10^{22} \text{ m}^{-2}$  ( $E > 0.1 \text{ MeV}$ ), the drop of the ILSS is not very pronounced, especially in 90° direction, where a reduction of a few percent is observed. However, both materials are well above the ITER specification of ~ 45 MPa [10].

## SUMMARY

Results on the ITER mock-up insulation, manufactured by industry, are compared with a similar material fabricated under laboratory conditions. Both materials were exposed to the ITER design fluence of  $1 \times 10^{22} \text{ m}^{-2}$  ( $E > 0.1 \text{ MeV}$ ), in order to check their radiation resistance.

The results may be summarized as follows:

- Both the UTS and the ILSS of the mock-up insulation are higher by up to 50 % compared to the laboratory samples. The enhanced strength is related to the higher number of thinner glass fiber tapes and does not indicate a “better” material quality.
- Under dynamic load the mock-up material survives a slightly higher number of cycles at a given relative load. In addition, the scatter of the results at a certain load is lower, which demonstrates the excellent impregnation quality as well as the material homogeneity.
- Irradiation to a fast neutron fluence of  $1 \times 10^{22} \text{ m}^{-2}$  ( $E > 0.1 \text{ MeV}$ ) confirms the results expected from previous tests on comparable CE/epoxy blends. At this neutron fluence the reduction of the mechanical strength is negligible, both under tensile and interlaminar shear load.

The mechanical characterization demonstrates that the industrial fabrication of an ITER like insulation is feasible, and even more importantly, that the quality of the material is not worse than when fabricated under laboratory conditions.

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