Assessment of the Mechanical Properties of Candidate ITER Magnet Insulation Systems before and after Reactor Irradiation

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Abstract. The ITER design fluence of fast neutrons is 1×10^{22} m^{-2} (E>0.1 MeV). At this fluence conventional glass-fibre / Kapton insulations impregnated with DGEBA epoxy resins are at the edge of disintegration. This was the reason for the Atomic Institute to engage in measuring mechanical properties of sample materials from magnet manufacturers and alternative materials under static and cyclic loads at ambient and low (77 K) temperature. Starting points were the insulation systems used by the suppliers of the ITER TF Model Coil (TFMC). These two systems of the DGEBA-type are very similar and have been successfully used by two companies for many years for many superconducting and conventional magnets. However, it turned out that both systems start to disintegrate after irradiation to the ITER design fluence at ambient temperature (~340 K) in the TRIGA reactor, Vienna. After consulting specialists of chemical and magnet industry, a program was set up in agreement with EFDA and ITER comprising about 10 systems of conventional and advanced insulation systems containing cyanate ester. Samples of these systems were prepared and part of them were irradiated and tested at 77 K in screening tests. Based on these tests, the most promising systems were selected for more detailed investigation comprising also fatigue tests. For economic reasons the cyanate ester content should be kept as low as possible due to the higher price compared to traditional epoxy resins. Therefore, the optimal percentage of the cyanate ester/epoxy blend is of great importance. This work presents initial results obtained on innovative cyanate ester/epoxy blends, which are alternative materials for the coil insulation of the ITER magnets. The mechanical strength was characterized under static load in tension and interlaminar shear at 77 K prior to and after irradiation, to a fast neutron fluence of 1×10^{22} m^{-2} (E>0.1 MeV). In order to simulate the pulsed operation of ITER, fatigue measurements were performed in the load and strain controlled mode as well.

1. Introduction

The magnet system is a key component of ITER. It has to withstand high thermal and pulsed loads without significant degradation in a radiation environment due to the fast neutrons from the D–T fusion process. Neutron irradiation primarily affects the magnet insulation, particularly the resin used to impregnate the combined glass-Kapton insulation. At the ITER design fluence of fast neutrons, i.e. 1×10^{22} m^{-2} (E>0.1 MeV) conventional DGEBA epoxy resins show a significant degradation of their integrity and mechanical strength. This was the reason for ITER to start R&D activities in this field quite early on. Many samples from suppliers in the U.S., Japan and Europe were investigated at the TRIGA reactor, Vienna, Austria, the FRM Munich, Germany, the IVV-2M reactor, Zarechny, Ekaterinburg, Russia, IPNS Argonne, USA, as well as in a pure ^{60}Co γ-radiation and a 2 MeV electron accelerator at JAERI, Takasaki, Japan [1]. The Atomic Institute of the Austrian Universities (ATI) overtook these investigations after having performed calibration and scaling experiments before these facilities became no longer accessible [2]. The mechanical properties (i.e. in tension and inter-laminar shear) of sample materials of magnet insulation systems and alternative materials were investigated under static and cyclic load at low temperature (77 K) before and after irradiation.

The insulation systems used by the suppliers of the ITER TF Model Coil (TFMC) served as a starting point for the more recent research programs. These two DGEBA-type systems are very similar and were successfully used for many years by two European companies for the fabrication of numerous superconducting and conventional magnets. However, they performed poorly after irradiation to the ITER design fluence at 5 K in the FRM Munich, Germany, and at ambient temperature (~340 K) in the TRIGA reactor, Vienna, Austria [3, 4]. Therefore, a research programme comprising more then 10 systems of conventional and advanced insulation systems was set up in agreement with EFDA and ITER after having consulted specialists of chemical and magnet industry [5]. In addition a large number of samples were irradiated and tested, which were provided without exact material description by companies or institutes in Japan and the U.S. [6]. Some important conclusions were drawn from these investigations, e. g. that some radiation resistant systems were not suitable for vacuum-pressure-impregnation or that cyanate ester (CE) had a very high radiation resistance [5, 6].

2. Technical Requirements on the Insulation System

The insulation system has to fulfil the requirements of the ITER magnet technical specifications concerning the mechanical and electrical strength over the whole expected ITER operation period, which includes also the radiation dose corresponding to the ITER design fluence of fast neutrons. On the other hand, the system has to be suitable for the impregnation of large volumes over long distances, i.e. the resin has to have a low viscosity (< 100 mPa·s) and a long pot-life of many hours. This had to be taken into account when selecting new candidate resin systems. The filler materials foreseen for the ITER insulation is a combination of glass fabrics and polyimide tapes, which are applied in a sandwich-like manner. The glass fabric has to be made of boron-free glass fibres, because boron has a high neutron cross-section, which would not be acceptable in a neutron environment like in ITER or the irradiation facility of the TRIGA reactor at ATI.

3. Test Equipment and Characterization Methods.

Static and dynamic tests are 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 the DIN 53455 and the ASTM D638 standards. The inter-laminar shear strength (ILSS) was assessed by the short-beam-shear (SBS) test according to the ASTM D2344 standard. Span-to-thickness ratios of 4:1 and 5:1 were used. For the simulation of the pulsed ITER operation, tension-tension fatigue measurements (ASTM D 3479) were done at a frequency of 10 Hz in the load and strain controlled mode up to 10^6 cycles at R ratios of 0.1 (load controlled) and 0.3 (strain controlled) [7]. For each data point 4 or more samples were measured. Double lap shear samples were used to assess the interlaminar shear strength under fatigue load [8].

The test geometries were analyzed by FE calculations leading to correction factors for obtaining the real shear strength values [9]. Another analysis was made to describe the combined shear-tensile/compression behavior of laminated insulation compounds [10].

Because of the build-up and the manufacturing process the materials have anisotropic properties. Therefore, test specimens were cut parallel (0°) and perpendicular (90°) to the wrapping direction of the reinforcing glass fiber tapes. For tensile tests mostly the 90° direction was investigated, because the UTS in 0° direction is mainly dominated by the strength of the glass fibers, whereas the 90° samples better characterize the integrity of the compound.

4. Insulation systems and sample preparation

The sample materials were produced with boron-free glass-fibre tapes and Kapton foil wound as sandwich in several layers half overlapped on steel plates. These plates were vacuum pressure impregnated with the resins according to specific prescriptions shown in table 1 and subsequently pressed to an insulation thickness of 4 mm before the curing cycle.

TABLE 1: RESIN SYSTEMS (SUPPLIER OF ALL COMPONENTS: HUNTSMAN)

Test No. Resin type	Component 1	Component 2	Additives	Viscosity Curing cycle	Remarks
TFMC 1 DGEBA	Araldite F	HY905		< 80 mPa s at 80°C 100 -135°C, ~ 12 h	Used by Ansaldo Superconductors
TFMC 2 DGEBA	MY745	HY905	DY073	< 80 mPa s at 80°C 90 – 105°C, ~ 12 h	Used by Alstom
T1 CE (cyanate ester)	AroCy L10		Mn-AANP*	< 80 mPa s at 40°C 80 – 160°C, ~ 12 h	Proposed by Huntsman
T1 CE (cyanate ester)	AroCy L10		Mn-AANP*	< 80 mPa s at 40°C 80 – 160°C, ~ 12 h	Sample without Kapton
T2 DGEBA + CE	PY306 (60%)	AroCy L10 (40%)	Mn-AANP*	~100 mPa s at 40°C 80 – 160°C, ~ 12 h	Proposed by Huntsman
T3 DGEBA	MY790-1	HY1102		< 80 mPa s at 80°C 120 + 140°C, ~ 12 h	Proposed by Huntsman
T4 DGEBA filled	CW229	HW229	Filled with Wolastonit	2000 mPa s at 60°C 110 – 140°C, ~ 12 h	Proposed by Huntsman as filler
T5 DGEBA	MY790-1	HY5200		~500 mPa s at 40°C 110 – 140°C, ~ 12 h	Proposed in report of Oxford Instrum.
T6 'Orlitherm' DGEBA	LY1025/CH	HY906	OH44 from Alstom/CH	< 70 mPa s at 80°C 100 – 130°C, ~ 12 h	Used by ABB/BNG for W7-X coils
T7 DGEBA	MY790-1	HY1102		< 80 mPa s at 80°C 120 °C, ~ 12 h	T3 not fully cured
T8 DGEBA + CE	PY306 (70%)	AroCy L10 (30%)	Mn-AANP*	<100 mPa s at 70°C 80 – 160°C, ~ 12 h	Ordered by ATI For blend optimisation
T9 DGEBA + CE	T6 'Orlitherm' (70%)	AroCy L10 (30%)	Mn-AANP*	<100 mPa s at 70°C 80 – 160°C, ~ 12 h	Blend of AroCy with best DGEBA
T10 DGEBA + CE	PY306 (80%)	AroCy L10 (20%)	Mn-AANP*	<100 mPa s at 70°C 80 – 160°C, ~ 12 h	Ordered by ATI For blend optimisation
T11 DGEBA + CE	T6 'Orlitherm' (80%)	AroCy L10 (20%)	Mn-AANP*	<100 mPa s at 70°C 80 – 160°C, ~ 12 h	Blend of AroCy with best DGEBA
			* Mn-acetyl acetonate in nonylphenol		

All sample materials except for the two TFMC materials were produced by Marti Supratec (CH). The same materials and the same build up were used as far as possible. In the beginning, some samples were produced using remaining glass fabric tapes from the ITER TF model coil manufacture. For the later sample production, new tapes had to be ordered, which were different in thickness resulting in a reduced number of layers.

5. Results of static screening tests

Not all the materials listed in table 1 will be discussed in the following. The results of the most relevant tests are compiled in FIG.1, where the ultimate tensile strength perpendicular to the wrapping direction (UTS90) is summarized, and in FIG.2, which shows the results of the short beam shear tests parallel to the wrapping direction (ILSS0). We show here the relative values before and after neutron irradiation to $1x10^{22}$ m^{-2}. Some variations in the initial strength can be explained by the different build-up mentioned above. Therefore, the attention should be primarily paid to the relative change due to irradiation. The results can be summarized as follows:

(a) The DGEBA based insulation systems show a significant, some of them a dramatic degradation of their mechanical properties after irradiation to the ITER design fluence. While the best system retains a reasonable mechanical strength, other systems (e.g. those used for TFMC) were close to disintegration [5].

(b) Samples impregnated with pure CE do not show any measurable degradation [5, 6]. However, this material is quite expensive and delicate in its application due to possible exothermic reactions. It also turned out that the bonding to the Kapton surface is less good than with the systems under (a) and (c).
(c) Following a suggestion by the resin supplier, one series of samples was impregnated with a blend of CE with DGEBA resins in the proportion 40:60. In order to find the limits of such blended systems, two further systems were added by ATI with 30:70 and 20:80 mixtures. It turns out that these blended systems also do not show significant degradation after irradiation [8]. Their application is less delicate than that of pure CE. The bonding of the CE/DGEBA blends to Kapton is excellent. As the price of the blended systems becomes reasonable, they are interesting and very promising candidates for the ITER magnet system.

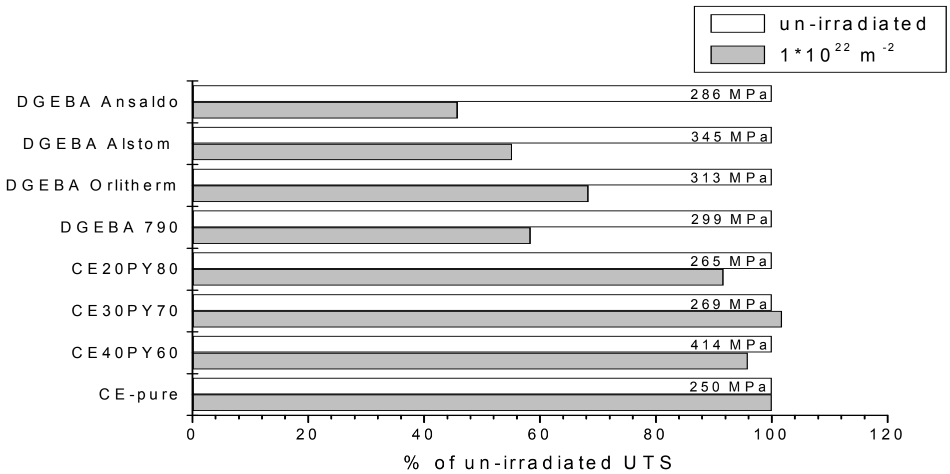

FIG.1: Ultimate tensile strength (UTS90) in 90° direction (i.e. perpendicular to the wrapping direction). Only one of the DGEBA resin systems (Orlitherm) keeps a reasonable strength after exposure to the ITER design fluence, but all the CE based systems (pure and blends with DGEBA) show no or almost no degradation.

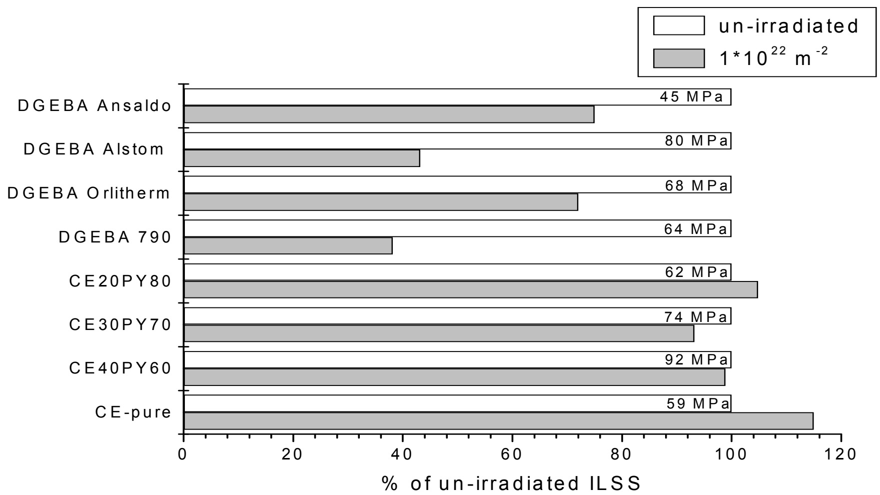

FIG.2: The inter-laminar shear strength (short beam shear test – SBS) in 0° direction (i.e. parallel to the wrapping direction) confirms the results of FIG.1. The DGEBA resin system of Ansaldo seems to degrade less, but starts already at very low values due to an out gassing glue used in their system. The Orlitherm system still keeps a reasonable strength after exposure to the ITER design fluence, while all the CE systems (pure and blends with DGEBA) show no or almost no degradation.

6. Fatigue tests in load and strain controlled modes

The fatigue tests were done originally in the load controlled mode, which is easier to perform in the servo-hydraulic MTS 810 testing device. However, the deformations and consequently the stresses in the insulation of the ITER TF coils are imposed by the deformations of the steel case and the radial plates as well as the conductor. The stainless steels proposed for the TF steel case and radial plates have 0.2% yield strength of about 1200 MPa. To load the steel to this level, one has to apply a strain of about 0.8% (FIG.3). The much weaker insulation compound has to follow the deformations of the steel. Therefore, it was proposed by EFDA/ITER to investigate the fatigue behaviour in the strain controlled mode. FIG.4 shows the results of stress and strain controlled tests with a minimum to peak stress or strain ratio R of 0.5. The data show that both modes lead to very similar results [7], at least in the ITER relevant range up to ~ 10^5 cycles.

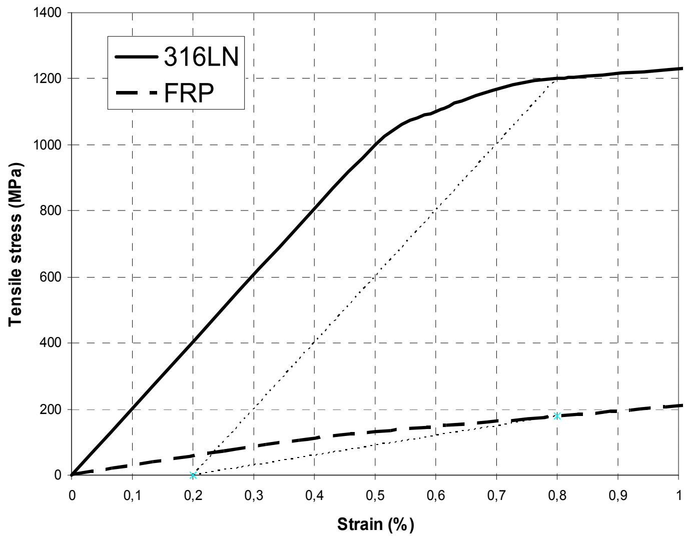

FIG.3 Typical stress strain diagrams of AISI 316LN steel and of an insulation material (FRP). To stress the steel to the 0.2% yield strength, a strain of about 0.8% has to be applied. As the insulation has to follow the steel it would be exposed to the same strain.

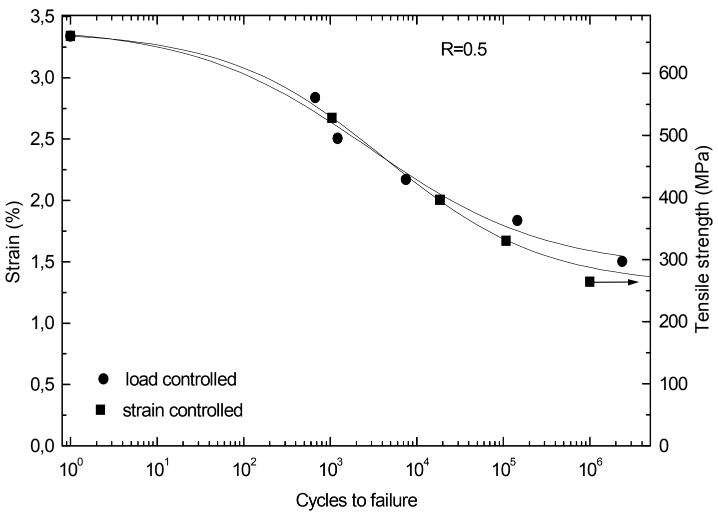

FIG.4 The load and strain controlled tensile tests on a standard insulation material executed with a minimum to peak stress or strain ratio R of 0.5 and 0.3 deliver very similar results [7], at least in the ITER relevant range up to ~ 10^5 cycles.

7. Characterisation of the systems under cyclic load

FIG's 5 to 7 show the stress-lifetime diagrams of the DGEBA samples TFMC1 (FIG.5), T6 (FIG.6) and the CE systems T2 (FIG.7) measured in the load controlled tension-tension mode in the 90° direction [8]. The shape of the curves before irradiation is similar for all systems. A rapid decrease is observed between 0.8 and 0.4 $\sigma_{max.}$ A pronounced life endurance limit is found at 0.2 and 0.35 σ_{max} depending on the insulation system.

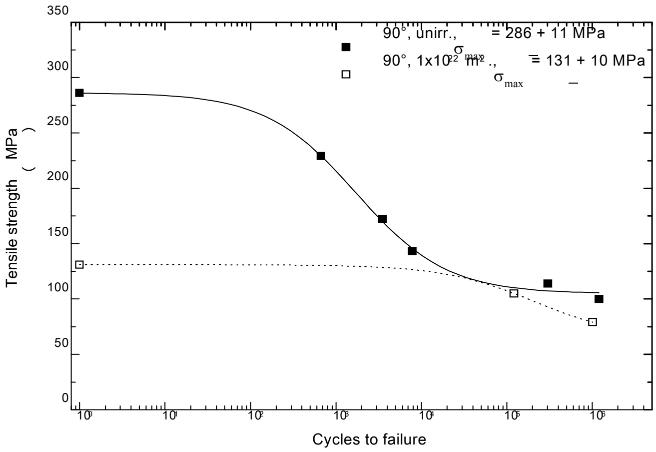

FIG.5 The tensile fatigue behaviour of the DGEBA system used for the ITER TF Model Coil (TFMC1) shows a dramatic drop after irradiation to less than 35% and disintegrates during the fatigue tests. The second system TFMC2 shows a similar behaviour. Therefore these, systems are not suitable for use in an ITER environment.

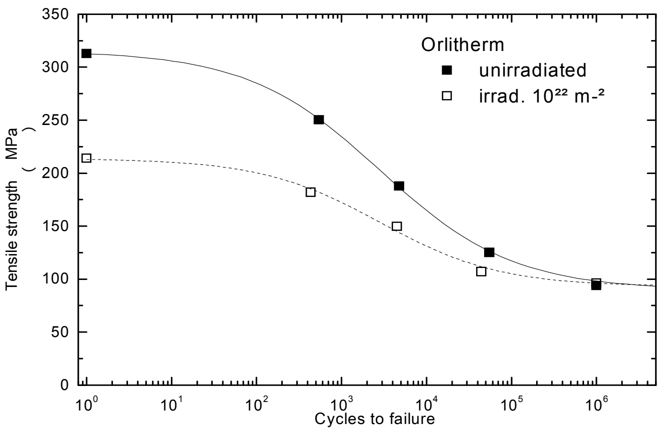

FIG.6 In the most radiation resistant industrial DGEBA system (T6) the tensile strength drops to about 70% after irradiation, while the fatigue limit remains unchanged.
This system, which is used for part of the W7-X coils, could just fulfil the ITER requirements. If industry would like to use other systems, those had to be qualified on test samples after irradiation as described in this paper.

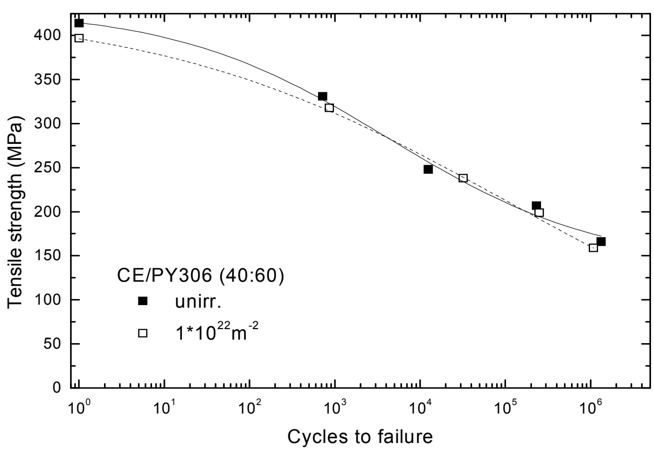

Fig. 7 The equivalent test on the CE/DGEBA (40:60) blend (T2) shows that this system remains nearly unaffected by irradiation.
The other CE based systems (T1, T8, and T10) show a similar behaviour at a slightly lower level.
The samples impregnated with pure CE behave less good, as they show signs of bad bonding to the Kapton tapes.

After irradiation up to the ITER design fluence

- the industrial DGEBA system TFMC1 shows a dramatic degradation and disintegration during cycling resulting in a deformed Wöhler curve.
- the most radiation resistant DGEBA system (T6) shows much less, but still significant degradation reaching practically the same fatigue limit after 10^6 cycles.
- the four CE based systems are nearly unaffected by irradiation.

8. Extrapolated mechanical behaviour of insulation systems in all directions

According Pahr et al. [10] the shear-compression strength limits of insulation laminates can be calculated over the whole range of combined loads. Using the points available from previous [11, 12, 13] and recent measurements in a compressive/tensile vs. shear strength plot the behaviour shown in FIG.8 can be extrapolated. The area inside the stress limits shrinks for DGEBA systems significantly after exposure to the ITER design fluence, while all CE based systems are expected to keep their mechanical strength. The graph shows also that the insulation systems can overtake quite high shear compressive loads, while they cannot overtake significant tensile loads perpendicular to the insulation layers. Such loads have to be avoided by using de-bonding agents in such regions. The ITER acceptance limits should be revised accordingly.

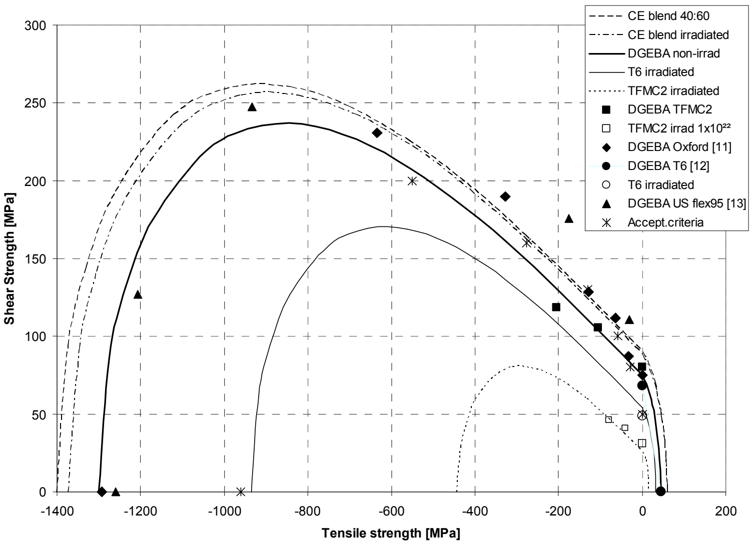

FIG.8 The real loads on the insulation system in the ITER TF coils are combinations of shear and compressive or very low tensile stresses. Pahr et al. showed that the strength of the insulation can be plotted as shown in the figure. Data measured on samples of the ITER TFMC insulation show a significant shrinkage of the useful area after exposure to the ITER design fluence. Nearly no change is expected in case of the CE systems.

9. Conclusions and suggestions

Only one of the DGEBA materials investigated so far keeps a reasonable strength after irradiation, but shows nevertheless significant degradation. The pure CE system presents difficulties in magnet production, does not show very good bonding to Kapton and is expensive. Blends of DGEBA resin PY306 and the CE AroCy L10 are applicable almost in the same way as DGEBA resins and become reasonable in price due to the blending. They show excellent mechanical properties with almost no change after irradiation to the ITER design fluence. It is, therefore, recommended to continue the investigations to complete the data base and to develop and qualify the application of these materials further based on large size mock-ups fabricated by industry. The goal should be to create a full specification of materials and the application procedure to the ITER TF coil insulation, which can then be overtaken by industry.

10. Acknowledgements

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