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CARE-NED Work Package 4 EDMS815074

Insulation Development – Conventional Insulation

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1. ACKNOWLEDGEMENTS

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Contents

1. Introduction

This report represents a deliverable under Work Package 4 of the CARE-NED programme, Insulation Development.

A programme of materials technology work has been carried out in order to economically address the particular problems of insulation materials for niobium-tin, wind and react accelerator magnets. While we were unable to manufacture model magnets due to budget constraints, the programme has nevertherless been able to add to the body of knowledge by strategically targeting particular problem areas.

We have approached the problem by performing screening tests on candidate materials. The three test methods chosen are:

-Electrical breakdown

-Short beam shear

-Interlaminar fracture toughness

An extensive literature survey was performed.

In addition we have used thermo-gravimetric analysis to measure the very small weight loss arising from degradation of organic fibre coatings in the same environment as niobium-tin magnet insulation experiences during the reaction cycle.

We have addressed the particular problem of industrialising the manufacturing process of niobium tin magnets. Niobium tin magnets are currently produced using labour-intensive methods. Such magnets are relatively small and are usually solenoids. Scaling this technology to produce 10 metre long accelerator-quality, high field dipole magnets represents a number of challenges.

The challenge for this work package is to:

- produce an insulation system that can be applied in the industrial coil manufacturing setting,

- is compatible with the heat treatment,

- meets the agreed Insulation Specification.

-Develop test techniques and samples which allow quantitative comparison of insulation mechanical properties

2. Insulation Specification

Appendix 1 is the Insulation Specification agreed between the project collaborators.

3. Insulation review

A review of niobium-tin magnet insulation was presented at the 2005 WAMS meeting at CERN, see Appendix 2.

4. Fibre type

For wind-and-react niobium tin applications where the glass-fibre taped cables are exposed to the reaction temperatures, the fibre has to survive superconductor heat treatment temperatures with little change in properties.

The most common glass fibre, E-glass, contains boron oxides which act as a flux and reduce melting temperature of the glass. For general purpose electrical insulation the advantage is reduced material cost. For this application a major disadvantage is that boron reacts with thermal neutrons which results in fibre-matrix disbonds (1). S-glass contains no boron oxides so has a higher melting temperature, making it a more expensive product.

Quartz fibre is an alternative material (2), however it can be difficult to weave and it is not easily available. The extra cost of quartz has not yet been justified so S-glass remains the fibre of choice.

Since the insulation system takes up space in the magnet and so reduces current density, it is desirable to make the insulation thin, so long as it is reliable. This requires the use of thin fibre tapes. The NED Insulation Specification calls for 0.4 mm total insulation thickness, this means nominally 0.1 mm thick tape. The issue of tape thickness is complicated by the applied pressure when the thickness is measured since the fibres within the tape will pack together. In any case such a thin tape is not easy to source in s-glass fibres.

JPS Composites (3) have woven suitably thin S-glass fibre tapes for this project.

The sequence of weaving and sizing is critical to success of niobium-tin insulation. To enable weaving, more than 1% sizing is required to lubricate the fibres and keep fibre damage to a minimum. If left in place, this sizing degrades during heat treatment and compromises electrical properties.

Conventionally the sizing is removed after weaving and the desized tape is wrapped around the cable. Because the tape has no lubrication or protection it tends to tear.

4.1 Fibre sizing

Glass fibres are coated with a mixture of organic materials immediately after fibre drawing. This is known as a “sizing”. The sizing can perform a number of functions; lubricant and protection for glass fibre to facilitate weaving, to enable the matrix material to wet out the fibre during impregnation, functional groups for chemical bonding from the inorganic fibre to organic matrix.

Sizing formulations are generally proprietary and can vary depending on final application. Formulations are proprietary to manufacturers. Sizing is important to

Nb_3Sn processing because most sizing materials are organic and will degrade to some degree during the heat treatment. This can result in carbon residues on the glass surface, which reduces electrical breakdown strength. The carbon colours the composite black when it is wetted with epoxy resin.

Figure 1 illustrates this effect. The darkest coloured composite at the back of the Figure contains carbon residue after a heat treatment at 660 ºC in vacuum. Both the front and middle composite meet the NED electrical breakdown specification.

The grey coloured composite (middle) contained up to 0.4 % of a novel high temperature sizing and has also been heat treated at 660 ºC in vacuum.

The white coloured composite (front) had no heat treatment and provides a reference.

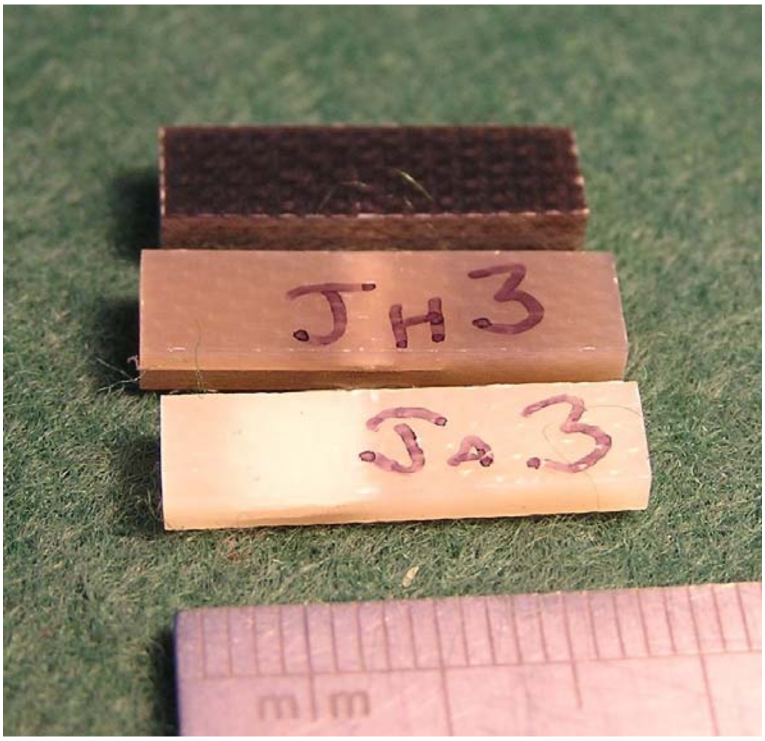


Figure 1 Illustrating contamination due to carbon residue from sizing decomposition. The laminates are short-beam-shear samples, after testing.

Thermo-gravimetric analysis, TGA, has been used to characterise the behaviour of the sizing at temperatures in two gas environments, oxidising and inert.

To determine the total amount of organics present, a sample is heated in air. The weight change is recorded To determine the effect of the niobium-tin reaction cycle, a sample is heated in argon gas.

The Perkin Elmer TGA “Pyris 1” instrument is designed to have high sensitivity to weight changes with time and/or temperature. The sample is suspended in a furnace in a platinum crucible. The balance mechanism and sample furnace are sealed and purged with a gas. The furnace is heated at 40 ºC per minute to 700 ºC. The weight and temperature are recorded. Typically 10 mg samples were used and weight changes were of the order of 0.1 mg.

Figure 2 shows an example result. It shows thermal degradation of a conventional sizing. Most of the weight loss occurs between 300 ºC and 400 ºC. The test method is under development. A critical part of setting up the machine is obtaining a flat baseline so that buoyancy and other effects are negated. Then one can be confident that apparent weight changes are not machine artefacts.

This test will be useful for Quality Assurance for batches of glass fibre tape in a production process.

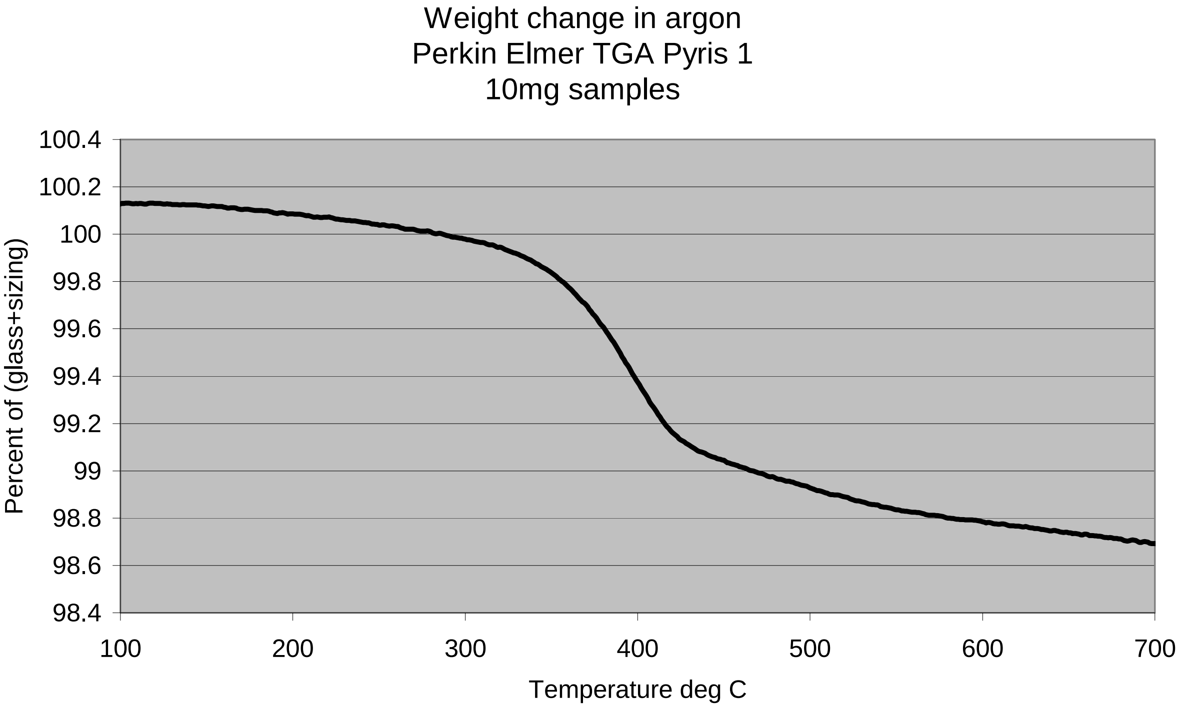


Figure 2, Weight loss vs temperature for a conventional sizing

JPS Composites have supplied a number of samples to the programme, in S-glass and quartz. They specialise in high temperature sizing treatments. Because some lubrication is required to weave glass fibres, the order of sizing, heat cleaning and weaving is critical.

The first sample provided by JPS, named “S-glass/PI #37”, was from a 1 yard wide roll of satin weave fabric. Although this is unsuitable for insulating a cable, where one would typically use 12 mm wide tape, it was suitable for producing a laminate for mechanical and electrical testing. Its light grey colour suggested that although it contained some carbon residue it was not enough to degrade electrical properties.

Transferring the manufacturing process of this material to a thin tape is now taking place. Material was not available to test in time for the NED project.

This technology promises to be very useful for insulation of react-and-wind niobium tin magnets.

5. Matrix materials

To bond the insulation fibre and cable into a monolith of accurate dimensions, a polymer matrix is used. Historically this has been an epoxy resin which is introduced as a liquid by a vacuum assisted technique. The intention is to fill all the voids in the mould.

The factors affecting choice of matrix material are:

- Viscosity (initial) and viscosity/time characteristics to allow sufficient time for the magnet mould to fill
- Radiation hardness – influenced by the chemistry of the matrix(4).
- Toughness –A tough matrix is desirable to avoid cracking
- Electrical properties – all polymeric matrices are likely to have excellent electrical insulation properties
- Thermal conductivity – likely to be poor, and not vary greatly between candidates
- Thermal expansion – likely to be high, and not vary greatly between candidates

For a detailed consideration of epoxy resins for vacuum impregnation purposes, see (5).

Alternative candidates

Tetra-functional epoxies

Evans has shown (6) that epoxies with functionalities of 3 or 4 are more radiation stable. An epoxy such as TGDM (tetraglycidyl DiaminoDiphenylMethane) such as Ciba MY722, is an alternative to epoxies with lower functionalities. This material was included in the test programme.

Cyanate Ester

The fusion community has studied the potential of cyanate ester materials, which show promise as radiation hard matrices. However their high cost, up to ten times that of epoxies, has led the community to test a blend of cyanate ester and epoxy (7).

Tooling is being developed to enable cyanate ester materials to be processed. Compared to most epoxies, closer control of the cure process, in particular to avoid hazards associated with exotherm, is required.

BMI

Chichili et al (8) studied Matrimid, a bismaelimide polymer, as a potential matrix. He concluded that the material required closer control of the mixing and impregnation process compared to the control material, an epoxy.

6 Radiation

The NED project is aimed at producing a high-field magnet design, ultimately for an LHC upgrade. The dipole magnetic field deflects charged particles into the midplane of the magnet. In a cos-theta magnet design, many coil turns are located near the midplane and so the magnet insulation experiences a very high power deposition. Mokhov et al (9) used the MARS 14 code to model the problem and calculated a peak power density of 13mW/g in the superconducting coils. Compared to the baseline LHC quadrupoles, this is more than 20 times the peak power density.

Mokhov considered the heat load and radiation dose in a block coil. The block coil design places the coils further from the beam than a cos-theta design. Even so the doses are challenging, 20 to 50 MGy over 10 years. He suggested that a cos-theta dose would be ten times higher (10).

Open-midplane designs have been proposed by Gupta et al (11). The major advantage of the open midplane approach is the reduced radiation dose and heat load and hence cooling requirements.

The power dissipation in the winding is important because it can limit magnet performance due to cooling limits. The radiation dose is potentially damaging to insulation materials. Sensitive materials are the epoxy matrix materials. Large doses may degrade the mechanical properties and lead to magnet degradation.

A number of variables influence the radiation dose including the optics layout (dipole first, etc), beam screens, and magnet design.

Most radiation studies of materials have been performed in fission reactors with a predominantly gamma dose. The available information on irradiation by other species, particularly energetic neutrons, (12) suggest that linear extrapolations would be invalid. Therefore irradiation to the full dose with expected species is important.

Radiation studies were removed from the NED programme initially due to lack of funding.

The target station at the ISIS spallation neutron source at CCLRC has a mix of gamma and neutron species. An opportunistic attempt has been made to irradiate a range of NED candidate composite materials, epoxy resins with S-glass fibre, in the target station. However activation of the samples and a lack of active handling equipment has prevented assessment of them. At present we do not know why the samples were so highly activated.

There remains an urgent need to compare the performance of available matrix materials since the doses that a NED magnet will see in service are at or beyond the expected limit of today’s materials.

Ideally irradiation would take place at low temperatures to freeze in evolved gases. Currently there is no facility that provides the correct combination of parameters so that the performance of any candidate materials can be confirmed.

Radiation-induced gas evolution remains a real concern for such high radiation doses. At low temperature the evolved gases are frozen into the matrix, but on warming, such as in a quench, the pressure of the gas represents a real danger.

Since ITER also has magnets exposed to high radiation doses, some groups have carried out some irradiation and testing of epoxies and alternative materials. The radiation has been carried out at 77 K in a fission reactor at ATI Vienna. This does not closely replicate the LHC upgrade conditions but may give an indication of relative performance of different materials. Bittner-Rohrhofer (13) reported that a blend of epoxy and cyanate ester improved interlaminar shear strength by a factor of 3 compared to a conventional epoxy after irradiation at 1x10^22 m2 neutron fluence in fission reactor radiation conditions.

7 Materials Testing

Most testing has been performed using a standard glass-fibre epoxy laminate of 3 mm thickness for mechanical tests and 0.4 mm thickness for electrical breakdown.

8.1 Electrical testing

Electrical breakdown testing to BS7831:1995 has been performed on thin laminates. A high voltage source of 12 kV was used. A laminate thickness of 0.4 mm was tested, the same as the total insulation specification between cables in a NED magnet design.

The test equipment was set to cut out at a predetermined current of up to 1 mA.

It was straightforward to detect the presence of carbon as only a few hundred volts was required to cause breakdown.

Results are presented in Appendix 3.

7.2 Mechanical testing

7.2.1 Short Beam Shear Testing

Short beam shear testing is a standard test applied to composites, ASTM D2344. Samples are easy to prepare and the test is very useful for assessing changes in material processing. Results are presented in Appendix 3.

7.2.2 Interlaminar fracture testing

The fracture properties of the insulation matrix are important because reliability of the magnet will partly depend on having a tough matrix that resists cracking. At low temperatures the heat capacity of all materials is so low that heat liberated by cracking could cause a quench. The test procedure in ASTM D5528 was followed.

See Appendix 3 for testing results of Interlaminar fracture and short beam shear.

Figure 3 shows a laminate being tested in interlaminar fracture. A starter crack is formed by a release film that was inserted during laminate manufacture. The testing machine pulls the test piece apart in a double cantilever arrangement. On crack propagation, a sudden load drop is seen and the crack extension and load are noted.

Figure 4 shows a different type of behaviour, the crack is not propagating through the epoxy resin but instead though the glass fibre. This may indicate some degradation of the glass fibre after reaction heat treatment.

Figure 3 Interlaminar fracture testing to ASTM5528 of a non-heat treated laminate

Figure 4 Interlaminar fracture test of a laminate contaminated with carbon from sizing decomposition, note the black colour and crack propagating through the glass fibre.

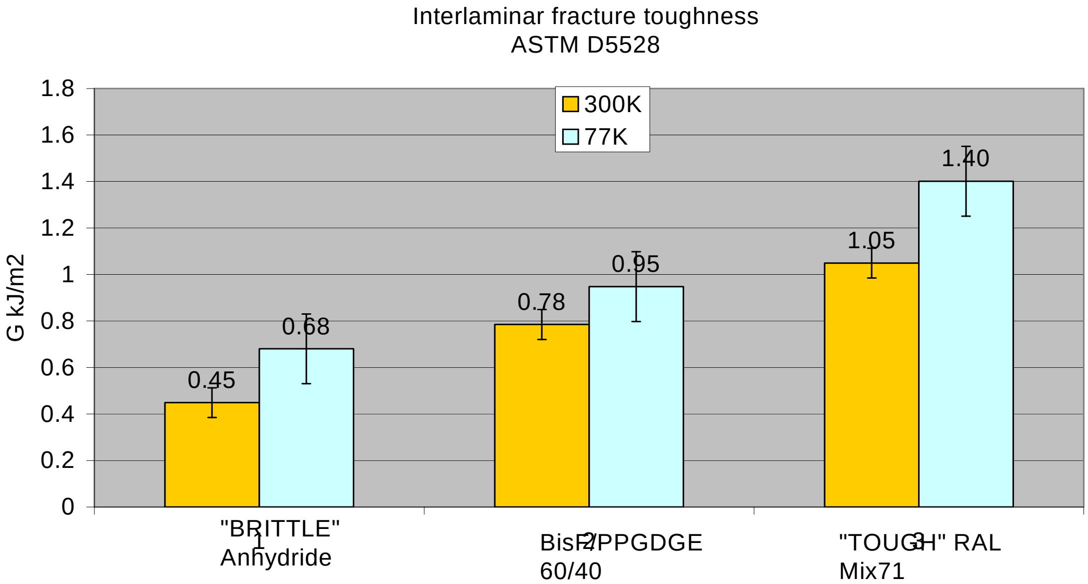


Figure 5a Results of Phase 1 Interlaminar testing, showing work of fracture at 300K and 77K for three epoxy resins

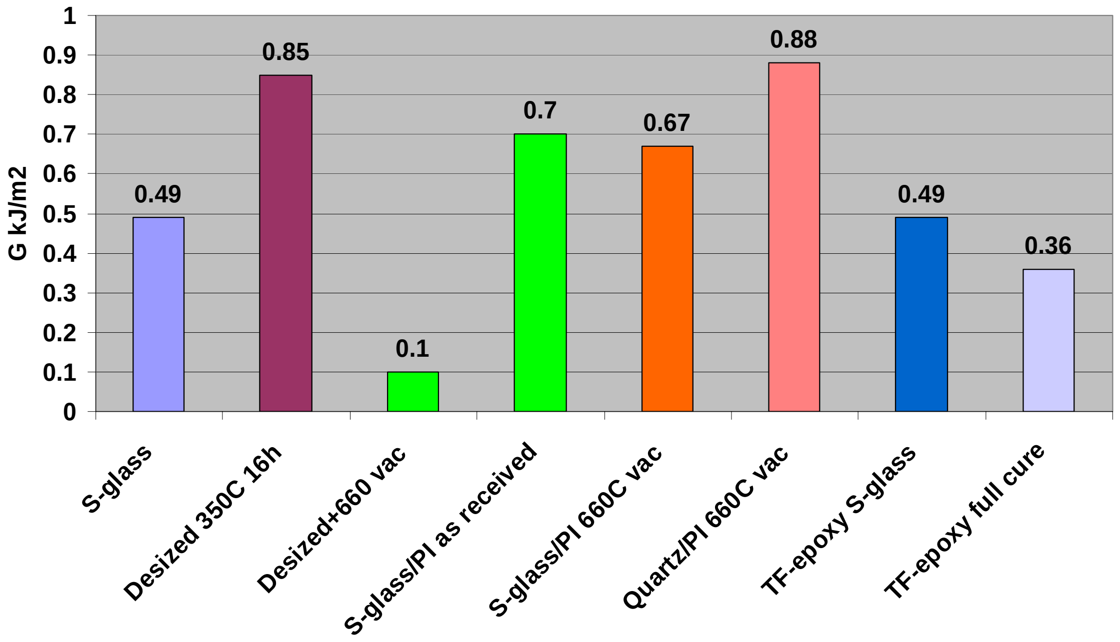


Figure 5b Results of interlaminar fracture testing showing work of fracture, G, on a range of specimens at 300 K.

Figure 5a shows the range of results obtained with 3 different epoxy resins and E-glass. These tests were carried out to gain experience of the test and are not meant to represent candidate insulation materials. Figure 5b shows effect of heat treatment on S-glass fibre laminates. Most data in figure 5b uses DGEBF/DETD epoxy. This was chosen as a representative radiation-hard epoxy. The S-glass sample gives a control. A desized sample gave a surprisingly high work of fracture given that there should be little bonding between the desized fibres and the epoxy. In any case this result is not directly relevant to the magnet case because, after reaction heat treatment the work of fracture is very low as shown by the third data point. The fourth and fifth columns labelled “S-glass/PI” refer to S glass fabric from JPS Composites with a small quantity of high temperature sizing. This performed very well both before and after heat treatment, showing little degradation. Both results are above the control S-glass result. The sixth column shows a quartz fabric with the same sizing as the S-glass/PI, which gave the highest result. Owing to shortage of material only a heat-treated result was possible. Quartz may be an attractive insulation fibre however it will be expensive and even more difficult to weave than S-glass.

The last two columns are for a different epoxy, TF stands for Tetra-Functional. This is Ciba MY722 epoxy with DETD hardener. This is believed to be better than other epoxies in terms of radiation hardness. However the very chemistry that lends radiation hardness can also lead to a brittle material, and this is borne out by the lower fracture toughness if the material is completely cured, i.e. to its maximum crosslink density.

Table 1: Formulations of epoxies and hardeners used

Resin system reference	Epoxy resin (pbw*) / example commercial designation	Hardener (pbw) / example commercial designation	Other component/ example commercial designation
RAL # 1	DGEBA (100) Huntsman MY750	MTHPA (85) Huntsman HY918	Accelerator (0.5) Huntsman DY073
RAL #230	DGEBF (100) Dow DER354P	DETD (21) Ethacure 100	PPGDGE (40) Dow DER732
RAL#71A	DGEBA (100) Huntsman MY750	POPDA (55) Vantico HY5922	None
RAL #227	DGEBF (100) DOW DER354P	DETD (26) Ethacure 100	None
TF	TGDM (100) Vantico MY722	DETD (35) Ethacure 100	none

Note: * = parts by weight

7.2.3 Cable stack tests

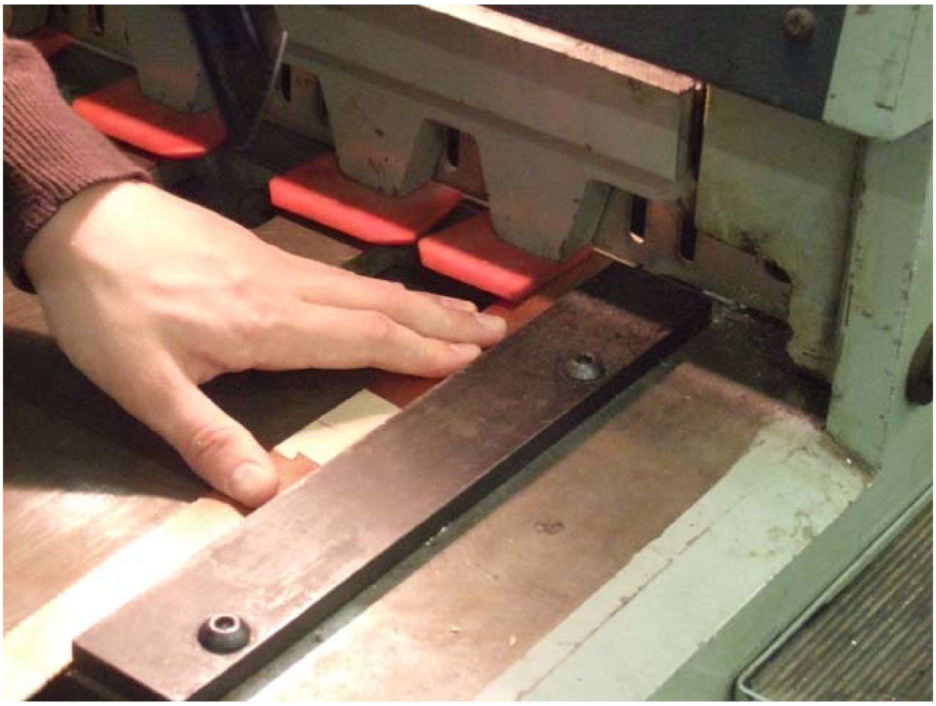
Insulated cable stack tests are a useful test vehicle as a step between testing glass fibre-epoxy laminates alone, and making and testing coils (14). This geometry has been used by Reytier to determine compression modulus and thermal contraction coefficients on stacks of ten cables. Here we intend to use a similar test to study short-beam-shear, and interlaminar fracture toughness, using a smaller number of cables.

The aim in these tests was to subject the insulation to preparation and stresses more representative of a real magnet matrix. This test procedure is under development.

Specimen preparation

An existing NbTi cable from the ATLAS project was used for the test as it was easily available. The cable is approximately 29 mm wide and 3 mm thick. These dimensions are similar to those for a full-size NED magnet. The cable was cut into approximately 200 mm lengths using an industrial guillotine. The cable was taped with PVC electrical tape around the cutting point prior to cutting. This helped the cable retain its shape, see figure 6.

Figure 6: Cutting of ATLAS cable using a guillotine



After cutting the cables were twisted, this made wrapping the cable in glass tape difficult and the wrapped cable would no longer fit in the mould tool. To remove some of the distortion the cable was annealed. Annealing tooling was produced; this allows up to 8 lengths of cables to be annealed at once. The tooling, shown in figure 7, consists of three plates of Stainless steel between which the cable is placed. The cable is then compressed by tightening the bolts that run through the plates. A simple handle was added for ease of handling. The cable was annealed for 330 °C for 4 hours in a vacuum furnace.

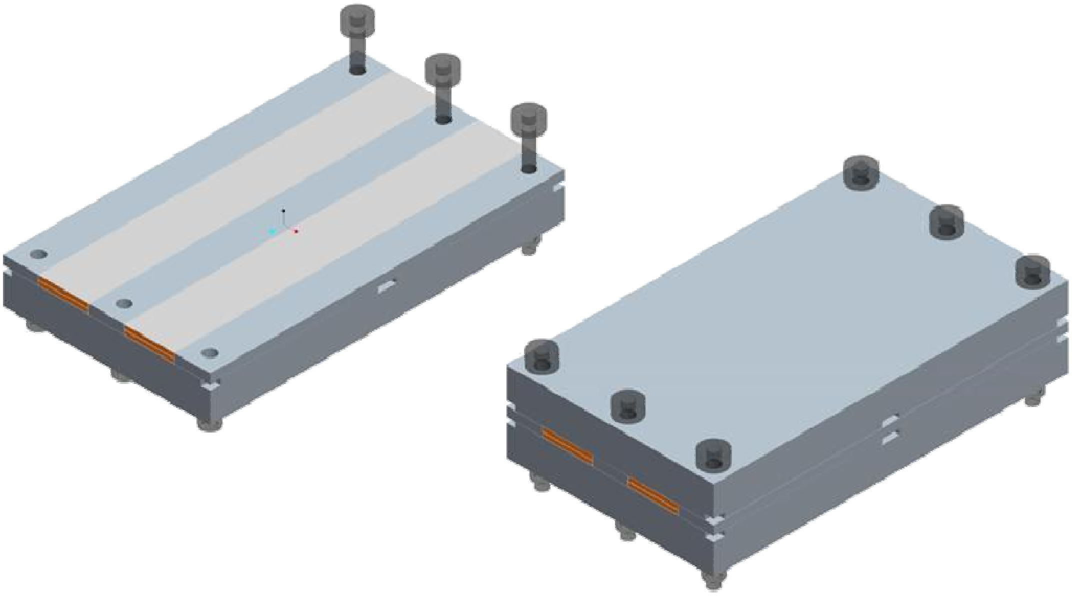
Figure 7: Cable annealing tool

After annealing the cable was lightly cleaned using PFSR solvent and from then onwards was only handled using clean nitrile rubber gloves. To provide baseline data and to easily prove the tooling, this initial test was not heat treated at the niobium-tin reaction temperature. The cable was wrapped in a half-lap pattern with Hiltex conventionally-sized S-glass tape. Superglue was used to hold the tape in place whilst wrapping. This is shown in figures 8 a &b.

Figure 8 a & b: Wrapping of Atlas Cable with tape

After wrapping the cables were assembled into the tool, see figure 9. A small piece of release film was placed between one set of cables, this provided a crack initiator for fracture testing. As no heat treatment took place conventional release film was included between the tool and wrapped cables to help extract the samples from the mould. The outer tool was treated with a wax release agent.

Figure 9: Wrapped cables in Stack test mould



The assembled tool was vacuum impregnated using RAL mix 71 A, see Table 1. This is one of the toughest resins used in magnet production and would provide a benchmark. This resin gels at 40°C and is then fully cured by heating to 80°C for 2 hours. After curing the samples were extracted from tool. This was fairly straightforward because of the release treatments used on the tooling; it could be more problematic on a mould that experiences 660°C. Boron nitride release agent would be necessary for high temperatures.

Testing

Short beam shear

The samples were tested in short beam shear in liquid nitrogen following ASTM2344. The samples were cut to nominal size using an abrasive wheel. The sample geometry in ASTM is length 6 x thickness, span 4 x thickness and width 2 x thickness. The thickness was ~5.5mm. The samples were tested in three point bend, using the set up shown in figure 10, immersed in liquid nitrogen. The results from the short beam shear test are shown in figure 11.

Figure 10: Short beam shear set up

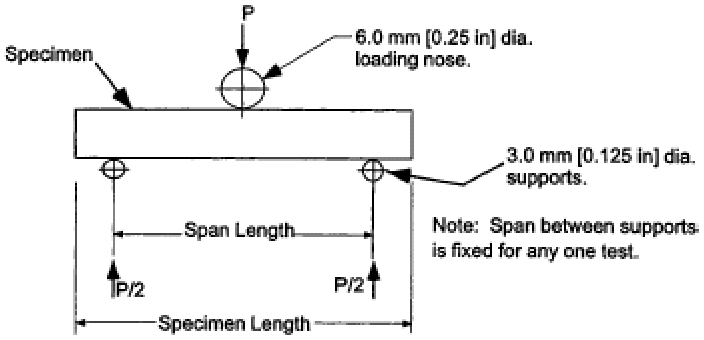
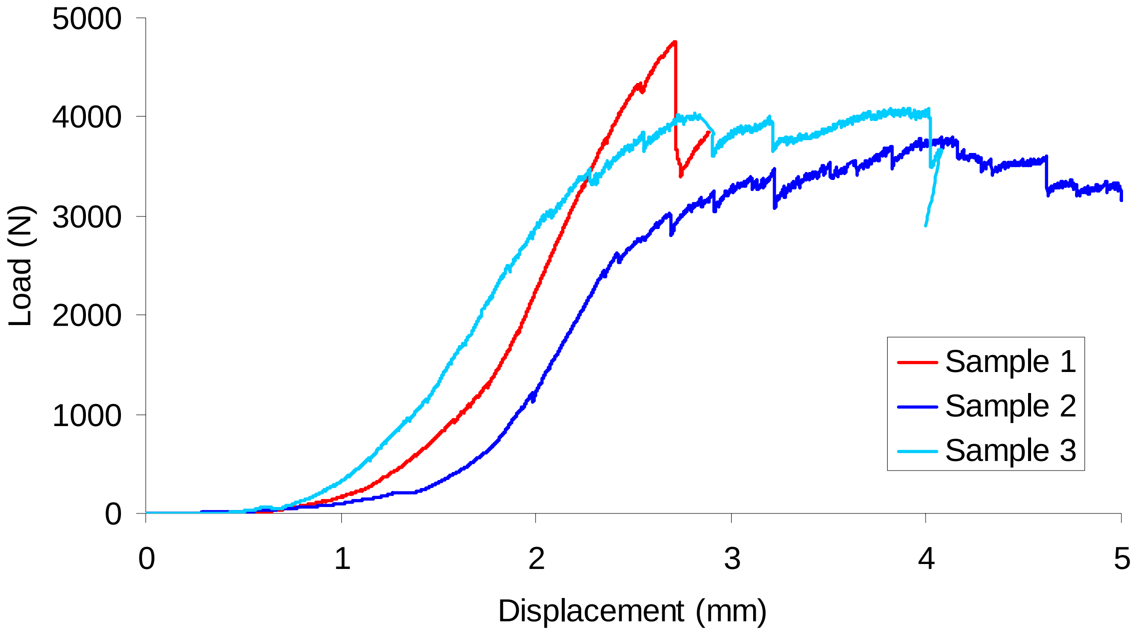
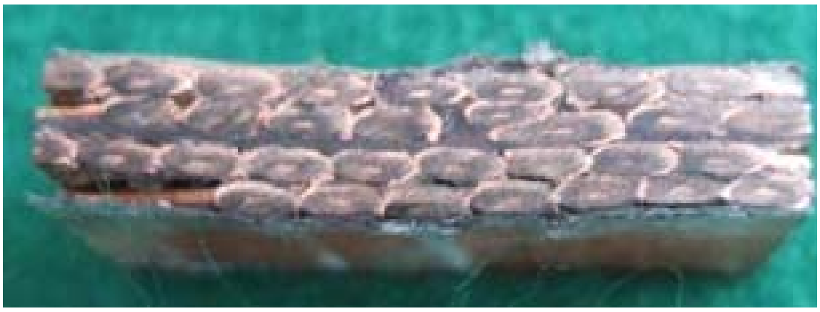


Figure 11: Load vs displacement from short beam shear testing. The 3 specimens are nominally the same.



As the load was applied during test, cracking sounds were made by the samples. These noises were accompanied by small drops in load. The samples failed in shear and there appeared to be delamination between the layers of copper cable within the samples. The bond between the copper and the glass appeared to be poor. Samples after testing are shown in figure 12.

Figure 12: Sample after shear testing. Note how poor the bond appears between the insulation and cable



Work of Fracture

An interlaminar fracture test was performed on one cable sample. The test is based on ASTM D5528. At this stage is is unclear whether absolute results are valid because the test would normally use an all-composite specimen. However the mode of failure provides useful information and the test may be useful for ranking insulation systems and changes to the process.

For example, the stack tests have shown that the bond to the copper cable could be improved.

8 Recommended insulation system for short racetrack coil.

The next stage of development in the NED programme is to make short racetrack coils as a vehicle for testing conductor and different insulation schemes.

There is a key difference between the choice of matrix material for a racetrack model magnet, a 1 metre long “Fresca test facility upgrade” dipole and that for a LHC-upgrade dipole. That is, radiation hardness.

8.1 Matrix

The test magnets will not be exposed to a high radiation dose, therefore a established, tough epoxy widely used for cryogenic applications can be chosen such as RAL # 71A, see Table 1. The only disadvantage of such a system is the relatively short pot life compared to anhydride-cured epoxies. The impregnation tool needs to be carefully designed to allow good access for the resin. The tool should be qualified with a low cost “dummy coil” prior to committing a reacted niobium-tin magnet pole.

8.2 Fibreglass tape

The S-glass fibreglass tape chosen for the model magnets is an S-glass tape from JPS Composites, woven with 18 ends and 26 double picks with a small amount of high temperature sizing. The manufacturing process for such a material is being set up as of early 2007, see section 4.1.

9 References

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Appendices

Appendix 1 NED Insulation Specification

Appendix 2 Nb3Sn Accelerator Magnets Insulation Review

Appendix 3 Insulation Development for the Next European Dipole

Appendix 4 Vacuum impregnation of compacted glass fabric Canfer S. J., Ellwood, G E.

Appendix 1: Insulation Specification

CARE-NED-IDI-Document RAL-001	**NED Insulation Specification**	**Version 2.4**
Simon Canfer, CCLRC	**16 July 2004**	**EDMS 548037 v.6**

Introduction

This specification applies to insulation for the Next European Dipole (NED) design. The dipole may be of cos-theta, or other winding design configuration.

The dipole will measure up to 10m in length. It is intended that the design should be compatible with industrial production, ultimately for production in hundreds of units for application in a particle accelerator. The first application of such a dipole is likely to be for an LHC Interaction Region Upgrade. Radiation loads are taken from such an application as they are likely to represent the worst-case.

The NED design is based on Nb_3Sn superconductor in a wind and react scheme. That is, the insulation system will be heat-treated together with the superconductor. The background to the specification is given in Appendix 2 - Nb_3Sn Accelerator Magnets – Insulation Review .

There are two insulation schemes under consideration:

“Conventional” which includes vacuum impregnation of inorganic fibres with an organic material, for example S-glass/epoxy.

“Innovative” which includes novel schemes such as inorganic fibres with an inorganic matrix. This may be followed by impregnation with an organic material. An inorganic scheme would have superior radiation tolerance.

The Insulation Work Package for NED will encompass specification and testing of insulation materials. This report defines the basic insulation specification developed in consultation with members of the NED collaborators. Some refinement of the specification may be made as the magnet design proceeds. Explanatory notes are given in Appendix 1.

The specification will form the basis of the testing plan currently under preparation. Some tests will be carried out before insulation heat treatment and some after heat treatment. The majority of tests will be carried out on the final product, after impregnation (if applicable) and again after irradiation. The testing plan will detail test methods.

SPECIFICATION

1	**GENERAL**	**Design**	**Failure**
1.1	Insulation thickness per cable	0.2mm	
1.2.1	Winding compatibility: Capable of being applied to the cable and formed into a dipole winding by a semi-automatic winding system	Minimal fraying or abrasion during winding	
1.2.2	Conductor bend radius minimum	20mm	
1.3	Compatible with Nb3Sn heat treatment cycle	Minimal degradation of basic components	
1.4	Thermal cycles to low temperature: 300K – 4.2K	10	50

1.5	Running cycles: ramp to max compressive stress	100	500
1.6	For conventional organic insulation scheme and innovative scheme if applicable: ability to be impregnated with a liquid of viscosity 200 mPa.s	200mPa.s	

Mechanical properties: design stresses are before irradiation

2	**MECHANICAL**	**Design**	**Failure**
2.1.1	Applied conductor winding load	500N	
2.1.2	Compression during heat treatment	20MPa	40MPa
2.1.3	Coil re-shaping after heat treatment before impregnation	20MPa	40MPa
2.1.4	Compressive stress after completion of coil fabrication – at 300K and 4K.	200MPa	400MPa
2.2	Shear: Short-beam shear strength at 4K	50MPa	100MPa
2.3	Tension: Transverse tensile strength of insulation laminate at 4K	25MPa	50MPa
2.4	Fracture, need to know properties at 300K and 4K, specification to be determined	TBD	

3	**THERMAL**	**Design**	
3.5	Transverse thermal contraction (to match conductor contraction and modulus). Integrated thermal contraction [300K to 4K]	0.003 to 0.004	
3.6	Thermal conductivity at 4.2K	50mW/m/K	

4	**ELECTRICAL**	**Design**	**Failure**
4.1	Breakdown voltage inter-turn tested in helium at 300K	1000V 2500V/mm	2000V 5000V/mm

5	**RADIATION**	
5.1	The failure properties above must be achieved following doses expected during 10 years' running. See notes for detailed explanation.	
5.2		**Range**
5.2.1	Dose	**50 to 600 Mgy**
5.2.2	Fluence >0.1MeV	**2.5 to 30x10^{16}cm^{-2}**
5.2.3	Neutron dose at high energy (>10MeV)	**TBD**

Appendix 1. Discussion and References

1 General

1.1 Generally the desire is to minimise insulation thickness. However, practical insulation thickness is determined by several factors ; electrical safety of the coil during quench ; thickness constraints of the basic insulation materials ; allowance for abnormal cable features or inclusions.

There is a minimum thickness determined from the breakdown voltage of the insulation. A safety factor above this must be allowed for.

The thickness of commercially available fibre tape or braid determines an absolute lower limit of ~0.12mm per conductor, 0.24mm inter-turn. The fibre can be compressed to roughly 65% of this before crushing.

Taking all factors into account a thickness of 0.4mm between turns is considered a practical thickness for design.

1.2

1.2.1 The insulation must be compatible with a degree of automation to reduce winding time and cost for production magnets. The insulation will be applied to the cable as a double wrap tape or braid using a specialised machine. The cable will be wound onto the coil former under tension with clamping and forming around the coil ends.

1.2.2 A cos-theta coil design requires the tightest bend radii. Given the large cable dimensions proposed for NED this is a demanding specification for the cable and the insulation which must accommodate the tight bend radii and the associated loads imparted by the cable . Other coil designs (e.g. block) will allow greater bend radii and be less demanding.

1.3 Nb_3Sn is the candidate conductor for high-field coils, and wind-and-react the most suitable production technique for small bend radii. React-and-wind coils have not, to date, delivered reliable high field performance. Consequently, the insulation material, or the relevant component of it, or precursor, must be compatible with the Nb_3Sn reaction cycle ~ 10days @ ~700C.

The glass fibres will become more brittle/fragile during the heat treatment phase due to re-sizing and some changes in the crystal structure. It should retain sufficient strength to withstand the post heat treatment compression and any necessary coil shape adjustment ie: item 2.1.3.

1.4 The design value for current LHC dipole specification is 40 cycles from 300K – 4.2K – 300K. This has been scaled down for this specification in order to be compatible with the test programme

1.5 The design value for current LHC dipole specification is ~10000 power ramp cycles. This has been scaled down for this specification in order to be compatible with the test programme

1.6 The insulation should have sufficient porosity to allow a low viscosity resin to reliably impregnate the coil structure (Canfer et al, A definition of the parameters that influence and control the flow of resin during vacuum impregnation of magnets and other structures, Proc. ICMC Vol 48, 2002, ed B Balachandran et al)

2 Mechanical

2.1.1 Typical conductor winding load – 500N in the straight sections and 300-350N in the coil heads (A. Devred).

2.1.2 Typical compressive stress required to compact insulation to required thickness and maintain coil shape prior to impregnation.

2.1.3 Typical compressive stress required to correct coil shape after heat treatment.

2.1.4 To prevent conductor movement during operation a preload of the coil structure will be applied at room temperature eg: the collaring applied to LHC dipoles. For a cos-theta design this preload stress could be up to 200MPa . More detailed analysis of the coil structure for NED may allow some reduction of this parameter.

2.2 The design aim should be to limit shear stresses to 25MPa (A. den Ouden). The proposed specification for the insulation laminate is therefore 50MPa.

2.3 The design aim will be to eliminate or at least minimise transverse tensile stresses by the application of pre-compression – see section 2.1.4. For the conventional insulation approach the cable structure will be fully bonded to the insulation. In this case transverse tensile stress can be applied to the insulation laminate. For the innovative insulation approach where the centre of the cable is not bonded minimal transverse tensile stress will be applied to the insulation.

2.4 High fracture toughness is required for stability, as a crack can release sufficient energy to initiate coil quenches. Stability is more important than a particularly high value for fracture toughness.

3 Thermal

3.1 To minimise differential thermal stresses within the cable/insulation matrix and to minimise thermal contraction of the overall coil pack. In practise this is achieved by using a relatively high fibre to matrix ratio, which will also make the modulus of the insulation closer to that of the conductor.

3.2 To maximise heat transfer due to beam loss and during quench conditions. This is a typical value for an epoxy material.

4 Electrical

4.1 The basic turn to turn voltage specification is derived from a worst case quench scenario with a typical NED cable – coil temperature 400K, coil current 14000 amps, 30 metre turn length. The resulting turn to turn voltage is estimated to be ~250V. A design electric strength of 1000V is proposed allowing a 4x safety factor. This is a critical parameter and in practise, should be a modest value relative to the dielectric strength of the insulation laminate.

5 Radiation

5.1 These are preliminary ideas for a radiation dose specification and we expect them to be developed as design work progresses. The amount of damage is largely dependent upon the dose (energy deposited in the material) but it is to a lesser extent dependent upon the nature of the radiation especially gamma vs neutron irradiation, and on the number of high-energy neutrons. Various designs of dipole gives different maximum radiation doses. The approach we have taken is to use as a minimum requirement the radiation expected in the “block” design (the one that gives the lowest dose) but to set as a target the radiation expected in the “cos theta” design (the one that gives the highest dose).

5.2 Doses from N. Mokhov, SC Magnets in high radiation environment at supercolliders, Erice Nov.2003 http://supercon.lbl.gov/erice/.

4.2.3 High energy neutrons are known to cause significant damage to organic materials. Above about 10MeV neutrons can liberate “knock-on protons” which are highly damaging owing to their large mass and charged nature. Low energy neutrons do not liberate knock-on protons. (Abe et al, Irradiation effects in KAPTON polyimide film from 14-MeV Neutrons and Cobalt-60 gamma rays, Influence of radiation on material properties: 13th international symposium (part II) ASTM STP 956, F A Garner ed, ASTM 1987 pp669-681)

APPENDIX 2.

NB$_3$SN ACCELERATOR MAGNETS

Insulation Review

1. Introduction

Electrical insulation is one of the most challenging issues governing the engineering exploitation of Nb$_3$Sn conductors. This is especially true for accelerator magnet applications where the requirement for high current operation and small bending radii dictate the wind and react manufacturing route. The status of insulation development for accelerator magnets has been reviewed by Devred(1).

In this paper we present a brief review of insulation techniques and consider the critical issues and limitations in relation to the technical challenge presented by future high field dipoles for accelerator application. We present the key issues and use these to define a preliminary insulation specification for a 15T magnet to be studied within the NED programme(2). From the basic specification we outline a series of studies to be undertaken under the auspices of NED and look forward to see how we might improve insulation and magnet performance by improving our knowledge of materials.

2. Insulation Techniques for Accelerator Magnets

In this section we review the status of insulation technology and look at new or innovative developments which are underway. For convenience we refer to these as conventional and innovative.

2.1 Conventional

The conventional approach, established over some 10-20 years has been to use a glass or quartz tape wrap or a braid to insulate the basic cable. In some cases the glass insulation has been supplemented by mica film inserted between the turns eg: the University of Twente 11T dipole(3) . In the earliest applications the tape or braid was applied to the cable with its commercial sizing intact. However, it was found that removal of this sizing at the Nb$_3$Sn heat treatment stage was difficult and could lead to poor electrical resistance levels between turns. Removal of the sizing is now usually done before applying the insulation to the cable. This does make the insulation stage more difficult and does make the insulation more fragile for winding.

Tape insulation is applied as a double layer wrap to give adequate overlap. Braid insulation can be applied using a specialised machine or for short lengths ~100m the braid can be applied as a sleeve to the cable (5).

After insulation the cable is wound using a conventional machine but great care is required in handling of the cable, clamping and forming around the tight bends at the magnet ends. After winding the coil is clamped in a mould and undergoes the heat treatment process of ~1 week at 650C-700C. This heat treatment is carried out in a vacuum or inert (Argon) atmosphere. During heat treatment there is a risk of damage due to expansion movements. This risk will be increased for the long coils required for future accelerators.

After heat treatment the glass is extremely fragile and great care is required if the coil is to be transferred from a heat treatment mould to an impregnation mould. The final stage in coil manufacture is vacuum impregnation with epoxy. This is a relatively complex process with some risk of failure. It also represents a significant cost for large scale magnet production. Impregnation with epoxy means that helium is excluded from the winding and can play no part in coil stability or heat removal (except at the coil surfaces).

2.2 Novel/Innovative

While many variants/improvements on the conventional insulation route have been developed, the basic process remains similar to the one developed for some of the earliest Nb_3Sn filamentary magnets.(4)

2.2.1 Sizing

Significant advances have been achieved by improvements in 'sizing' or in fact by 're-sizing'. Sizing is applied to the glass filaments by the manufacturer to allow commercial handling processes to be used to fabricate tapes.

The commercial sizing is organic and it is not easy to find the precise composition due to commercial secrecy. One approach has therefore been to remove the commercial sizing as a first step and to replace this with a known sizing material to aid conductor wrapping and coil winding. Such an approach has been successfully used by the Berkeley Group using Palmitic Acid to re-size insulation braid(5).

Apart from this improvement the other steps in magnet manufacture closely follow the procedure given in Section 2.1.

2.2.2 Inorganic/Ceramic Route

A more radical approach is to replace the organic sizing with an inorganic or ceramic precursor. This approach is being developed at CEA Saclay and LBNL. In this approach the glass tape is impregnated with a ceramic precursor which fully penetrates the tape fibres. The tape is used to wrap the cable in the conventional way for coil winding.

The standard heat treatment to form the Nb_3Sn also achieves the heat treatment of the ceramic to give a robust insulation and a structurally complete coil. The coil will still have some porosity and the aim is to remove the impregnation stage. For this to be possible the insulation bonded with ceramic must have sufficient mechanical and electrical integrity for magnet assembly and operation. If some porosity can be maintained then helium will penetrate the winding to aid stability.

This route can also be finished by a vacuum impregnation. Significant advances have been made in tape preparation. See Fig. 1.

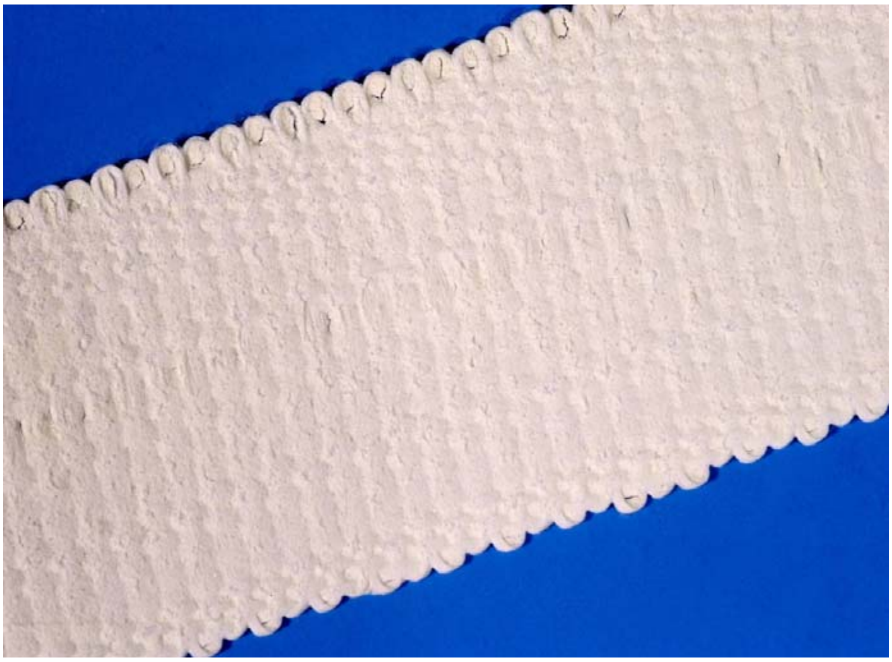

Figure 1: Ceramic precursor on glass fibre tape produced by Saclay (Cortesy of Francoise Rondeaux)

3. Insulation – Key Issues - Specification

In this section we address some of the key insulation issues and limitations and develop a target specification for a high field magnet.

3.1 Electrical Insulation Strength

The conductor insulation must have sufficient electrical integrity to withstand turn-to-turn voltages generated during a quench. Ground insulation i.e. to the coil support structure, will require a higher specification in terms of breakdown voltage but this can be achieved by additional insulation layers. In this context we consider only the insulation on the cable.

We estimate that the maximum turn-to-turn voltage generated in a quench of a 15m accelerator dipole will be ~250V which equates to an intrinsic voltage of ~600V/mm for a typical insulation thickness of 0.4mm turn to turn.

Allowing for a safety factor of 4 these values translate into 1kV turn to turn or 2-3kV/mm in the insulation laminate. The dielectric strength requirements are therefore not especially demanding when compared with standard laminate (G10) materials which exhibit 20-30kV/mm. However, these properties do have to be achieved through a complete winding, heat treatment, impregnation, coil assembly and thermal cycle process.

While dielectric properties are not so demanding the need for integrity is very high. Insulation failure will have serious consequences because of the high stored energy density in a 15T dipole.

3.2 Thickness

Insulation thickness is important for 3 reasons:

(i) current density

Typically, the insulation will occupy ~20% of the winding space and will impact on current density overall. Current density improvement is therefore a driver to reduce insulation thickness

(ii) thermal contraction

In a dipole winding the insulation will affect the azimuthal thermal contraction of the winding block. If the insulation is essentially a glass/epoxy laminate the transverse thermal contraction will be strongly dependent on glass content e.g. 50% glass by volume will give an integrated thermal contraction $6\text{-}7 \times 10^{-3}$ while 60% glass by volume will give $\sim 4 \times 10^{-3}$ which is much closer to the conductor properties.

Low glass content will affect the pre-compression required at room temperature. However, the desire to compress the winding heavily at the impregnation stage to achieve high glass content must be tempered with the risk of crushing insulation and creating a fault. Over-compression at this stage may also prevent resin penetration which can also lead to reduced mechanical and electrical properties.

(iii) winding modulus

Insulation thickness will affect the winding modulus and will influence movement under magnetic loads. Again the desire is to compress to reduce insulation thickness, increase glass content and hence modulus.

The above arguments are drivers for reducing insulation thickness. However, as discussed in Section 3.1 insulation integrity is paramount for production magnets. A very thin insulation, if it could be realised, would have little tolerance for cable imperfections and may be prone to damage at high loads. So the simple geometric spacing provided by the insulation is in itself an important factor which should not be discarded.

In our view a robust insulation which facilitates coil manufacture with high integrity is more important than 10% improvement in current density.

Typically, for S glass, the minimum commercial tape thickness is ~0.1mm giving an insulation thickness on the cable of ~0.2mm with double overlap. The thickness can be reduced by using a single tape wrap with mica insulation inserted between turns(3). Tape thickness is not so much a fundamental property of the materials but more one of commercial availability. In principle, tapes of 0.06-0.08mm could be made and used for winding.

Braid offers potential reductions in thickness because it is nominally a single layer.

3.3 Mechanical Properties

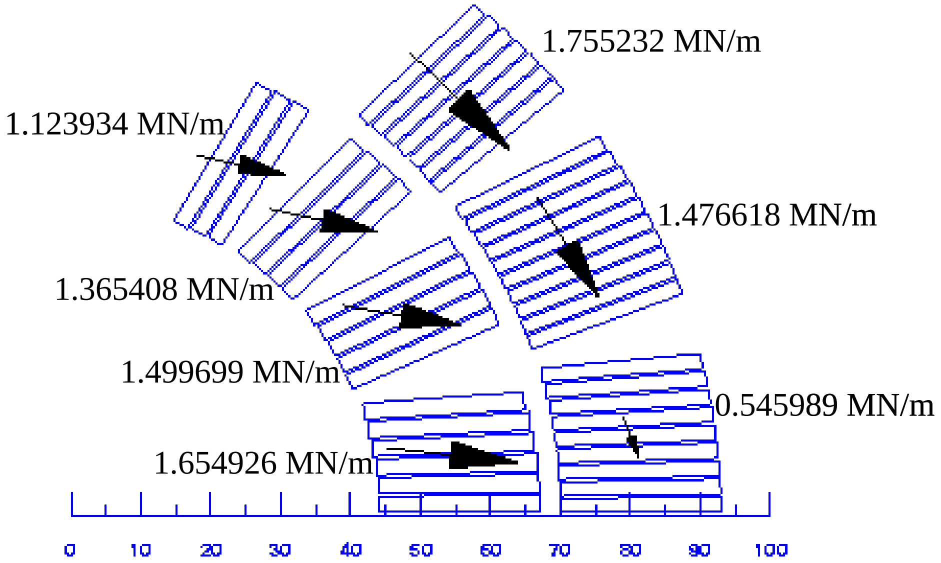

Figure 2: Mechanical forces loads on a 15T, 88mm aperture dipole(6).

The mechanical properties of the insulation must be compatible with all phases from cable taping, through winding, heat treatment, impregnation, assembly (collaring ~200MPa), cool down (thermal stresses) and magnet operation. We have already commented on the winding requirements. In this section we will address the operational requirements. In a cos-theta or block type design the principal loading in the straight section will be compressive, applied through the cable and insulation laminate thickness see Fig.2. Typically, magnetic loading will be ~150-200MPa (average) with high local stresses due to the cable structure.

For a standard, G10 type, laminate a compressive strength of 600-800MPa through the thickness is readily achievable. For an insulation laminate made in-situ with heat treatment, i.e. magnet fabrication conditions, ultimate compressive strengths are not known.

The design of a high field dipole will aim to eliminate tensile stresses and minimise shear stress in the straight sections. However, it is clear that localised shear and tensile stresses will be present in the straight sections and it is difficult to eliminate more global shear stresses in the coil ends.

The insulation should therefore have capacity to resist shear and tension loads. The scale of these stresses should form part of the NED magnet design study.

3.4 Radiation Hardness

For an LHC upgrade or future high energy accelerator applications increased radiation hardness of the insulation will become important. Currently the insulation material is the limiting factor in radiation hardness of magnets.

Conventional epoxy-glass materials are limited to doses of 10^8Gy. Dipoles used in interaction region applications may receive doses in excess of this, although the dose can be reduced by an order of magnitude by design, for example a block coil design as opposed to a cos-theta. The block coil design moves the sensitive winding away from the damaging particles(9).

Cyanate-ester resins, although expensive, have received attention by the fusion community and may offer increased radiation resistance compared to epoxies. However they are still organic materials and will be susceptible to radiation induced damage, particularly by high-energy neutrons.

An inorganic material should offer the ultimate solution to radiation stability, hence the interest of CTD and Saclay in this area.

Testing of radiation hardness should be a part of the NED programme as it will be a critical factor in the design and useful lifetime of the dipole.

3.5 Thermal Conductivity

Thermal conductivity of the insulation is a parameter which can be important for magnet operation. Thermal conductivity between cables will have some influence on stability (MPZ) but is unlikely to be high enough for the effect to be significant in magnet performance.

Thermal conductivity will also influence the peak temperatures due to steady state or transient beam losses. For a laminate of glass/epoxy the thermal conductivity will be dependent largely on the epoxy content. Typically, a laminate will have transverse thermal conductivity ~0.05W/m/K. There is not much scope to improve this in a Nb_3Sn dipole.

3.6 Helium Porosity

An insulation system that allows penetration of helium to the internal part of the cable is an objective of the innovative, ceramic bonded insulation. For the 'conventional' insulation with epoxy impregnation, helium porosity is not feasible.

Overall, the benefits of porosity with its potential for improved conductor stability will need to be balanced against the mechanical properties required at 15T. Porosity will reduce the mechanical properties of the insulation/winding and could lead to movements or stress cracking under high stress conditions.

4. NED Insulation Development Programme

An R&D programme for insulation development has been set up under the overall Next European Dipole Programme (NED).

Two R&D strands are planned:

(i) conventional insulation development by CCLRC, Rutherford Appleton Laboratory

(ii) innovative insulation development by CEA Saclay. This programme will build on the existing CEA developments with the aim of meeting NED specification levels

4.1 R&D Programme Overview

The programme will include all facets of insulation development from magnet design and analysis to define mechanical requirements and specification through to testing of large scale samples.

Generic tests will be developed to characterise materials and process routes. These tests will include; electrical insulation, thermal contraction, and a range of mechanical tests including tensile, compressive, shear and work of fracture.

In addition special test samples will be developed to give better simulation of real winding behaviour e.g. compression stacks and short beam shear. These samples will be characterised in standard testing machines with capability to operate at 300K, 77K and 4.2K.

For characterisation of epoxy systems specialised equipment such as a Dynamic Mechanical Analyser and a Differential Scanning Calorimeter will be used. A DMA can be used to evaluate temperature dependence of mechanical properties. A DSC can evaluate parameters such as reaction kinetics and Tg (glass transition temperature).

4.2 Materials Improvement

The aim of the programme will also be to develop new processes and tests with the objective of improved performance.

Work of fracture is a materials property that is not well understood in resin and composite materials at low temperatures. Fracture properties are significant for magnet performance because crack propagation can induce quenches and crack propagation is determined by fracture toughness. A better understanding of fracture properties in insulation materials could lead to enhanced magnet performance. Reducing or eliminating cracking at relatively low stresses has more relevance to magnet stability than improving the ultimate strength.

Historically epoxy resins have been formulated for use at low temperature by either using high levels of filler to reduce thermal contraction, or by making the epoxy inherently tough. High filler loadings are not compatible with vacuum impregnation processing routes. There are opportunities to improve fracture properties of the basic resin systems using recently developed materials such as:

(i) dendritic hyperbranched polymers, which have been shown to be able to double the interlaminar fracture resistance of epoxy based composites and to reduce the internal stress level by as much as 80% with only 10phr of modifier. These property improvements were obtained without affecting the viscosity, and thus the processibility, nor the glass transition temperature of the epoxy resin (7).

(ii) Nanofillers (clay)

Layered clays were used as nanoparticle fillers in fibre reinforced polymeric materials (epoxy composites). Transverse cracking in response to cryogenic cycling was significantly reduced when nanoparticle fillers were used at concentrations much lower than those used for traditional fillers e.g. 5phr. Nanoclays have been shown to modify traditional fibre reinforced composite materials and enhance their resistance to thermal cycling induced stresses (8) .

5. Summary

Insulation for Nb_3Sn magnets remains one of the most challenging issues for the engineering exploitation of Nb_3Sn conductors in future accelerators. The design, processing route, materials properties and end product are totally interdependent. The aim through NED is to achieve a better specification for insulation in a 15 Tesla dipole magnet – this will be an interaction between magnet design, fabrication route and materials.

The aim is also to explore and develop new materials characteristics for critical application.

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APPENDIX 3 INSULATION DEVELOPMENT FOR THE NEXT EUROPEAN DIPOLE

PRESENTED AT ICMC 2005

INSULATION DEVELOPMENT FOR THE NEXT EUROPEAN DIPOLE

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ABSTRACT

Electrical insulation is one of the most challenging issues governing the engineering exploitation of niobium-tin conductors. This is especially true for future accelerator magnets manufactured by the "wind-and-react" route. Applications such as the LHC Interaction Region upgrade or in the longer term an LHC energy upgrade will require magnets to operate at high fields in demanding thermal and radiation environments. The Next European Dipole (NED) programme is aimed at the development of a large aperture (up to 88mm) high field (up to 15T conductor peak field) superconducting dipole magnet relying on niobium-tin conductors. Conventional insulation development is being addressed in two key areas, the engineering requirements for large scale magnet manufacture and the improvement of materials properties for magnet operation and performance. The paper will review the status of insulation technology for niobium-tin accelerator magnets and define the special requirements for NED. Particular emphasis will be placed on the development of fibre sizing technology and its influence on magnet manufacture and electrical/mechanical performance of insulation laminates.

KEYWORDS: Polymers, epoxy, S-glass fibers, insulation, fracture, materials testing
PACS: 84.71ba, 81.70q

INTRODUCTION

NED is a 3 year Joint Research Activity (JRA-3) embedded in the Integrated Activity CARE (Coordinated Accelerator Research in Europe). NED focuses on research and development on advanced accelerator magnet technology for existing and future facilities by laying the foundation for an integrated European effort towards bringing Nb_3Sn technology to maturity and boosting the competitiveness of European laboratories and industry.

Insulation development is one work package of the NED Joint Research Activity. This paper reviews the current state of the art in insulation technology and presents experimental results on a novel fibre sizing material, which shows promise for use in Nb_3Sn insulation applications.

Accelerator magnets for the Large Hadron Collider are close to the practical limits for NbTi superconductor. Future upgrades to LHC beyond 10 Tesla will require the use of alternative superconducting materials, the most promising material is Nb_3Sn. Nb_3Sn superconductor is very brittle and strain sensitive [1]. As a result, high-field accelerator magnets can only be manufactured by the so-called "wind-and react" route. The Nb_3Sn compound is formed from precursors, and after winding the magnet it is subjected to a heat treatment process which forms the Nb_3Sn compound. This solid state diffusion process typically takes place at temperatures of up to 973K (700ºC) for some days in a vacuum or argon atmosphere, to avoid oxidising the copper stabiliser. This implies that the insulation system must also experience the heat treatment since it is applied to the superconducting cable as part of the winding process.

Electrical insulation is one of the most challenging issues governing the engineering exploitation of Nb_3Sn conductors.

INSULATION TECHNIQUES FOR NIOBIUM-TIN ACCELERATOR MAGNETS

In this section we briefly review the status of insulation technology and look at new or innovative developments that are underway. For high field accelerator magnets the Rutherford cable is typically insulated using a glass fibre tape. The insulation needs to be thin to maintain a high superconductor content and overall current density. This in turn dictates the use of thin, 150um, glass fibre tapes.

Conventional Insulation

The conventional approach, established over some 10-20 years has been to use a glass or quartz tape wrap or a braid to insulate the basic cable. In some cases the glass insulation has been supplemented by mica film inserted between the turns, for example the University of Twente 11T dipole [2]. In the earliest applications the tape or braid was applied to the cable with its commercial sizing intact. However, it was found that removal of this sizing at the Nb_3Sn heat treatment stage was difficult and could lead to poor electrical breakdown strength between turns. Removal of the sizing is now usually done before applying the insulation to the cable. This makes the insulation stage more difficult and the insulation more fragile for winding.

Tape insulation is applied as a double layer wrap to give adequate overlap. Braid insulation can be applied using a specialised machine or for short lengths ~100m the braid can be applied as a sleeve to the cable [3].

After insulation the cable is wound using a conventional machine but great care is required in handling of the cable, clamping and forming around the tight bends at the magnet ends, particularly for cos-theta magnet designs. After winding the coil is clamped in a mould and undergoes the heat treatment process of, typically, one week at 933K (660ºC). Heat treatment is carried out in a vacuum or inert (argon) atmosphere. During heat treatment there is a risk of damage due to expansion movements. This risk will be increased for the long coils required for future accelerators.

After heat treatment the glass is extremely fragile and great care is required if the coil is to be transferred from a heat treatment mould to an impregnation mould. The final stage in coil manufacture is vacuum impregnation with epoxy. This is a relatively complex process with some risk of failure. It also represents a significant cost for large scale magnet production. Impregnation with epoxy means that helium is excluded from the winding and can play no part in coil stability or heat removal (except at the coil surfaces).

While many variants/improvements on the conventional insulation route have been developed, the basic process remains similar to the one developed for some of the earliest Nb_3Sn filamentary magnets[4].

SIZING

The commercial sizing applied after glass filament drawing is a mixture of organic materials. The sizing has many functions: to enable high-speed weaving by providing a protective layer, to protect the glass surface from damage and fracture and also to protect weaving machines from the abrasive nature of the glass. Some manufacturers use a starch size for weaving purposes, which is removed before a functional sizing is applied, depending on the application. A functional sizing includes materials to chemically couple the glass to the matrix in order to increase fibre-to-matrix bond strength, and wetting agents. The sizing is a thin coating that is approximately 1-2% of the weight of the fibre[5].

A typical commercial glass sizing is not intended for high-temperature use, such as experienced during the Nb_3Sn reaction cycle. Commercial sizings degrade during heat treatment but do not oxidise in the vacuum or argon environment. A carbon residue is left on the glass. This compromises electrical breakdown strength and cannot be tolerated because high voltages can be generated during a magnet quench. Therefore the most common approach is to remove the sizing from the glass fibre before cable wrapping.

Desizing

A typical approach is to remove the sizing by heating the glass tape to 623-723K (350-450°C) in air before winding the cable[3,6]. The effect of removing the sizing is to leave the glass fibre vulnerable to damage during winding. The lack of a functional sizing and a wetting agent is likely to reduce the fibre to matrix bond strength.

Resizing

Arkan [3] reports the use of palmitic acid to reinstate the lubrication function of a size. Palmitic acid is removed during heat treatment and is reported to leave little carbon residue.

Sizing is very desirable for ease of cable wrapping and magnet winding. This will be especially so if larger scale magnet production is required for a future particle accelerator. The ideal sizing material would provide protection to the fibres during tape winding, and provide compatibility between the inorganic fibre and organic matrix. This is considered to be essential for high shear strength and fracture toughness in the final insulation. We have therefore taken the approach of looking for a sizing that survives heat treatment.

A literature search identified polyimide materials as a promising sizing material for this application. Polyimide materials are amongst the most thermally stable polymers because their chemical structure is highly aromatic. A family of polyimide materials have been developed at NASA Langley Research Centre for high temperature composite applications. They have been commercialised, for example by UBE [7]. Hydrosize Technologies Inc. market a polyimide material for use as a sizing for polyimide resin transfer moulding applications [8]. It is rated to 773K (500°C) in air. Since the Nb_3Sn reaction takes place in an oxygen-free environment to avoid oxidising the conductor's copper stabiliser, it was thought that the maximum service temperature could be increased and that this sizing might therefore be useful for Nb_3Sn insulation.

INNOVATIVE INSULATION

A more radical approach is to replace the organic sizing with an inorganic or ceramic precursor. This approach is being developed at CEA Saclay[9] and separately by CTD Inc[10]. In this approach the glass tape is impregnated with a ceramic precursor, which fully penetrates the tape fibres. The tape is used to wrap the cable in the conventional way for coil winding.

The standard heat treatment to form the Nb_3Sn also achieves the heat treatment of the ceramic to give a robust insulation and a structurally complete coil. The coil will still have some porosity and the aim is to remove the impregnation stage. For this to be possible the insulation bonded with ceramic must have sufficient mechanical and electrical integrity for magnet assembly and operation. If some porosity can be maintained then helium will penetrate the winding to aid stability. The innovative programme is not reported in this paper.

EXPERIMENTAL STUDIES

A specification for the NED insulation material has been formulated. It includes mechanical, electrical, thermal and tentative radiation resistance parameters [11].

Since the parameter space for candidate insulation materials is so large, three screening tests have been used to economically test key material properties, namely interlaminar fracture toughness, shear strength, and electrical breakdown strength. The screening tests have been carried out at a temperature of 77K rather than 4K for expediency. More comprehensive testing of a few candidate materials to the specification will be carried out towards the end of the NED Insulation Development Programme.

Work of fracture is considered to be an important material property as fracture can lead to magnet instability and quenches. A standard test method was chosen, ASTM D5528, which can be applied to composites made using heat treated glass fibre and used to give a quantitative measure of work of fracture. It can also be performed at low temperature.

Standardised composite laminates were produced using a process representative of magnet insulation, with the exception that S-glass fibre fabric measuring 150mm x 150mm was used, and not tape; and that the laminates measured approximately 3mm in thickness to enable mechanical testing. Heat treatment was carried out in a stainless steel tool in a vacuum furnace.

Interlaminar Work of Fracture Testing

Interlaminar fracture test ASTM D5528 has already been shown to be a useful test at low temperature by Shindo et al [13]. Shindo found that work of fracture (G) increases at 77K then falls back to approximately room temperature values at 4K. Therefore room temperature is a suitable environment for screening test purposes. The test yields many results per sample and sample preparation is relatively straightforward.

Two sets of tests were performed. Firstly, in order to apply this technique to our current programme it was necessary to perform validation test studies. Secondly the effect of heat treatment and a polyimide sizing was investigated.

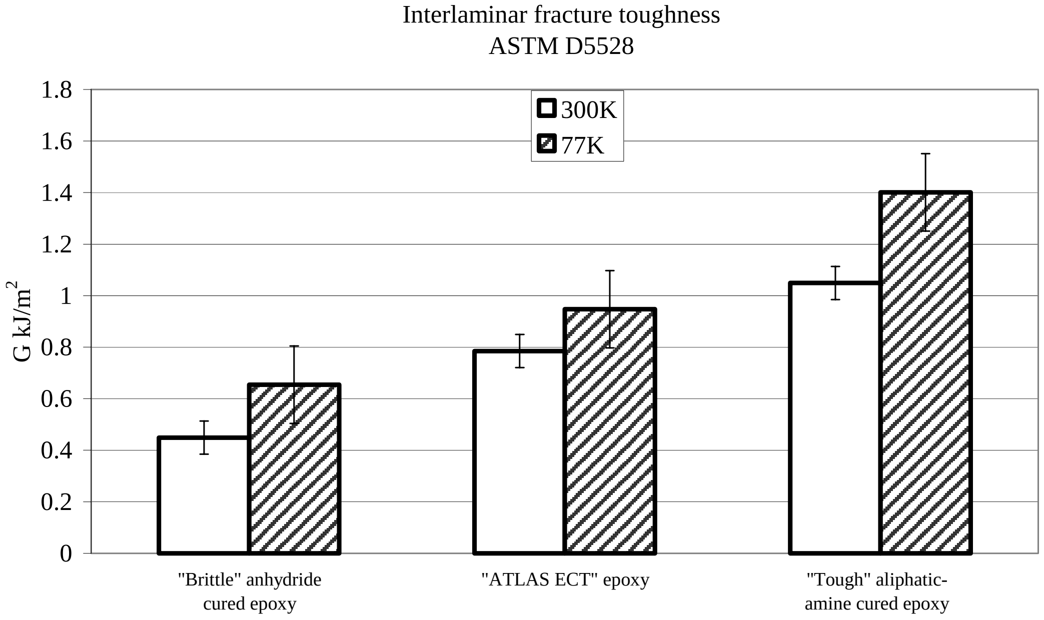

FIGURE 1. Work of fracture (G) validation test results. E glass fabric reinforcement with three epoxy systems, tested at 300K and 77K.

Work of Fracture Validation Tests

Fracture testing was carried out on known tough and brittle resin systems to gain experience of the test technique and to replicate Shindo's results. Tests were performed at ambient temperature and at 77K in liquid nitrogen. All validation tests were made using E-glass fibre. Results of these validation tests are presented in FIGURE 1.

The "brittle" epoxy is DGEBA epoxy resin with an acid anhydride curing agent (e.g. HY918, Huntsman), and is considered the standard epoxy for superconducting magnets as it has many production advantages. The tough epoxy is DGEBA with an aliphatic amine curing agent (e.g. Huntsman HY5922). This has a shorter working time compared to the anhydride. The results in the center of the chart are for an epoxy formulated for the ATLAS End Cap Toroids which combines toughness with sufficient working time for impregnation of large magnets [12]. It uses DGEBF epoxy (DER354P, Dow), PPGDGE flexibiliser, (DER732, Dow) and an aromatic amine curing agent, DETDA (Ethacure 100, Albemarle).

The results enable a quantitative comparison of the toughness of these resin systems to be made for the first time.

Effect Of Polyimide Sizing And Heat Treatment On Work Of Fracture

The effect of desizing the glass was investigated. Three types of fabric were used. S-glass from SP Systems was used as a reference material. JPS Glass supplied S-glass and quartz fabric, both treated with a polyimide sizing.

Laminates were produced by vacuum impregnation of the glass fibre with an epoxy resin at 313K (40°C). An epoxy matrix was chosen which is both relatively radiation stable, and has low viscosity so it is an epoxy that could be suitable for this application[12]. The epoxy is DGEBF with DETDA curing agent. Curing was performed in a press at a pressure of 1MPa and temperature of 353K (80ºC) for 16 hours followed by 403K (130ºC) for 24 hours. The fibre content of laminates was 50 to 55vol%.

TABLE 1 summarises the results obtained.

Laminate 1 was used as a reference material; the work of fracture is as expected from a brittle matrix. The short beam shear strength of 94MPa is close to the NED specification of 100MPa [11]. Laminate 2 contained desized S-glass and showed a surprisingly high work of fracture result. Laminate 3, desized then heat treated to simulate the Nb_3Sn process, failed through the glass layers and not in an interlaminar fashion. This is an invalid test result so a quantitative result is meaningless. This result indicates that the glass was adversely affected by the heat treatment.

Laminate 4 provides reference data for polyimide sized S-glass before heat treatment. It is higher than the commercially sized S-glass (0.70 compared to 0.49kJ/m2), which is encouraging considering that the polyimide sizing was not intended for compatability with epoxies, but for a polyimide matrix. Results for laminate 5, after heat treatment, show the same work of fracture and an increase in shear strength. This may be due to the vacuum heat treatment completing the curing process of the water-based polyimide sizing.

Quartz fibre showed the highest work of fracture, 0.88kJ/m2.

The fracture surfaces were examined using SEM. The polyimide-treated glass shows little evidence of fibre breakage compared to the reference S glass (laminate 1), see FIGURES 2 and 3.

Short Beam Shear Testing

Short-beam shear testing to ASTM D2344 was performed to examine the fibre to matrix bond strength. Results are presented in TABLE 1.

All laminates that had been heat treated at 933K (660°C) failed in a tensile or partly tensile manner. In contrast, all non-heat treated laminates failed in shear. This indicates that the strength of the glass has been affected by the heat treatment.

Laminate 5, JPS glass with polyimide sizing after heat treatment, shows the highest shear strength. The mode of failure is partly shear.

Quartz fibre exhibited a low shear strength compared to the S-glass, (64 compared to 98MPa).

TABLE 1 Interlaminar fracture and short-beam-shear test results of laminates with S-glass fibre and DGEBF/DETDA epoxy matrix

Laminate	Fabric	Sizing	Heat	Work of fracture, G	Short beam shear

Reference			treatment	kJ/m2 at 300K	strength MPa at 77K (failure mode)
1	S-glass	Commercial (as received)	None	0.49	94 (S)
2	S-glass	Desized	350°C 16hrs air	0.85	97 (S)
3	S-glass	Desized	350°C 16hrs + 660°C 60hrs in vacuum	Invalid result, glass fracture	69 (T)
4	JPS S-glass	Polyimide	None	0.70	89 (S)
5	JPS S-glass	Polyimide	660°C 60 hrs in vacuum	0.67	98 (S+T)
6	JPS Quartz	Polyimide	660°C 60 hrs in vacuum	0.88	64 (T)

Short beam shear test failure mode: T = Tensile S = Shear

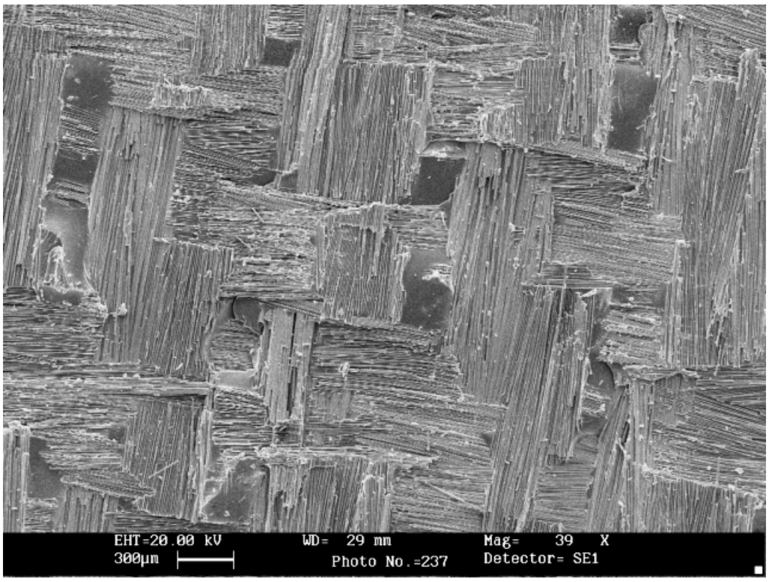

FIGURE 2. Fracture surface of laminate ref.4, desized and heat treated

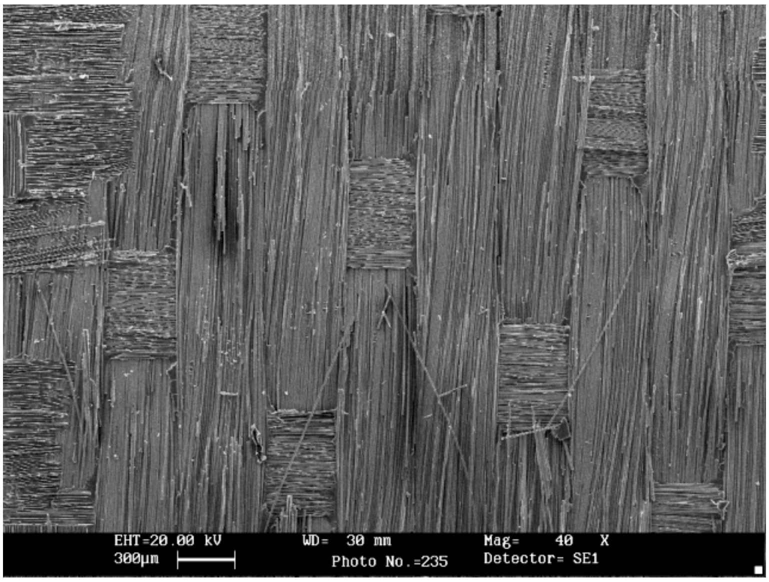

FIGURE 3. Fracture surface of laminate ref. 6, polyimide sizing and heat treated

Electrical Breakdown Testing

Electrical breakdown test to BS7831 is included because of the tendency for conventional sizing materials to degrade to carbon, and reduce electrical breakdown strength. A key specification requirement is breakdown strength of 5kV/mm. A G10 GRP material without carbon should withstand 30kV/mm so this is a modest specification.

Tests were carried out on laminates 0.4mm in thickness, manufactured at the same time as the thicker laminates for mechanical tests.

Electrical breakdown test results are presented in TABLE 2.

TABLE 2: Results of electrical breakdown test to BS7831

Fabric	Heat treatment	Colour of laminate	Breakdown voltage kV/mm
S glass	Desized + 660°C vacuum	White	>30
S glass	660°C vacuum	Dark grey	2.5
S glass +polyimide sizing	660°C vacuum	Light grey	>30

The polyimide-sized glass has high electrical breakdown strength after heat treatment. The colour change is from white to light grey, confirming that there is little carbon residue arising from thermal degradation.

CONCLUSIONS

A polyimide glass sizing material promises to solve many of the practical problems facing insulation manufacture for niobium-tin superconducting magnet applications.

Three test methods have been used to economically screen materials for insulation applications, interlaminar fracture, short beam shear and electrical breakdown voltage. The polyimide sizing on S-glass delivers improvements in all three parameters.

ACKNOWLEDGEMENT

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2. VACUUM IMPREGNATION OF COMPACTED GLASS FABRIC

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The Next European Dipole (NED), will be a large aperture, high field superconducting dipole magnet using Nb_3Sn Rutherford-type cables. The prepared coil will be vacuum impregnated with an epoxy resin in a mould. To enable mould closure, the glass fibre wrap will be heavily compacted before and during vacuum impregnation. To investigate the effect of these compression loads on the mechanical properties of glass fabric composites, panels were prepared in which the glass was compacted at stresses up to 10 MPa. The short beam shear strength was measured at 77 K and reduced with higher compaction stress during impregnation.

INTRODUCTION

NED is a Joint Research Activity (JRA-3) embedded in the Integrated Activity CARE (Coordinated Accelerator Research in Europe). NED focuses on research and development on advanced accelerator magnet technology for existing and future facilities by laying the foundation for an integrated European effort towards bringing Nb_3Sn technology to maturity and boosting the competitiveness of European laboratories and industry.

Insulation development is one work package of the NED Joint Research Activity. High field magnets feature high stresses. CCLRC have focused on developing "conventional" insulation for Nb_3Sn cable, where conventional refers to glass fibre and organic matrix such as epoxy resin. A new glass fibre sizing material has been shown to improve mechanical performance of insulation for the Nb_3Sn application [1]. A summary of the other activities in the NED project may be found in reference [2].

A typical manufacturing route for a wind-and-react accelerator magnet is by wrapping or braiding glass fibre tape around cables prior to winding the magnet and heat treatment. Upon winding and heat treatment completion, this is followed by vacuum impregnation with an organic matrix in a mould of accurate dimensions. To hold the coil turns in place and control their geometry and positioning a force is applied to the coil and hence to the glass fibre to force mould closure. The objective of this work was to determine the effect of this stress on the resulting glass-epoxy composite and to determine a recommended maximum stress.

A compaction pressure of 0.1 MPa is widely used in vacuum bagging applications, i.e. atmospheric pressure. For high performance applications it is desirable to increase the fibre fraction beyond that obtainable using atmospheric pressure. Autoclaves are commonly used to increase this pressure in prepreg applications, to 0.68 MPa or 100 psi for example [3]. However the effect of higher compaction pressures on laminates produced by vacuum impregnation is not well characterised. In particular, it is hypothesised that the resin may not be able to enter tightly crimped areas of glass where fibre bundles cross and local stresses are high.

There are two types of voids that occur in fibre reinforced plastics. These are voids along individual filaments (within fibre bundles or tows) and voids between laminates. There are two main causes of voids. The first is entrapment of air during impregnation. A viscous resin will struggle to penetrate between tightly packed bundles of filaments. The second cause is volatiles arising from the resin system. Low molecular weight components of the resin system may be volatile at the curing temperature and vacuum pressure [4].

S2-glass will be used in any magnet exposed to ionising radiation but for these initial investigations, E-glass plain weave fabric was used.

EXPERIMENTAL PROCEDURES

A series of glass fibre-epoxy laminates were manufactured using a variation of vacuum-impregnation known as vacuum infusion. 32 layers of E-glass plain weave cloth were placed inside a nylon vacuum bag and sealed with vacuum bagging tape. Two pipes were inserted to allow connection to a vacuum pump and to epoxy resin, via valves.

A screw-driven mechanical testing machine (Testometric AX500 model) was used to apply a known stress to the glass cloth. Epoxy resin was allowed into the bag whilst a controlled stress was applied to the glass fibre. The matrix material consisted of an unmodified DGEBA epoxy resin, with a molecular weight 400 aliphatic amine hardener and a piperazine accelerator. The resin was cured under pressure.

To check the resin flow, a laminate was manufactured using a thick transparent acrylic block to apply the pressure, allowing resin flow to be observed under stress. Even at 10 MPa, resin was observed moving through the laminate.

RESULTS

Figure 1 shows the appearance of the cured laminates at 1 to 10 MPa pressure Areas of white coinciding with fibre bundles crossing are visible, and increase in size as stress increases. At 10 MPa, the complete laminate appears white. Up to 4MPa, higher pressures resulted in thinned laminates. The 4 MPa laminate measured 4.3 mm in thickness, but the 10 MPa laminate measured 4.9 mm. This suggests that there is insufficient resin penetration into the fibre bundles and that the glass is springing back when the load is removed.

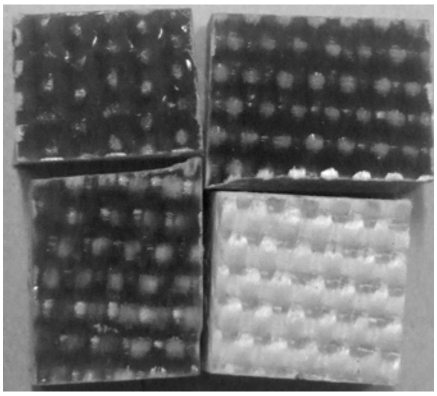

Figure 1 Comparison of laminates 1 MPa (top left), 2 MPa (top right), 3 MPa (bottom left) and 10MPa (bottom right)

DENSITY, GLASS FIBRE FRACTION AND VOID CONTENT

Density of samples of laminates were determined according to BS2782-6 1991 Method 620A. Samples of material are weighed in air and weighed again suspended in water.

Void content was determined according to EN ISO 7822:1999. This is a loss on ignition method to burn away the organic material and thus determine the weight of fibre and resin. A theoretical composite density can then be calculated based on the measured weight percentages of fibre and resin. The difference between theoretical and actual densities must be due to voids. This method relies on highly accurate measurements and even though a 4-place balance was used, it was found that the standard deviation of results was high. Only one meaningful void content result was determined: at 10 MPa applied stress, the estimated void content is 4.9% with a standard deviation of 2.7. This is consistent with the accuracy of the method given in EN ISO 7822 (2.5% by volume) [5].

Figure 2 shows the results obtained as a function of compaction pressure. Density peaks at 3 MPa. The drop in density at higher applied stresses could be due to void content. This coincides with increased opacity of the laminate. The glass fibre content plateaus above 3 MPa, at around 82% weight percent. This is probably due to reaching the packing limit of the glass fibres. Typical values are in the range 66 to 72 weight percent for a G10 or G11 laminate [6], so our values are unusually high.

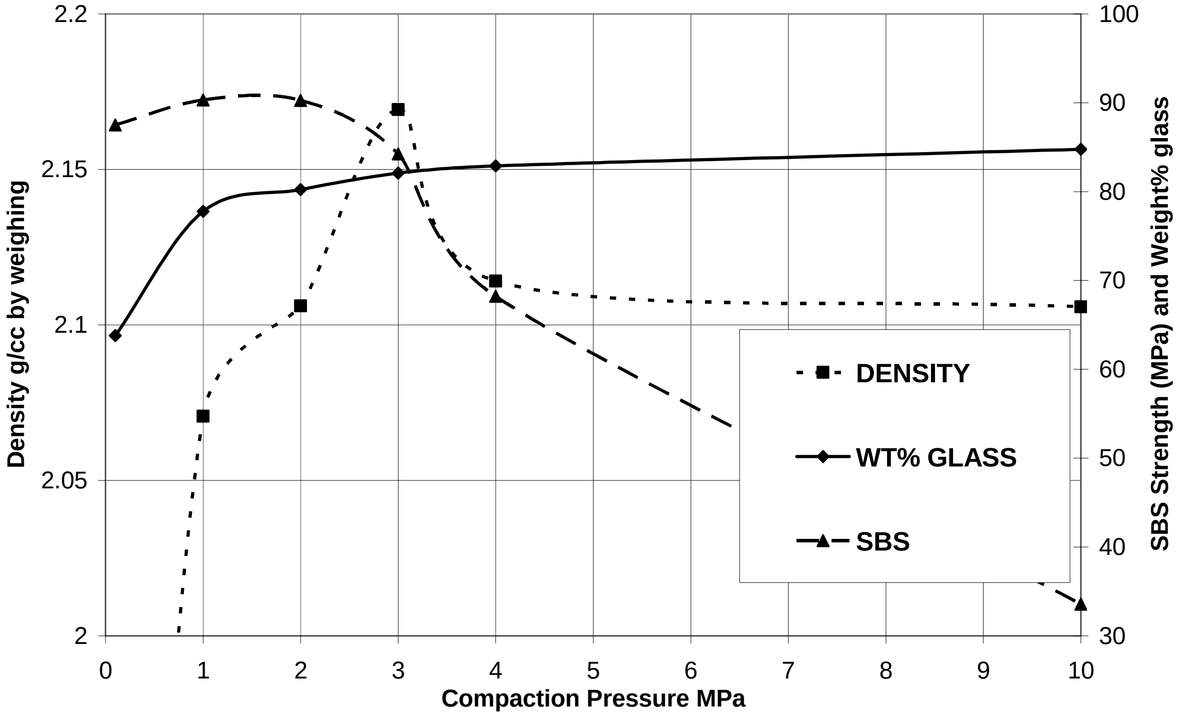

Figure 2 Composite material density and weight-percent glass as a function of compaction pressure during manufacture

SHORT BEAM SHEAR STRENGTH

Shear strength was measured in short beam shear immersed in liquid nitrogen at 77 K, based on the method given in ASTM D2344. Test pieces were machined to 3-mm thickness, 6-mm width and 18-mm length. Failures were all inter-laminar. Figure 2 shows short beam shear strength as a function of applied stress during impregnation.

A sharp drop in shear strength occurs above 2 MPa which correlates with the opaque appearance and is thought to be caused by lack of resin penetration inside fibre bundles. The NED specification is for 100 MPa shear strength but this will be using S2 glass fibre which has higher strength than E-glass. Earlier results from this programme measured a maximum short-beam-shear strength of 97 MPa with S2 glass [1].

PHOTOMICROGRAPHY

Sections of glass fibre-epoxy were cut using a diamond saw and polished using diamond compound. Both 0.1-MPa and 1-MPa samples were easy to polish. The 4-MPa sample was not easy to polish as fibre ends were not supported by epoxy resin (see Figure 3). At 0.1 MPa, approximately one third of the area is resin-rich volumes between fibre bundles. At 1 MPa, the resin rich areas are absent. At 4 MPa, many individual fibres were observed sprung out of the bundles. This suggests that the bundles are not completely impregnated with epoxy.

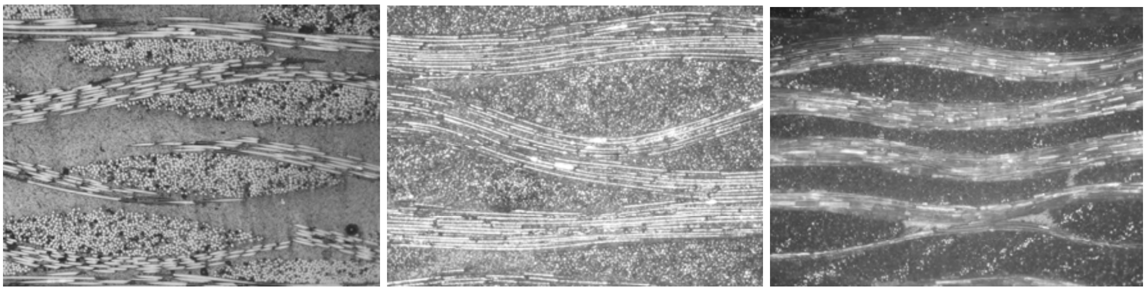

Figure 3 Micrographs of laminates at 0.1 MPa (left) compared to 1 MPa (middle) and 4 MPa (right; 100x magnification)

CONCLUSION

Applied compaction pressures up to approximately 2 MPa has a beneficial effect on short beam shear strength and glass content of epoxy-glass fibre laminates produced by vacuum infusion. Above an applied stress of 2MPa, the glass content does not increase significantly and shear strength is reduced. At a pressure of 10 MPa the laminate is of poor quality and shear strength is reduced to one third of a high quality laminate. All results suggest that visual inspection is a good guide to laminate quality. These results provide useful data for magnet insulation systems, but further investigations are required on insulated cable stacks that are more representative of actual coil configurations.

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