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VACUUM IMPREGNATION OF COMPACTED GLASS FABRIC

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Abstract

The Next European Dipole (NED), will be a large aperture, high field superconducting dipole magnet using Nb₃Sn 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.

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The Next European Dipole (NED), will be a large aperture, high field superconducting dipole magnet using Nb₃Sn 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 Nb₃Sn 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 Nb₃Sn 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 Nb₃Sn 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.

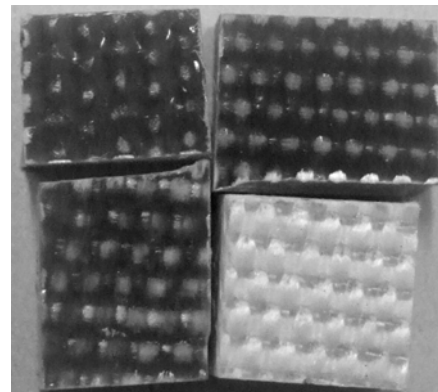


Figure 1 Comparison of laminates 1 MPa (top left), 2 MPa (top right), 3 MPa (bottom left) and 10MPa (bottom right)

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.

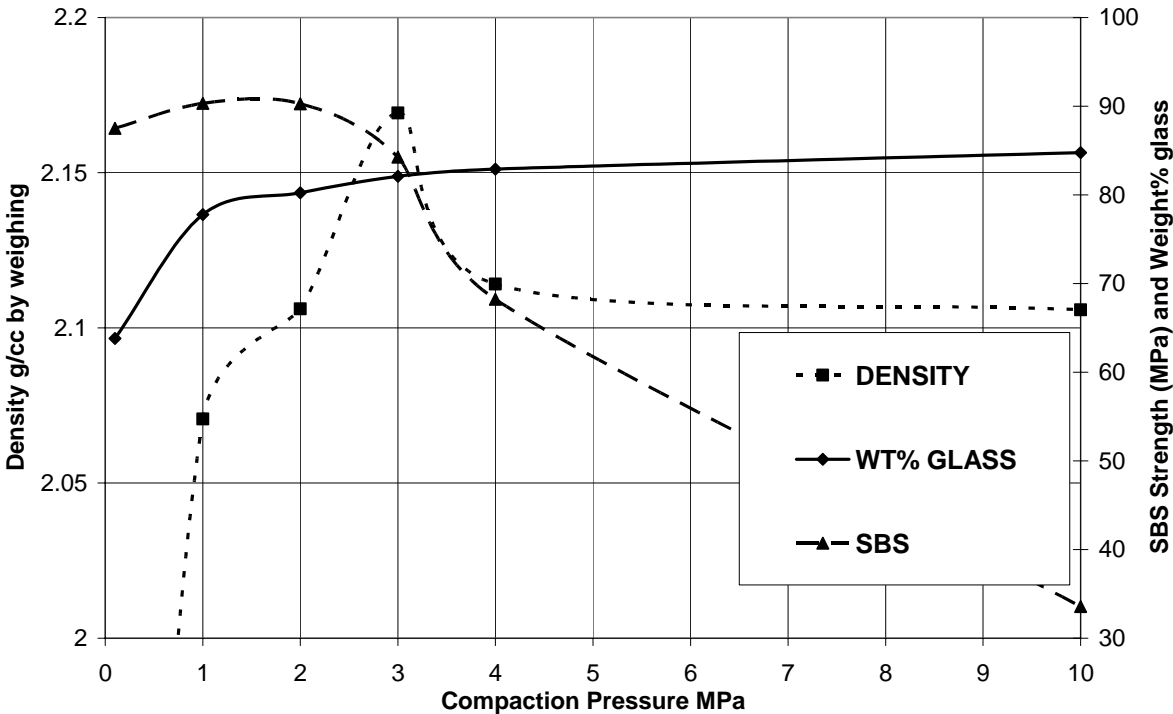


Figure 2 Composite material density and weight-percent glass as a function of compaction pressure during manufacture

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.

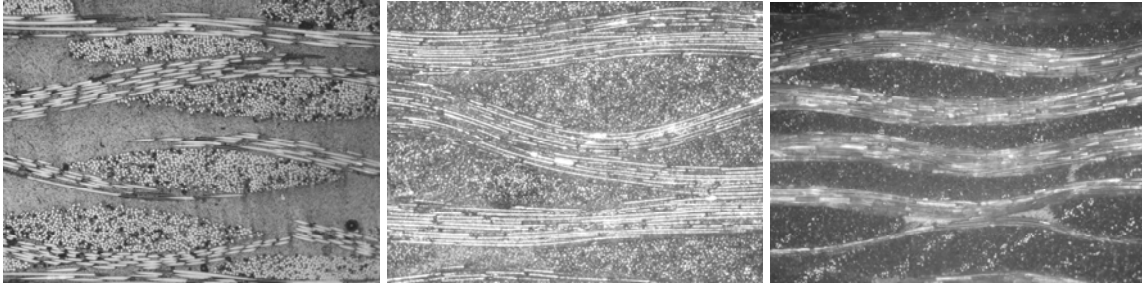


Figure 3 Micrographs of laminates at 0.1 MPa (left) compared to 1 MPa (middle) and 4 MPa (right; 100x magnification)

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.

ACKNOWLEDGEMENT

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REFERENCES

1. Canfer S.J. et al, Insulation development for the next European dipole, *Advances in Cryogenic Engineering* (2005), 52 298-305
2. Devred A., Overview and status of the Next European Dipole (NED) joint research activity, *Superconductor Science and Technology*, (2006), 19 S67-S83
3. Campbell F.C., *Manufacturing Processes for Advanced Composites*, Elsevier, (2004)
4. Jud N.W.C., Wright W.W., "Voids and their Effects on the Mechanical Properties of Composites - An Appraisal" *SAMPE Journal* (1978), 14
5. BS EN ISO 7822:1999 Textile glass reinforced plastics- Determination of void content, BSi (1999)
6. Kasen M.B. et al, Mechanical, electrical, and thermal characterization of G-10CR and G-11CR laminates between room temperature and 4K, *Advances in Cryogenic Engineering* (1980), 26 235-243