Resins for Superconducting Magnet Construction – An Overview of Requirements, Processing and Properties

David Evans
Advanced Cryogenic Materials Ltd
Brunel House
3, Norman Avenue, Abingdon, Oxon, OX14 2HQ UK.

David Evans276@btinternet.com

Abstract. The resin used for bonding and insulating superconducting magnets can be a major factor in ensuring reliable and stable operation of the completed assembly. There are many resin systems and available to magnet designers and engineers. The selection of the correct material depends on the application technique selected, the processing requirements and the end properties required of the cured resin. Available techniques for magnet construction and bonding include vacuum impregnation (VPI), so called ‘wet winding’ and the use of pre-impregnated fabrics. The advantages and disadvantages of these techniques are considered and the processing requirements for each are discussed. Materials selection requires an understanding of the processing characteristics such as viscosity and ‘useable lifetime’ but these must be married with the properties required of the cured resin. A low viscosity and long useable life is a frequent requirement for VPI processing of large magnet systems and this may be difficult to match with high thermal shock resistance that may be required to minimize cracking possibilities in resin rich regions. Examples are presented of resin systems resistant to cracking and structural features that enhance this parameter. For many magnets that may operate in an ionising radiation environment, radiation stability is an important requirement. Radiation stable systems are described and structural features that promote such stability are considered, along with the difficulty of matching all competing requirements.

1. Introduction
Of the three possible techniques for magnet insulation / bonding, wet winding and use of pre-impregnated materials introduce the resin during the winding procedure. Vacuum impregnation applies the resin only after winding has been completed. All three techniques predominantly use epoxy resins. Whatever technique is selected for coil manufacture, the resin system selected for bonding must have an overall ‘suitability for purpose’ and have the characteristics believed to be necessary. The insulating / bonding materials may be required to withstand high mechanical loads, high electrical stresses and be subjected thermally induced stresses as a result of cooling and possible thermal cycling that could result in resin cracking or delamination.

Since the vacuum impregnation technique (VPI) is the dominant procedure selected for magnet construction, most reports have concentrated on this technique, and there have been reports on optimising the procedure [1],[2] but the limited use of the other techniques and the infinite number of variables, limits the opportunities for any form of ‘normalising’.

The aim of any insulation / bonding technology is to ensure full “wetting” with resin of all surfaces to be bonded and to minimise, so far as is reasonably possible ‘voids’ within the insulation. It was reported [3] that the partial discharge signal in a composite manufactured in air was an order of magnitude higher than for composite insulation produced by vacuum impregnation. It was also
claimed that the existence of voids in electrical insulation should not be tolerated unless the sole purpose of that insulation was mechanical support.

2. Available Techniques for Coil bonding / Insulation

There is not a single correct method for insulating and bonding a particular coil. Rather, the choice between vacuum impregnation, wet winding or pre-impregnated materials depends on many factors and the perceived advantages and disadvantages of the two techniques are set out below (Tables 1, 2 and 3.) the decision on which route to employ may depend on coil geometry, numbers to be built (and therefore amortised costs) and a number of local factors – but the final decision can often be a compromise.

2.1 Vacuum Impregnation

A liquid resin is infused between conductors that have been previously wrapped (or coated) with an insulating material to provide turn and layer insulation. The conductor may be wrapped with a glass tape (or a glass tape/polymer film combination), or coated with an inert (inorganic material). For the VPI process the coil is sealed within a vacuum tight enclosure, heated and the pressure reduced to allow a period of out-gassing, followed by filling with a fully de-gassed resin. After filling, the pressure on the resin / coil assembly is raised and the prevailing atmospheric pressure (or an over-pressure, if considered necessary) drives the resin into the structure.

2.2 Use Pre-pregs

Pre-pregs are fabrics or tapes that have been pre-impregnated with resin and in most cases, partially cured. These materials are used, under tension, to wrap or cover conductors and so provide the necessary insulation and the resin for bonding. Such materials may be applied only after reaction of niobium / tin coils. Pre-pregs need to be consolidated to encourage resin flow during winding and resin flow that occurs during the gel / cure phase after winding may lead to overall dimensional changes in the coil. tool to ensure all surfaces are bonded. Pre-pregs offer only limited resin formulating properties (hot curing only) but radiation stable materials are readily available.

2.3 Wet Winding

The insulation is applied to the pre-insulated conductor (after reaction in the case of niobium tin coils) or by wrapping (say) glass tape onto the conductor and at the same time wetting the tape with liquid resin. Adding a layer of dry glass fabric between layers of conductor and wetting the glass before winding the next conductor layer is another variation on the wet wiring theme.

From the Discussion above and considering the vast majority of magnet coils are vacuum impregnated, the remainder of this paper is devoted to aspects of the VPI process

3. Techniques for VPI

Within the general vacuum impregnation process, there are three variations on a VPI theme that may be used: These three variations on an impregnation theme are outlined below.

3.1 Flood filling

The coil complete with some tooling to define dimensions etc, is placed within a tank to hold resin and the complete system placed within a vacuum chamber. After evacuation at elevated temperature for some period of time (See [1] and [2]) depending on the nature of the coil assembly, liquid resin is allowed to completely cover the tool and at this stage, atmospheric pressure may be applied and used to force the resin into the free space within the coil assembly. If a pressure greater than atmospheric is required, the ‘vacuum’ chamber has to be pressurised.

3.2 Vacuum Impregnation with Vacuum Chamber

The coil is contained within a tool that is sufficiently well sealed to prevent resin leakage under the operating conditions (i.e. using atmospheric pressure or greater) but need not necessarily be vacuum tight. The coil and tooling assembly is placed inside a chamber and evacuated. Resin is introduced directly into the tool, with the valve on the evacuation line open. When resin appears in the ‘riser’, this outlet valve is closed and atmospheric pressure is then available to force the resin into the coil structure. Over-pressure may be applied if required. If resin is able to leak from the tool at this stage,
either there will be insufficient pressure generated to ensure the structure is thoroughly impregnated, or drainage of resin could result in starvation of the coil. The vacuum in the chamber should be maintained until the coil is fully impregnated and resin is no longer flowing.

3.3 Vacuum Sealed Tool

Similar to the technique described in Section 3(b), the major difference being that the tool is vacuum tight and an external vacuum vessel is not used. Assuming the mould is sealed and remains so throughout the procedure, coils may be successfully impregnated using this technique.

4. Materials for The VPI Process

For coil impregnation, the ideal system would have the following characteristics:

(a) Adequate ‘useable life’ - Low viscosity, to be maintained if possible for many hours
(b) Resistant to cracking when subjected to thermal shock
(c) Low curing temperature (<130°C).
(d) Low shrinkage on cure
(e) Low thermal contraction, related to that of the conductors
(f) Adequate room temperature mechanical strength and resistance to creep under the loads from clamping forces
(g) Adequate electrical properties, reliably reproducible, to provide insulation
(h) Good adhesion to conductor and / or insulation – possibly also to tooling
(i) Resistance to ionising radiation up to the doses anticipated - if this is a design requirement

It is unlikely that a single resin system will meet all of these criteria and inevitably, a compromise is required. While it is necessary for designers of superconducting magnets to take into account the technology of impregnation and insulation materials as they are today, there are also opportunities to blend and formulate materials for specialist applications.

4.1 Viscosity, Useable life and Toughness

The ‘useable life’ of a mixed resin / hardener system is defined by many suppliers as the time for the viscosity to double, at the operational temperature, from that of the initially mixed value. This loose definition doesn’t provide guidance for magnet engineers and the ‘useable life’ required would depend very much on the details of the magnet system that is to be vacuum impregnated.

There is not a simple definition of ‘useable life’ since this is usually defined by the needs of the particular application. Essentially, ‘useable life’ is the time for the viscosity to rise to a level at which it is no longer ‘suitable for purpose’ and this point will be different for different applications.

The viscosity achieved immediately after mixing resin and hardener is influenced by temperature, a higher temperature lowers this initial viscosity but also increases the reaction rate and so shortens the usable life. The ‘useable life’ is usually quoted ‘at the processing temperature’, which is an optimised temperature to maximise the working life while achieving a sufficiently low viscosity for the work involved.

The mass of resin involved may also have a significant influence on the working life. Preparing a large batch of mixed resin and hardener may reduce the working life of the material relative to resin prepared by automatic proportioning and mixing in a dedicated resin dispensing machine.

The viscosity of the resin system will determine the rate of flow of the resin into the magnet and the extent to which the resin will flow within the structure. The time for which the resin remains ‘useable’ is determined by the rate of change of viscosity at the processing temperature and the value that is defined as the ‘upper limit’ for the process.

Hacker et al [4] described low viscosity as 20 - 100 mPa.s and the upper limit for flow was defined by Green et al [5] as 500 mPa.s

More generally, it is regarded that the upper limit of viscosity for impregnation is reached when the resin viscosity has risen to 1000 mPa.s providing the initial viscosity is less than 200MPa.s. Therefore the time for the viscosity to rise from its initial value (at the prevailing temperature) to 750 mPa.s may be termed the "useable life". However, there is not a fixed and immutable value for an upper limit to viscosity, since this will depend on largely on the nature of the coil.

The rate of change of viscosity, at a fixed temperature for an epoxide resin / hardener system (with accelerator if appropriate) can be determined and from this the ‘useable life’ may be estimated.

In general, for impregnation, high addition, low viscosity hardeners, such as some of the acid anhydrides, form the most efficient impregnating systems i.e. low initial viscosity and long ‘useable life’. In order to minimize the gel / cure temperatures for these systems, an accelerator is generally
used and the rate of addition of such a component will strongly influence the rate of viscosity change (Figure 1).

If the upper limit of viscosity is taken as a conservative 500 mPa.s, the highest level of added accelerator shows a useable life of ~17 hours and the lowest >35 hours. Figure 1 shows the rate of change of viscosity of diglycidyl ether of bisphenol F (DGEBA) resin with MTHPA acid anhydride hardner (typically, Huntsman HY 917) and with varying levels of added accelerator.

4.2 Toughness
Most anhydride cured resins are inherently brittle – along with many other curing agents that have long ‘useable life’ and therefore require elevated temperature curing. An aliphatic amine hardener, typically Huntsman HY 5922, generally known as “D400’ or POPDA – (Polyoxypropylene diamine) when combined with DGEBA resin, has outstanding thermal shock resistance and has found widespread use for the impregnation of superconducting magnets, notwithstanding the relative short ‘useable life’ (Figure 2).

Figure 1. Viscosity Change of DGEBA Resin with Acid Anhydride Hardener and Various Levels of Accelerator (Huntsman DY 073-1)

Figure 2. Change in Viscosity at 40°C of DGEBA Resin with Curing Agent POPDA
Many applications have used raised injection pressures to increase the speed of resin flow into the structure. Tensile and compressive properties of the brittle acid anhydride cured resins and the tough POPDA cured resin system are similar at 4K.

Colyer et al [6] measured the work of fracture of epoxy resins and reported that for the tough POPDA system the work of fracture was approximately a factor of five greater at 4K than the acid anhydride cured system and showed how this parameter could be useful in defining differences in the ‘cracking potential’ of resins.

The work of fracture was used by Evans [7] to estimate the ‘allowable’ thickness of resin, that would not crack due to differential thermal movements, when restrained between conductors in a superconducting coil.

The work of fracture of epoxy resins is an important parameter in assessing the cracking potential of resin in a given i.e. if the stored strain energy in the resin exceeds the work of fracture, then there is a possibility of forming cracks:

\[
\frac{\sigma^2 t}{2E} \geq 2\gamma_p
\]  

(1)

where \( \sigma \) is the stress, \( t \) is the material thickness, \( \gamma_p \) is the work of fracture and \( E \) is Young’s modulus.

Equation 1 may be re-written to define the minimum thickness of resin - at the given stress - which will have the necessary stored strain energy for crack formation

\[
t \geq \frac{4E\gamma_p}{\sigma^2}
\]  

(2)

The stress in the resin when constrained between conductors in two dimensions may be simplistically estimated from:

\[
\text{Stress} \ (\sigma) = \frac{\int_{4K}^{300K} E \alpha \ dT}{1-2\mu}
\]  

(3)

Where \( \mu \) = Poisson’s Ratio and is taken to have the value of 0.3

A calculation based on equation (3) and using ‘average’ values for young’s modulus can only approximate to the true condition because both \( E \) and \( \alpha \) vary with temperature and the value for Poisson’s ratio is uncertain. In addition, the calculation does not take into account strain induced in a resin due to the volume change during cure, which in itself could lead to damage during cooling from the cure temperature. However, even this simplistic estimation of stress is a useful starting point in trying to understand and predict the overall effect of cooling the resin/conductor combination.

Using the above equations to calculate the induced stress and from that estimate the ‘maximum allowable thickness between conductors for two resin systems:

(i) A ‘brittle’ acid anhydride cured DGEBA resin

(ii) The same resin cured with POPDA (D400)

Young’s Modulus (E) is similar at 4 K for the two resins (3000 MPa at RT and 6000 MPa at 4K) - and is “averaged” over the temperature range. The contraction coefficient (\( \alpha \)) is also taken to be “constant” and is derived from the total contraction (RT to 4K) divided by 290 and subtracting the contraction coefficient for copper (1.0 x 10-5/K) from that of the resin (3.8 x10-5/K). The temperature range (dT) is taken as the difference between the Tg and the operational temperature (4K).

Tg for Resin (i) is taken as 120°C , (dT ~390K) and for resin (ii) as 40°C (dT ~ 310K)

For resin (i):

\[
\text{Stress} \ (\sigma) = 124 \text{ MPa}
\]

and for resin (ii)

\[
\text{Stress} \ (\sigma) = 90 \text{ MPa}
\]
The work of fracture of resin (i) was estimated [6] as 72J/m² and for resin (ii) as 360 J/m².

Using these values in equation 2 to estimate the maximum allowable thickness (E and σ in N/m²):

\[
\text{Resin (i)} \quad t = 8.4 \times 10^{-5} m (0.084 \text{ mm})
\]

\[
\text{Resin (ii)} \quad t = 8.0 \times 10^{-4} m (0.8 \text{ mm})
\]

To increase toughness for low temperature applications, there is a range of additives that may be used with epoxies that fully react into the system. These materials are preferred to other diluents that literally just dilute the resin and ‘plasticize’ by their presence among the long chain molecules of the resin. Other so called flexibilizers have only one reactive group within the molecular structure and these function by the one reactive group combining with the growing epoxy chain, so preventing further growth of that chain. i.e. these are “chain stoppers”, limiting the molecular weight of the cured system and limiting cross-link density.

In Figure 3 details are presented of DGEBF resin but cured with an aromatic amine (DETD – typically Huntsman HY 5200). The unmodified resin / hardener system offers a high degree of radiation stability and shows a ‘useable life’ of ~7 hours (at 50°C), based on an ‘upper limit of viscosity of 500mPa.s.

When the DGEBF resin is blended with a flexibilising resin, polypropylene glycol diglycidyl ether (PPGDGE, typically Dow Resin DER 732 a di-epoxide), not only does the toughness increase dramatically but the rate of change of viscosity is reduced so that the ‘useable life’ of the widely used “60/40” blend of DGEBF / PPGDGE is > 30 hours [8].

The resin PPGDGE and curing agent POPDA both have the same repeating ‘polyoxypropylene’ unit in the structure and it is this feature that confers the toughness on resin systems.

Evans and Zhang [9] reported work of fracture on the resin systems defined in Figure 3 and these data are presented in Table 1. Results for DGEBF /POPDA at 4K differ by approximately a factor of two from that in [6] but the value for an anhydride cured resin is similar. The blend of DGEBF with PPGDGE (60/40 is shown to have a work of fracture three times that of the DGEBF / MTHPA system at RT and nearly twice that at 4K.

![Figure 3. Effect of Added Flexibilising Resin (PPGDGE) on ‘Useable life’ of DGEBF Resin Cured with DETD (Ratios are in pbw of DGEBF/PPDGE/Hardener DETD)](image-url)
Table 1. Work of Fracture of Resin Systems at Three Temperatures [9]

| Resin System | RT (J/m²) | 77K (J/m²) | 4.2K (J/m²) |
|--------------|-----------|------------|-------------|
| DGEBF/POPDA  | 103       | 199        | 61          |
| DGEBF/MTHPA* | 899**     | 525        | 67          |
| DGEBF/PPGDGE | 2241**    | 396        | 13          |
| DGEBF/PPGDGE | 353       | 359        | 10          |
| DGEBF/PPGDGE | 310       | 249        | 9           |
| DGEBF/PPGDGE | 205       | 110        | 3           |

* MTHPA – methyl tetrahydrophthalic anhydride – typically Huntsman HY 917
** Results at RT are geometry dependent - no single value for the work of fracture

A ‘low’ Tg (but sufficiently raised to maintain acceptable room temperature properties) along with a high work of fracture, combine to increase the cracking index. Calculated values for the induced thermal stress (equation 3) and ‘cracking index’ (maximum allowable thickness without cracking – equation 2) are presented in Table 2.

Table 2. Calculated “Cracking Index’ at 4K For Four Resin Systems

| Resins       | Thermal Stress (MPa) | Cracking Index (mm) |
|--------------|----------------------|---------------------|
| DGEBA / POPDA | 4K                   | 0.36                |
| DGEBA / MTHPA | 124                  | 0.07                |
| DGEBA / PPGDGE | 124                  | 0.07                |
| DGEBA / PPGDGE | 101                  | 0.19                |

5. Outgassing and Resin Flow & Distribution

5.1 Coil Outgassing

The importance of resin de-gassing was explained in [1] and [2]. The only additional comment here is to emphasize that a coil structure is a very efficient de-gassing medium. When the advancing resin front is spread as a thin film, with many nucleation points, if the resin has not been fully out-gassed, any residual gas will certainly be release into the coil structure. The need to ensure complete de-gassing of resin prior to impregnation cannot be over-stated.

Another procedures include purging the evacuated coil structure with a dry gas as an aid to accelerating the removal of chemically absorbed water molecules. Frequently dry nitrogen is specified for this purpose but others prefer to use carbon dioxide on the basis of a claim that this gas is more soluble in resin than nitrogen. Increased solubility was said to be an advantage because pockets of the purge gas that may be trapped within the structure would more readily dissolve in resin and so minimise the risk of void formation. The comparative figures for the solubility of nitrogen and CO₂ on which the above conclusion was based were not available.

Adsorption of species onto a surface may take place as either: (a) Physical adsorption or (b) chemical adsorption. When physical adsorption occurs, gas molecules are attracted and held by weak van der Waals forces with low binding energies (<40 kJ/mol). Adsorption does not require energy for
activation. When chemical adsorption occurs, there is a definite (exothermic) chemical reaction between the gas and the absorbing surface.

The binding energies for chemically absorbed species can vary anywhere between 80 kJ/mol and 800 kJ/mol. When the binding energies are low (physical adsorption) the rates of desorption are high and the desorbed molecule is readily removed by pumping. When binding energy is high, the rates of desorption are low.

Elsey [10] found that when pumping metal structures, de-gassing occurred quickly but once the pressure had been reduced to a low value, the major residual gases being removed were water vapour and carbon dioxide.

Work by Bakaeva et al [11], compared the adsorption of the technically non-polar CO\textsubscript{2} with non-polar argon on glass surfaces. It was found that the pattern of adsorption of CO\textsubscript{2} mirrored that of water vapour but there was no indication of adsorption of non-polar gases such argon or nitrogen.

Siperstein and Gubbins [12] measured the heat of adsorption of argon and krypton on glass at 14 – 18 kJ/mol at room temperature.

Baptist and Levy [13] confirmed a chemical reaction between a freshly broken glass surface and CO\textsubscript{2}.

Battjes et al [14] showed that when exposed to the gas, surfaces that were coated in amino-silane coupling agents, became coated with a strongly adhering layer of a white powder. This powder was shown to be an ‘amine carbamate’, the product of the interaction between the amine and CO\textsubscript{2}.

Details of the reaction between CO\textsubscript{2} and Bisphenol A epoxy resin were documented by Khoshkish et al [15]. It was demonstrated that carbon dioxide reacted with epoxy groups to form ‘cyclic carbonates’.

The apparent high solubility of carbon dioxide in water is well known and this is due to chemical action (to form carbonic acid) and not just simple solubility. Carbon monoxide is around a factor of 60 less soluble in water than carbon dioxide.

5.2 Resin Flow & Distribution

The rate of resin flow through packed glass fabric is governed by the permeability of the glass fabric, the resin viscosity and pressure difference. The volumetric flow rate, \( A \) the cross-sectional area, \( \eta \) the fluid viscosity, \( K \) the permeability, and \( \Delta P \) the pressure difference over the length \( L \). Permeability, in turn, is defined as that property of a porous material, which characterizes the ease of fluid to flow through the material by the applied pressure gradient.

The complicated and non-uniform micro-structure of the compacted insulation, where the local permeability may vary by more than an order of magnitude, will lead to non-uniform flow of resin (race tracking) and the presence of un-wetted regions in the coil as the resin flows past.

The manner in which the permeability of compacted glass cloth varies with the degree of compaction was demonstrated by Hammond [16] and it was shown that permeability of glass fabric compacted to 60 volume % was a factor of approximately twenty less than the same fabric compacted to 40 volume %.

6. Closing Remarks

The various methods of coil bonding with epoxy resins, wet-winding, use of pre-pregs and vacuum impregnation VPI have been reviewed and the various options within the VPI process considered in detail. To complement these various procedures, the material requirements have been considered and discussed, including resin viscosity and the concept of ‘useable life’ outlined.

The resistance of resins to cracking at low temperatures has been discussed along with a summary of some published data, to show how the induced thermal stress may be minimized and toughness maximized by modification of resin formulation.

Factors influencing resin flow into compacted insulation structures have been outlined and evidence is presented that carbon dioxide reacts with resin and is chemically adsorbed onto most solid surfaces. It is confirmed that carbon dioxide is not more soluble in resin than dry nitrogen and apparent solubility of the gas has been confused with a chemical reaction.
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