FERMILAB-CONF-22-518-TD

ICMC 2021 IOP Publishing

IOP Conf. Series: Materials Science and Engineering 1241 (2022) 012010 doi:10.1088/1757-899X/1241/1/012010

Characterization of Candidate Insulation Resins for Training Reduction in High Energy Physics Magnets

A E Haight[1], M M Haynes[1], S T Krave[2], J L Rudeiros-Fernandez[3] and T Shen[3]

[1] Composite Technology Development, Inc., 2600 Campus Drive, Suite D, Lafayette, CO 80026

[2] Magnet Systems Department, Mail Stop 315, Fermilab, PO Box 500, Batavia, IL 60510-5011

[3] Lawrence Berkeley National Laboratory, 1 Cyclotron Road Mailstop 47R0112, Berkeley, CA 94720

Email: andrea.haight@ctd-materials.com

Abstract. Superconducting magnets are critical components in particle accelerators and are used to generate and sustain the large magnetic fields needed for High Energy Physics programs. One significant issue with current epoxy insulated Nb_3Sn magnets is the long training process required before stable magnet performance can be realized. It is believed that training can be significantly reduced by addressing magnet quenching through improvements in the epoxy electrical insulation. In this work, two approaches for insulation modification have been undertaken: (1) addition of thermally conductive fillers to help with quench management and (2) development of insulation resins with high strain capability at cryogenic temperatures. This paper will discuss the characterization of these insulation systems to verify their performance prior to evaluation in subscale Nb_3Sn canted cosine theta accelerator dipole magnets.

1. Introduction

Superconducting magnets are critical components in particle accelerators and are used to generate and sustain the large magnetic fields needed high energy physics experiments. Nb_3Sn magnets are the current focus for upgrades to the Large Hadron Collider and are being considered for future, larger machines such as the 100 TeV class Future Circular Collider (FCC). One significant issue with current epoxy insulated Nb_3Sn magnets is the long training process required before stable magnet performance can be realized. It is believed that training can be significantly reduced by addressing magnet quenching through improvements in the epoxy electrical insulation. Modification of epoxy insulation for improved magnet performance has been reported extensively in the literature [1]-[7]. The potential of alternative systems has also been discussed [8].

The main objective of this work is to identify the new resin systems with the most potential for reducing the number of training cycles required for Nb_3Sn superconducting magnets. Two approaches are being considered: (1) addition of thermally conductive fillers to CTD-101K type insulation to better manage heat dissipation during quench and (2) development of toughened resin systems that will exhibit reduced cracking during cooling and energization of the magnet. This paper discusses the characterization of these resin systems in preparation for their evaluation in subscale Canted Cosine Theta (CCT) [9] magnets.

Content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI.
Published under licence by IOP Publishing Ltd

This manuscript has been authored by Fermi Research Alliance, LLC under Contract No. DE-AC02-07CH11359 with the U.S. Department of Energy, Office of Science, Office of High Energy Physics.

2. Experimental Methods

2.1. Resin Viscosity
Resin viscosity was determined using a Brookfield DV2T viscometer with automated data acquisition. In order to be classified as suitable for vacuum pressure impregnation (VPI), a viscosity below 300 mPa-s and working times of at least 4 hours were sought.

2.2. Mechanical Testing

2.2.1. Neat Resin Tensile Test
Neat resin tensile tests of high strain resin candidates were conducted in accordance with ASTM D638 using a Type IV strain-gaged specimen with a gage length of 35.6 mm. Specimens were tested at both 77K and 293K.

2.2.2. Compression and Short Beam Shear Tests
Laminates were fabricated using Style 6781 S2 glass fabric and the resins developed during the course of the program. Resins included: CTD-101K (reference), CTD-155, CTD-155GN, and CTD-701X. All laminates were fabricated using a vacuum pressure impregnation (VPI) process using a shimmed closed mold and the number of plies and mass of resin necessary to produce laminates with a nominal 50% fiber volume fraction with a laminate thickness of approximately 3.2 mm. Resin transfer was along the length of the laminate. Impregnation pressure was adjusted as needed to accommodate the higher volatility of the CTD-701X resin system. Fiber volume fraction was determined using weights of glass and resin in the laminate.

Short beam shear testing was conducted in accordance with ASTM D2344, nominal specimen dimensions were 28 mm x 6.35 mm x 3.2 mm with a span of 15 mm. The dominant failure mode in all cases was interlaminar shear.

Compressive properties were evaluated using methods established in evaluating ITER resin candidates [10]. Nominal specimen dimensions were 6.35 mm x 6.35 mm x 3.2 mm. Laminates were fixtured using 25 mm platens and tested in the through-thickness direction at both 77 K and 293 K. This configuration was chosen in part because it more closely represents conditions observed in magnet applications. Through-thickness deflections were determined using extensometers. Modulus is reported as “chord modulus,” which we define as the slope of the chord drawn between any two specified points on the stress-strain curve; chord modulus calculations are recommended for non-linear materials [11].

2.3. Thermal Expansion
Thermal expansion tests were performed on two or three axes of at least three specimens of each type. Uni-axial strain gages were mounted on the test specimens, one in each direction to be evaluated. An identical strain gage was mounted on a Copper NIST reference standard. A single silicon diode temperature sensor is mounted between one test specimen and the reference material, and all specimens reside within the same vertical plane during testing. The specimens and standards were cooled to cryogenic temperature using either liquid helium or liquid nitrogen and held at cryogenic temperature for a minimum of 5 minutes prior to initiation of the warming cycle. Thermal expansion is measured via strain variance relative to the NIST standard. Thermal expansion data is normalized relative to room temperature and averaged across the three data files.
Shear compression testing was conducted at 77K using the method described in the work of Simon, *et al* [12]. Specimens were fabricated by placing plies of S2 glass cloth between two grit-blasted steel buttons in the impregnation fixture, followed by impregnation with the resin of choice. Steel was chosen as the substrate to facilitate comparison to prior data. Specimens were then placed at an angle in the test fixture for testing. Several angles were tested for each resin type to establish the “envelope” for the system.

IOP Conf. Series: Materials Science and Engineering 1241 (2022) 012010 doi:10.1088/1757-899X/1241/1/012010

3. Results and Discussion

3.1. Evaluation of Thermally Conductive Resin Systems

Thermally conductive resin evaluation focused on a system utilizing the CTD-155 resin system with varying levels of nanoscale filler. All early testing was conducted at the highest possible volume loadings of fillers (35 volume percent) but lower filler contents were evaluated due to the lower resin viscosities that can be achieved. Filled resins were formulated at 15, 20, and 35 volume percent (vol %) of nanofiller and were evaluated for viscosity/pot life and thermal conductivity.

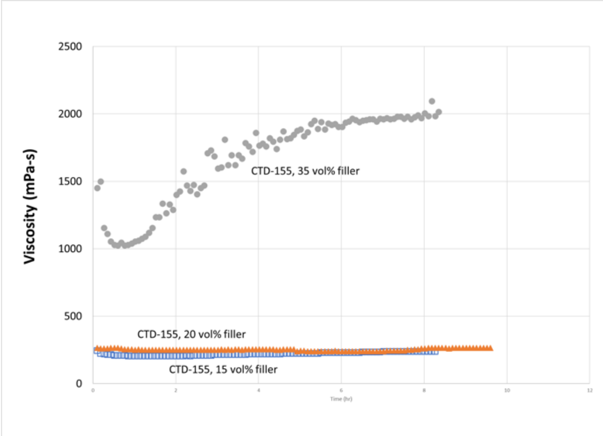


Figure 1. Pot life at 60°C for CTD-155 with various levels of nano-alumina filler.

Viscosity curves obtained at 60°C are presented in Figure 1. The viscosity of the 35 vol % formulation is not at all suitable for the commonly employed VPI processes, whereas both the 20 vol % and 15 vol % systems are suitable.

The thermal conductivity of these resin systems was also verified by Thermtest, Inc. (Fredericton, NB Canada) using the Physical Properties Measurement System (PPMS) by Quantum Design to ensure that significant thermal conductivity was not lost relative to the 35 vol% system. The thermal conductivity of the 20 vol % specimen was 0.4 W/m-K at 90 K (the lowest temperature available at the time of testing), corresponding to that of the 35 vol % system measured during an earlier phase of the program. It is recognized however, that this temperature is not necessarily relevant to a magnet application and that thermal conductivity enhancement at 4K is extremely difficult. Additional efforts on characterizing the thermal conductivity of these materials are described in the paper by Adams, *et. al* in these proceedings [13].

3.1.1. Laminate Evaluation. The 20 vol % CTD-155 resin, hereafter referred to as CTD-155GN, was then used to manufacture a laminate reinforced with S2 glass (50% fiber volume fraction) using a standard VPI process. Generally, the concern using filled resin systems is that the particles will get filtered out by the reinforcement; nanometer-sized particles were used for this work in the hope that the filtering issue can be avoided. This does indeed appear to be the case; inspection of the laminates using optical microscopy shows a uniformly impregnated laminate with no evidence of particle filtering, as shown in Figure 2.

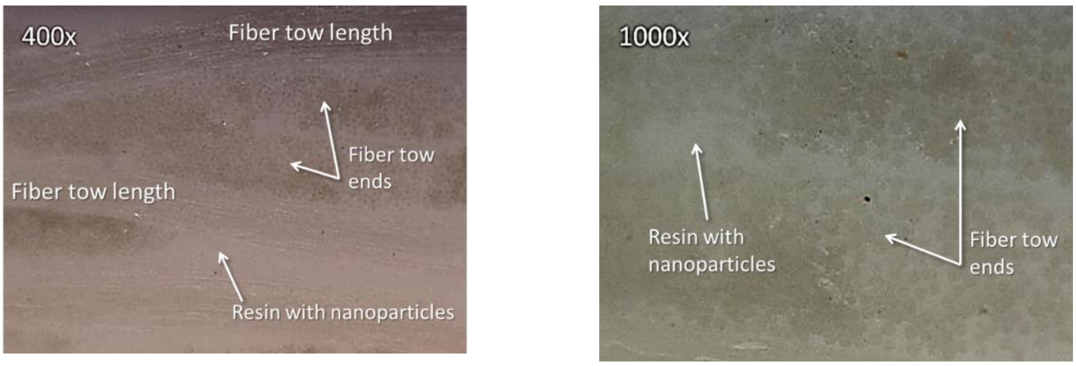


Figure 2. Optical microscopy of S2 glass/CTD-155GN laminate. Notice the uniformity of the laminate; no particle separation visible, resin area is a uniform milky white color with no “clear” spots or voids.

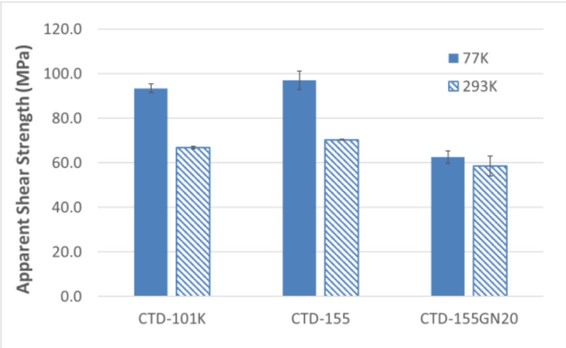


Figure 3. Comparison of shear performance of S2 glass laminates using CTD-155 resin variants. Data for CTD-101K laminates is included for comparison.

Mechanical properties of the laminates were then evaluated at 77 K and at 293 K; results of the short beam shear testing are presented in Figure 3. While the apparent shear strength of the nanoparticle-filled laminate is somewhat reduced relative to laminates manufactured with the unfilled resins, the resulting strength of 60 MPa is more than sufficient for the intended magnet application; modeling indicated that a minimum shear strength of 35 MPa would be required in the insulation to withstand magnet shear stresses [14].

3.1.2. Shear Compression Testing. Since magnet stresses are not strictly limited to shear or compressive stresses, we also conducted shear-compression testing for new resins under development in this work. This test is designed to more accurately mimic the performance of the insulation system in a magnet configuration. Typically, several angles are evaluated to define the shear-compression “envelope” for the insulation system. Results for the CTD-155GN system compared to the often-used CTD-101K insulation system are shown in Figure 4.

The shear compression performance for the CTD-155GN laminate is a bit lower than that of the CTD-101K laminates. We believe that this is due to the larger fraction of adhesive failures in the CTD-155GN specimens, where it appears that the particle loading may have an influence on adhesion to the steel substrates. In contrast, most of the CTD-101K failures were cohesive in nature. The photograph inset of Figure 4 shows both cohesive (A1, A4) and adhesive (A3, A6, A8) failures of the CTD-155GN specimens.

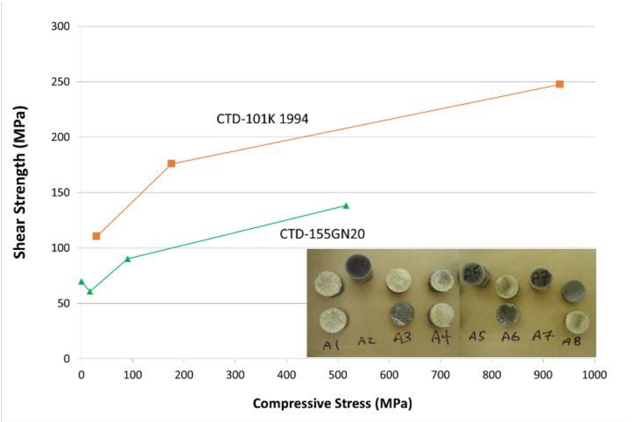


Figure 4. Shear compression test results for S2 glass/CTD-155GN as compared to results for S2 glass/CTD-101K. Inset photograph shows failure surfaces in the test specimens.

3.1.3. Thermal Expansion. Thermal expansion of the CTD-155GN was evaluated over the temperature range 77-293 K. Thermal expansions of the unfilled CTD-155 resin (neat resin and laminate) were included for comparison. The results for both S2 fabric reinforced laminates and neat resin are provided in Figure 5.

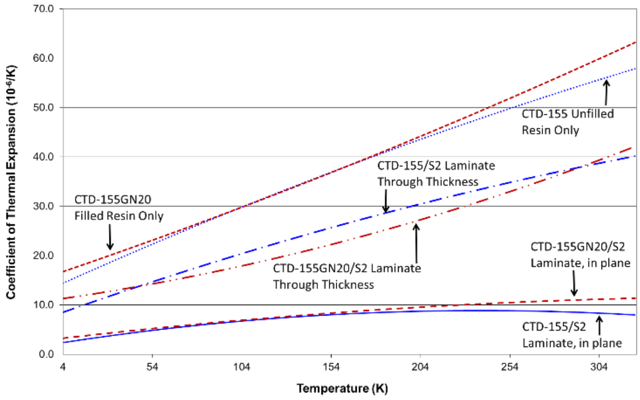


Figure 5. Comparison of thermal expansion for CTD-155 and CTD-155GN20 laminates and unreinforced resins.

As expected, the in-plane thermal expansion of the laminates is lower than the through-thickness thermal expansion of the laminate and both are lower than that of the neat resins. Interestingly, the thermal expansion of the unfilled resin and the nanoparticle-filled resin are not significantly different; one might expect that the filled resin would show lower thermal expansion. It may be that the filler content is lower than is normally used to manage thermal expansion, or that nanofillers do not have as large an effect on thermal expansion as micron-scale fillers, or a combination of the two. Additional studies are underway to better understand these results.

3.2. Evaluation of High Strain Resin Candidates

Several higher strain resin systems were developed during this program in an effort to reduce long training cycles in magnet systems. The systems evaluated to date and a few basic characteristics are listed in Table 1.

Table 1. High strain resin candidates for magnet applications.

Name	Description	T_g, °C (tan δ)	Viscosity at 60°C (mPa·s)	
			Initial	**7 hours**
CTD-101K	Baseline anhydride-cured epoxy resin	150 (avg QA)	70	100
CTD-103K2	Long pot life 2-part formulation similar to CTD-101K	141	61	61.5 (4.5 hr)
CTD-103LT	Lightly toughened version of CTD-103K2	126	24	71
CTD-155	Anhydride-cured epoxy with reactive rubber toughener	147	125	127
CTD-701X	Extremely low viscosity, tough [15] polyolefin resin system	131	25 (@ 25°C)	70 (90 min, 25°C)
CTD-7.1E	Low viscosity, toughened amine-cured epoxy resin system	72	225	Approx. 2hr

Of these, the most interesting system is CTD-701X, which showed neat resin elongation at failure as high as 5% at 77 K (Figure 6), whereas most of the other systems (e.g., CTD-101K) exhibited elongation at failure on the order of 2% at 77 K.

The following sections discuss a more comprehensive characterization of the CTD-701X system to support the production and testing of a subscale CCT magnet.

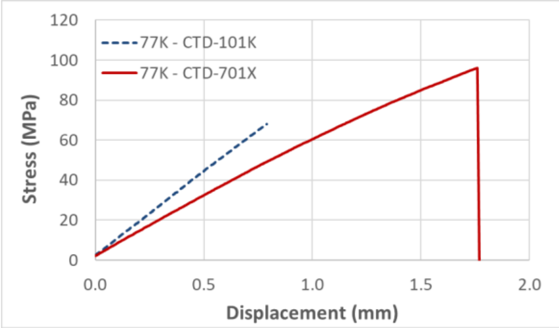


Figure 6. Neat resin stress-deflection curves at 77 K for CTD-101K and CTD-701X

3.2.1. Viscosity and Pot Life

The viscosity and pot life of the CTD-701X were evaluated at 25 °C and the results are shown in Figure 7. The initial formulation showed a significant acceleration in the viscosity increase beginning at approximately 3 hours. An alternative catalyst, that requires an elevated temperature cure (1 hour at 40°C followed by a 1 hour post cure at 120°C), was then evaluated at 25 °C. In this case, the viscosity remained stable over more than 8 hours.

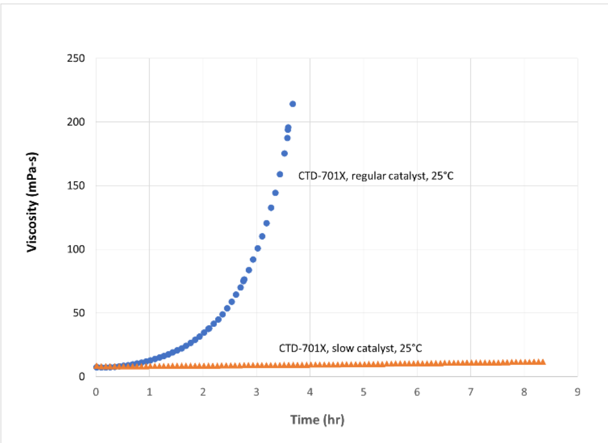
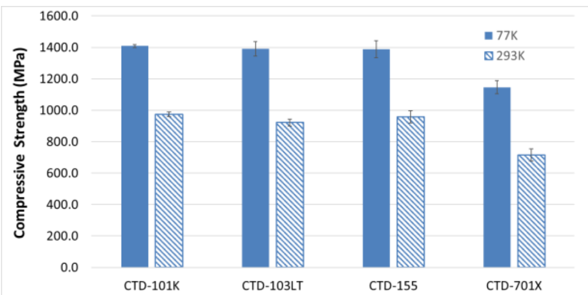


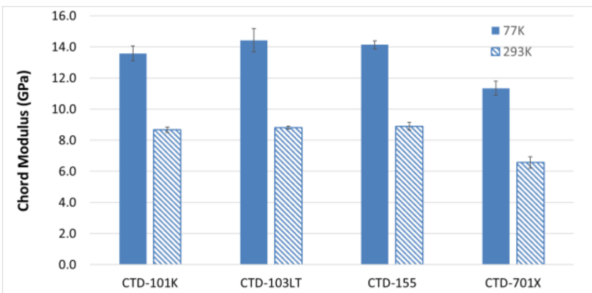
Figure 7. Pot life of CTD-701X at 25°C with two different catalyst systems.

3.2.2. Laminate Mechanical Properties. Shear and compressive properties of laminates fabricated with the high strain candidate systems were also evaluated. Compressive properties are shown in Figure 8 and shear properties are shown in Figure 9.

The compressive properties show very little variation regardless of the resin system. However, there were some differences noted in the shear properties. The epoxy systems (CTD-101K, CTD-101LT, and CTD-155) are largely equivalent, but the CTD-701X has significantly lower shear strength, likely due to the different chemistry (polyolefin vs epoxy) and potentially reduced compatibility with the fiber sizing in the glass laminate. However, the shear strength does appear to be sufficient for the subscale CCT magnet application based on modeling results as described above.



(a)



(b)

Figure 8. Compressive properties of S2 glass-reinforced laminates fabricated with high strain resin candidates (a) compressive strength and (b) chord modulus. Data for CTD-101K laminates are included for comparison.

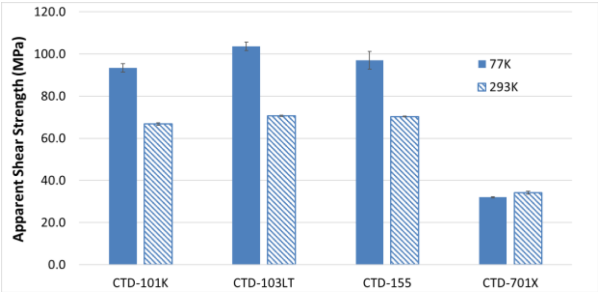


Figure 9. Apparent shear strength of S2 glass-reinforced laminates fabricated with high strain resin candidates. Data for CTD-101K laminates are included for comparison.

3.2.3. Shear Compression Testing. Shear compression testing as described in Section 3.1.2 was also conducted for laminates using the CTD-701X resin; the results are shown in Figure 10. The CTD-701X results are slightly lower than those observed for the CTD-101K system but are higher than those discussed earlier for the CTD-155GN system. All CTD-701X specimens showed cohesive failures.

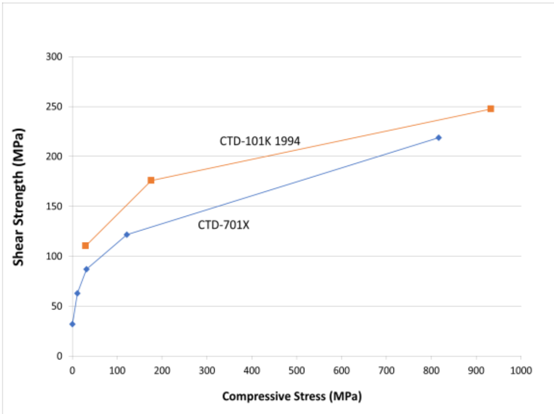


Figure 10. Shear compression test results for S2 glass/CTD-701X as compared to results for S2 glass/CTD-101K.

3.2.4. Thermal Expansion. Thermal expansion of the CTD-701X was evaluated between 4 K and 293 K. The results for both S2 fabric reinforced laminates and neat resin are provided in Figure 11.

The thermal expansion of this resin system, both with and without reinforcement, is somewhat higher than that observed for the commonly used epoxy resins, including CTD-101K. This trend is also expected in the through-thickness direction, which was not evaluated in this work. The presence of the S2 glass reinforcement does significantly curtail thermal expansion of the resin, however, so there is no reason to expect this material to be problematic in a magnet application provided that care is taken to avoid significant pockets of neat resin in the finished magnet.

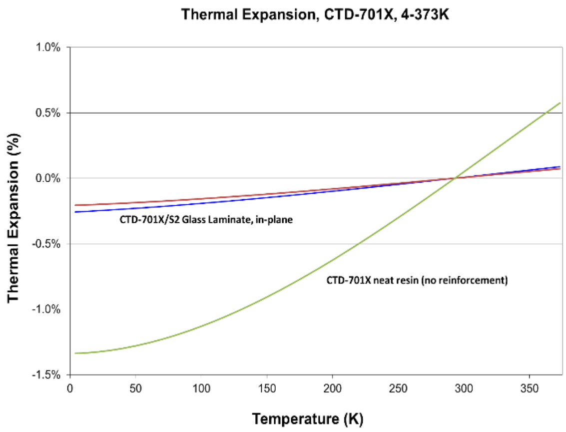


Figure 11. Thermal expansion of CTD-701X with and without reinforcement.

3.3. Results of Stack Testing [15]

Additional work evaluating these materials in thermal cycling as well as in four and ten-stack configurations also supported recommending materials for subscale CCT evaluation. Two particular results stood out from that work: (1) thermal cycling resulted in only one sample of six exhibiting any cracking over 10 thermal cycles and (2) the CTD-701X resin system in a cable-type configuration (4-stack) showed a higher short beam strength (160% at 77 K) relative to CTD-101K in a comparable configuration. Note that this is different from the laminate-only result presented here and was a large contributor to the decision to pursue subscale CCT magnets with this resin system.

4. Conclusions

This paper has presented results of characterization activities for two different resin systems developed as candidates for reducing training in high energy physics magnets. This characterization effort focused on understanding the resin systems as well understanding the properties of S2 glass laminates fabricated with these resins so to better understand how to apply them in a subscale CCT magnet system.

Based on the testing described herein, the following conclusions can be made for the thermally conductive system CTD-155GN: (1) it is possible to make a high quality composite using a resin system with 20 vol% nanoscale filler (2) thermally conductive formulations are unlikely to offer any improvements in heat dissipation when operating at 4 K, but are valuable for high-temperature superconducting magnets working above 20 K, and (3) addition of nanoscale fillers does not offer any significant improvement in thermal contraction performance for the CTD-155GN system. Thus, testing of this material in a Nb-Ti or Nb_3Sn magnet application is not justified at this time. However, the application of this insulation system for magnets operating at higher temperatures (e.g., magnets based on high temperature superconductors) might be worth considering.

For the high strain system CTD-701X, lower mechanical properties were noted. The compression modulus and strength are lower but are sufficient for superconducting accelerator magnets, for which the design transverse pressure to Rutherford cable is now no larger than 200 MPa. The interlaminar shear strength and the shear strength determined by shear/compression testing are also lower and in the territory of potentially causing more interface failures, and perhaps quench, in superconducting accelerator magnets. However, it is felt that this system provides a critical and useful evaluation of the importance of this parameter on the quench training of superconducting accelerator magnets. The work on four and ten-stack configurations described by Krave *et al*. [15] and summarized in Section 3.3 also supports this evaluation. The extreme resistance to cracking and improved short beam strength in stacks described by Krave *et al*., coupled with favorable processing (low viscosity and long pot life, resin transfer at ambient temperature) as shown in this work, led to a decision to evaluate this resin system in a subscale CCT magnet. The results of the CCT magnet performance will be reported elsewhere.

5. References

[1] Yin, S, Arbelaez, D, Swanson, J. and Shen, T, 2019 *IEEE Trans. Appl. Supercond.*, **29(5)**, 7800205, doi: 10.1109/TASC.2019.2898124.
[2] Evans, D. and Morgan, J. "Epoxide resins for use at low temperatures," in *Nonmetallic Materials and Composites at Low Temperatures*. Boston, MA, USA: Springer, 1982, pp. 73–87.
[3] Fabian, P, Munshi, N and Denis, R. 2002, *AIP Conf. Proc.,* **614**, 295–304.
[4] Reed, R, Evans, D and Fabian, P, "Development of a new resin system for the US ITER central solenoid model coil," in Advances in Cryogenic Engineering Materials. New York, NY, USA: Springer, 2000, pp. 227– 234.
[5] Reed, R P and Evans, D, 2004, *AIP Conf. Proc*., **711(1)**, 209– 216.
[6] Munshi, N A *et al.*, 2013 *IEEE Trans. Appl. Supercond.,* **23(3)**, 7700104.
[7] Zhang, Z and Evans, D, "Low-temperature deformation and fracture behaviour of epoxide resins," in Advances in Cryogenic Engineering Materials. New York, NY, USA: Springer, 2000, pp. 251–257.
[8] Toplosky, V J and Walsh, R P, 2006, *AIP Conference Proceedings* **824**, 219.
[9] Arbelaez,D, Bogdanof, T, Brouwer, L, Caspi, S, Dietderich, D, Rudeiros Fernandez, J L, Ferracin, P, Gourlay, S, Hafalia, R, Krutulis, M, Marchevsky, M, Maruszewski, M, Prestemon, S, Reynolds, M, Shen, T, Swanson, J, Teyber, R, Turqueti, M, Vallone, G and Wang, X, "Status of the Nb_3Sn Canted-Cosine-Theta Dipole Magnet Program at Lawrence Berkeley National Laboratory", submitted for publication in *IEEE Trans. Appl. Supercond.*
[10] Fabian, P E, Schutz, J B, Hazelton, C S, and Reed, R P 1994 *Adv. Cryo Eng*. **40**, 1007.
[11] https://www.admet.com/youngs-modulus-tangent-chord-modulus-testing-tips/#:~:text=Chord%20modulus%2D%20Slope%20of%20the,recommended%20for%20non%2Dlinear%20materials.

[12] Simon, N, Reed, R, and Walsh, R, 1992 *Adv. Cryo. Eng. (Mater)* **38,** 363.
[13] Adams, J, Haight, A E, Michael, P C, Bromberg, L, and Brisson, J G, *IOP Conference Series: Materials Science and Engineering, Advances in Cryogenic Engineering*, in press.
[14] Tengming Shen, private communication, August 20, 2020.
[15] Krave, S, Shen T, and Haight A. 2021, *IEEE Trans. Appl. Supercond.* **31(5)**, 400190.

Acknowledgments
The authors would like to acknowledge funding of this work through U. S Department of Energy Small Business Innovation Research (SBIR) Program, Grant No. DE-SC0018701.