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Authors

Cheng, DW
Dietderich, DR
Godeke, A
[et al.](#)

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Characterization of Insulating Coatings for Wind-and-React Coil Fabrication

D. W. Cheng, D. R. Dietderich, A. Godeke, A. R. Hafalia, M. Marchevsky,
S. O. Prestemon, P. K. Roy, G. Sabbi, and C. A. Swenson

Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

Abstract. Electrical insulation breakdown between conductor and coil parts and structures is a limiting factor in the performance of high-field magnets. We have evaluated various insulation coatings for possible application in both Nb₃Sn and Bi-2212 coil fabrication. Such coatings must be robust to maintain structural integrity and provide adequate voltage standoff after the wind-and-react coil fabrication process. Such processes are characterized by reaction temperatures of 650°C in an inert atmosphere for Nb₃Sn and 890°C in a pure oxygen atmosphere for Bi-2212, and down to cryogenic temperatures when coils are in service. We present a method of testing standardized samples and report the performance characteristics of oxide layers produced (or applied) by plasma-spray, surface conversion, and “paintable” coatings in common areas of voltage breakdown in coil parts. We also address material compatibility and durability during high-temperature heat treatment and cryogenic shock. Suitable coatings selected in the testing process will be instrumental in improving the performance of future wind-and-react coils.

Keywords: surface coatings, testing, adhesion, voltage breakdown.

PACS: 46.50.+a, 46.80.+j, 41.85.Lc, 74.25.Ld, 81.40.Np, 62.20.mm

INTRODUCTION

The Superconducting Magnet Program at the Lawrence Berkeley National Laboratory (LBNL) has a long history in the development of high-field superconducting magnets for accelerators and HEP colliders. As magnetic field requirements have increased, high-field magnet fabrication technology has matured from using NbTi superconductors to Nb₃Sn and high temperature superconductor (HTS) Bi-2212 type conductors in order to enable higher fields [1]. Due to their strain sensitivity, these latter superconductors require “wind-and-react” fabrication techniques and this fundamental change in magnet fabrication requires a coil’s structural materials to withstand extreme conditions throughout its lifecycle—from high-temperature reactions to cryogenic temperatures of 4 K and below.

While typical structural materials (stainless steels, titanium, etc.) have proven their ability to withstand these conditions, electrically insulating materials used in these coils have not always fared as well. In fact, despite having superconducting cables vacuum impregnated in an epoxy/fiberglass matrix, recent experience has shown that insulation breakdowns between the conductor and metallic coil structures are one of the limiting factors in the performance of magnets during testing [2].

As a result, in addition to optimizing fabrication processes to ensure more robust magnet coils, employing insulating surface coatings has also been explored as additional protection against voltage breakdown to magnet structures. These coatings must not only withstand the extreme conditions during the coil processing, but must also retain their integrity and insulating properties throughout all phases in the coil lifecycle. This paper presents a test methodology for insulating coatings that will aid in determining whether their performance characteristics are compatible with wind-and-react coil fabrication technology. Additionally, we report on preliminary results of some coatings that were tested and discuss possible directions for further research.

EXPERIMENTAL PROCEDURE

While many standardized test methods for measuring properties of surface coatings already exist in publications by organizations such as the American Society for Testing and Materials (ASTM), a unified test methodology particular to superconducting coil fabrication technology requires a framework in order to produce reliable data.

Based on the conditions present during the lifecycle of a coil, one can develop this framework for processes and measurements steps.

Coil Lifecycle

Table 1 shows the fabrication steps and environmental conditions present during the lifecycle of typical wind-and-react superconducting coils. For the coils mentioned herein a rectangular (or keystone) Rutherford type cable is used, which has been insulated with either a braided S2 fiberglass or alumina-silica (mullite) weave.

TABLE 1. Typical steps and conditions in the lifecycle of two types of superconducting magnet coils, Nb₃Sn and Bi-2212.

Process Step	Nb ₃ Sn-Type Conductors	Bi-2212-Type Conductors
1. Fabricate and insulate Rutherford cable	√	√
2. Wind 1st layer of coil	√	√
3. Matrix-cure in heat press	CTD-1202 @ 150 °C	n/a
4. Wind 2nd layer of coil	√	√
5. Package coil in reaction fixture	√	√
6. React coil	~650 °C in Argon	~890 °C in Oxygen
7. Vacuum impregnate with epoxy	CTD-101K @ 125 °C	CTD-101K @ 125 °C
8. Instrument coil (strain gages, voltage taps, etc.)	√	√
9. Electrical QA	Hipot to >1000V, L/R	Hipot to >1000V, L/R
10. Preload and cool-down magnet structure	<200 MPa compr. limit	<60 MPa compr. limit
11. Magnet test	<10 MPa tension	<10MPa tension

Steps 2 - 5 describe the winding and curing process where the insulated Rutherford cable is wound around a center pole to form the coil structure. The curing step incorporates a ceramic matrix reinforcement, CTD-1202, which is an inorganic polymer binder that basically “glues” the turns of a Cosine- Θ coil in place for the winding of the second layer as well as for handling. This step is generally not needed for block, or “double-pancake”, style coils.

The high temperature reaction cycle differs depending on which conductor is used. While the copper-based Nb₃Sn conductors need to be reacted in a pure argon atmosphere at around 650 °C, the silver alloy-based Bi-2212 type conductors require a high reaction temperature of 890 °C in a pure oxygen environment. These reacted coils must then be vacuum impregnated with epoxy to prevent movement due to Lorentz forces and to distribute the stresses during operation. This also creates a monolithic structure that can be handled safely without damage to the conductor. The standard epoxy used in this vacuum impregnation step is CTD-101K, which is cured at a temperature of 125 °C.

In operation, a superconducting magnet structure will be preloaded and then cooled down to liquid helium temperatures where increased preload takes place as the structure thermally contracts around the coils. When energized, the Lorentz forces of the magnet may at times overcome the compressive preload forces, causing the bond interface between the coil’s center pole and the cable turns to experience tensile and shear loading conditions. It has been suggested in [3] that tensile loads approaching 10 MPa cause breakage of the bond interfaces, sometimes to the point of complete separation.

Finally, when coil quenches occur during magnet training and/or operation, instantaneous voltage potentials of hundreds of volts are present between grounded structures and the superconductor, which must be withstood by the insulation and epoxy matrix in the coil.

Testing Matrix

Table 2 lists steps that are used to measure critical parameters in the performance of surface coatings and standards that may be applicable. These tests are described below.

After a surface coating is applied, the thickness of the layer needs to be measured. It is a critical parameter because uniformity and repeatability may affect the overall dimensional build quality of a magnet coil. Large variances on real parts could result in either not enough coverage or cause over-compaction of the conductor, which has been reported in [4] to be a factor in the performance of magnets.

An initial hi-pot test is performed at the same time in order to quantify the surface coating's dielectric strength. In fact, the hi-pot test is also repeated after several steps to determine how factors such as the high temperature reaction cycle affect the coating's dielectric strength.

The reaction environments of both types of superconductors present their own set of challenges. At 650 °C in argon, any hydrocarbon molecules that may be present in the system may carbonize—leaving behind a carbon residue that is conductive at room temperature. Additionally, it has also been reported that the CTD-1202 polymer binder reacts with the braided cable insulation during the high temperature reaction process [5], therefore, examining a coating's compatibility with the binder would be prudent. For HTS, on the other hand, many typical structural materials (certain stainless steels, Ti alloys, etc.) will oxidize—and these reactions often call for exotic superalloys (i.e., INCONEL®, etc.) to withstand these conditions. Moreover, with the silver-alloy being close to its melting point, chemical compatibility is a major concern.

Lastly, a surface coating's adhesion must be measured as well. This can be accomplished by using an adhesion testing device and/or thermal cycling a sample in liquid nitrogen. On cool-down any significant thermal contraction mismatches between relatively weak bond interfaces may cause cracking and/or flaking off of the coating from the substrate.

TABLE 2. Test matrix for evaluating performance of surface coatings used in superconducting magnet coil fabrication and some applicable standards.

Process Description	Applicable Standards
Measure coating thickness	ASTM Standards B 244, D 1400, D7091; ISO 2360
Hipot (dielectric strength)	ASTM Standards D 3755, D 149, D 3151
Apply CTD-1202 matrix binder	(For Nb ₃ Sn-type reactions only)
High temperature reaction cycle	(Nb ₃ Sn or Bi-2212 Superconductor specific conditions)
Bond Test, adhesion	ASTM Standards C 633, D 6677, D 3359, D 4541, D 2370; ISO 4624
Cool down test	77 K in LN ₂

Sample Coupon Design

Figure 1 shows a typical 3 mm thick-by-50 mm square test coupon that is coated using various techniques and processes. We leverage the fact that the coatings industry already has developed field test devices that are not dependent on specific sample geometry in order to measure parameters such as thickness and adhesion. These flat, coated coupons can easily be sent through the different coating process steps, followed by the battery of tests and environmental conditions. Afterwards, they can be examined using non-destructive methods or destructively pull-tested, or even sectioned for microscopy as necessary.

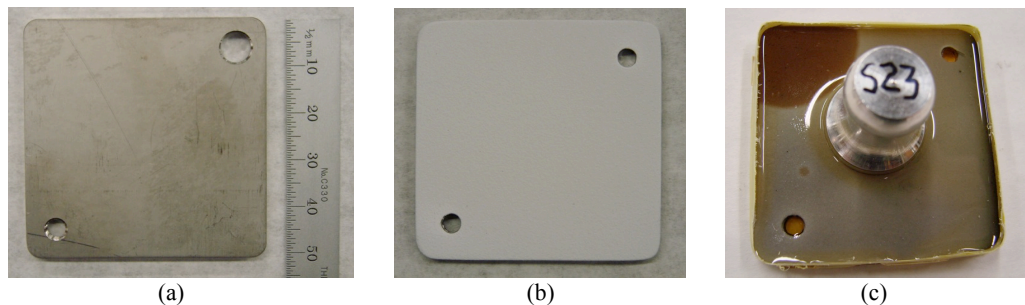


FIGURE 1. An (a) uncoated sample coupon, (b) a sample that has been plasma-sprayed, and (c) a sample bonded to a pull-test dolly in preparation for testing.

Surface Coatings Tested

Coupons were fabricated out of 300-series stainless steels and Ti-6Al-4V—some of the commonly used structural materials for wind-and-react superconducting coil fabrication. Table 3 lists the substrate samples, and their surface coating treatments applied.

The powder used for the plasma spray application was Baystate PP131, which is a 99.47% Al₂O₃ powder. The other sprayed-on coating is called CERAKOTE™ C-6010, applied much like a powder-coating application, except with a room temperature cure. According to the manufacturer, C-6010 is part of a family of protective and

decorative high temperature coatings that they produce for engines, firearms, or exhaust systems, whose primary components are alumina and silica. This particular product is what they describe as their electrical barrier coating that withstands >650 °C after a room temperature cure, which is beneficial since parts do not experience thermal excursions during processing.

Another proprietary coating called CeraFuse™, which is a surface oxide conversion produced through a micro-arc-oxidation (MAO), was also evaluated. Used in the aerospace industry for surface treating turbine blades, for instance, it produces a hard and dense ceramic surface on alloys of titanium, aluminum, and a few other refractory metals. While similar in function to hard anodizing, it is fundamentally different in its chemistry and electrical processing and properties.

TABLE 3. Coatings applied to (or produced on) base substrate materials.

Substrate Material	Plasma Spray	CERAKOTE™ C-6010	CeraFuse™/MAO
304 Stainless	✓	✓	-
316 Stainless	✓	✓	-
Ti-6Al-4V	✓	✓	✓

Test Equipment and Apparatus

As mentioned previously, off-the-shelf test equipment devices were utilized where possible. For measuring coating thickness, we used a DeFelsko Positector 6000 thickness gage that is commonly used in automobile body paint applications. Virtually any metallic substrate can be used since the device uses both magnetic and eddy current principles to measure coating thicknesses between 0 and 1500 microns.

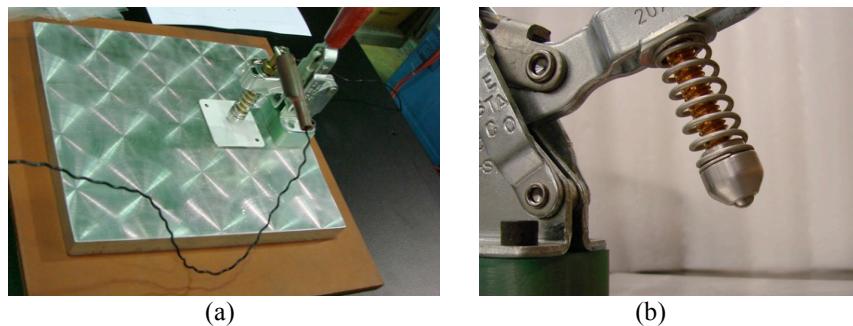


FIGURE 2. (a) Clamping rig for performing hipot measurements, and (b) a closeup of the electrode.

The hi-pot measurements are conducted using a Vitrex 944i Dielectric Analyzer. A simple spring-loaded rig was also designed and built so that the clamping force for each sample was repeatable. The electrode is a 6.35 mm ball tip, which can be seen in Fig. 2.

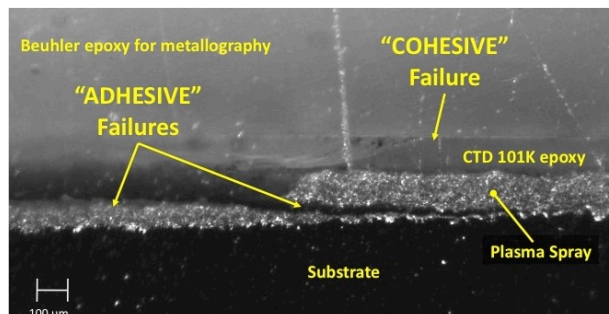


FIGURE 3. Micrograph of a plasma sprayed sample after a pull-test that reveals both “cohesive” and “adhesive” failures.

Adhesion testing is performed using a DeFelsko PosiTest AT tester. A 20 mm diameter 6061 aluminum “dolly” is bonded to the surface in a vacuum impregnation process using the standard CTD-101K, which mimics

construction of a real magnet coil. With this particular dolly diameter, the device is capable of pulling up to 20 MPa using a pull rate of 0.2 MPa/sec. Adopting the terminology described in ASTM C633 for adhesion of thermal spray coatings, bond failures are classified as either “cohesive” or “adhesive”. Figure 3 shows a sample micrograph of a coupon sample after the pull-test. “Cohesive” failures occur in the bond interface of the pull specimen and the surface coating, which reveals more about the bond adhesive than it does about the surface coating. “Adhesive” failures, on the other hand, occur at the surface coating/substrate interface, and these results allow us to determine the bond strength of the surface coating.

Coated samples are heat-treated by placing them in either a tube furnace or a furnace retort in either pure argon or pure oxygen, depending on which heat treatment cycle is being employed, and held at temperature for at least ten hours. After removing the samples from the furnace, they are once again subjected to a hi-pot test to determine the effect of the heat treatment on the surface coating’s dielectric strength.

The pull-test dollies are bonded to the coupon samples after the furnace cycle. CTD-101K is used in a vacuum impregnation process to ensure that no air bubbles are in the bond interfaces. Prior to the pull tests, these bonded samples were thermal-cycled twice in a LN₂ bath to simulate the temperature excursions that a coil would experience. It should be noted, however, that these rapid cool down cycles (on the order of -100 K/min) and warm up cycles (on the order of ~10K/min) subject these samples to greater temperature gradients than would be experienced in a full magnet assembly.

RESULTS AND DISCUSSION

Thickness and Dielectric Strength, Pre-Heat Treatment

Table 4 shows the surface coatings that were tested as they were received; each type of coating had 2 to 6 coupons available to test. At this point in time, the different substrate materials are not taken into account. The manually sprayed samples (plasma spray & CERAKOTE™ C-6010) had the thickest average application, but also had the largest variance. It should be noted, however, that the manufacturer’s recommended thickness for CERAKOTE™ is actually closer to 25 μm thick, but the thicker coat was accommodated as a trial. The CeraFuse™ coating thickness exhibits less variation since it is a surface oxide conversion produced using controlled process parameters, as opposed to a manual operation for the other two processes.

The dielectric behavior of the coatings varied from about 3 kV/mm in the CeraFuse™ samples to 64 kV/mm with the CERAKOTE™ samples. For comparison, the dielectric strength of air at standard conditions is commonly accepted as 3 kV/mm. The relatively low value in the CeraFuse™ samples suggest that the electrochemical nature of the process leaves pores in the surface that weaken its voltage breakdown performance. While the CERAKOTE™ seemed to have the highest as-processed dielectric performance, a promising observation is the nearly three- to seven-fold increase (from as low as 5.1 kV/mm to 38 kV/mm) in dielectric strength of a plasma sprayed surface when the CTD-1202 binder is applied. Because no similar increase was seen in the case where the binder was applied to the CERAKOTE™ surfaces, it appears that the binder is conveyed into the porous plasma sprayed surface by capillary action, which increases the dielectric strength of the resultant sample.

TABLE 4. Measured thicknesses of surface coatings and their dielectric strength before heat treatment.

Surface Coating	Meas. Thickness, μm (min. / max.)	Diel. Strength kV/mm (min. / max.)
Plasma Spray	91 / 168	5.1 / 13.1
Plasma Spray w/ CTD-1202	102 / 168	29.0 / 38.0
CERAKOTE™ C-6010	20 / 83	24.6 / 64.4
CERAKOTE™ C-6010 w/ CTD-1202	20 / 40	29.5 / 43.7
CeraFuse™	22 / 38	3.0 / 8.3

Samples Heat Treated at 665 °C in Argon

The first heat treatment cycle performed was the Nb₃Sn reaction in argon, which contained representatives of each surface coating on both stainless and titanium alloy samples. They were placed on a layer of heat-cleaned S2 fiberglass before they were inserted into the furnace retort, and reacted with at 665 °C for 48 hours in argon.

Figure 4a shows the CERAKOTE™ C-6010 and plasma-sprayed samples as they were removed from the furnace. Upon visual inspection, the corners, where the CTD-1202 binder was applied on the plasma-sprayed samples, were discolored with a brown hue - while the binder applied to the CERAKOTE™ C-6010 showed evidence of having been molten and “pooled up” during the reaction before solidifying. This seems to confirm the observation from the hi-pot tests that the CERAKOTE™ C-6010 coating does not soak up the binder as the plasma-sprayed surface did.

Structurally, however, upon minimal handling most of the CERAKOTE™ C-6010 coatings de-bonded in relatively large patches, which is shown in Fig. 4b. The plasma-sprayed and CeraFuse™ samples, on the other hand, seemed to survive the heat treatment structurally sound. Additionally, the CERAKOTE™ C-6010 samples came out conductive. In the absence of oxygen, a substance in the coating likely carbonized at reaction temperatures, leaving behind a conductive coating.

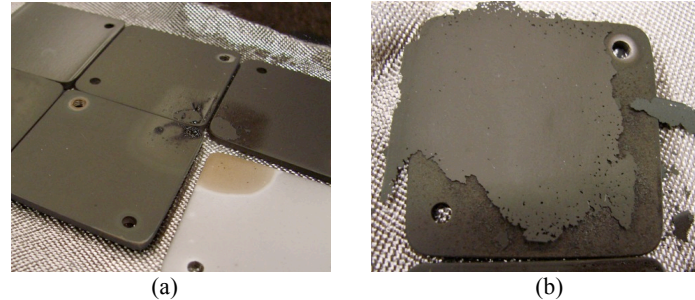


FIGURE 4. (a) Coated samples, as removed from the Nb₃Sn heat treatment cycle. The darker samples on the left are coated with CERAKOTE™ C-6010; at bottom right is a plasma-sprayed sample. The discolored corners are areas where the CTD-1202 binder was applied. (b) A heat-treated CERAKOTE™ sample flaking off in a large sheet, after only minimal handling.

Samples Heat Treated at 890 °C in Oxygen

Based on the observations of the carbonized CERAKOTE™ samples from the Nb₃Sn reaction, four CERAKOTE™-treated samples, three stainless and one Ti-6Al-4V, were placed into a tube furnace to be reacted at 890 °C in oxygen. Those coating samples came out both structurally sound and non-conductive. However, when these samples’ dielectric strength was measured, they showed an average reduction in strength of about 97%, from 46 kV/mm before reaction to 3.1 kV/mm after reaction. More interestingly, however, the titanium sample emerged from the furnace with a curvature, while the stainless samples had remained flat.

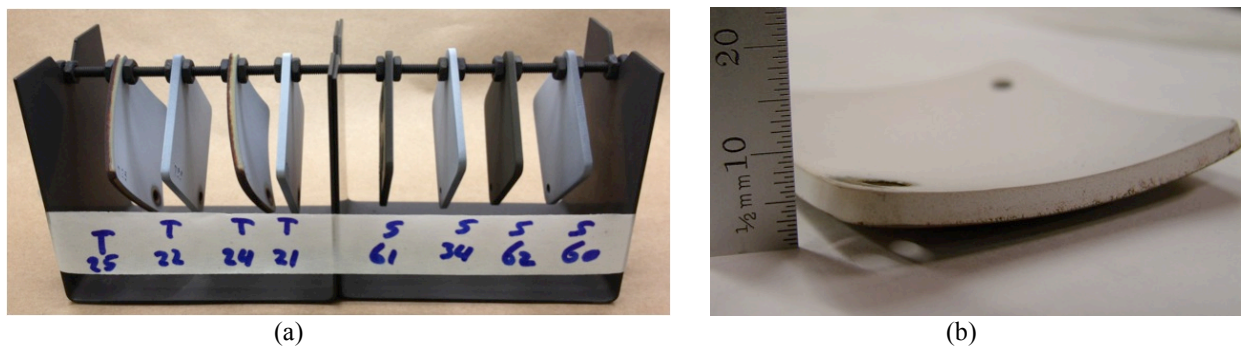


FIGURE 5. (a) Ti 6Al4V (“T”) and 316 stainless (“S”) samples, coated with CERAKOTE™, as pulled from a 890 °C reaction cycle in oxygen. From left to right, samples T25, T22, T24, T21, S61, S34, S62, and S60 were all coated on one side only. The balance of samples were coated on both sides. (b) A close-up view of a deformed Ti-6Al-4V sample coupon.

A second reaction cycle was performed using several more stainless and Ti-6Al-4V samples processed with CERAKOTE™. This time, however, half of the samples were coated on both sides, while the others were coated only on one, as before. Figure 5 shows the samples after the reaction cycle. The stainless samples came out flat, regardless of which sides were coated. The titanium samples with only one side coated came out with the same

curvature as in the first reaction. However, the titanium samples that had both sides coated came out flat. This result suggests that the CERAKOTE™ coating somehow acts as a barrier for the diffusion of oxygen into the titanium alloy, and the differential growth of the oxide layer on one side causes the sample to deform. Additionally, this result implies that this test methodology could be expanded to also measure how coatings function as diffusion barriers in Bi-2212 reactions.

Adhesion Testing

The final series of tests performed on these samples was for adhesion properties. The surviving plasma spray and CeraFuse™ samples from the Nb₃Sn heat treatment had pull-test dollies bonded onto them using CTD-101K epoxy. Prior to conducting the pull test, the bonded samples were first thermal cycled twice in LN₂, being allowed to warm up to room temperature between cycles. A summary of the tests is shown in Table 5.

TABLE 5. Summary of adhesion tests performed on heat-treated (650 °C in argon) samples, bonded with CTD-101K epoxy.

Sample Coupon	LN ₂ Cycle	Bond failure type	Bond Strength (MPa)
Cerafuse™	Adhesive failure	N/A	N/A
Plasma spray	✓	Cohesive	5.5 - 5.8
Plasma spray w/CTD-1202 binder	✓	Adhesive / cohesive	5.5

The first of three CeraFuse™ samples was found to have de-bonded from its dolly after the vacuum impregnation step. Then after the second LN₂ thermal cycle, a second one had its dolly separate. Finally, during preparations for the pull test, the third CeraFuse™ sample also separated from its dolly; see Fig. 6a. So while it appeared that the CeraFuse™ surface coating survived the Nb₃Sn heat treatment cycle intact, the oxide layer appeared to be weakly bonded to the Ti-alloy substrate at that time, as all failures were completely “adhesive” in nature.

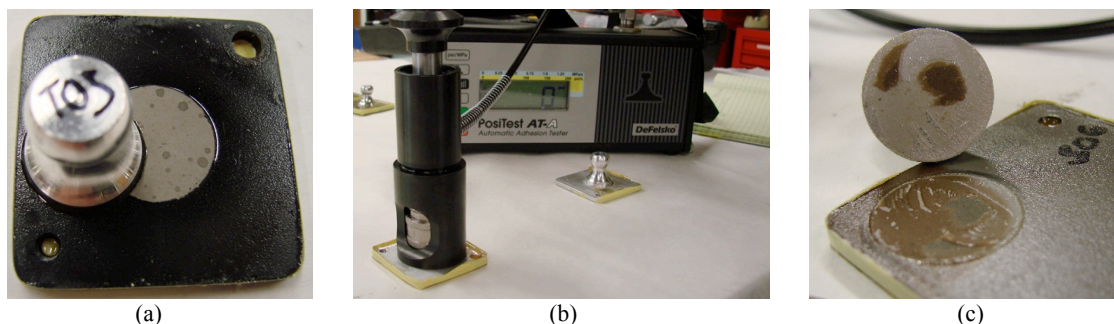


FIGURE 6. (a) Ti 6Al4V sample with a CeraFuse™ coating that broke during preparations for adhesion testing. (b) Adhesion test setup with the sample coupons. (c) Stainless 304 sample, plasma-sprayed with CTD-1202 binder. Note the combination of adhesive and cohesive failures in the failure surface.

In the end, pull tests were performed only on the plasma sprayed surfaces from the Nb₃Sn reaction after the LN₂ thermal cycles. These pull-test failures were primarily “cohesive” in nature, where the bond interface between the epoxy and the dolly fractured. One exception of this was one plasma sprayed sample that was also treated with the CTD-1202 binder. This one showed evidence of a partial “cohesive” failure in the surface (Fig. 6c). Nevertheless, each of plasma sprayed samples failed between 5.5 and 5.8 MPa, which seems to indicate that the CTD-101K epoxy releases below 6 MPa under these conditions. While not the focus of these experiments, this data point may be useful in the development of more specific epoxy systems and impregnation processes directed towards coil fabrication.

There are concerns in these results, however, which are described here. For ease of manufacturing and low cost, the standard pull test dolly supplied by the device manufacturer is made out of 6061 Aluminum. This is perfectly acceptable in most cases because their tests and test preparations occur at room temperature - where thermal mismatches between substrate and dolly are insignificant. In our tests, however, epoxy cure cycles of 125 °C and cool-down cycles to 77 K may cause undesired thermal stresses that may damage or weaken bond interfaces. To reduce this potential effect, dollies are being made of 316 stainless and Ti-6Al-4V for use in future testing.

Additionally, the bond interface in a real coil has layers of S2 glass insulation between the cable and the pole island. While this functions as a reinforced composite matrix, the glass may also serve to control the bond joint thickness, which was not controlled in these first adhesion tests. Therefore, in many respects, these initial adhesion test results require further examination to confirm these results.

CONCLUSIONS AND NEXT STEPS

We present a test methodology for evaluating insulating surface coatings for their compatibility with wind-and-react superconducting coil fabrication. We also report on the initial evaluation of coatings applied by spraying, plasma spray, or produced by micro-arc-oxidation, and their compatibility with Nb₃Sn-type and Bi-2212-type reaction cycles. Based on the currently small sample size that was reported on, it may be premature to draw definitive conclusions, but there are several trends that were observed, which open the door to further development.

Initial findings suggest that plasma sprayed surfaces have good potential when used in Nb₃Sn coil fabrication, as they have been shown to survive the heat treatment and cryogenic thermal cycles without detrimental effects. Their adhesion properties also appear to be greater than that of the standard CTD-101K epoxy system used in coil fabrication. However, because of the different thermal expansion coefficients between that of the aluminum pull-test dollies used and the coupon substrates, these initial results should be confirmed. Future experiments will incorporate the use of pull-test dollies made from the same materials as the coupons in order to eliminate this thermal effect.

The Cerafuse™ and CERAKOTE™ C-6010 coatings, while promising at the start, seem to suffer ill effects from either their chemical makeup or their structure after being subjected to the high temperature heat treatment cycles. Both coatings did not survive the heat treatment at 665 °C in argon, and the CERAKOTE™ samples only survived the heat treatment at 890 °C in oxygen with a significant loss in dielectric strength. Additional experiments will test the adhesion strength of these CERAKOTE™ samples.

For Bi-2212 reactions, material compatibility continues to be the primary issue that must be overcome with wind-and-react coil fabrication technology—in addition to durability, adhesion, etc. Our initial results show that the CERAKOTE™ coating appears to be a barrier to oxygen diffusion when applied to Ti-alloy samples. The implication of this result is that this test methodology can also be expanded to include tests of a coating's ability to function as a diffusion barrier in Bi-2212 reactions. Future experiments will be designed to measure and quantify this characteristic of the surface coatings that we test.

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