HIGH VOLTAGE BREAKDOWN TESTING OF SOL-GEL MgO-ZrO$_2$
INSULATION COATINGS UNDER VARIOUS COMPRESSIONS AT 298 K AND 77 K

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Abstract

High voltage breakdown (HV$_{bd}$) tests were carried out to investigate electrical properties of high temperature MgO-ZrO$_2$ insulation coatings on long-length stainless-steel 304 (SS) tapes under various pressures at room temperature (298 K) and liquid nitrogen temperature (77 K) for applications of HTS/LTS coils and magnets. Solutions are prepared Mg and Zr based precursors and insulated on SS substrates using reel-to-reel sol-gel technique. Thicknesses of coatings (7 and 12 µm for 4 and 8 dips) and stycasted ribbons were determined by using Environmental Scanning Electron Microscope (ESEM). HV$_{bd}$ and dielectric constant of ribbons have been presented graphically.

1. Introduction

Sol-gel ceramic-based insulation coating have been developed at National High Magnetic Field Laboratory (NHMFL) for fabricating turn-to-turn electrical insulation for high temperature superconductor (HTS)/low temperature superconductor (LTS) coils [1]. Sol-gel coatings were prepared using various precursors and solvent so that coatings are processed to apply to both high temperature and cryogenic temperature when needed. The advantages of sol-gel process and applications reported elsewhere [1,2,3]. ZrO$_2$ based coatings have ability of chemical stability, high resistivity, large relative ($\epsilon=20$) dielectric constant and good phase transformation [4,5]. Therefore sol-gel MgO-ZrO$_2$ insulation coatings are passionately applicable to HV$_{bd}$ tests for HTS/LTS.

Previous researchers at NHMFL studied electrical properties of MgO-ZrO$_2$ coating without applying pressure at 298 K, while insulating on Ag and AgMg/Bi-2212 superconducting tapes [3,6]. They showed that ZrO$_2$ provided sufficient turn-to-turn insulation after 13 dipping in this process [7]; MgO-ZrO$_2$ does the same with only 8 dipping. We studied electrical properties of sol-gel MgO-ZrO$_2$ insulation coated on SS tapes in our recently published paper [8]. In this work we studied HV$_{bd}$ and dielectric properties of the sol-gel MgO-ZrO$_2$ reel-to-reel insulation coatings as function under various pressures at 298 K and 77 K.

2. Experimental procedure

The MgO-ZrO$_2$ coatings were insulated on commercial 304-SS tapes by using sol-gel precursor dipping technique in air. The prepared solution consists of dilute and normal. The normal coating solution consists of 20 mol % MgO and 80 mol % ZrO$_2$, which was prepared using zirconium tetrabutoxide, isopropanol and acetone. Dilute solution is obtained by adding equal amount of isopropanol to normal solution. Zirconium tetrabutoxide and several Mg based
precursors were used as precursors, which was prepared as clear homogenous by stirring for an 8 h period at 100 rpm. The detail information on solution preparation and coating process can be found in Refs. [1-3]-[6-8].

Conductor substrate SS tapes were cleaned with acetone in air. Then SS tape was coated by MgO-ZrO₂ solution and the coating process was repeated 4 and 8 dips. Coating thickness is regulated with dip numbers, viscosity of solution, wetting and withdrawal rate. By cutting the tapes as 10 cm long, two kinds of samples were produced for each 4 and 8 dips. First, ribbons were made without stycast; the second, the gap of the layer was filled with stycast (2850 FT/11 Black, Emerson & Cuming). The thickness of coating and stycast were determined by using ESEM, as seen in Fig 1. Ribbons were placed for testing the experimental set up as shown in Fig.2, in which HVₜ₟ and capacitance was measured by Model 200-02R High Voltage Power Supply and 161 Analog Digital Capacitance Meter, respectively. For each measurement, the process was repeated at least three times. The dielectric constant of samples was calculated by using Eq. (1) without taking into account effects of strain.

\[
\varepsilon_r = \frac{C(d_i + d_s)}{\varepsilon_0 A},
\]

where \(\varepsilon_r\) is dielectric constant of insulation coatings, \(\varepsilon_0\) permittivity of free space \(8.854 \times 10^{-12} \text{ F/m}\), \(A\) is coated area in the sandwich, \(d_i\) is twice the coating thickness, and \(d_s\) is the stycast

![Fig.1. The thickness of insulation and Staycast of 8 dips samples.](image1)

![Fig.2. Experimental set up HVₜ₟ tests](image2)

| Table I. The average values of HVₜ₟, electric strength and dielectric constant |
|--------------------------------|-------|-------|-------|-------|
| Dip Number                  | Room Temp. (298K) | Liquid Nitrogen Temp. (77K) |
| Without stycast (WOS)       |       |       |       |       |
| HVₜ₟ (kV)                   | 0.18  | 0.35  | 0.53  | 1.21  |
| Electric Strength (kV/mm)   | 13.2  | 14.8  | 38.44 | 49.4  |
| Dielectric Strength, P=0    | 3.52  | 4.6   | 2.79  | 3.94  |
| With stycast (WS)           |       |       |       |       |
| HVₜ₟ (kV)                   | 0.85  | 0.65  | 2.72  | 2.78  |
| Electric Strength (kV/mm)   | 5.74  | 4.22  | 18.24 | 18.00 |
| Dielectric Strength, P=0    | 32.90 | 27.30 | 27.60 | 24.60 |
thickness, which is zero for the cases without stycast ribbons. Under the stress, the epoxy between the layer fills the porosity of the insulation coating of the layers and air is leaked or/and trapped into pores, so that it is extremely hardened; therefore, HV$_{bd}$ may not appreciably change with increasing pressure on them.

2. Results and discussion

As expected, coating thickness, crack in coatings, dopant materials, stycast thickness, pressure and temperature influence electrical, mechanical properties as well as surface morphology and structure of insulations. Also, porosity, coating thickness, cracks; dopant, viscosity, and withdrawal speed influence insulation properties.

Table I and Fig.3 show the HV$_{bd}$ of 4 and 8 dips for WS and WOS ribbons at 298 K and 77 K. HV$_{bd}$ values increased with increasing of the coating thickness and temperature. It is clear that the HV$_{bd}$ of WOS samples for 4 dips are lower than that of 8 dips samples, as expected. HV$_{bd}$ values of WOS ribbons are concerning 0.18, 0.53 kV for 4 dips, and 0.35, 1.21 kV for 8 dips for the former at 298 K and the latter 77 K. HV$_{bd}$ practically increases by 3 times for WOS samples, when cooling from 298 K to 77 K. HV$_{bd}$ of samples WS for 4 dips are about 0.85 kV at 298 K and 2.72 kV at 77 K for WS samples. At these temperatures the HV$_{bd}$ values of the ribbons of 8 dips are 0.65 and 2.72 kV, respectively. HV$_{bd}$ values of WS samples increase almost by 3.3 times for 4 dips and by 4.3 times for 8 dips, when cooling from 298 K to 77 K.

Fig.3. HV$_{bd}$ compression with the ribbons of WS and WOS for 4 and 8 dips at 298 K and 77 K.
Fig. 4. Dielectric constant varies with dip numbers, temperature and pressure with epoxy samples. It is constant with epoxy impregnation.

As shown in Fig. 4 dielectric constant values increased with increasing number of dipping and pressure for WOS samples at 298 K. This increase at 77 K is lower than that at 298 K. Dielectric constant values of WS ribbons at 298 K are slightly higher than at 77 K. This is probably due to thicker stycast, increased porosity, pinhole and/or higher value of crack cavity. Dielectric constant values of WS ribbons are about 7.4 and 6.8 times higher than that of WOS at 0 pressure at 298 K and 77 K, respectively. Normally, in spite of the fact that the dielectric constant of ZrO$_2$ was 20 [5], it was measured to be 32.9 and 27.5 for 4 and 8 dipped WS ribbons at 298 K.

**3. Conclusion**

MgO-ZrO$_2$ was coated on SS-304 tapes at 4 and 8 dips by using reel-to-reel sol-gel technique. Test ribbons were formed of WS and WOS. Thickness of insulation was determined by ESEM, which were about 7 and 12 μm for insulation for 4 and 8 dips, respectively. HV$_{bd}$ tests were performed with these samples under various stresses at 298 K and 77 K. HV$_{bd}$ values vary with numbers of dipping. WOS ribbons were cooled from 298 K to 77 K. Depending on this, HV$_{bd}$ values increased by about 3 times. The highest values of HV$_{bd}$ is 2.78 kV for 8 dips for WS at 77 K. Stycast increased the HV$_{bd}$ values by 4.7 and 2 times at 298 K, and by 5 and 2.3 times at 77 K for the former 4 dips and the latter 8 dips in which stycast thickness for 4 dips is thicker than 8 dips. The highest HV$_{bd}$ was 2.78 kV at 8 dips for WS at 77 K.

Dielectric constant depends on capacitance, temperature, pressure, coating and stycast thickness. Dielectric constant values for WOS ribbons gradually increased with increasing numbers of dipping as well as increasing pressure at 298 K. But this trend is considerably lower
and adjacent to each other with the number of dipping at 77 K. On the other hand, the dielectric constant values for WS samples are about constant with increasing pressure, but decreasing with increasing number of dipping, since insulation thickness for 4 dips becomes thicker than that for the other samples. The dielectric constant increased by about 7.4 and 6.8 times with stycast at 298 K and 77 K. Dielectric constant of $\text{ZrO}_2$ is given by about 20 in the literature [5], and in this work, WS ribbons values are found to be 27-32 at 298 K. $\text{HV}_{bd}$ and dielectric constant depend on dip number, temperature and epoxy impregnation.

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