

## **HIGH VOLTAGE BREAKDOWN PROPERTIES of SOL-GEL MgO–ZrO<sub>2</sub> INSULATION COATINGS UNDER COMPRESION at 300 K and 77 K**

**O. Cakiroglu**

**Hasan Ali Yucel Education Faculty, Istanbul University,  
Beyazit, 34452 Istanbul, Turkey**

**L. Arda**

**Faculty of Engineering and Natural Sciences, Bahcesehir University,  
Besiktas, 34349, Istanbul, Turkey**

### **ABSTRACT**

*High voltage breakdown (HVbd) tests were performed to investigate electrical properties of high temperature MgO–ZrO<sub>2</sub> insulation coatings on long-length stainless steel (SS) ribbons by sol-gel process for applications of HTS/LTS coils and magnets technologies. After solutions were prepared from Mg and Zr based precursors, solvent and chelating agent, the coating were fabricated on SS substrates using reel-to-reel sol-gel technique. Electrical properties of the samples such as breakdown voltage, electrical strength and dielectric constant under varying stress, at Room Temperature (RT) and 77 K were investigated. Coating thicknesses for 4, 8, and 9 dipping were about 7, 12, and 13  $\mu\text{m}$  respectively. The pressure from 0 GPa to 0.54 GPa was applied on to test couples, the stycast thicknesses between the layers were varied 32–20  $\mu\text{m}$ . Surface morphologies of insulated samples were examined by SEM.*

**Keywords:** High voltage breakdown; Sol-gel; MgO–ZrO<sub>2</sub>

### **1. INTRODUCTION**

MgO–ZrO<sub>2</sub> coatings have been insulated on SS-304 tapes using a reel-to-reel continues sol-gel technique. Sol-gel ZrO<sub>2</sub> was prepared using zirconium tetrabutoxide, acetyl acetone, and isopropanol. Of these insulation methods, reel-to-reel sol-gel technique is the most suited one, as reported elsewhere [1,2,3,4]. Also the sol-gel technique is very attractive low temperature processing technique for preparation of complex oxide composition with high homogeneity [5,6]. ZrO<sub>2</sub> based coatings are suitable for high temperature insulation due to its chemical stability, high resistivity, and large relative dielectric constant. Sol-gel MgO–ZrO<sub>2</sub> insulation coatings are most suited to HVbd tests for HTS since MgO content in ZrO<sub>2</sub> seems to increase HVbd, and hence positively affect electrical properties of insulation coating at the high temperature required for processing and the cryogenic temperatures for the operations [3,7,8].

In this study, sol-gel MgO–ZrO<sub>2</sub> insulation was coated on SS-304 tapes with dips of 4, 8, and 9 times. Three kinds of test samples were made of each of these tapes. Test couples were prepared with the tape with stycast (WS), without stycast (WOS), and with stycast prepared under pressure (WSUP). Pressure in the WSUP samples varied from 0 GPa to 0.54 GPa, and stycast was left to cure for 8 h. The electrical properties of the test couples, such as HVbd and dielectric constant were then investigated at 298 K and 77 K, and under various pressures. Surface morphology of arcing spots of the samples was examined using ESEM.

### **2. EXPERIMENTAL**

MgO – ZrO<sub>2</sub> precursor solutions were prepared as explained in reference [3] and 5  $\mu\text{m}$  thick and 5

mm SS ribbons were coated using sol-gel dip coating system with an in-line three zone furnace. Thicknesses of the coatings were varied by varying number of dip coatings. The coating processes were repeated 4, 8, and 9 times in this work and 10 cm long coated SS ribbons were cut from total long samples. As seen in Table 1, the dip coating is composed of dipping, withdrawal, and heating. Individual 10 cm long ribbons were made into couples with and without stycast, which is a cryogenics temperature compatible epoxy, to form a capacitance and tested under varying pressures at RT and 77 K. Finally microstructures of the coatings were investigated by ESEM.

Table 1. Sol-Gel MgO-ZrO<sub>2</sub> insulation coating parameters

Insulation	Number of Dip	Furnace Temp. (°C)	Withdrawal Speed (m/min)	Thickness (μm)
MgO-ZrO <sub>2</sub>	Total Dip #: 9 3 Dip Dilute 4 Dip Normal 2 Dip Dilute	550, 570, 580 580, 610, 630 550, 570, 580	0.65	13
MgO-ZrO <sub>2</sub>	Total Dip #: 8 3 Dip Dilute 3 Dip Normal 2 Dip Dilute	550, 570, 580 580, 610, 630 550, 570, 580	0.65	12
MgO-ZrO <sub>2</sub>	Total Dip #: 4 1 Dip Dilute 2 Dip Normal 1 Dip Dilute	550, 570, 580 580, 610, 630 550, 570, 580	0.65	7

The test couples were placed for testing the experimental set up as shown in Fig. 2. The resistance, HVbd and capacitance were measured at least three times.

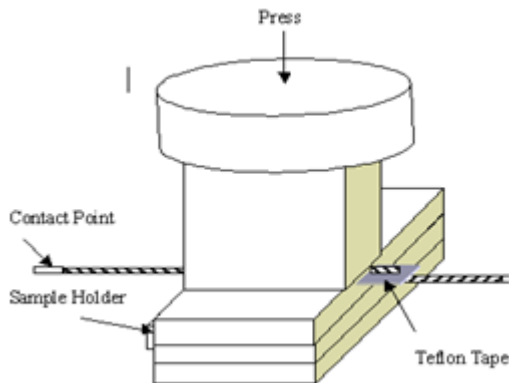


Figure 1. Experimental set up for HV breakdown tests.

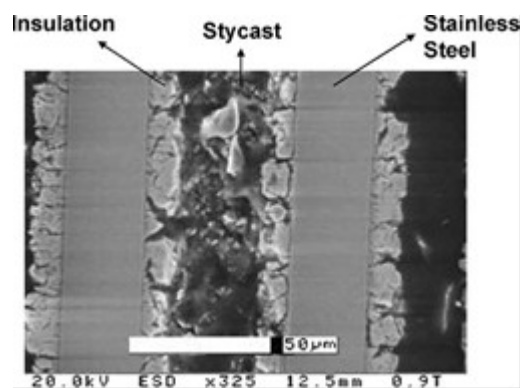


Figure 2. The insulation, stycast and stainless of eight times dipped substrates. The scale bar is 50 μm.

### 3. RESULTS AND DISCUSSION

Figure 3 shows the  $V_{bd}$  for 4, 8 and 9 dip samples at RT and 77 K without epoxy. The  $V_{bd}$  at RT and 77 K are 0.5 kV and 1.3 kV, respectively. It was seen that there is small drop in  $V_{bd}$  at low pressures, but then it levels off.

The electrical strength (ES) at 77 K and RT for 8 and 9 dip samples at 77 K and RT are 50 kV/mm and 15 kV/mm, respectively. Although the ES of the 4 dip sample is about the same with those of the 8 and 9 dip samples at RT, its ES at 77 K is much lower than those of 8 and 9 dip samples.

Figure 5 shows the dielectric constants, which are calculated using the formula in the inset of the plot, versus compression at 77 K and RT for 4, 8 and 9 dip samples without epoxy. These were calculated without taking into effects of strain hence the change in the capacitor gap and thermal contraction due to cooling.

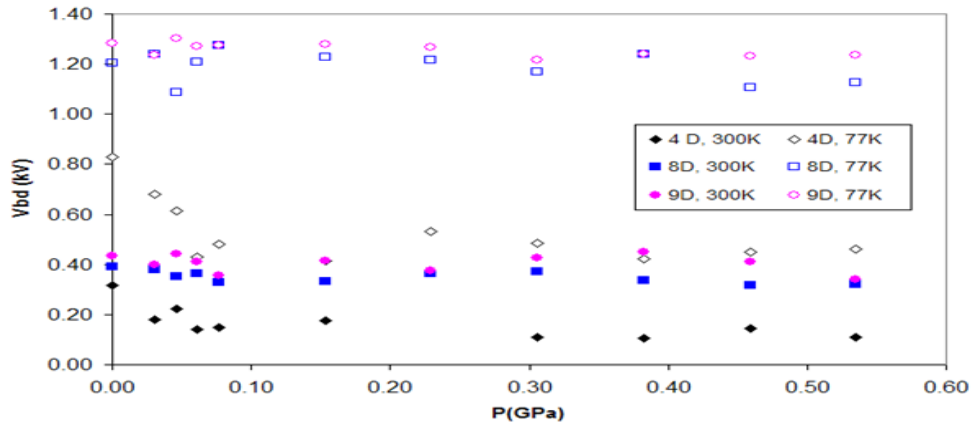


Figure 3. Voltage breakdown values as a function of compression at 77 K and RT for 4, 8 and 9 dip sol-gel Mg O-ZrO<sub>2</sub> coatings on SS ribbon without epoxy

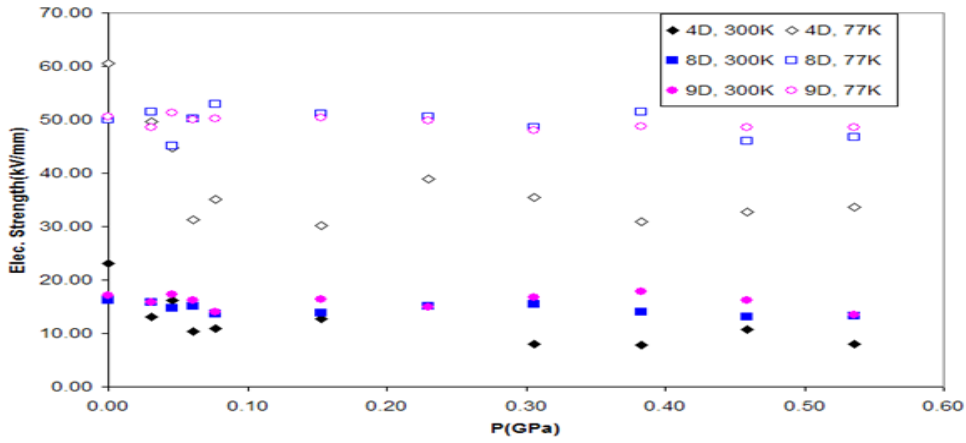


Figure 4. Electric strength as a function of compression at 77 K and RT for samples

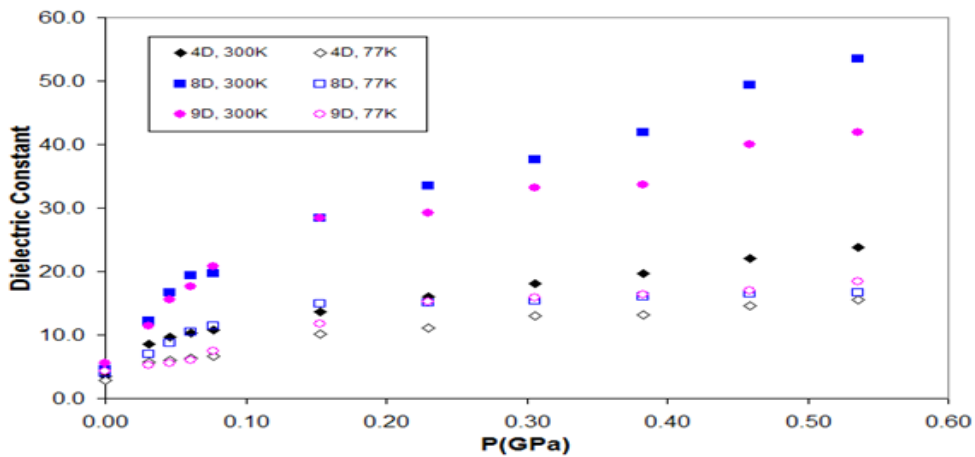


Figure 5. The dielectric constants, which were calculated, from the measured capacitance of the couples using the formula in the inset of the plot

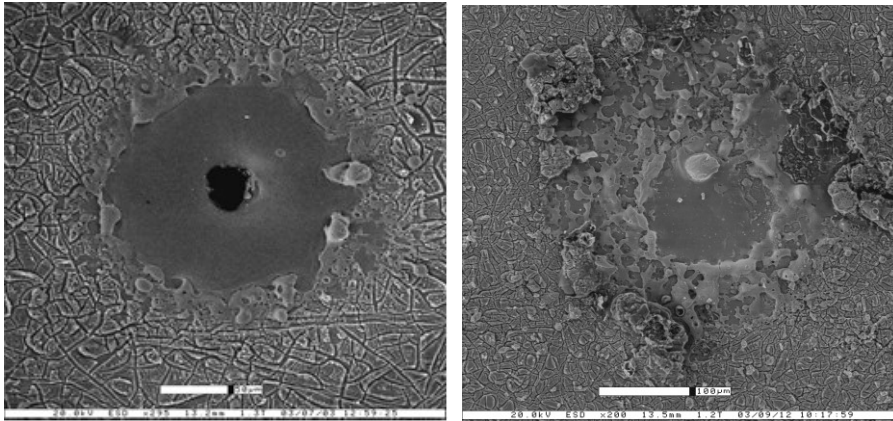


Figure 6. SEM Micrographs of the arching spots for a 4 dip sample without epoxy which was tested at 77 K and zero pressure with a  $V_{bd}$  of 0.8 kV (left, the scale bar is 50 micro meters), and for an 8 dip sample without epoxy which was tested at Rt and zero pressure with a  $V_{bd}$  of 0.4 kV (right, the scale bar is 50 micrometers).

#### 4. CONCLUSIONS

SS ribbons (25 micron thick, 5 mm wide) were coated with MgO-ZrO<sub>2</sub> by sol-gel process in three different thicknesses.  $V_{bd}$  test couples were formed with and without stycast.  $V_{bd}$ , capacitance measurements were carried out at RT and 77 K. SEM pictures shows that MgO-ZrO<sub>2</sub> coating was molten at RT and 77K at the points of arching in the test couples without epoxy.  $V_{bd}(s)$  were 0.4 kV, 1.3 kV at RT and 77 K and ES(s) were 15 kV/mm, 50 kV/mm at RT and 77 K respectively.  $V_{bd}(s)$  at RT were 0.8 kV, 0.4 kV, with and without epoxy and  $V_{bd}(s)$  at 77 K were 3 kV, 1.3 kV, with and without epoxy respectively.

**ACKNOWLEDGMENTS:** The present work was supported by the Research Foundation of Scientific Research Center (BAB) of Istanbul University

#### 5. REFERENCES

- [1] Mutlu, I.H., and Hascicek, Y.S., Advances in Cryogenic Engineering 44, 233 (1998).
- [2] Celik, E., Mutlu, I.H., E, Avci, and Hascicek, Y.S., Advances in Cryogenic Engineering 46, 895 (1999).
- [3] Cakiroglu O., Arda L., and Hascicek, Y.S. Physica C422 (2005) 117–126
- [4] Mutlu, I.H., Celik, E., and Hascicek, Y.S., Physica C 370, 113 (2002).
- [5] Celik, E., Mutlu, I.H., Hascicek, Y.S., Physica C, 370, 125 (2002).
- [6] Celik, E., Mutlu, I.H., E, Avci, and Hascicek, Y.S., Scripta Materialia 47, 315 (2002).
- [7] O. Cakiroglu, L. Arda, C. Boyraz, and O. A. Sacli. IEEE Transactions On Applied Superconductivity, Vol. 18, No. 2, June 2008.
- [8] Kim DH., Park HJ., Choi J., Lim H M. Journal of the Korean Ceramic Society. 52(5) 2015, Pages 366-373
- [9] Emam W I., Mabied A F., Hashem H M., El-Shabiny AM., Ahmed Farag I S. Journal of Solid State Chemistry. Volume 228, 2015, Pages 153–159.