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Dielectric properties of TiO_2/Epoxy nanocomposites in liquid nitrogen environment

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Abstract. The application of superconductivity technology can solve many problems in power industry. Compared with the conventional technology, superconductivity technology has many superior properties. However, the epoxy resin material used for superconducting equipment insulation is often in liquid nitrogen environment, and the low temperature will bring many changes to the insulation properties of the epoxy resin, which brings new problems to the insulation design of the superconducting equipment. In this paper, a novel epoxy resin nanocomposite was developed. We used titanium dioxide particles with average size of 10nm and 35nm mixed with epoxy resin to obtain different doping amount of samples, and the samples were tested at room temperature and liquid nitrogen environment separately. Through experiments, it was found that the addition of TiO2 nanoparticles enhanced the breakdown strength of the epoxy resin compared to the pure epoxy samples. The breakdown strength under liquid nitrogen conditions was higher than the breakdown strength at normal temperature. The dielectric properties test showed that the dielectric constant of epoxy resin in liquid nitrogen was lower than that in room temperature, and the addition of TiO2 nanoparticles increased the dielectric constant of the epoxy resin. Dielectric loss was greatly influenced by temperature and was significantly higher than normal temperature under liquid nitrogen conditions.

1. Introduction

As superconducting material prices gradually decrease and properties continue to increase, superconducting technology is maturing. Superconducting cables [1], superconducting transformers [2], superconducting magnetic energy storage systems, and other equipment have been successively developed. Since the design and manufacture of high-voltage insulation combines the disciplines of electronics, thermals and mechanics, which will bring new problems and difficulties in low temperature condition. The complex environmental condition of liquid nitrogen will bring new problems in material selection, structural design and analysis method. Therefore, it is important to study the dielectric properties of insulation materials in liquid nitrogen environment.

As a kind of commonly used polymer dielectric material, epoxy resin is widely used as solid sealing dielectric materials of electrical and electronic equipment due to its excellent physical and chemical properties, mechanical properties and thermal properties, etc[3-5]. Research on nano-TiO_2 filled epoxy resin has made some progress in recent years. Birgit Bittmann prepared nano-TiO2/EP nanocomposites and the test results showed that when the doping amount of nano-TiO_2 was 1wt%, the mechanical properties of composites were best[6].Bernd Wetzel tested the properties of nano-TiO_2-Al_2O_3/epoxy resin composites and found that the mechanical properties of the composites were significantly improved[7]. However, most of the studies focused on the mechanical properties of the composites, and the dielectric properties of the nano-TiO_2/EP composite in liquid nitrogen environment had not been studied. This paper studies the effect of particle size, doping concentration

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and temperature on the dielectric properties of epoxy resin, and provides reference for the design of insulation system of superconducting equipment.

2. Samples

2.1. Materials
The epoxy resin we chose was liquid bisphenol F type epoxy resin. The nanoparticles used were titanium dioxide powder with an average particle size of 10 nm and 35nm. The curing agent chose was DETDA. We used silane coupling agent for the modification of nanoparticles. In addition, we also need some thinner to reduce the viscosity of the liquid and increase the toughness of samples.

2.2. Samples preparation
The specific preparation steps are as follows:

(a) Add the modified nanoparticles to the epoxy resin solution and stir the mixture for 30 minutes at 50°C;

(b) The mixed solution was sonicated at 50°C for 2 hours;

(c) Add an appropriate amount of curing agent and thinner (the ratio of the mass ratio of the epoxy resin, thinner, and curing agent were 50:30:21) to the mixture;

(d) The mixed solution was stirred for 30 minutes at room temperature;

(e) Place the mixed solution to the vacuum drying oven and remove the bubbles of the solution;

(f) Pour the mixed solution into preheated molds;

(g) Put the molds into the vacuum drying oven and continue removing the bubbles for 6 hours to ensure the bubbles are removed cleanly;

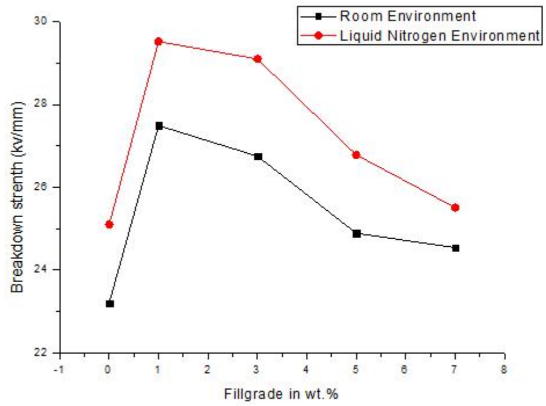
(h) Place the samples in the vacuum oven at a temperature of 60°C for 24 hours and then at a temperature of 150°C for 20 hours;

(i) Open the vacuum oven and take out the samples with doping loadings of 1%, 3%, 5% and 7% per weight with the thickness was 1mm.

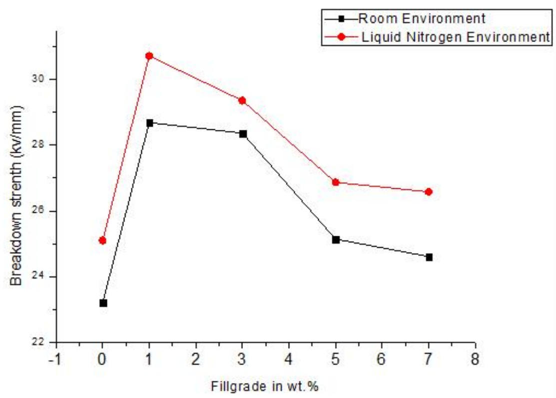
3. Results and discussions

3.1. Lightning impulse electric strength test results
When the voltage increases to a certain value, the insulating material is broken down, and its insulation properties is lost, which leads to a short circuit between the electrodes. The phenomenon that the insulating material loses the insulating properties due to the large electric strength is called electrical breakdown. In the required test environment, the breakdown voltage when the insulation material is broken is called the breakdown voltage. The ratio of the breakdown voltage to the distance between the electrodes is called the electrical strength.

This paper tested the lightning impact electrical strength of TiO_2/EP nanocomposites, the results were shown in figure 1. From figure 1, we can see that with the increase of nano-TiO_2 doping amounts, the electrical strength increases firstly and then decreases. When the nano-TiO_2 doping amount is 1wt%, the electrical strength of the TiO_2/EP nanocomposites reaches the maximum. The breakdown strength of the TiO_2/EP nanocomposites under room temperature is lower than that in liquid nitrogen environment.



(a)10nm-TiO_2/EP



(b)35nm-TiO_2/EP

Figure 1.Breakdown strength of TiO_2/EP nanocomposites

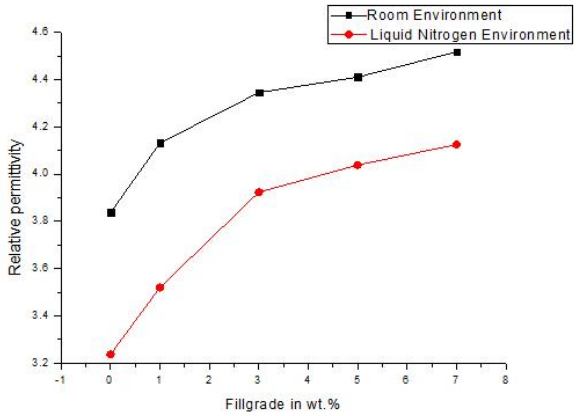
3.2. Dielectric properties test results

The dielectric property refers to the phenomenon that a polymer molecule responds under an applied electric field. It can be characterized by two major parameters of dielectric constant and dielectric loss. The dielectric properties of epoxy resin composites can be tested according to the relevant standards. The LCR tester is selected as the measuring device, which can stably and accurately measure the parameters of inductors, capacitors and other components. The electrode used in the experiment is a plate electrode consisting of a pair of plate electrodes. The dielectric loss factor of the epoxy resin composite samples can be directly measured by the device, and the relative dielectric constant can be obtained according to the following formula.

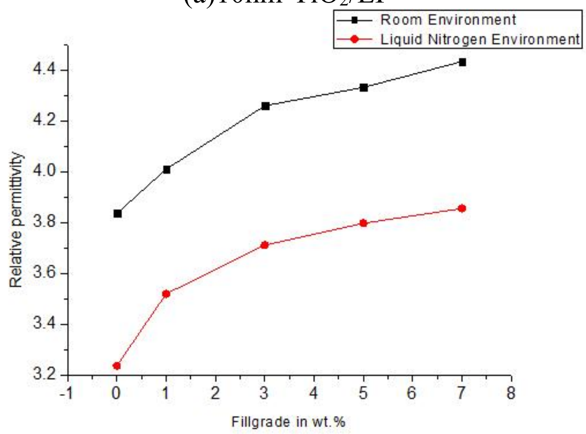
$$\varepsilon_r = \frac{Cd}{S\varepsilon_0} \quad (1)$$

C is the capacitance value of the sample, d is the thickness of the sample, S is the area of the sample, ε_0 is the vacuum permittivity and its value is $8.854187817\times10^{-12}$F/m.

Figure 2 shows the dielectric constant of TiO_2/EP nanocomposites at a test frequency of 0.05 V and a frequency of 50 Hz. It can be seen from the figure that the dielectric constant of composites at room temperature is higher than that in liquid nitrogen environment. The dielectric constant of the epoxy resin doped with 10nm-TiO_2 particles is higher than that of the epoxy resin doped with 35nm-TiO_2 particles. The dielectric constant of TiO_2/EP nanocomposites will increase with the increase of doping amount. This is because as the content of nano-TiO_2 increases, the number of polar functional groups in the composite increases and the applied electric field increases. As a result, the degree of polarization of the composite material has increased, eventually resulting in an increase in its dielectric constant.



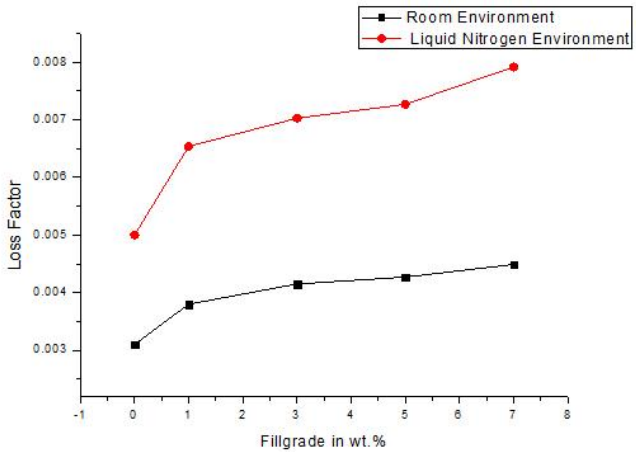
(a)10nm-TiO_2/EP



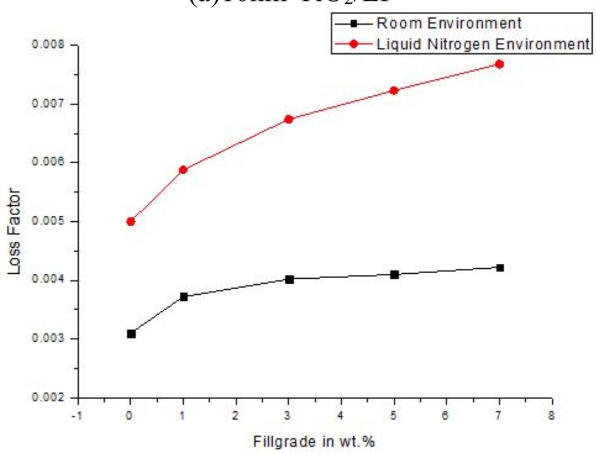
(b)35nm-TiO_2/EP

Figure 2.Dielectric constant of TiO_2/EP nanocomposites

As can be seen from the following two figures, the dielectric loss of composites at room temperature is lower than that in liquid nitrogen environment. The dielectric loss of TiO_2/EP nanocomposites increases with the increase of the doping amount. The reason of this phenomenon may be the number of conductive carriers in the composite increases with the increase of the amount of doping. Under the influence of the applied electric field, carriers have the characteristic of directional migration and eventually lead to heat loss of the dielectric. Therefore, with the increase of nano-TiO_2 doping, the dielectric loss of composites increases.



(a)10nm-TiO_2/EP



(b)35nm-TiO_2/EP

Figure 3.Dielectric loss of TiO_2/EP nanocomposites

4. Conclusions

The electrical properties of the TiO_2/EP nanocomposites are studied in this paper. The lightning impact test shows that adding TiO_2 nanoparticles will increase the breakdown strength of the epoxy resin, and the breakdown strength of the composite material in the liquid nitrogen environment is slightly higher than the normal temperature environment. Dielectric properties test results show that the addition of TiO_2 nanoparticles will increase the dielectric constant of the epoxy resin, and as the doping amount increases, the dielectric constant of the composite material also increases. The dielectric constant of the TiO_2/EP nanocomposites will drop significantly under liquid nitrogen environment. The cryogenic condition has a great influence on the dielectric loss of the TiO_2/EP nanocomposites, and the dielectric loss of all samples will increase significantly at low temperature.

Acknowledgments

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References

[1] Masuda T and Yumura H 2009 A new HTS cable project in Japan IEEE Trans on Applied Superconductivity vol 19 pp 1735-1739

[2] Cheon H G, Kwag D S and Choi J H 2007 Electrical insulation design of a 154kV class HTS transformer Journal of the Korea Institute of Applied Superconductivity and Cryogenics vol 9 pp 53-56
[3] B X Du 2002 Temperature Effects of Surface Discharge Characteristics of Epoxy Resin Laminate Trans IEEE vol 122 pp 415-420
[4] F O ldervoll 2002 High Electric Stress and Insulation Challenges in Integrated Microelectronic Circuits IEEE Electrical Insulation Magazine vol 18 pp 16-20
[5] N Yoshimura, S Kumagai and B X Du 1997 Research in Japan on the Tracking Phenomenon of Electric Insulating Materials IEEE Trans on Electrical Insulation Magazine vol 13 pp 8-19
[6] BIRGIT B, FRANK H and ALOIS K 2009 Ultrasonic Dispersion of Inorganic Nanoparticles in Epoxy Resin 2009 Ultrasonics Sonochemistry vol 16 pp 622-628
[7] WETZEL B, ROSSO P and HAUPERT F 2006 Epoxy Nanocomposites-Fracture and Toughening Mechanisms Engineering Fracture Mechanics vol 73 pp2375-2398