Dielectric properties of MgO/Epoxy nanocomposites in liquid nitrogen environment

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Abstract. As the epoxy resin material of superconducting equipment is often in low temperature environment, the insulation properties of epoxy resin changes with temperature accordingly which brings new problems on insulation design of superconducting equipment. This paper studied the effect of nano-MgO particles on the dielectric properties of epoxy resin in liquid nitrogen condition. We used magnesium oxide particles with average size of 50nm and 80nm mixed with epoxy resin to obtain different doping amount of composite dielectric and prepared pure epoxy resin samples as comparison. The test results showed that the addition of nanoparticles enhanced the breakdown strength of the epoxy resin compared to the pure epoxy samples. The breakdown strength in liquid nitrogen condition was higher than the breakdown strength at normal temperature. Dielectric properties test results showed that the dielectric constant of epoxy resin in liquid nitrogen environment was lower than that at normal temperature, and the addition of nanoparticles decreased the dielectric constant of epoxy resin. The dielectric loss was greatly influenced by temperature, and it will decrease significantly in low temperature environment. The research provides important reference value for the insulation design of superconducting equipment.

1. Introduction

With the development of superconducting power technology, superconducting transformers[1], superconducting cables[2], superconducting magnets and other superconducting power devices had been greatly improved in application level and voltage level. The high voltage insulation technology in low temperature environment is the research difficulties of the current superconducting application technology. In the aspect of design and manufacture, it integrates multidisciplinary content, such as electricity, thermals, and mechanics. Therefore, it is of great significance to study the dielectric properties of materials in liquid nitrogen environment[3].

Epoxy resin has good electrical properties, thermal properties and processing properties, which is widely used in the electrical field. In recent years, the application of nanometer materials in the field of epoxy resin had obtained some achievements. About the study of nano-MgO filled epoxy resin, Thomas A mainly focused on the effect of nano-MgO on space charge, DC conductance and dielectric properties of epoxy resin[4]. Du.B X found that nano-MgO can improve the DC resistance tracking properties of epoxy resin[5]. J. Yoshida found that the magnesium oxide particles can reduce the space charge (SC) density of polymers[6]. T.Andritsch studied the space charge properties of MgO-epoxy nanocomposites and investigated the difference at room temperature and high temperatures[7]. However, there were few studies on the dielectric properties of nano-MgO/EP composite dielectrics in liquid nitrogen condition. This paper researched the effect of particle size, doping concentration and temperature on the dielectric properties of epoxy resin, which provided reference for the insulation system design of superconducting equipment.
2. Samples

2.1. Materials
We chose liquid bisphenol F type epoxy resin (RAL230) from Rutherford Appleton Laboratory. The nanoparticle used was magnesium oxide powder with an average particle size of 50nm and 80nm, its purity was about 99.9%. The curing agent used was DETDA. We used silane in the form of Glycidoxypropyltrimethoxysilane to deal with the surface of nanoparticles. In addition, thinner was also needed to reduce the viscosity of the liquid and increase the toughness of samples.

2.2. Samples preparation
Nano-MgO/EP composites were prepared by mechanical dispersion method. The nanoparticles were modified by KH-560 firstly so that the particles can disperse in the host epoxy resin evenly. Due to the nanoparticles were exposed to air and absorbed moisture easily, the nanoparticles needed to be dried at 120°C for 12 hours. To reduce the viscosity of the epoxy resin, it was stored at 50°C for about 30 minutes. The dried MgO nanoparticles were added to the epoxy resin and mixed for 30 minutes. After the mixture was sonicated for 2 hours, add the curing agent and thinner (the ratio of the mass ratio of the epoxy resin, thinner, and curing agent were 50:30:21). After stirring evenly and defoaming, pour the mixture into the moulds sprayed with release agent. Then the samples were stayed in a vacuum drying oven for 24 hours at 60 °C and 20 hours at 150 °C. In this study, we prepared four kinds of nano-MgO/EP composite dielectrics samples with doping loadings of 1%, 3%, 5% and 7% per weight, and the thickness was 1mm. Neat epoxy was also prepared for reference.

3. Results and discussions

3.1. Lightning impulse electric strength test results
The lightning impulse voltage test platform used a ball electrode with a diameter of 20mm. The samples were wafers with the diameter of 100mm and the thickness of 1mm. The breakdown test results were analyzed by a two-parameter Weibull distribution function. Before the test was started, the samples were soaked in a liquid nitrogen atmosphere for about 20 minutes firstly, and then the samples were tightly sandwiched between the electrodes. Due to the low temperature of liquid nitrogen, the samples would be brittle and cannot be over-tightened. Otherwise, the samples may be broken or deformed. After the sample was fixed, put the electrode to the liquid nitrogen cryogenic container slowly. The sample can be tested when the liquid nitrogen became relatively stable. The result is shown in Figure 1.
environment is 25.10kV/mm, an increase of about 8.2%. The breakdown strength of the composite material in the liquid nitrogen environment is slightly higher than that in the normal temperature environment. The performance of the epoxy resin composite material with 50nm-MgO particles added in the liquid nitrogen environment is the best.

From the figure 1, it can be concluded that the electric strength increases firstly and then decreases with the increase of the doping amount of nano-MgO particles, but all are greater than the electric strength of pure epoxy. When the nano-MgO doping amount is 3wt%, the electric strength of the nano-MgO/EP composite dielectric reaches the maximum. This shows that a slight amount of nano-MgO doping can increase the electrical strength of the epoxy resin. However, as the MgO doping amount increases, the electrical strength of the epoxy resin decreases. This may be because when the nano-particles are doped, the interaction areas formed between the particles and the epoxy matrix are far apart and no overlap is formed. At this point, the interaction zone can be seen as isolated cells. Carriers can hardly overcome barriers to participate in conduction, so the electrical frequency of the power frequency increases. But when the doping amount of nanoparticles is increased, the spacing between adjacent particles decreases. It will form many local conduction channels inside the sample. Under the action of an electric field, carriers travel along these conductive paths, resulting in a decrease in the electrical strength.

3.2. Dielectric properties test results

Dielectric properties refer to the response of polymer molecules to an applied electric field, which can be characterized by dielectric constant and dielectric loss. The dielectric properties were measured at a test frequency of 50Hz. During the test, the sample was applied a bias voltage of 0.05V.

![Dielectric constant of MgO/EP nanocomposites](image)

From the figures 2, it can be seen that the dielectric constant of MgO/EP nanoparticles is obviously decreased in the liquid nitrogen environment. The dielectric constant of the pure epoxy resin composite is 3.237 in liquid nitrogen environment, a decrease of about 15.6% compared with the dielectric constants at normal temperature.
Figure 3 shows that when the doping amount of MgO particles is more than 1wt%, the dielectric loss of composite dielectrics increases with the increase of the doping amount of nano-MgO particles. It may be because of the overlap of interaction regions, the formation of local conduction channels inside the samples and the increase of conductance loss. Low temperature has a great influence on the dielectric loss of composite dielectrics, and the dielectric loss of all the samples decrease significantly in liquid nitrogen environment.

4. Conclusions
After adding a certain amount of nano-MgO particles to the epoxy resin, the breakdown strength properties can be improved. With the increase of the doping amount of nano-MgO particles, the breakdown strength of the composite dielectric increases firstly and then decreases. When the doping amount of nano-MgO particles is 3wt%, the breakdown strength of the composite dielectric reaches the maximum. The breakdown strength of the composite dielectrics in liquid nitrogen environment is greater than that at room temperature. After a certain amount of nano-MgO particles are added to the epoxy resin, the dielectric constant of composite dielectrics decreases. Low temperature will decrease the dielectric constant of composite dielectrics. The dielectric loss of the MgO/EP nanocomposites increases when it is placed in liquid nitrogen environment. These experimental data provide a reference for the insulation design of superconducting equipment.

Acknowledgments
This research was financially supported by China Electric Power Research Institute innovation foundation (Grant NO.DG83-18-001).

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