Hot Pressing Recovery of Epoxy Insulating Material Cured with Anhydride based on Triethanolamine

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Abstract. Scholars in the electrical field hope to use catalysts to improve the curing system of acid anhydride epoxy insulating materials, so as to solve the problem that a large number of waste insulating materials can not be effectively recycled. One of the most important research directions is to select a suitable catalyst to realize the recovery of epoxy by hot pressing. Therefore, we prepared epoxy samples based on the published triethanolamine catalytic anhydride cured epoxy formula. Then, hot pressing recovery tests were carried out at 180, 200, 220 and 240°C to test the insulating properties of the samples, and to study the changes of dielectric constant, dielectric loss and breakdown field strength with the hot pressing recovery temperature. The results show that with the increase of hot pressing recovery temperature, the dielectric constant of the sample increases, the dielectric loss decreases first and then increases, and the breakdown field strength increases first and then decreases. The insulation properties of the samples recovered by hot pressing at 200°C are the best. Triethanolamine catalyzed anhydride curing epoxy can realize hot pressing recovery of insulating materials.

Keywords: Triethanolamine, Anhydride cured epoxy, Hot press recovery, dielectric properties, Electrical strength

1 Introduction

Epoxy cured with acid anhydride has the advantages of good thermal stability, high mechanical strength and good insulation performance, and has been widely used in the field of electrical equipment [1]. However, the material is difficult to recycle, resulting in the accumulation of a large amount of waste epoxy insulating materials, which pollutes the environment. In order to solve this problem, the scholars in the electrical field hope to use catalysts to improve the curing system of acid anhydride epoxy insulating materials [2]. One of the most important research directions is to select a suitable catalyst to realize the recovery of epoxy by hot pressing [3]. However, a large number of

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existing research contents mainly focus on the selection and exploration of catalysts, and there are few reports on the investigation of hot pressing recovery conditions of epoxy insulating materials [4-5]. Therefore, it is necessary to study the hot press recovery temperature of the catalyzed anhydride cured epoxy.

Dielectric properties are important parameters that reflect the insulation properties of insulating materials. Therefore, studying the dielectric properties of epoxy is of great significance for evaluating the insulation status of transformers[6]. At present, researchers at home and abroad have conducted a lot of research on the dielectric properties of epoxy. Kajal et al.[7] studied the curing mechanisms of different epoxy curing systems and analyzed the effects of different curing agents and coupling agents on the dielectric properties of composite materials. Ibrahim, Joshi et al.[8-9] studied the impact of nanoparticles on the dielectric properties of epoxy insulation materials by adding nanosilver and nanosilica respectively. Zhang Yanqing et al.[10] studied the dielectric properties of epoxy /montmorillonite composite materials. At present, most of the research on the dielectric properties of epoxy focuses on the influence of modified materials on the dielectric properties. At present, there are few researches on dielectric properties of epoxy insulating materials after hot pressing recovery. In addition, the hot pressing recovery temperature is different, and the state of the material after hot pressing is also different, which leads to changes in performance [11]. Therefore, it is of great significance to study the change of dielectric properties of the epoxy cured by anhydride after catalysis at different temperatures after hot pressing recovery for the later iteration of transformer insulation materials.

In this paper, we prepared epoxy wafer samples using a published formulation of an anhydride cured epoxy resin catalyzed by triethanolamine [12]. The hot pressing recovery test was carried out at 180, 200, 220 and 240°C. After hot pressing recovery, the samples obtained at each temperature were collected and their dielectric properties and breakdown field strength were tested. The effects of different hot press recovery temperatures on the dielectric properties and breakdown field strength of the recovered epoxy were studied to provide experimental basis for the application of the new type of acid anhydride cured epoxy in the electrical field.

2 Experiment

2.1 Materials

Bisphenol A diglycidyl ether: Shanghai Anaiji Chemical Co., Ltd., purity 98%. Methyl hexahydrophthalic anhydride, triethanolamine: Chengdu Kelong Chemical Co., Ltd., analytical grade.

2.2 Apparatus

Broadband dielectric spectrometer: German NOVOCONTROL company, model NEISYS. Vacuum drying oven, electric heating constant temperature blast drying oven: China Thermo Fisher Company. Power frequency breakdown platform. Plate vulcanizing machine.

2.3 Sample preparation

Bisphenol A diglycidyl ether and methyl hexahydrophthalic anhydride were added into the glass container according to molar ratio 1:1, and vacuumed at 80°C for 10min [13]. After mixing evenly, add triethanolamine at 0.1 molar ratio and continue mixing evenly for a short time. The mixture was poured into the metal mold after preheating and spraying release agent, and cured according to the curing process of 100°C 2h+130°C 2h+170°C 2h. After curing, the sample of a new type of acid anhydride cured epoxy with a thickness of 1 mm was obtained.

2.4 Sample Recovery by Hot Pressing

The 2.2g powder sample was put into the round metal mold, and the sample was hot-pressed for 5h at a pressure of 10MPa by a plate vulcanizer, and the temperature was set at 180, 200, 220, 240°C. A complete circular material is obtained after hot pressing recovery.

2.5 Dielectric Property Test

A wideband dielectric spectrometer with a diameter of 40mm was used to test the electrode in the frequency range of 1~106Hz, and the dielectric properties of epoxy samples were obtained.

2.6 Breakdown Field Strength Test

The test AC breakdown voltage is composed of power frequency AC power supply, protection water resistance, voltage divider and ball gap electrode unit. The electrode unit uses a ball electrode (copper material) with a diameter of 20mm. The electrode and the sample are immersed in 25# transformer oil.

3 Results and Discussion

3.1 Hot Press Recovery

The principle of sample hot pressing recovery is shown in Figure 1. There is a trans-esterification reaction catalyzed by the tertiary nitrogen structure in the epoxy insulating material prepared by us. The activity of this reaction is low in the daily working environment. Under high temperature conditions, this reaction will be activated, and the damaged molecular fragments can be crosslinked again, so that the material can be hot processed.

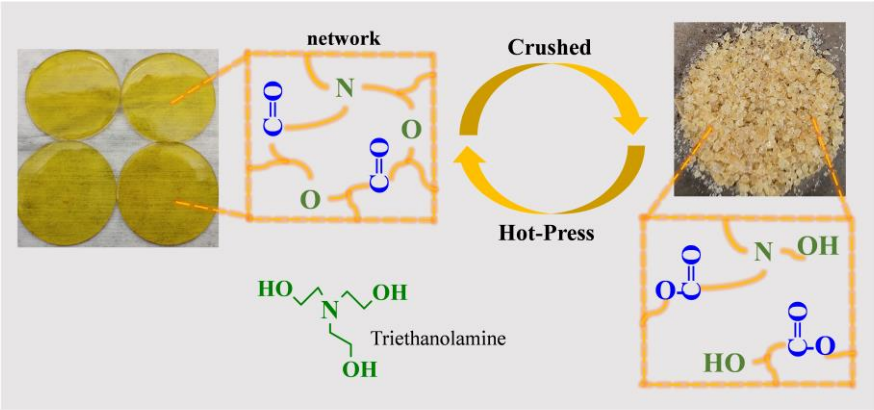

Fig. 1. Hot press recovery principle

3.2 Permittivity

The dielectric constant spectra of epoxy recovered by hot pressing at different temperatures are shown in Figure 2. The dielectric constants of epoxy samples recovered by hot pressing under the conditions of 180, 200, 220 and 240°C at power frequency 50 Hz are shown in Table 1.

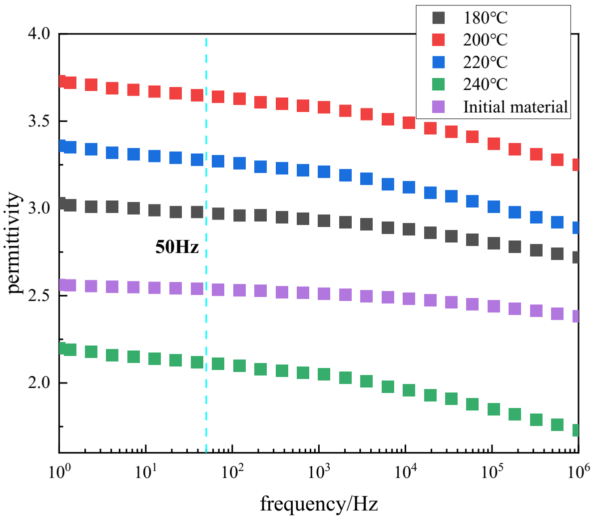

Fig. 2. The dielectric constant of the material

It can be seen from FIG. 2 that in the frequency range of 1~106Hz, the dielectric constant of the sample decreases with the increase of frequency. This is because relaxation polarization loss decreases with increasing frequency, while displacement polarization increases, resulting in dielectric constant decreasing with increasing frequency.

As can be seen from Table 1, at the same frequency, the dielectric constant of the sample increases first and then decreases with the increase of hot pressing recovery temperature. This is because when the hot pressing temperature is below 200 ° C, the polymer is constantly cross-linked and reshaped, resulting in an increase in the number of dipoles inside the material, and therefore an increase in the dielectric constant. With the increase of hot pressing temperature, the weak point on the molecular chain of epoxy insulation material is broken by heating, and the dipole concentration decreases, resulting in the decrease of dielectric constant.

Electrical equipment requires that the dielectric constant of epoxy insulating materials is in the range of 2-4. The above results show that the dielectric constant of hot pressing recovered materials can meet the requirements of insulating materials.

Table 1. Dielectric properties analysis table of materials

Hot pressing temperature /°C	Dielectric constant at 50Hz	Dielectric loss at 50Hz
180	2.99	0.00652
200	3.61	0.00417
220	3.42	0.00754
240	2.25	0.00858
Initial material	2.53	0.00394

3.3 Dielectric Loss

The dielectric loss spectra of epoxy recovered by hot pressing at different temperatures are shown in Figure 3. The dielectric losses of epoxy samples recovered by hot pressing at 180, 200, 220 and 240°C at power frequency 50 Hz are shown in Table 1.

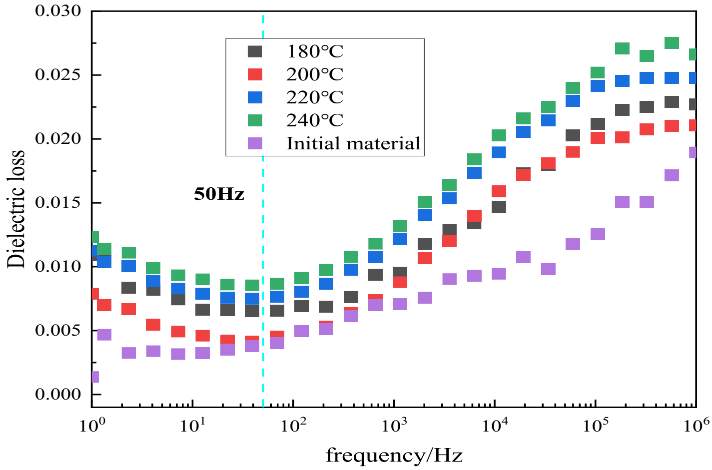

Fig. 3. Dielectric loss of material

As can be seen from FIG. 3, in the frequency range of 1~106Hz, the dielectric loss of the sample decreases first and then increases with the increase of the electric field frequency, and the loss peak appears near the frequency of 105Hz. This is because in the low frequency region, the dielectric loss is completely determined by the conduction loss, which is inversely proportional to the frequency. In the frequency range of 102~105Hz, with the increase of frequency, the relaxation loss increases, and the dielectric loss also increases.

In Table 1, at the same frequency, the dielectric loss of the material decreases first and then increases with the increase of the hot pressing recovery temperature. This is because when the hot pressing temperature is below 200 ° C, the polymer is continuously reshaped from the crosslinking, resulting in the reduction of free radicals within the material, the reduction of polarization loss, and therefore the reduction of dielectric loss. With the increase of hot pressing temperature, the chain segment of epoxy deteriorates, the polar groups are easier to rotate, the relaxation polarization loss increases, and the dielectric loss increases.

Electrical equipment requires that the dielectric loss of epoxy insulating materials should be less than 0.006. The above results show that the materials recovered by hot pressing at 200°C can meet the requirements of insulating materials, while the dielectric properties of materials recovered at other temperatures are significantly degraded.

3.4 Breakdown Field Strength

The breakdown field strength of epoxy recovered by hot pressing at different temperatures is shown in Figure 4.

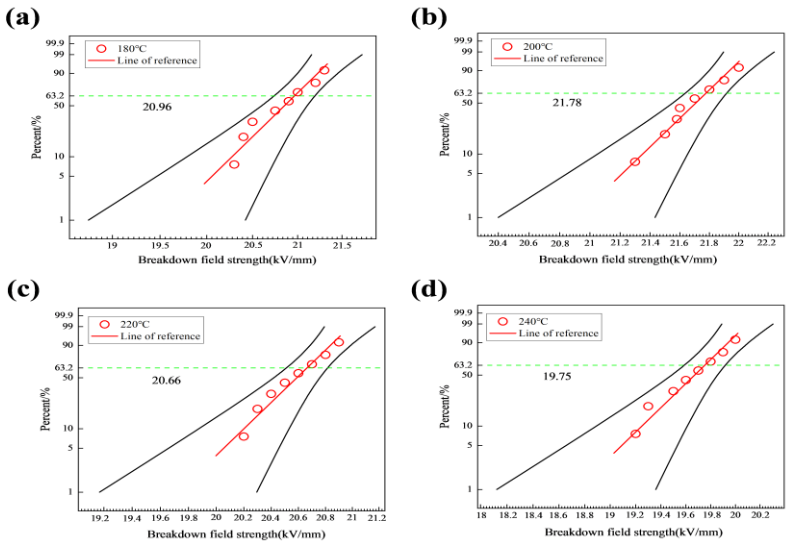

Fig. 4. Breakdown field strength of material

As can be seen from FIG. 4, with the increase of hot pressing recovery temperature, the breakdown field strength of recovered samples first increases and then decreases. This may be because when the hot pressing temperature is below 200 ° C, the interior of the material is constantly cross-linked and reshaped, the chain segment tends to be complete, and the insulation capacity is improved. When the hot pressing temperature continues to increase, the molecular chain of the resin matrix breaks or the molecular weight distribution area widens, and the electron passability in the molecular chain increases, resulting in a decrease in the breakdown field strength.

4 Conclusion

(1) At the same frequency, with the increase of hot pressing recovery temperature, the dielectric loss of triethanolamine catalyzed anhydride cured epoxy sample first decreased and then increased, and the breakdown field strength first increased and then decreased.

(2) When the hot pressing recovery temperature is 200°C, the insulating performance of the recovered material is the best, and it can still meet the requirements of insulating materials.

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