



Yuanxin Yao¹, Meng Huang ^{1,*}, Jie Ma^{1,2}, Yanxiao Su¹, Sheng Shi¹ and Chunhe Wang¹

- State Key Laboratory of New Energy Power System, School of Electrical and Electronic Engineering, North China Electric Power University, Beijing 102206, China
- ² State Grid Beijing Urban Power Supply Company, Beijing 100031, China
- Correspondence: hm2016@ncepu.edu.cn

Abstract: The hot pressing parameters and fiber ratio have an important influence on the dielectric properties of aramid insulating paper. In order to deeply explore its influence and the mechanism behind it, aramid insulating papers were made with different hot pressing temperatures and pressures as well as fiber ratios. Its tightness, dielectric constant, and AC breakdown strength were tested, and its microstructure was analyzed by scanning electron microscopy. It was found that with an increase in hot pressing temperature, pressure, and fibrid content, the overall dielectric constant of the insulating paper showed a slight upward trend, while the tightness and AC breakdown strength continued to increase. Hot pressing temperature and pressure have a synergistic effect on the dielectric properties of insulating paper. The effects of these two parameters on the dielectric properties of insulating paper are similar, while the AC breakdown strength is greatly affected by the fiber ratio. In this paper, the influence mechanism by which the microstructure and fiber crystallinity of insulating paper is affected in the hot pressing process is discussed.

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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Keywords: aramid insulating paper; dielectric constant; breakdown strength; hot pressing; fiber ratio

1. Introduction

Electric energy occupies an extremely important position in production and life. Due to the uneven distribution of power resources in China, power transmission is developing in the direction of high voltage and large capacity [1]. Aramid insulating paper has high electrical and mechanical properties and excellent high-temperature resistance. It has been used in power transformers to improve the oil-paper insulation performance of transformers and prolong their service life [2,3], and as a potential substitute for insulating paper in oil-immersed transformers.

Aramid insulating paper is made from aramid chopped fiber and aramid fibrid, and it is prepared by wet forming and hot pressing. An increase in shear rate and aramid solution concentration affects the shear force and double diffusion process, which in turn affects the properties of the fibrids, such as their length, specific surface area, and crystallinity. When they are larger, the fibrids have a larger film-like structure, which helps increase the bonding force and bonding area between fibers, reduce the number of pores in the paper, and thereby improve the mechanical strength, breakdown strength, and dielectric strength of the fibers. The chopped fiber is used as the skeleton material, which is evenly dispersed in the paper, and the precipitating fiber plays the role of filling and bonding [4]. During the hot pressing process, the surfaces of the precipitating fibers and the chopped fibers are softened by heat, the bonding force between the fibers is increased, and the fibers are connected more tightly. Therefore, a change in temperature and pressure during the hot pressing of aramid insulating paper has a great influence on the structure of the insulating paper, which will directly affect its dielectric properties.

Hot pressing technology is the most critical link after fiber forming. Due to technical confidentiality issues, there is not much published literature on hot pressing technology



outside China [5]. Some patents published by DuPont mention the hot pressing process of aramid paper [6–8], and these are significant for guiding the analysis of the hot pressing process of aramid paper. In China, the research and development of aramid paper has been carried out since the 1970s. At present, Yantai Taihe New Materials, Shanghai Shengou Group, and other companies have realized the industrial production of aramid paper, and relevant research institutes have carried out research on the preparation and structure of aramid insulating paper. Lu Zhaoqing et al. [9] found that hot pressing has a great influence on the performance of paper, i.e., that it can enhance the bonding force between fibers and greatly improve the mechanical properties of aramid paper [10]. Zhao H et al. [11] measured the molecular weight and crystallinity of fibers and found that lower crystallinity and wider molecular weight distribution range were the main reasons for the low mechanical properties of domestic aramid paper. Li Jinbao et al. [12] studied the effects of hot pressing temperature and pressure on the mechanical and compressive strength of aramid paper. The compressive strength of aramid paper first increased and then decreased with the increase in temperature and pressure. Yang Bing et al. [13] used orthogonal tests to analyze the influence of various parameters on the mechanical properties of insulating paper during the hot pressing process.

At present, the influence mechanism of the hot pressing process affecting the mechanical properties and structure of insulating paper has been studied, but different hot pressing parameters are likely required for different insulating papers.

In this paper, aramid insulating papers with different hot pressing temperatures and pressures as well as fiber ratios were produced in order to study the variation law of the dielectric properties of the meta-aramid insulating paper and the hot pressing process. Its tightness, dielectric constant, and AC breakdown strength were tested, and its microstructure was analyzed by scanning electron microscopy.

2. Experimental Details

2.1. Preparation of Insulating Paper Samples

The beating degree of the selected fibrids was 55.3°SR, and the length of the chopped fibers was 6 mm. The insulating paper samples were prepared using the laboratory synthetic fiber insulating paper preparation platform. The steps were as follows:

Firstly, set the basis weight of the paper to 90 g/m^2 , calculate and weigh the fibers according to the ratio of chopped fiber mass to precipitating fiber mass ratio of 1:1.5. Secondly, use a fiber disintegrating machine to decompose and disperse precipitating fiber and chopped fiber to obtain pulp. Thirdly, prepare the pulp using a sheet former to obtain a wet sheet of insulating paper, and vacuum dry it; Finally, transfer the insulating paper to the hot pressing equipment and process the aramid insulating paper according to a certain hot pressing temperature and pressure. Perform the hot pressing to obtain the insulating paper, and seal and store the aramid insulating paper in a sealed bag for subsequent experiments.

Aramid fibrids have an amorphous structure, and the glass transition temperature of amorphous aramid polymer molecules is about 280 °C. Considering the effect of pressure, the fibrid can also recrystallize when its temperature is lower than the glass transition temperature. Too high a temperature and pressure will destroy the chemical bonds of aramid macromolecules and reduce the relative molecular mass [14], so in the test process, the hot pressing temperature was set to 220–260 °C, and the pressure was set to 8–16 MPa. In aramid insulating paper, the precipitant fiber content usually accounts for more than half. In order to clearly observe the difference in the influence of different fibrillar fiber content on the insulating paper, the ratio of chopped fiber to precipitant fiber was set to 2:1, 1.51, 1:1, 1:1.5, and 1:2. During the experiment, except for the hot pressing temperature, hot pressing pressure, and fiber ratio, other process parameters and steps were kept the same.

2.2. Preparation of Oil-Impregnated Insulating Paper Samples

The prepared insulating paper was cut to a suitable size and dried in a vacuum drying box at 105 $^{\circ}$ C for 24 h. The dried insulating paper was then immersed in oil with

filtered transformer oil, placed in a vacuum drying box at 90 $^{\circ}$ C for 24 h, and used when the temperature dropped to room temperature.

2.3. Dielectric Constant Test

The size of each sample was 10 cm \times 10 cm, and the dielectric constant was measured by Haefely 2830/2831 precision liquid–solid dielectric analyzer. The applied voltage was 1 kV, and the value was taken after the indication was stable. Each sample was measured three times, and the result was recorded as an average value.

2.4. AC Breakdown Characteristic Test

The power frequency breakdown strength of the aramid insulating paper was tested according to the standard ASTM D149-2009. The size of each sample was 5 cm \times 5 cm, and the electrode was a symmetrical stainless steel cylindrical electrode with a diameter of 25 mm. The number of each test sample was 10, and the average value was taken as the final result.

3. Results

3.1. Tightness of Paper

3.1.1. Influence of Hot Pressing Temperature

The change in the tightness of the aramid insulating paper is shown in Figure 1. When the hot pressing temperature increased from 220 °C to 240 °C, it can be seen from Figure 2 that the tightness of the insulating paper increased greatly, from 0.56 g/cm³ to 0.61 g/cm³. After that, with the further increase in the hot pressing temperature, the tightness gradually became stable.

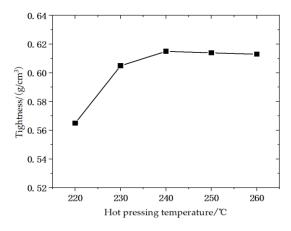


Figure 1. Tightness of insulating paper with different hot pressing temperatures.

3.1.2. Influence of Hot Pressing Pressure

Figure 2 shows the test results for the tightness of the aramid insulating paper under different pressures. With the increase in hot pressing pressure, the tightness of the aramid paper increased continuously. When the pressure reached 14 MPa, the tightness settled at a stable value of 0.58 g/cm^3 .

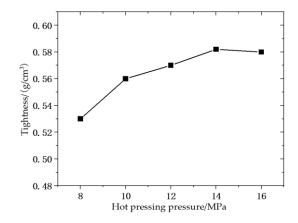


Figure 2. Tightness of insulating paper with different hot pressing pressures.

3.1.3. Influence of Fiber Ratio

Figure 3 shows the change in the properties of the aramid insulating paper after changing the fiber ratio. With the increase in the precipitant fiber content, the tightness increased significantly, from 0.46 g/cm^3 to 0.56 g/cm^3 .

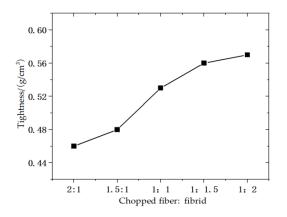


Figure 3. Tightness of insulating paper with different fiber ratios.

3.2. Dielectric Constant

3.2.1. Influence of Hot Pressing Temperature

The change in the dielectric constant of the aramid insulating paper before and after oil immersion is shown in Figure 4. It can be seen from Figure 4 that the hot pressing temperature has little effect on the dielectric constant. With the increase in the hot pressing temperature, the overall dielectric constant showed only a slight increase.

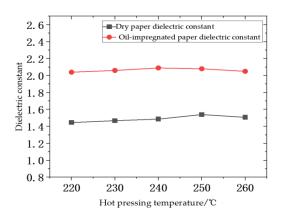


Figure 4. Dielectric constant of insulating paper with different hot pressing temperatures.

3.2.2. Influence of Hot Pressing Pressure

Figure 5 shows the test results for the dielectric constant of the aramid insulating paper before and after immersion in oil under different pressures. It can be seen from Figure 5 that when the hot pressing pressure was low, the dielectric constant increased significantly before and after oil immersion, and that the dielectric constant did not change much with the increase in the hot pressing pressure.

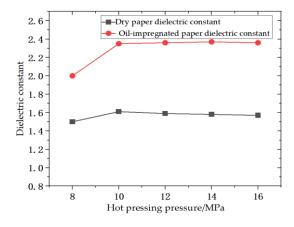


Figure 5. Dielectric constant of insulating paper with different hot pressing pressures.

3.2.3. Influence of Fiber Ratio

Figure 6 shows the change in the properties of the aramid insulating paper after changing the fiber ratio. With the increase in the precipitant fiber content, the dielectric constant of the aramid paper before and after oil immersion showed an upward trend.

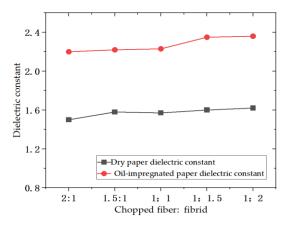


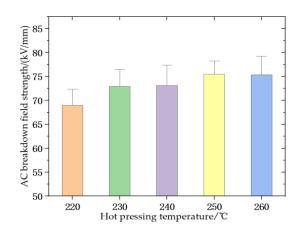
Figure 6. Dielectric constant of insulating paper with different fiber ratios.

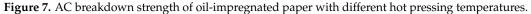
3.3. AC Breakdown Strength

3.3.1. Influence of Hot Pressing Temperature

The AC breakdown strength test results for the oil-impregnated insulating paper are shown in Figure 7.

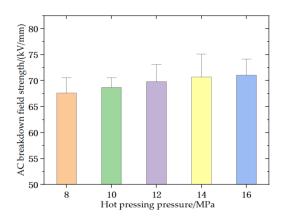
With the increase in the hot pressing temperature, the AC breakdown strength gradually increased. When the hot pressing temperature increased from 220 °C to 250 °C, the AC breakdown strength increased rapidly, from 68.95 kV/mm to 75.48 kV/mm. The breakdown strength tended to stabilize.

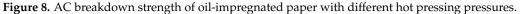




3.3.2. Influence of Hot Pressing Pressure

Figure 8 shows the AC breakdown strength test results for the oil-impregnated paper under different hot pressing pressures.





When the hot pressing pressure increased from 8 MPa to 14 MPa, the AC breakdown strength increased from 67.59 kV/mm to 70.67 kV/mm, an increase of 4.5%, and the growth rate slowed down when the hot pressing pressure was greater than 14 MPa.

3.3.3. Influence of Fiber Ratio

Figure 9 shows the AC breakdown strength test results for the oil-impregnated paper under different fiber ratios.

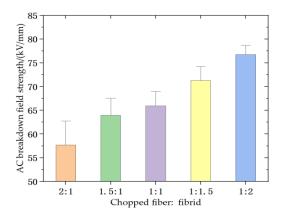


Figure 9. AC breakdown strength of oil-impregnated paper with different fiber ratios.

When the fibrid content increased, the AC breakdown field strength of the oil-impregnated paper increased from 57.7 kV/mm to 77.3 kV/mm, and when the ratio of chopped fibers to fibrids was 1:2, the AC breakdown field strength increased from 57.7 kV/mm to 77.3 kV/mm. The breakdown strength did not show a slowing trend.

4. Discussion

4.1. Variation of Tightness

Aramid insulating paper can be regarded as a composite insulating material composed of insulating paper and air or transformer oil. The dielectric constant of insulating paper is related to the content of the pores in the paper and the degree of polarization of its own material [15]. Through the adjustment of the fibrid preparation process, the change in the fibrid morphology changes the connection structure of the fibers in the aramid insulating paper, thereby affecting the change in the dielectric constant and the properties of the fibers themselves. To a certain extent, the tightness can represent the change in the pores in the paper.

It can be seen from Figure 10 that under a higher temperature and hot pressing pressure, and with a higher fiber ratio, the surface of aramid insulating paper has fewer pores, a smoother surface, and a tighter structure. Each variable, whether it is hot pressing temperature, hot pressing pressure, or fiber ratio, affects the fiber pores of the aramid paper, resulting in changes in tightness.

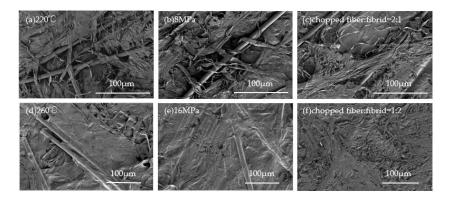


Figure 10. Micromorphology of insulating paper with different hot pressing temperature, pressure, and fiber content.

4.2. Variation of Dielectric Constant

It can be seen from the experimental data that the dielectric constant of aramid insulating paper increases with an increase in hot pressing temperature, pressure, and precipitating fiber content. In the process of changing the preparation process, there were two main changes in the aramid insulating paper. On the one hand, the preparation process changed the structure of the paper sheet, which caused changes in the process in the insulating paper. On the other hand, the fiber material itself changed due to the preparation process.

With the increase in hot pressing temperature and pressure, the tightness of the insulating paper increased and the porosity decreased, which increased the value of the dielectric constant of the tested sample. While changing the structure of the paper sheet, the parameters of hot pressing also affected the properties of the fiber itself. After hot pressing, the insulating paper could recrystallize the fibers, thereby changing the crystallinity of the insulating paper. Increasing the hot pressing temperature and pressure alone could increase the crystallinity [16]. In order to facilitate an intuitive analysis, XRD analysis was carried out on the aramid insulating paper with different hot pressing temperatures. The diffraction images are shown in Figure 11. The aramid insulating paper appears at the diffraction angle 20 of 18.3°, 22.6°, and 27.3°. The sharp characteristic peaks indicate the existence of several different crystal types in the aramid insulating paper. The crystallinity of the aramid insulating paper was calculated by peak fitting. Hot pressing temperatures of 220 °C and 260 °C resulted in relative crystallinities of 66.3% and 76.2%, respectively. The increase in the hot pressing temperature increased the crystallinity of the fiber and decreased dielectric constant of insulating paper, which is beneficial. The change in the dielectric constant of the aramid insulating paper can be considered the result of the combined effect of the pores and the crystallinity of the paper, but when the hot pressing parameters are changed, the change in the dielectric constant of the insulating paper is roughly the same as the change in the tightness, and it has an upward trend, which is similar to the crystallinity. This shows that in the process of changing the preparation process, the content of the pores of the insulating paper plays a dominant role in the change in the dielectric constant.

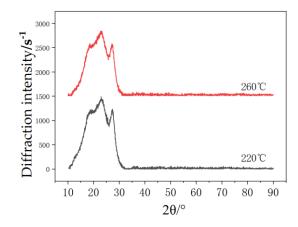


Figure 11. XRD diagrams of aramid insulating paper.

Since the precipitating fiber acts as a binder in the insulating paper, when the precipitating fiber content increases, the fibers will bond more tightly, and the number of pores in the insulating paper will be reduced, so that the tightness and dielectric constant of the insulating paper become uniform. This result is consistent with the experimental results obtained by Liao Ruijin et al. [17]. At the same time, the change in the fibrid content changes the material of the insulating paper. When the crystallinity of the fibrids is low, the crystalline structure can limit the orientation polarization and relaxation movement of the aramid macromolecules, which is beneficial for reducing the dielectric constant of the insulating paper [18]. When the fiber ratio is changed, the precipitating fiber content not only affects the pores in the paper, but it also affects the polarization degree of the insulating paper itself, which in turn affects the change in the dielectric constant.

4.3. Variation of AC Breakdown Strength

Usually, the pores in the paper can be regarded as the starting point of discharge. Under the action of the external electric field, the electrons at the pores are prone to form electron avalanches, and further development leads to the breakdown of the insulating paper [19]. It can be understood intuitively that the AC breakdown strength of insulating paper increases with an increase in hot pressing temperature, pressure, and precipitating fiber content. In order to further analyze and discuss the test results, a scanning electron microscope (SEM) was used to observe the micro-morphology of the aramid insulating papers with different preparation processes. The results are shown in Figures 12 and 13.

Figure 12 includes $50 \times$ and $300 \times$ magnification images for different preparation processes. It can be seen from Figure 12 that when the hot pressing parameters and the precipitating fiber content are low, there are many holes between the chopped fibers and the precipitating fibers in the insulating paper, and individual chopped fibers exist in isolation. The images clearly show the outlines of single fibers and the free state of the precipitating fibers. In Figure 12d,e, the hot pressing parameters for preparing the aramid paper were low, and the precipitating fibers showed a ribbon-like shape, which failed to fully fill the pores and wrap the chopped fibers. In Figure 12f, the chopped fiber content is high, there are not enough fibrids to fill the pores in the paper, and there are large pores in some areas. When the hot pressing parameters and the fibrid content increase, the fibrids have better wrapping properties for the chopped fibers, as shown in Figure 13. At this time, the fibrids wrap around the chopped fibers, and the fibers are thus tightly bound [20]. When the hot pressing temperature and pressure increase, the softening area and degree of the precipitating fibers increase, and therefore they can better fill the pores between the fibers. The increase in the hot pressing pressure increases the softening degree of the precipitating fibers and leads to slight deformation and migration, filling the pores between the fibers and making the surface of the paper flatter, thus further improving the breakdown strength of the insulating paper. Following an increase in the precipitating fiber content of the insulating paper, the bonding area and bonding force between the fibers are increased, the number of pores in the paper is reduced, and the AC breakdown field strength of the insulating paper is increased.

By analyzing the change trend of the AC breakdown strength of the insulating paper, it can be seen that when the hot pressing temperature, pressure, and precipitating fiber content increase, the AC breakdown strength increases by 9.5%, 5.2%, and 32.3% respectively. Increasing the precipitant fiber content has a more obvious effect on the improvement of the AC breakdown field strength, and increasing the hot pressing parameters contributes more to the surface flatness of the insulating paper. The process of hot pressing aramid insulating paper is the result of the synergistic effect of hot pressing temperature and pressure. During the hot pressing process, the precipitation fiber acts as a binder. Under high temperature conditions, the precipitation fibers are softened by heat, and with the action of pressure, the softened precipitation fibers undergo slight deformation and migration, so that the fibers are tightly connected, reducing the number of pores in the paper and increasing the breakdown field strength.

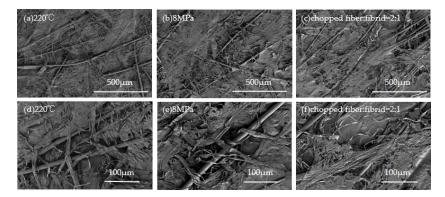


Figure 12. Micro-morphology of insulating paper when hot pressing temperature, pressure, and precipitating fiber content are low.

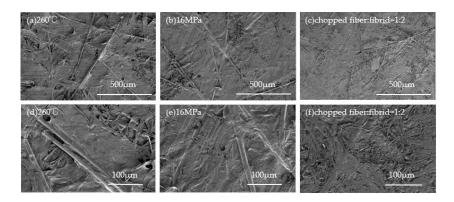


Figure 13. Micromorphology of insulating paper when hot pressing temperature, pressure, and precipitating fiber content are high.

Changes in the hot pressing parameters and fiber ratio of insulating paper can have a great influence on the performance of the insulating paper. When the hot pressing parameters are low and the precipitating fiber content is low, there are a lot of pores between the fibers. After the oil immersion treatment, the internal pores are filled with transformer oil. Under the action of the external electric field, the electric field strength at the pores is relatively large and it is easy to form a discharge point, resulting in the breakdown of the insulating paper [21]. When the hot pressing parameters and the precipitating fibers content is increased, the precipitating fibers are softened by heating and undergo slight deformation and migration, filling the pores in the insulating paper and causing the number of pores and discharge points in the paper to reduce or disappear, thereby increasing the AC breakdown field strength. In addition, within the set experimental parameters, the change trend of the AC breakdown strength is more obvious when the temperature is changed, compared with the pressure. During the hot pressing process, the thermal softening process of the precipitating fibers is mainly affected by temperature [20], and its change has a great impact on the internal structure of the insulating paper, which in turn has a great impact on the AC breakdown strength.

5. Conclusions

- (1) With the increase in hot pressing temperature and pressure, the tightness of the aramid insulating paper gradually increased, and the increase range was larger, while the overall dielectric constant showed a slight upward trend. The AC breakdown strength of the aramid insulating paper increased continuously with the increase in hot pressing parameters, and then slowed down and stabilized.
- (2) During the hot pressing process, the change in the dielectric constant of the aramid insulating paper was jointly affected by the crystallinity of the fibers and the pores in the paper. Hot pressing temperature and pressure had a synergistic effect on the dielectric properties of the insulating paper, and the two had a similar effect on the dielectric properties. Within the parameters studied in this paper, hot pressing temperature had a greater impact on the breakdown strength of the insulating paper.
- (3) With the increase in fibrid content, the tightness of the aramid insulating paper increased significantly. The dielectric constant of the aramid paper before and after oil immersion showed an upward trend, and the AC breakdown field strength of the oil-impregnated paper gradually increased.

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Data Availability Statement: The data presented in this study are available on request from the corresponding author. The data are not publicly available due to the confidentiality of the project involved.

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