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Study of Polyamide Coating on Aluminum Substrate by Different Methods

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STUDY OF POLYAMIDE COATING
ON ALUMINUM SUBSTRATE BY
DIFFERENT METHODS

By

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The Office of Graduate Studies has verified and approved the above named committee members.
This manuscript is dedicated to my deceased grandparents, my parents, my brother’s family and my sister’s family with my deepest love. I also give my appreciation to all those people who generously helped me before. Without their years of care and sacrifices, I wouldn’t have achieved my goal. I will continue to furthest my goal under their care. God bless them!
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ABSTRACT

A 3.5MVA High Temperature Superconductor (HTS) transformer has been in design at FAMU-FSU College of Engineering since 2002. The coils of the HTS transformer use stainless steel sheathed Bi2223 HTS wire. In order to make insulation around the HTS transformer coils, research has been conducted. The results of this research are the main contents of this thesis.

Since Siemens constructed the first HTS transformer in 1997, several HTS transformer projects have been developed around the world. In these HTS transformers, coils generally were wrapped by insulation materials. This insulation method is not ideal since wrapped insulation has disadvantages: displacement of insulation materials, low efficiency and variable insulation effect. In order to find a better approach, we wanted to investigate polymer coatings around HTS transformer coils.

Polyamide 6 coatings were made on aluminum substrate utilizing dipping, brush and spray methods. After which, their properties were studied, including density, breakdown strength, dielectric constant and dissipation factor. Through comparison of properties, spray coatings show the best insulating effect. Our research also included influence of heat treatment and vacuum treatment on spray coatings. Heat and vacuum treatments effect on breakdown strength is insignificant. Vacuum treatment can greatly increase the density of the coating. Heat treatment influences the dielectric constant and dissipation factor. But the breakdown strength of spray coatings is only one fifth of that of commercial polyamide film. And the
density of spray coatings is about 60% of that of commercial polyamide film. All studies about polymer coating were conducted at room temperature.
CHAPTER 1

INTRODUCTION

1.1 High temperature superconductor transformers

1.1.1 Review of high temperature superconductor transformers

In order to find a new means to produce good electrical insulation coatings around coils of high temperature superconductor (HTS) transformers, research has been carried out over the past two years [1]. This thesis is a conclusion of previous research.

Transformers are a common and important power apparatus. Even though manufacturing conventional transformers is a mature industry, the discovering of HTS in 1986 has brought new opportunity and challenge to this mature industry. Since then, many projects have been carried out to study the application of HTS transformer around the world. Major HTS transformer projects among them are listed in Table 1.
<table>
<thead>
<tr>
<th>Year</th>
<th>Country</th>
<th>Rated power</th>
<th>HTS material</th>
<th>Electrical insulating</th>
</tr>
</thead>
<tbody>
<tr>
<td>1994~2002</td>
<td>Sweden, (ABB)</td>
<td>630 kVA 10 MVA</td>
<td>B,2223</td>
<td>*</td>
</tr>
<tr>
<td></td>
<td>[2, 3]</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1999~[4,5]</td>
<td>USA, (WES)</td>
<td>1 MVA 5/10 MVA 35/70 MVA</td>
<td>B,2221</td>
<td>*</td>
</tr>
<tr>
<td>2000 [8]</td>
<td>Germany, (Siemens)</td>
<td>100 kVA</td>
<td>B,2223</td>
<td>Kapton</td>
</tr>
<tr>
<td>2002 [9,10]</td>
<td>Germany, (Siemens)</td>
<td>1 MVA</td>
<td>B,2223</td>
<td>Electrically insulating thermoplastic material using an extrusion process developed at Siemens</td>
</tr>
<tr>
<td>1997~[11-15]</td>
<td>Japan (Kyushu University)</td>
<td>500 kVA 800 kVA 1 MVA 3 MVA</td>
<td>B,2223</td>
<td>Glass-fiber tape, Polyvinyl fluoride (PVF) tape</td>
</tr>
<tr>
<td>2002 [18]</td>
<td>Europe</td>
<td>14 kVA</td>
<td>B,2223</td>
<td>Insulating paper</td>
</tr>
</tbody>
</table>

ASEA-Brown-Boveri (ABB); Waukesha Electric Systems (WES);  

*Don’t find information about insulation of HTS coils.
The advantages of HTS transformers over conventional transformers have been fully recognized after ten years’ investigation of HTS transformers. In Europe, ASEA-Brown-Boveri (ABB)’s first HTS transformer at 630 kVA was taken into operation in the power grid at the spring of 1997 and operated successfully for one year. In 2002 Japan, a 22 kV/6.9 kV-1 MVA HTS transformer was connected to a power grid and tested. The test data also proved HTS transformer’s applicability in practice [2, 19].

Until now, nearly all HTS transformer have utilized Bi2223 or Bi2221 wire which is also called the 1st generation HTS wire. With the appearance of the 2nd generation commercial YBCO HTS wire, HTS transformer coils will probably utilize YBCO instead of Bi2223 in the future. Research about YBCO HTS transformers has only recently begun [5, 20, 21]. Compared with Bi2223 wire, YBCO wire has advantages, including larger critical current density and lower cost. In the application of HTS transformers, cost is still a challenge [5]. At present, the price of Bi2223 wire is $200-300/kAm. It is predicted that the price of $50/kAm of HTS wire can bring commercial benefit to HTS applications [6]. The 1st generation Bi2223 HTS wire was expected to reach such a low price in the future. Comparably, the 2nd generation commercial YBCO HTS wire is expected to reach $10/kAm with larger potential of reducing price [7]. YBCO wire may provide a way to overcome this cost challenge.

A 3.5 MVA HTS transformer has been under construction in the Center for Advanced Power Systems (CAPS) at Florida State University since 2002 [1]. This HTS transformer was designed to work under vacuum with no liquid cryogen. Bi2223 high strength reinforced wire made by American Superconductor Corp. (ASC) was planed for the windings of the HTS transformer.
1.1.2 High temperature superconductor materials and Bi2223 high strength reinforced wire

Superconductivity phenomenon was first observed by Dutch physicist H. Kammerlingh Onnes in 1911. Until 1986, many superconductor materials had been found. Later, so called low temperature superconductor (LTS) were discovered because the highest critical temperature of these superconductor materials is 23 K. Such low critical temperature restricts application of superconductor due to very high cooling cost. LTS main applications are in the fields of magnetic resonance imaging machines, high-energy physics and nuclear fusion. Commercial application of LTS materials is very limited.

The discovery of high critical temperature superconductor by Georg Bednorz and Alex Muller in 1986 opened a new era. The superconductor material found by Georg Bednorz and Alex Muller, lanthanum-barium-copper oxide (La$_{2-x}$Ba$_x$CuO$_4$) ceramic, is a ceramic material with a critical temperature of 30 K. Since then, many ceramic superconductor materials with higher critical temperature have been found. To date, the highest critical temperature superconductor is mercury-barium-calcium-copper oxide (HgBa$_2$Ca$_2$Cu$_3$O$_8$) ceramic with a critical temperature of 135 K. Since superconductor materials found after 1986 have higher critical temperature, they are called high temperature superconductor (HTS).

With the discovery of more HTS with higher critical temperature, applications of HTS have attracted more attention than LTS, particularly in the commercial field. At present, Bismuth Strontium Copper Oxides (BSCCO), with 110 K critical temperature, and Yttrium-Barium-Copper Oxide (YBCO), with 92 K critical temperature, are regarded as the best candidate HTS materials for power applications. For BSCCO, it is mainly applied in two forms: Bi$_2$Sr$_2$Ca$_2$CuO (B$_{2221}$) and Bi$_2$Sr$_2$Ca$_2$Cu$_3$O (B$_{2223}$). At present, B$_{2223}$ has been more applied than B$_{2221}$ since its critical temperature is 20 K higher than B$_{2221}$. B$_{2223}$ commercial wire appeared and power applications based on B$_{2223}$ began with satisfying prospects. YBCO commercial HTS wire was recently developed in 2003. As prior mentioned, one of the advantages of YBCO wire is low cost, only one-half to one-fifth of
B\textsubscript{2}2221 wire. It makes YBCO wire more suitable for commercial applications. To present, the high cost of HTS application hasn’t been solved.

The above part is the brief about discovery, development, present status and future of HTS materials. The following focuses on HTS transformer application. Since our HTS transformer project was planned to use B\textsubscript{2}2223 material, the below discussion points to B\textsubscript{2}2223 wire.

Conventional transformer’s coils are made from common metal wire, copper or aluminum. And HTS transformer’s coils are made from HTS wire, B\textsubscript{2}2223 wire for our case. This is the basic difference between conventional transformers and HTS transformers. But the basic difference leads to many technical challenges. Compared with copper, HTS materials have new properties [Table 2]. HTS materials can’t be used as wire directly since they are ceramic materials. In prior applications, HTS material has been sheathed with silver or stainless steel by powder-in-tube (PIT) technology. In addition, HTS is a brittle crystal material. It is needed to pay more attention on them than on copper in the application. External influences in the process of transformer constructing and operating, i.e. excess mechanical stress and excess temperature, probably damage crystal structure of HTS materials, and consequently degrade the superconducting property of HTS wire.

American Superconductor’s (ASC) B\textsubscript{2}2223 high strength reinforced wire was planed to be used in CAPS’ HTS transformer project [Table 3]. This is because such HTS wire can provide both superior current capacity and mechanical robustness among American Superconductor’s B\textsubscript{2}2223 HTS commercial wires. B\textsubscript{2}2223 high strength reinforced wire is a multi-filamentary composite (MFC) architecture which is reinforced with stainless steel. It can withstand higher mechanical tensile stress than silver sheathed HTS wire. The robustness of B\textsubscript{2}2223 high strength reinforced wire meets the demand of and is a good option for HTS transformers.
Table 2 Properties comparison of B\textsubscript{2}223 and copper

<table>
<thead>
<tr>
<th>B\textsubscript{2}223</th>
<th>Copper</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ceramic material</td>
<td>Ductile metal</td>
</tr>
<tr>
<td>Can’t be used directly as wire, needs to be sheathed by silver or stainless steel carefully before application</td>
<td>Can be used as wire directly</td>
</tr>
<tr>
<td>Brittle crystal structure, fragile, attention needed when applying</td>
<td>Tough crystal structure, stout</td>
</tr>
</tbody>
</table>

Table 3 Properties of B\textsubscript{2}223 high strength reinforced wire [22]

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (avg.)</td>
<td>0.31 (± 0.02mm)</td>
</tr>
<tr>
<td>Width (avg.)</td>
<td>4.2 (± 0.2mm)</td>
</tr>
<tr>
<td>Critical Tensile Stress</td>
<td>265 MPa**</td>
</tr>
<tr>
<td>Critical Tensile Strain</td>
<td>0.4%*</td>
</tr>
<tr>
<td>Critical Bend Dia.</td>
<td>70 mm***</td>
</tr>
<tr>
<td>Min. I\textsubscript{c}</td>
<td>115 A* - 125 A* - 135 A*</td>
</tr>
</tbody>
</table>

* at 77K, self-field, 1 µV/cm; ** With 95% I\textsubscript{c} retention; *** Without back tension
1.1.3 Present insulation materials of HTS transformer coils

Figure 1 shows a simple transformer diagram. For our HTS transformer project, both coils are B\textsubscript{2}2223 wire. The aim of insulation research is to make a coating around the HTS coils which provide an electrical insulation effect between turns of coils.

![Figure 1 Illustration of transformer coils](image)

From table 1, we know that most HTS transformers have an applied polymer film or insulating paper as an insulation layer for the HTS coils. The insulation films were wound around HTS coils. The disadvantages of this film-wrap insulation technique are:

- Easy to damage crystal structure of HTS materials due to mechanical stress during wrapping resulting in a decrease in quality;
- Displacement of insulating films sometimes happens in the process of winding HTS wires as coils. Insulation quality is thus negatively affected. Mechanical stress of the whole insulation film is not uniform due to displacement [23];
- Since overlap of wrap-film exists, the thickness of the insulation is not uniform. Variable thickness of the insulation layer varies the electrical insulating effect for different sections of coils. Inconstant mechanical influence due to variable thickness of film hurts the crystal structure of HTS wire;
• Wrap of insulation material is not a productive means and does not adapt to industry production;

Another insulation coating method used in HTS transformer coils has been developed at National High Magnetic Laboratory (NHMFL), Tallahassee FL. [24-27]. The ceramic insulation coatings were made utilizing sol-gel for the high magnetic field coils. Even though this ceramic coating has successfully been applied at NHMF, its application in HTS transformer is limited by the following disadvantages:

• Generally, ceramic materials have a large dielectric constant and ceramic coating should bring great capacitance to HTS transformer. Consequently transformer operation is affected badly. Dielectric constants of ceramic coatings developed at NHMFL are larger than 10 when number of dipping is 20 [25];

• During the coating process, ceramic needs to be heated at temperature from 300 °C to 500 °C. American Superconductor Company, provider of HTS wire for CAPS’ project, introduced that treatment temperature stays below 130 °C in order to preventing from damaging HTS wires.

• Insulation islands exist for ceramic coatings. Insulation effect of the ceramic coatings is not constant;

• Ceramic coating method is a wind-and-react process, i.e. wind HTS wire into coil at first and then make ceramic coating on the surface of HTS coil. Our HTS transformer coil, however, was designed and made by react-and-wind process, i.e. make an insulation coating on HTS wire and then wind HTS wire into coil;

Based on above review of insulation methods for HTS transformer and special requirements of CAPS’ HTS transformer project, a different approach was under consideration. That is to make polymer coating around HTS wire before making HTS coils. This is also the goal of our insulation materials study: to find a better practical insulation means for HTS transformer coils.
1.2 Polymer and coating methods

1.2.1 Introduction to polymer and polyamide 6

The chemical elements in polymers are mainly carbon, hydrogen, nitrogen and oxygen, which are very common. Atoms in polymers are bonded by covalent bonds. In a covalent band, the electrons are locally held by two neighbor atoms. Most polymers have nonmetal properties, for example: low conductivity, low specific gravity, and low dielectric constant. Such nonmetal properties make polymers a good electrical insulation material. From the 1970’s, people have been discovering the conductive polymers. Since then, polymer applications have been widely expanded. Now, conductive polymers and photoconductive polymers are being applied.

Even though elements in polymers are common, properties of polymers are special, making them complicated to study. Two reasons cause polymer’s complexity. First, polymers are large molecules in which many, on the order of thousand, monomers are bonded together by covalent electron pairs. Second, polymer molecule has many spatial changes.

This thesis is about polyamide (PA), known commonly as nylon, which was invented in 1930’s by Wallace Carothers at DuPont. According to different chemical formulas, there are many kinds of nylon: nylon 4, nylon 5, nylon 6, nylon 66, and so on. Among them, nylon 6 and nylon 66 have the most applications. The nylon studied in this thesis is nylon 6 (PA6) since it is easily dissolved at room temperature. Its formula is:

\[
\begin{array}{c}
\text{O} \\
\text{H}
\end{array}
\]

\begin{align*}
\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{C} \overset{\text{N}_n}{\rightarrow} \\
\text{H}
\end{align*}

nylon 6

The nylon 6 film and powder used in the research was obtained from Goodfellow Company. Some properties are given in Table 4.
1.2.2 Coating methods

Coating is an old technique. Until now, many coating methods have been successfully applied in industry. A simple introduction to coating methods is given here. Coating methods given here are also studied in this research in order to find a good candidate coating method for PA6. It should be pointed out that coating methods listed here are common and economic methods.

(a) Brush

Brush is the simplest coating means. It is often applied in both industry and residence even now. If you paint your house, you should have enough experience about brush coating. Its disadvantage is its low-efficiency and is not suitable for high production applications.

(b) Dip

Dip is also a common and simple coating way. In this method, the part is dipped into solution for a while and then taken out of solution for drying. Sometimes drying is conducted in an oven. After drying, the coating is shaped on the surface of the part.

(c) Spray

In this method, tiny solution droplets are pushed to this surface of substrate by pressured gas. More solute particles are attached on the surface of substrate and solvent should throw...
away when solution droplets hit the part. Finally the coating is created on the surface of substrate.

No coating method is an all-purpose means. Every method has its appropriate application. Advantages and limitations of coatings are listed in Table 5.

Table 5 Comparison of common coating methods [29]

<table>
<thead>
<tr>
<th>Coating Methods</th>
<th>Advantages</th>
<th>Limitations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spray</td>
<td>Fast, adaptable to varied shapes and sizes; low equipment cost</td>
<td>Difficult to obtain complete coating of complex parts and to obtain uniform thickness and reproducible coverage</td>
</tr>
<tr>
<td>Dip</td>
<td>Thorough coverage even on complex parts such as tubes and high-density electronic modules</td>
<td>Viscosity and pot life of dip must be monitored; speed of withdrawal must be regulated for consistent coating thickness.</td>
</tr>
<tr>
<td>Brush</td>
<td>Brushing action provides good “wetting” of surface, resulting in good adhesion, Lowest equipment cost</td>
<td>Poor thickness control; not for precise applications; high labor cost.</td>
</tr>
</tbody>
</table>

1.3 Electrical parameters

1.3.1 Electrical breakdown phenomena

Under low voltage, a significant current passes through dielectric materials. When voltage is high enough, insulation material will suddenly conduct a substantial current. Such phenomenon is called dielectric breakdown, and the voltage is called the breakdown voltage $V_b$. Breakdown voltage per unit thickness, also called dielectric strength $E_b$, is the most important parameter for dielectric with unit thickness material, particular for power application. It is the maximum voltage that dielectric can withstand. When breakdown occurs, the dielectric does not function as an electrical insulator and the apparatus should be damaged. Breakdown voltage depends on not only dielectrics themselves but also on test environments, for example: temperature, pressure and humidity. Electrode properties
are also related to breakdown voltage, including the shape (flat, round) and material of electrodes (different metals).

Even though it has been studied for less than one century, the mechanism of breakdown is still unclear. Several mechanisms of the breakdown mechanical are:

- **Thermal breakdown**

  There is a small current passing through the dielectric before breakdown happens. Such small current produces Joule heat locally and temperature increases locally. The dielectric structure is damaged when the temperature is too high. Finally, the dielectric can’t withstand the applied voltage. Breakdown happens.

- **Electronic avalanche breakdown**

  A small amount of free electrons in the dielectric obtain enough energy from the external electrical field and hit other electrons bonded locally by dielectric atoms. When bonded electrons become free electrons under this effect, they also obtain enough energy under the external electrical field. Then they hit other bonded electrons too. Finally, breakdown happens.

- **Discharge**

  Under external electrical field, partial discharge takes place inside insulation materials, particularly inside cracks, pores and cracks. Initially, the effect of discharge is minor. With time, discharge damages the structure of materials. Cracks become larger. Dielectric is eroded at last and it loses the effect of insulation. Discharge also takes place at the surface of dielectric if the surface is contaminated by dirt, water and so on.

- **Electromechanical breakdown**
The force between two charges is determined by the equation of \[ F = \frac{Q_1 Q_2}{4\pi \varepsilon_0 \varepsilon_r R^2}, \]
where \( \varepsilon_0 \) is constant, permittivity of free space; \( \varepsilon_r \) is relative permittivity of medium; \( Q_1 \) and \( Q_2 \) are the charges and \( R \) is the distance between two charges. Under external electrical field, the dielectric is compelled by the attractive force \( F \) of the electrodes [Figure 2]. As the thickness of the dielectric decreases and consequently the attractive force \( F \) increases. At last, the structure of the dielectric deforms when the external field is very high. This mechanism is called electromechanical breakdown.

![Figure 2 Electromechanical breakdown](image)

Electrical breakdown is a complicated phenomenon. No single mechanism takes effect. It is impossible to predict breakdown voltages before an experiment is conducted.

1.3.2 Dielectric constant and dissipation factor

Any electrical insulation material has a capacitance effect. Considering the final purpose of research, which is to make a coating around the HTS transformer coil, such capacitance effect inherent with dielectric affects transformer’s operation.
For a planar capacitor filling with dielectric material, the capacitance is determined by:

\[
C = \varepsilon_r \varepsilon_0 S/D \quad (1)
\]

Where \( S \) is the area of electrode; \( D \) is the thickness of dielectric; \( \varepsilon_0 \) is a constant, its name is permittivity of free space and its value is \( 8.854 \times 10^{-12} \text{ F/m} \); \( \varepsilon_r \) is called the dielectric constant or relative permittivity. Note that \( \varepsilon_r \) is dimensionless and only related with insulation material.

Equation (1) gives us a method to measure the dielectric constant. Insert a piece of flat dielectric material into two electrodes and measure the capacitance \( C \) of this planar capacitor. Area of electrode \( S \) and thickness \( D \) are easily found, so we can calculate the dielectric constant \( \varepsilon_r \).

Under an AC external electrical field, another new dimensionless parameter, dissipation factor (DF), appears in order to indicate how much power is dissipated by insulation material. It should be admitted that the dissipation factor is much more difficult to understand than the dielectric constant. Explanation of dissipation factor based on the view of measurement is given below [Figure 3] as most textbooks did. Appendix A explains the dissipation factor from the view of energy. Dissipation factor is also called loss tangent (\( \tan \delta \)) or loss factor. Comments about Figure 3 can give a clear understanding:

- The dot over \( C \) and \( \varepsilon_r \) indicates that \( C \) and \( \varepsilon_r \) are complex number;
- \( j \) is imaginary unit and also denoted by \( i \) somewhere;
- \( \varepsilon_r' \) is dielectric constant, same as \( \varepsilon_r \) above noted;
- \( G \) is conductance of resistor in circuit model of insulation material;
- \( C_0 \) is capacitance of capacitor in in circuit model of insulation material;
- \( C_0 \) is the capacitance between electrodes when insulation material is taken away, i.e. vacuum exists between electrodes;
Both dielectric constant and dissipation factor are functions of frequency. Usually dielectric constant decreases with the increase of frequency. Different from dielectric constant, the curve of Dissipation factor vs. frequency has peaks at special frequencies only related with the insulation material electrical property. In this thesis, both dielectric constant and dissipation factor are measured in the range of from 20 Hz through 1M Hz which is limited by LCR meter.

The basic origin of dielectric constant and dissipation factor is electronic polarization under external electrical field. Because polarization mechanisms are very complicated, relationship between polarization and external electrical field hasn’t been fully recognized. A general survey is given here. Generally, less polarization is induced under higher frequency. So dielectric constant decreases under higher frequency. At special frequencies, one kind of polarization mechanism is resonant to external field. That is why there are peaks on the curve of dissipation factor vs. frequency. Reference [30] provides more information about this issue.
CHAPTER 2

EXPERIMENT

2.1 Preparation coating

PA6 powder and film was obtained from Goodfellow Company. Their physical properties are described in table 6. A PA6 solution of 10mg/ml was made utilizing PA6 powder dissolved in formic acid (+96%). Heating at 40 ℃ can speed the process of solving. The solution is a colorless liquid with low viscosity.

<table>
<thead>
<tr>
<th>Table 6 Brief of properties of commercial PA6 film and powder</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>PA6 film</strong></td>
</tr>
<tr>
<td>Color</td>
</tr>
<tr>
<td>Transparent</td>
</tr>
</tbody>
</table>

In our study, coatings were made on aluminum foil utilizing the following methods: spray, brush and dip. Before coating, aluminum foil (thickness = 254 µm) was cleaned by the below steps:

1. Put aluminum foil in ultrasonic bath of Toluene for 15 minutes;
2. Put aluminum foil in ultrasonic bath of Acetone for 15 minutes;
3. Put aluminum foil in ultrasonic bath of Methanol for 15 minutes;
4. Wash with deionization water and dry by pressured nitrogen gas;

For spray method, approximately 276 kPa (40 PSI) of pressured nitrogen gas was used. Because formic acid is a weak acid with sharp smell, the whole process of coating was performed in a fume hood. Heat treatment conducted in an oven and vacuum treatment lower than -50 kPa were applied after making coatings.

2.2 Measurement of DC breakdown strength

The setup for measuring DC breakdown strength is showed in Figure 4. The function of the sourcemeter was to drive the output of the DC high voltage supply at a designed DC sweep rate. When the sample can’t withstand the high voltage applied on it, electrical breakdown phenomenon happened. A breakdown current passed through sample. The breakdown current, at the same time, triggered the output voltage to drop to zero when the high voltage supply worked under current trip operation. The whole process was recorded and displayed on the screen of a digital oscilloscope [Figure 5]. A 10MΩ current-limit resistor and a high voltage probe with ratio of 1000:1 were applied to provide protection to equipments. The current-limit resistance was related with max. output voltage and current of high voltage supply and decided by below equation (2):

\[
R = \frac{\text{maximum output voltage}}{\text{maximum output current}} = \frac{30 \text{ kV}}{3 \text{ mA}} = 10 \text{ M}\Omega \quad (2)
\]

Under the control of a sourcemeter, the high voltage supply can create voltages with rich waveforms, for example, voltages with different sweep rates, square waveform, triangle waveform, and trapezoid waveform and so on. The combination of sourcemeter and high voltage supply is versatile and can be used in other research related with high voltage.

As known, the electrical breakdown phenomenon is a complicated issue. And it is related with not only materials but also test conditions. So below regulations were made in order to make measured data comparable. The breakdown voltage was measured in silicon oil, the current criterion current was 30 µA, DC high voltage sweep rate was 35 V/second, and the diameter of the brass electrodes was 6.35 mm.
Figure 4 Diagram of measure DC breakdown voltage

Figure 5 Waveform of DC electrical breakdown voltage
2.3 Measurement of dielectric constant and dissipation factor

The setup for measuring dielectric constant and dissipation factor is simple, as showed in Figure 6. Good contacts between electrodes and sample is necessary in the measurement of dielectric constant and dissipation factor. From our experience, brass electrodes can’t give us good contacts to our sample and the data isn’t reasonable (for example, calculated $\varepsilon_r$ is less than 1, whereas $\varepsilon_r$ should be greater than 1.). Mercury is a unique liquid state metal at room temperature. It is the best candidate electrode material with good contact. Mercury electrodes were applied for measuring dielectric constant and dissipation factor. Their diameter is 6.35 mm. Measurement of dielectric constant and dissipation factor was also conducted in a fume hood because mercury is dangerous to the human body.

![Figure 6 Diagram of measure DF and $\varepsilon_r$](image)

2.4 Measurement of coating’s thickness

The thickness was measured by a micrometer with 1.27 µm (0.05 mil.) accuracy. The thickness of aluminum substrate was measured at first (254 µm) and the sample’s thickness (coating + substrate) was measured [Figure 7]. The difference between two measurements is the thickness of coating. Thicknesses of coatings are in the range of 5 µm through 10 µm. Compared with micrometer’s accuracy of 1.27 µm, the coatings’ thickness is in the same order-of-magnitude. In addition, coatings are not too thick themselves. In order to obtain more accurate measurements, it is necessary to handle the substrate carefully. Any mechanical small strength must deform the substrate. If so, some bumps appeared on the
surface of substrate. Careful handling of the substrate for keeping its thickness constant and flat surface is an important skill for this experiment.

Major specifications of used equipments are given in Table 7.

![Cross-sectional view of sample](image)

**Figure 7 Cross-sectional view of sample**

<table>
<thead>
<tr>
<th>Equipments</th>
<th>Model (Company)</th>
<th>Related Specifications</th>
</tr>
</thead>
<tbody>
<tr>
<td>SourceMeter</td>
<td>2410 (Keithley)</td>
<td>Source voltage from 5 µV to 1100 V; Max. sweep steps: 2500;</td>
</tr>
<tr>
<td>DC High voltage supply</td>
<td>PS/EH30P3 (Glassman High Voltage)</td>
<td>Output volt.: 0 ~ 30 kV; Output current : 0 ~ 3 mA; Current trip; Program: 0 ~ 10 V linearly control from 0 to 30 kV output voltage;</td>
</tr>
<tr>
<td>Digital oscilloscope</td>
<td>LP142 (LeCroy)</td>
<td>Max. input volt.: ± 400 V (DC + AC peak); Input RC: 1 MΩ ± 1.5%, 20 pF ± 2 pF;</td>
</tr>
<tr>
<td>High voltage probe</td>
<td>1600A (Keithley)</td>
<td>Ratio: 1000:1; Max. Volt. DC: 40 kV, AC 28 kV; Input resistance: Approx. 1000 MΩ;</td>
</tr>
<tr>
<td>Current-limit resistor</td>
<td>SR60-1005-KB (RCD Components Inc.)</td>
<td>Resistance : 10 MΩ; High power/high voltage, thick film resistor;</td>
</tr>
<tr>
<td>LCR Meter</td>
<td>1920 Precision LCR Meter (QuadTech)</td>
<td>Frequency range: 20 Hz to 1 MHz; Accuracy: ± 0.1% for capacitance, ± 0.001 for dissipation factor.</td>
</tr>
<tr>
<td>Micrometer</td>
<td>Digitrix II (Fowler Company)</td>
<td>Accuracy: 1.27 µm (0.05 mil.); Range: 25.4 cm (1 inch)</td>
</tr>
</tbody>
</table>
CHAPTER 3

RESULTS AND DISCUSSIONS

3.1 Appearances of coatings by different methods

Appearances of coatings by different methods (Table 8) are dissimilar. Here some comments to help better understand Table 8. Adhesion refers to bonding between coating and aluminum substrate. Good adhesion indicates that the coating does not separate from the substrate in air. Coating by dipping does not stay bonded to the substrate after drying by air. Because the polymer solution (PA6 + formic acid) is transparent, color coating indicates there are many bubbles inside coating. Bubbles reflect light and consequently the coating is not transparent. Robust means that whether the coating is easy to break. After air drying, the dip coating broke into pieces. Thicknesses of coatings by all three methods were from 5 µm ~ 10 µm. Generally, dip coatings can not stick on the substrate and are brittle after air drying. Most areas of the brush coating were white, indicating that the bubbles distribute unequally in brush coating. Comparably, spray coating is best among the above three coating techniques. This result is strongly related with the solution properties. For example: PA6 solution is a liquid with low viscosity.
3.2 Spray coating’s surface pictures by optical microscope

From the optical pictures (magnitude x 5) of coatings by spray (Figure 8), textures of substrate (Aluminum) are clearly demonstrated. And polymer coatings also conform to the texture of the substrate. Some small polymer clusters are formed on the surface of the spray coating, but they are too little to be seen by naked eyes.

Table 8 Appearance of coatings

<table>
<thead>
<tr>
<th></th>
<th>Spraying</th>
<th>Brushing</th>
<th>dipping</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesion</td>
<td>Good</td>
<td>Good</td>
<td>Bad</td>
</tr>
<tr>
<td>Color</td>
<td>Transparent</td>
<td>White</td>
<td>White</td>
</tr>
<tr>
<td>Robust</td>
<td>Good</td>
<td>Good</td>
<td>Bad</td>
</tr>
</tbody>
</table>

Figure 8 Coating surface pictures by optical microscope (x 5) (a) Coating, no heating; (b) Coating, heating at 80 °C for 3 hours; (c) Coating, heating at 100 °C for 3 hours; (d) Coating, heating at 120 °C for 3 hours;
3.3 Comparison of coating densities

The densities of spray and brush coatings are compared in Figure 9. Spray coating is looser than brush coating. But the density of spray coating after vacuum treatment approaches the brush coating’s density. Figure 9 also gives the density of PA6 commercial film. Because the process of making polymer films is different from the process of coating, commercial films are denser than coating. The comparison of densities also indicates that there are bubbles in the coating. But two different kinds of bubbles (Table 9) exist in spray coating and brush coating. Vacuum treatment can make spray coating much denser. Two possible ways to increases spray coating’s density by the vacuum treatment are: (1) decrease bubble number; (2) decrease bubble size. Further research is needed to conduct in order to find how vacuum treatment changes the density of spray coating.

When the size of an object is not less than the visible light wavelength (0.4 µm – 0.7 µm), the visible light is reflected by the object. So we can see the object. As mentioned in section 3.1, spray coating is transparent and brush coating is white color [Table 8]. It is concluded that the bubbles’ size in the brush coating is larger than the visible light wavelength. Spray coating, including both vacuum treatment and no vacuum treatment, have smaller size bubbles which size are under the visible light wavelength.
3.4 DC dielectric strength of coatings

DC $E_b$ of commercial PA6 film (thickness of 15 $\mu$m) is higher than coatings’ DC $E_b$ (Figure 10). Even though spray coating is looser than brush coating, it has larger DC $E_b$. It shows us that bubbles in the coatings greatly affect DC $E_b$. Bigger bubbles decrease the insulation quality of brush coating. In addition, vacuum treatment does not affect DC $E_b$ of spray coating. Electrical breakdown in solids is a very complicated process. Dielectric strength depends on many factors. For spray coating, bubbles are not the dominant breakdown factor. This is because DC $E_b$ changed little when the density of spray coating was greatly increased after vacuum treatment.
It is noticed that the PA6 coatings’ $E_b$ is only one fifth of that of the commercial PA6 film. Three reasons of such a lower $E_b$ are below:

- There are bubbles inside the coatings. The bubbles cause discharge easily as mentioned in section 1.3.1. So the $E_b$ of the coatings is lower than the commercial film.
- The commercial film is semi-crystalline polymer. And our coatings do not have crystal structure. So there are more impurities in the coatings than in the commercial films. More impurities cause a lower $E_b$.
- The $2.52 \times 10^3 \Omega \cdot \text{cm}$ of the solvent’s resistivity was measured. Compared with the $5 \times 10^{12} \Omega \cdot \text{cm}$ of the commercial PA6 film’s resistivity, the solvent is more conductive [28]. The conductive solvent was left inside the coatings after heat and vacuum treatments. Their existence leads to a lower $E_b$.

![Figure 10 Comparison of DC $E_b$ of PA6 coatings and commercial film](image)

3.5 Influence of heat treatment on spray coating

Heat treatment’s effect on dielectric constant of spray coating was studied (Figure 11). Notice that dielectric constants for 3 hours treatment and no heat treatment are similar.
Heat treatment pushes most solvent (96% formic acid and 4% water) out of the coating. Considering the properties of water and formic acid (Table 10), it concludes that only a little solvent exit in the coating and most solvent molecules exit as gas state in spray coating’s bubbles. But it is interesting that change route of \( \varepsilon_r \) depends on heating temperature (Figure 12). \( \varepsilon_r \) changes to higher value for 100 °C and 120 °C, different from 80 °C heating in which \( \varepsilon_r \) changes to lower value. When water molecules escape from coating at different velocities (different heating temperatures), they do different effect on PA6 coating’s dipole. Higher speed of water molecules brings more dipole to spray coating. It is unclear how water molecules effects dipole. But the difference of dipole due to escape of water disappears with longer heating time.

Heating temperature has different effect on dissipation factor (DF) of coating. For 100 °C and 120 °C, dissipation factor decreases. For 80 °C, dissipation factor remains. It shows that velocity of water molecule acts DF value differently. It is also interesting that change route of dissipation factor depends on temperature (Figure 13).

Compared with \( \varepsilon_r \) and DF of the commercial PA6 film (Figure 14), spray coating has larger values of \( \varepsilon_r \) and DF. In the frequency range of 20 Hz – 1 MHz, interfacial polarization and orientational polarization give major contribution to electrical dipoles in solids [30]. Orientational polarization depends on material (PA6) and interfacial polarization depends on structure of material (the bubbles in coating). Bubbles, invisible by naked-eyes in the spray coating, bring more interfacial dipoles which lead to higher \( \varepsilon_r \). The surface of the aluminum substrate is not very flat. This is another factor related with higher \( \varepsilon_r \) of the spray coating. The random structure in the spray coating leads more energy to be dissipated than polymer film under an external AC electrical filed. Dissipation factor (DF) indicates how much energy is dissipated. So the spray coating has a higher DF than the commercial film. Another reason of larger DF for the spray coating is that the solvent was still left inside the spray coating after heat and vacuum treatment. Solvent has a smaller resistivity than the commercial film as mentioned in the section 3.4. These solvent molecules dissipate more energy than PA6 molecules. For power applications, small \( \varepsilon_r \) and DF values are welcome.
Table 10 Properties of solvent (96% formic acid + 4% water)

<table>
<thead>
<tr>
<th></th>
<th>Water</th>
<th>Formic acid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dielectric constant</td>
<td>81</td>
<td>58</td>
</tr>
<tr>
<td>Boiling point</td>
<td>100 °C</td>
<td>101 °C</td>
</tr>
<tr>
<td>Volatility</td>
<td>Not easy</td>
<td>Easy</td>
</tr>
</tbody>
</table>

Figure 11 The dielectric constant and dissipation factor as a function of frequency for spray coating after heated at temperatures of (a) & (b) 80 °C, (c) & (d) 100 °C, and (e) & (f) 120 °C for heating time of ◊: no heating, □: 1 hour, Δ: 2 hours, and x: 3 hours
Figure 11 Continued
Figure 11 Continued

Figure 12 Change route of $\varepsilon_r$ when heat treatment
Figure 13 Change route of dissipation factor when heat treatment

Figure 14 Dielectric constant (a) and dissipation factor (b) as a function of frequency for PA6 commercial films
Heat treatment’s influence on DC $E_b$ of spray coating is very small (Figure 15). The effect of heat treatment is to push solvent out of the coating. From Figure 15, we can know that the solvent’s existence inside the spray coating is not a major electrical breakdown’s reason (section 3.4). But there is another explanation for the heat treatment’s influence on $E_b$. A very small amount of solvent molecules were still left inside of the spray coating after the heat treatment. Even though the amount of solvent molecules was little, it was a dominant factor of the lower $E_b$ among the breakdown mechanisms (section 3.4). So a small amount of solvent molecules inside of the coating causes a lower $E_b$. 

Figure 15 DC $E_b$ vs. heating time at $80 \, ^\circ C$
3.6 Scanning electron microscope (SEM) pictures of PA6 spray coating

SEM pictures of PA6 spray coating on glass substrate were taken because they have higher magnitude. There are bubbles inside coating (Figure 16) and polymer particle on surface (Figure 17). Even though glass is different from aluminum, the SEM pictures can support the above conclusions about internal structure and surface of spray coatings which were made on aluminum substrate.

![Figure 16 Cross-sectional SEM picture of PA6 coating](image)

Figure 16 Cross-sectional SEM picture of PA6 coating
3.7 Relationship between DC $E_b$ and coating’s thickness

Figure 18 gives the relationship between DC $E_b$ and PA6 spray coating’s thickness. It clearly shows that the DC $E_b$ decreased with the increasing of coating’s thickness. The possible reason is given below. The thicker the coating, the more imperfections there are inside coating. Imperfections reduce DC $E_b$. From the experience, it was difficult to make thicker coating than thinner coating. With the increasing of coating thickness (larger than 10$\mu$m), cracks are likely to appear on the surface of coating.
Figure 18 Relationship between DC $E_b$ and spray coating’s thickness
CHAPTER 4

CONCLUSIONS AND FUTURE DIRECTION

4.1 Summary of experiment

PA6 coatings were made by spray, brush and dip coating methods on aluminum substrate. Spray coating is best among them because of poor adhesion of dip coating and lower DC $E_b$ of brush coating. Spray coating was studied in detail. Vacuum treatment can increase density of spray coating, but does nothing about DC $E_b$. Influence of heat treatment on dielectric constant and dissipation factor of spray coating depends on heating temperature. Heating’s effect on DC $E_b$ of spray coating is small.

Compared with commercial PA6 film, spray coating has lower density, lower DC $E_b$, higher dielectric constant and dissipation factor. These differences are mainly due to the porous structure of coating and remaining solvent molecules.

4.2 Improvement of experiment

A simple mercury electrode was used in the measurement of dielectric constant and dissipation factor (Figure 16). It needs to be installed every time carefully when measuring a sample: first, fill mercury into brass support to make the lower part of electrode; second, lay sample (PA6 coating on aluminum substrate) on it; finally, put a piece of plastic tube with length of 1.25 inch and inner diameter of 0.25 inch on the sample vertically and fill
mercury into the tube. That is the upper part of the electrode. There are two disadvantages which need to be mentioned:

- This simple mercury electrode is not convenient, needs to be installed carefully every time;
- Mercury sometimes leaks from the bottom of plastic tube due to the following reasons: more mercury filled into the plastic tube sometimes; closure at the bottom of the plastic tube is not very well; if mercury leaks, reinstall the electrode;

Because mercury is dangerous liquid to the human body, the above disadvantages seem to be worse. A safer convenient mercury electrode is needed in the future.

![Figure 19 Simple mercury electrodes](image)

4.3 Further research
4.3.1 Research under low temperature

Studying properties of PA6 coating under low temperature, below 100 K, is the next step after research under room temperature is done. When temperature changes from room temperature to cryogenic temperature, coating probably can’t resist influence of variable temperature. Since materials generally shrink when cooled and extend when heated, cracks maybe appears on the coating. The coating even separates from the substrate since they have different thermal expansion properties. Only if coatings can withstand the influence of great temperature’s change, research under cryogenic temperature would be meaningful.

4.3.2 Difference of aluminum and stainless steel

HTS wire is sheathed by stainless steel and our PA6 coatings were made on aluminum substrate. The difference of their properties can affect PA6 coating. Because aluminum foil is cheaper than stainless steel, we used aluminum as the substrate at present time. Properties of coatings on stainless steel need to be studied.

4.3.3 Dissipation factor of coating

Compared with insulating materials applied at room temperature, dissipation factor is a more important parameter for materials operated under low temperature. Higher dissipation factor means that more energy is dissipated during transformer’s operation. The dissipated energy is converted into heat. In order to keep the operating temperature at lower than 100 K which is common temperature for HTS application, the heat has to be pumped out. This is a larger burden for cooling system and increases operating cost [Appendix B]. Kapton film which was used as insulation for major HTS transformers coils (Table 1) has a dissipation factor of 0.0018 at 1 kHz [31]. Compared with Kapton film, PA6 spray coating has a larger dissipation factor at 1 kHz, approximately from 0.06 to 0.1.

4.3.4 Complete study of heat treatment’s effect on dielectric constant and dissipation factor

The effect of heat treatment on dielectric constant and dissipation factor should be carried on to steady state. Previous investigation was only related with up to 3 heat treatment
hours. Additional experiments should been conducted to reveal the whole effect of heat treatment.

4.4 Outlook about polymer coating for HTS transformer coils

Because we are looking for an industrial coating method for HTS transformer coils, practical coating method(s) should be among common and economic coating methods, for example: dip, spray and brush, with different coating method suitable for different polymers.

HTS materials are different from common metals in great manner and are brittle crystal materials. In the research for HTS insulation, two constraints need to be considered: (1) treatment temperature. AMSC, the manufacturer of HTS wire for our HTS transformer project, recommended that high strength HTS wire used for our HTS transformer had better stay below 130 °C to avoid damaging the crystal structure; (2) mechanical strength. Extra mechanical stress on HTS wire also damages HTS wire structure.

Because HTS transformers operate at low temperature, low temperature’s influence on insulation materials should be the focus of research.

With the development and application of HTS, it is absolutely necessary to investigate HTS insulation. More and more people will be involved with the old research field of electrical insulation.
A definition about dissipation factor is given in chapter 1. That definition is a very common in textbooks and is based on the view of measurement. It’s not clear to understand dissipation factor’s meaning through this definition. Another explanation of dissipation factor is up here which is based on the view of energy and provides us a clear and deep meaning. A parallel circuit model is often used to study a practical planar capacitor [Figure A.1] under AC case. For this parallel circuit model, dissipation factor can be explained by equation A.1.

\[
\frac{\text{dissipated energy}}{\text{stored energy}} = \frac{V^2}{R} = \frac{X_c}{R} = \frac{1}{\alpha CR} \quad \text{(A.1)}
\]
Where: \( V \) is the AC voltage with angle frequency \( \omega \) across capacitor and resistor in parallel circuit model of planar capacitor;

The below deduction is to prove that equation A.1 is as the same as common definition of dissipation factor. The deduction begins from Figure 3:

\[
j \omega C = \frac{1}{R} + j \omega C_p
\]

\[
j \omega \varepsilon_0 \varepsilon_r \frac{S}{t} = \frac{1}{R} + j \omega \varepsilon_0 \varepsilon_r \frac{S}{t}
\]

\[
\varepsilon_r = \frac{1}{R} \cdot \frac{t}{j \omega \varepsilon_0 S} + \varepsilon_r = \varepsilon_r - j \frac{1}{R} \cdot \frac{t}{\omega \varepsilon_0 S}
\]

Because \( \varepsilon_r' = \varepsilon_r - j \varepsilon_r'' \), we can know below relationships:

\[
\varepsilon_r' = \varepsilon_r \text{ and } \varepsilon_r'' = \frac{1}{R} \cdot \frac{t}{\omega \varepsilon_0 S}
\]

It is emphasized that \( \varepsilon_r' \) is as same as \( \varepsilon_r \) which is in the equation (1).

From the definition of dissipation factor, we get:

\[
DF = \frac{\varepsilon_r''}{\varepsilon_r'} = \frac{1}{R} \cdot \frac{t}{\omega \varepsilon_0 \varepsilon_r S} = \frac{1}{\omega RC}
\]

Dissipation factor indicates how much percent energy is dissipated to heat in AC case. It has no unit. If dissipation factor of a planar capacitor is 0.1, it means that ratio of dissipated energy to store energy is 1:10. At last, it is noted that DF has meaning only under AC case. But dielectric constant has meaning under both DC and AC case.
APPENDIX B

The significance of dissipation factor of insulating materials used at cryogenic temperature is discussed in this appendix. A refrigerator flow diagram is showed in Figure B.1. It works under such way: a refrigerator pumps heat ($Q_C$) from a cool place with $T_C$ temperature to a hot place with $T_H$ temperature by consuming energy ($W$). The hot place absorbs heat ($Q_H$).

![Flow diagram of a refrigerator](image)

**Figure B.1** Flow diagram of a refrigerator

From thermodynamics knowledge, the performance coefficient $K$ of a refrigerator is determined by equation:
\[ K = \frac{T_c}{T_H - T_c} \quad (B.1) \]

Where: \( T_c \) is the temperature in a cool place;

\( T_H \) is the temperature in a hot place;

\( K \) is the performance coefficient; its definition is ratio of the heat pumped out from cool place to hot place by a refrigerator (\( Q_c \)) to the consumed mechanical work by the refrigerator (\( W \)), i.e. \( K = \frac{|Q_c|}{W} \).

When the temperature difference is small, the performance coefficient of a refrigerator is large. Under such case, the refrigerator can pump more heat with small expenditure of work. Under opposite condition with large temperature difference, the performance coefficient is small. A refrigerator operates with a low efficiency.

Table B.1 shows two cases. For case 1 in which cool place’s temperature is 100 K, the performance coefficient of the refrigerator is 0.5. Under case 1, the refrigerator only pumps 0.5 part heat when consuming each part energy. For case 2 in which the performance coefficient is 2, the refrigerator can pump 2 parts energy when consuming each part energy. Case 1 is similar with HTS condition in which heat is pumped out from under 100 K (HTS operation temperature) to 300 K (room temperature).

<table>
<thead>
<tr>
<th></th>
<th>Case 1</th>
<th>Case 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>( T_H )</td>
<td>300 K (room temperature)</td>
<td>300K (room temperature)</td>
</tr>
<tr>
<td>( T_C )</td>
<td>100 K</td>
<td>200 K</td>
</tr>
<tr>
<td>K ( (\text{performance coefficient}) )</td>
<td>0.5</td>
<td>2</td>
</tr>
</tbody>
</table>
From above explanations, we know that the cooling system has larger burden in the HTS applications. A smaller dissipation factor means less consumed heat of insulation materials and then decreases greatly the burden of the cooling system.
REFERENCES


BIOGRAPHICAL SKETCH

Huazhong Wang was born at Shenyang City, Liaoning Province, the People’s Republic of China in 1972. He received his bachelor’s degree of Science at the Department of Electrical Engineering, Liaoning University in the spring 1993. Since fall 2001, he has studied for the Master degree at the Department of Electrical and Computer Engineering, FAMU-FSU College of Engineering, Florida State University. He received his Master degree in summer 2004. During his graduate study, he was a research assistant in the field of insulation materials and conducted research under Dr. Jim P. Zheng’s supervision.