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Fluorinated PEEK and XLPE as Promising Insulation Candidates for the Propulsion System of All-electric Aircraft

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ABSTRACT
This article investigates the surface electrical properties of PEEK and XLPE before and after the fluorination modification. The samples are tested for surface conductivity, surface morphology, and surface anti-aging characteristics. The samples are also tested for flashover voltage with ramping voltage under 100 kPa, 20 kPa and 10 kPa. The results showed that the surfaces of PEEK and XLPE become slightly smoother with the increase of fluorination time, and the conductivity increased with the increase of fluorination time. After fluorination, the surface of PEEK shows significant hydrophilicity, while the surface hydrophobicity of XLPE is slightly reduced after fluorination, and there is a trend of recovery with the extension of fluorination time. After fluorination, the surface anti-aging performance is significantly improved. Compared with the untreated samples, the fluorinated PEEK has a higher flashover voltage at 100 kPa, and the flashover voltage is slightly increased at 10 kPa and 20 kPa, while the flashover voltage of XLPE after fluorination shows a downward trend especially under low pressures. This study provides a reference for fluorinated polymers as promising candidates for the insulation systems for all-electric aircraft.

Index Terms — Surface charge, XLPE, PEEK, fluorination, flashover, all-electric aircraft.

1 INTRODUCTION

ALL-ELECTRIC aircraft uses batteries as the power source for the propulsion system where the key electric components operate at medium and low direct current (DC) voltage [1, 2]. The all-electric aircraft is at a normal atmospheric pressure of 100 kPa when it takes off at full load on the ground. After the stratosphere reaches the cruising altitude, it will remain at about 35000-40000 feet, in a low-pressure environment of about 20 kPa. The hybrid-electric aircraft is designed with one propulsion system powered by a battery, and to satisfy the dense output power requirement during taking off, there would be very high current flowing in the cables, resulting in a temperature of as high as more than 120 °C. Working in extreme environments such as low air pressure and sudden changes in thermal conditions, the insulation performance of the propulsion system requires more robust and special assessment [3]. Insulating materials used in aircraft require light weight and erosion resistance in the bulk, while when considering the electrical property, surface partial discharges and flashover are more likely to occur at DC voltage in low-pressure environments. Additionally, a large amount of radiation at high altitudes is more likely to cause aging and degradation of insulating materials [4]. The above concerns require dielectric materials with enhanced properties in lighter weight, such as better erosion resistance, and stable electrical, chemical and physical properties especially under DC voltage at low pressures [5, 6]. PEEK, XLPE and PI are three common insulation materials used in all-electric aircraft. PI is only used for motor winding insulation, and it is easy to accelerate aging under high humidity conditions [7, 8]. XLPE and PEEK are common insulating materials in aircraft cables,
especially XLPE can better adapt to various harsh environments and ensure insulation safety. PEEK is a special engineering plastic. Due to its strong chemical inertness and corrosion resistance, its reinforced composites are widely used in cable original parts, wings and other locations. Polyether-ether-ketone (PEEK) is a special engineering polymer with excellent electrical and thermal properties. It is extremely strong, chemically inert and flame-retardant, resistant to high temperatures, and has excellent mechanical and insulating properties [9]. Cross-linked polyethylene (XLPE) has good chemical stability, good resistance to extreme temperature and excellent electrical insulation [10]. Moreover, high-purity highly homogeneous XLPE being developed in the last two decades [10, 11] has also a better behavior vs. the so-called dimensional effect, i.e. the fact that as insulation volume is enlarged, more defects are found and breakdown voltage tends to drop [12]. This problem is important in all-electric aircrafts, as the cumulated lengths of electrical cables are steadily increasing in aircrafts: up to 20 km in a helicopter, 40 km in a fighter, 500 km in a civil aircraft [13].

The contamination can be introduced in the following procedures: Oils may be introduced during the manufacturing process. During working, light objects are likely to be attached to the insulation surface due to the polarization. Due to the temperature variation which ranges from, the surface of insulation may accumulate water drops or films. This water attaches more dust during working of the aircraft. In terms of density, PEEK has 1.34 kg/dm³ [14], while the density of XLPE is relatively lower, only 0.95 kg/dm³. Excellent insulation and light weight characteristics make these two polymers potential candidates in light-weight aircraft insulation systems [15].

Surface fluorination is one of the most important means to adjust the surface insulation properties of polymer dielectrics. Due to its advantages such as simple operation, mature process, and low cost, surface fluorination provides a feasible way for the surface modification of insulation materials [12]. Guo et al. believed that direct fluorination treatment can greatly improve the flashover voltage of LDPE [16]. Li et al. found that the fluorination modification modifies traps on the surface of alumina epoxy composites, affecting the morphology and surface conductivity of the insulating surface, which result in a boosted flashover voltage in both air and SF₆ [17, 18]. Fluorination treatment has also been found to have the effect of optimizing surface electrical properties of EPR, but excessive fluorination will weaken the ability to resist electrical tracking. 240-360 minutes of fluorination can help ethylene propylene rubber (EPR) obtain the best surface properties [19, 20]. The surface fluorination of PEEK film has been successfully achieved by using the reduced polymer (PEEK-OH) as a versatile key-intermediate [21]. Space charge measurements on the surface fluorinated PE film revealed that the fluorinated layer can effectively block the charge injection [22]. A large number of preliminary studies have proved that fluorination modification has potential advantages in improving the surface insulation performance of PEEK and XLPE. However, no studies have been performed to evaluate the electrical properties of these two materials after fluorination under low pressure.

The development of future hybrid-electric aircraft requires a significant voltage boosting to satisfy the increase in output power and narrow space. For example, an all-electric propulsion system for a twin-aisle (e.g. the notional NASA N3-X) aircraft requires at least 50 MW during take off. NASA estimates that the future hybrid-electric aircraft would need to operate at a maximum voltage of 20 kV, and its insulation must be able to withstand up to 41 kV. Therefore, it is a desperate need for us to find a suitable insulation material that can work at high voltage at elevated amplitudes, while the surface property, including Surface flashover, Surface conductivity, Leakage current, FTIR spectrum, Water contact angle, SEM, EDAX, plays an important role.

In this paper, some PEEK and XLPE samples were fluorinated for various time lengths, the electrical properties of the surface were tested before and after fluorination, and the surface insulation performance under low air pressure was evaluated. The research in this article can provide guidance for the selection and performance optimization of novel insulation materials for all-electric aircraft.

## 2 EXPERIMENTAL SETUP

### 2.1 SAMPLE PREPARATION

The experimental samples are PEEK and XLPE square films with a thickness of 100 μm and a side length of 25 mm. The chemical composition, density, and the operating temperature of samples are shown in Table 1 [23].

<table>
<thead>
<tr>
<th>Mat.</th>
<th>Molecular structure</th>
<th>Density (kg/dm³)</th>
<th>Allowable temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>PEEK</td>
<td><img src="image1" alt="PEEK structure" /></td>
<td>1.34</td>
<td>-40 °C~250 °C</td>
</tr>
<tr>
<td>XLPE</td>
<td><img src="image2" alt="XLPE structure" /></td>
<td>0.93</td>
<td>-20 °C~130 °C</td>
</tr>
</tbody>
</table>

Before surface fluorination, samples were treated by ultrasonic processing in absolute ethyl alcohol and distilled water for 60 min, and then dried in a vacuum oven for 30 min at 70 °C. Surface fluorination was performed with F₂/N₂ mixture containing 12.5 % F₂ in volume for 15, 30 and 60 min at 50 °C and 0.1 MPa.

### 2.2 MEASUREMENT DESCRIPTION

In the flashover test, two finger electrodes were used. The distance between the two electrodes was 8 mm. The samples were placed on an epoxy board support and pressed tightly below two electrodes. The flashover test was performed in a chamber and the setup is shown in Figure 1. After air pressure was kept stable for 10 min, the voltage was increased at a ramp rate of 500 V/s, and the occurrence of flashover was detected by a current amplifier. The cooling down time between each flashover was 5 min. The experiment was carried out at 10 kPa, 20 kPa and atmospheric pressure (100 kPa), and each sample was measured 10 times at every single pressure. The temperature was 15 °C and the relative humidity was 10 % when measuring the flashover voltage.

In the test of the surface conductivity, the temperature was 14 °C, the relative humidity was 10 %, and the applied voltage was 0.83kV/mm.
The chemical composition of the sample surface was measured by FTIR (TENSORn) in the attenuated total reflection mode. The surface morphology and cross-sectional morphology of the sample before and after fluorination were measured by a field emission scanning electron microscope (Nova Nano SEM450). An EDAX energy spectrometer EDS (OCTANE PLUS) was used for element content analysis. An optical contact angle meter (DAS30) was used to measure the water contact angle, and 10 different points were selected for each sample for the measurement. In the surface aging test, the sample was subjected to continuous flashover treatment at 17 °C and RH = 15% for 1 minute. About 130 times of flashover occurred per minute.

3 RESULT

Figure 2 shows the FTIR spectrum of original samples and samples fluorinated for 15 min, 30 min, and 60 min. As shown in Figure 2 (a), the peak at 1645 cm⁻¹ of the PEEK sample corresponds to the tensile vibration of the C=O bond. The peaks at 1591 cm⁻¹ and 1510 cm⁻¹ correspond to the bone vibration of the R-O-R aromatic ring structure. The peak at 1214 cm⁻¹ corresponds to the asymmetric tensile vibration of the R-O-R structure. The peak at 832 cm⁻¹ corresponds to the plane bending vibration of C-H in the aromatic ring [20]. No new characteristic peaks appeared in samples after fluorination. However, as the fluorination time increases, the curve moves toward the direction of high wave number, and a blue shift occurs, indicating that the chemical groups of the fluorinated polymer become more stable.

Figure 2 (b) shows that for the XLPE, the peak at 2914 cm⁻¹ represents the -CH₃ asymmetric stretching vibration, the peak at 2846 cm⁻¹ corresponds to -CH₂- symmetric stretching vibration, and the peak at 1461 cm⁻¹ corresponds to the -CH₂- bending vibration. At 718 cm⁻¹, -(CH₂)ₙ-(n≥4) in-plane rocking vibration appears. The fluorinated samples showed a strong absorption intensity between 1000-2000 cm⁻¹. The in-plane rocking vibration of the -(CF₂)ₙ- group mainly appeared at 1090 cm⁻¹, and the bending vibration of the -CF- and -CF₂- groups appeared at 1734 cm⁻¹, indicating the formation of -CF-, -CF₂-, and -CF₃-groups.

Figure 3 shows the elemental composition of experimental samples, namely the atomic ratio of C, O and F of original samples and samples fluorinated for 60 min. With the increase of the fluorination time, the content of C element decreases, and the content of O atoms and F atoms increases. The C atom in PEEK decreased from 83.51 % to 78.99 %, the O atom increased from 16.49 % to 17.64 %, and the F element increased from 0 to 3.38 %. The C atom in the XLPE decreased from 99.66 % to 95.1 %, the O atom content increased from 0.34 % to 2.87 %, and the F atom content increased from 0 to 2.03 %. This observation indicates that PEEK had a displacement reaction with fluorine gas. It is believed that this is due to the presence of C-H on the benzene ring of PEEK, which may replace a small amount of H element during the fluorination process.

Figure 4 shows the SEM images of samples before and after the surface aging test. The surface texture of the original PEEK sample was uniform and slightly rough. After fluorination, especially after 60 min of fluorination, the
surface of the sample became very smooth and flat. The surface morphology of the XLPE samples before and after fluorination did not change much. Interestingly, for both materials, the local electro-erosion area on the surface of the sample after fluorination for 15 min under the same electrical aging conditions was lighter, and was replaced by a relatively small and dense dot-like substance. The traces of carbonization on the surfaces of both materials after 30 min fluorination were very inconspicuous, which were basically the same as those of the samples before aging. However, local carbonization traces can be found on the surface of the 60 min fluorinated samples.

Figure 5 shows the surface conductivity of experimental samples. The surface conductivity of PEEK before fluorination was $7.8 \times 10^{-16}$ S. It can be seen that as the fluorination time increases, the surface conductivity of PEEK increases first, but the increase is small. The highest surface conductivity of the sample after fluorination for 60 minutes was $2.4 \times 10^{-15}$ S. The conductivity of the original XLPE was $1.4 \times 10^{-16}$ S, and the conductivity of the XLPE sample increased proportionally to the fluorination time. The longer the fluorination time results in higher surface conductivity. The highest conductivity of the 60 min-fluorinated sample was $2.8 \times 10^{-15}$ S.
Figure 6 shows the Weibull distribution of the flashover voltage of experimental samples at 100 kPa, 20 kPa, and 10 kPa. Table 2, 3, and 4 represent the values of \( \alpha \) and \( \beta \) parameters of the Weibull distribution of flashover voltage at 100 kPa, 20 kPa, and 10 kPa, respectively, where \( \alpha \) is the scale parameter, and \( \beta \) is the shape parameter. Before fluorination, the flashover voltage of samples at 10 kPa was slightly lower than the flashover voltage at 20 kPa, and all of the values were much lower than the flashover voltage at 100 kPa. Because the \( \beta \) parameter of XLPE is larger than that of PEEK under the condition of 100 kPa, the breakdown voltage of PEEK is much more dispersed than that of XLPE. But under the condition of 20 kPa, the shape parameter of PEEK is larger, and the breakdown voltage of XLPE is much more dispersed than that of PEEK. Under the condition of 10 kPa, the shape parameters of the two materials are close to each other, and their breakdown voltage scatter is basically the same. In addition, PEEK has a lower flashover voltage, especially at low pressures.

After fluorination, the flashover voltage of PEEK increased, but the flashover voltage showed no clear relationship with the fluorination time. The flashover voltage of the untreated PEEK under 100 kPa was between 22 and 26 kV, and the flashover voltage increased significantly after fluorination, especially for the samples fluorinated for 15 min and 30 min, which was higher than 26 kV. As the air pressure decreased, this improvement after fluorination became less evident. In contrast to PEEK, fluorinated XLPE seemed to exhibit lower flashover voltages compared with the original sample. This characteristic was consistent under different pressures and was more pronounced as the pressure decreased. Under low pressure, it is challenging to identify which sample is better, especially at 10 kPa. The flashover curves of 60 min-fluorinated and nonfluorinated samples were almost identical.

4 DISCUSSION

Both PEEK and XLPE have sufficiently low surface conductivity before the fluorination. With the increase of fluorination time, the surface conductivity increases. This finding is different from that of epoxy resin before and after fluorination based on our previous research, where an increased surface conductivity of up to two orders of magnitude was obtained after short time fluorination, followed by a decreasing trend of surface conductivity with the increase of fluorination time [18].

The flashover voltage is one of the core parameters for...
assessing the insulating property. The flashover voltage can be affected by the voltage boost rate, gas type, pressure, sample characteristics, temperature, and humidity, etc. Between the two materials, PEEK has a lower flashover voltage, and the flashover voltage after different fluorination time is more scattered. On the contrary, the flashover voltage distribution of XLPE before and after fluorination is more concentrated. The difference between the two materials is clearer under atmospheric pressure. This observation is mainly determined by the characteristics of the materials. During the voltage ramping, there are complex physical electric field-driven processes at the bulk of the insulation, e.g., the homo charge injection, which is significantly important in mitigating the electric field strength in the triple junction [24]. Additionally, the homo-polar charges transporting along insulation bulk and surface are key elements affecting the “analogous ineffective region”, especially under temperature gradient [25]. The difference in the flashover voltage can be explained by the irregularity of the surface charge behavior of samples with different charge transport behaviors. In other words, the breakdown voltage of a gas gap is based on Paschen’s law. However, when an insulation is involved, the charge behavior inside the solid insulation before flashover occurs has particular influence based on the field- and temperature-dependent electric properties, including polarization, electrical conductivity, permittivity, nonlinear conduction, and other physical parameters.

The water contact angles of PEEK and XLPE before and after fluorination were measured as shown in Figure 7. The water contact angle of the original PEEK was around 73.44°, showing a water repellency property. After fluorination, the water contact angle of PEEK dropped rapidly. As the fluorination time increased, the water contact angle increased from 4° to 7°. The water contact angle of the original XLPE sample is around 106.1°. However, the water repellency of the XLPE decreased after fluorination, and with the extension of the fluorination time, the water contact angle increases from the initial value of 72° to 88°.

After fluorination, the hydrophilicity of PEEK is greatly improved, which indicates that a local continuous water film on the PEEK surface is easier to be formed. The randomly formed water film distorts the local field strength and affects the flashover voltage distribution, which we believe is the reason for the large dispersion in flashover voltage for PEEK before and after fluorination. After surface fluorination, the water contact angle of the material changes obviously, especially for PEEK material, the hydrophilicity of the material after fluorination is significantly enhanced. For XLPE materials, the change of water contact angle is not as obvious as PEEK, but the hydrophilicity is also enhanced. The decrease of water contact angle is due to the introduction of polar groups such as CHF groups and oxygen-containing groups in the fluorination process, and high polar groups are further formed with the increase of the number of broken chains. Because the size of fluorine atom is larger than that of hydrogen atom, the introduction of fluorine atom into the surface layer increases the molar volume of molecules, which changes the surface structure of the sample. In addition, fluorination reaction is always accompanied by molecular chain breaking. These physical and chemical changes lead to the changes in electrical properties on the surface of materials. The measurement results of surface conductivity also increase with the increase of fluorination time. The increase of conductivity can inhibit the accumulation of surface charge and accelerate the dissipation of charge, thus affecting the flashover performance.

At 20 kPa, the flashover voltages of PEEK and XLPE are reduced by 4-5 times compared with the atmosphere pressure condition, but the flashover values at 10 kPa and 20 kPa are not much different. Under low pressure conditions, the relative density of gas molecules near the triple joint decreases, resulting in a longer free travel length of electrons. As a result, the probability of sufficient kinetic energy accumulated between two adjacent collisions is increased, and the flashover voltage is decreased more easily. As a consequence, the weights of factors determining the flashover voltage at low pressure depend more on the gas property such as the gas adsorption energy and the gas molecular ionization energy, and the contributions of factors from the solid phase, i.e. charge injection, charge transport, are greatly reduced. This explains why the flashover voltage before and after fluorination of PEEK at low pressure is very similar. For XLPE, the flashover voltage decreased after fluorination. The reason might be due to the increase in the horizontal electric field component which is due to the increase in the surface conductivity after fluorination. As has already been known that an increase in the horizontal electric field component along the insulation surface causes the streamers more likely to develop into an arc.

Compared with the flashover voltage, the arc aging resistance characteristics of PEEK and XLPE after fluorination were greatly improved as shown in Figure 2. To further verify this finding, a continuous aging test for 4 minutes was performed and the leakage current and flashover voltage was measured every minute. To be frank, the aging performance of samples was checked by the leakage current after samples were arc aged for different time duration. However, we admit that the long-term aging performance of the insulation also plays a significant role in determining the insulating properties, including surface insulation performance under elevated DC and AC application, as well as polarity reversal test, etc. These measurements should also be considered and performed in the future. Figure 8 shows the measurement results of the leakage current of samples after...
aging with continuous flashover for different time duration. As shown in Figure 8 (a), the original PEEK exhibits a steadily increasing leakage current as the aging time increases. The leakage current of the fluorinated PEEK is relatively unstable after aging for a short time, while the leakage currents of the 30-minute and 60-minute samples decrease slightly with the extension of the aging time, and are eventually lower than the leakage currents of the original sample. As shown in Figure 8 (b), excepted the 60-minute fluorinated XLPE which shows a relatively stable leakage current after aging, the overall fluorinated XLPE shows a decreasing trend of leakage current with prolonged aging time, while the leakage current of the original XLPE shows firstly a slight decrease at 2 min aging and then continuous increase after the aging test at 3 min and 4 min.

Figure 8. Leakage current v.s. aging time duration of PEEK and XLPE.

PEEK, XLPE and PI are three common insulation materials used in all-electric aircraft. In our previous research, it was found that the arc resistance of PI material was greatly reduced under the condition of high temperature and surface pollution, which will limit the application of PI material in a harsh environment. At high temperatures, the surface flashover voltage of PI material will decrease by about 20% compared with PEEK and XLPE. PEEK material is not sensitive to temperature due to its strong chemical inertia. Even at the high temperature of 150 °C, its arc erosion resistance is still higher than that of other insulating materials. When there are pollutants on the surface, the arc resistance times of PI are reduced by 86% compared with those without pollutants. On the contrary, the arc resistance times of PEEK materials are not affected by surface pollutants [23]. XLPE material is a common insulating material for ground and submarine cables. It has good chemical stability and resistance to extreme environments. It is also a special insulating material for electric aircraft cables.

It is not accurate to judge the surface anti-aging performance only by the changes in the surface morphology. Based on the results from Figure 4 and Figure 8, 15, and 30 minute-fluorinated PEEK and XLPE show unstable performance in their arc aging resistance property. However, the performance of 60 minute-fluorinated PEEK shows good anti-aging performance, which is verified by a relatively stable decrease of leakage current with the aging time, while for XLPE, samples fluorinated for 60 min shows a reasonably stable leakage current. We presume that the improvement of the aging resistance for 60 minute-fluorinated samples is due to the sufficient change in the surface chemical composition. Additionally, the blue shift of the absorption peak displayed by FTIR indicates that the chemical group of PEEK has become more stable after 60 minute-fluorination as has already been verified in Figure 2. It should be noted that the reason why the fluorinated PEEK does not show the C-F structure in FTIR is that the C-F peak overlaps with some chemical group peaks of the sample itself at 1000 - 1100 cm$^{-1}$.

5 CONCLUSION

In this paper, the physical, chemical, and electrical properties of PEEK and XLPE after various fluorination times are reported. The focus of this work is experimental research on the surface characteristics of materials. However, the research on the bulk characteristics and corrosion resistance are equally important for insulation performance. We will study the material properties and corrosion resistance in the next work. Major conclusions are as follows.

1. After the fluorination, the electrical conductivity, water contact angle, arc aging resistance and flashover voltage of experimental samples can be modified. With the increase of the fluorination treatment time, the surface conductivity of PEEK and XLPE increased up to more than one order of magnitude, and the surface of PEEK became smoother, while the XLPE surface basically showed no significant changes.

2. For PEEK, at 100 kPa, the flashover voltage can be increased significantly after fluorination, and a slight increase of flashover voltage can be found at low pressures. However, for XLPE, the flashover voltage of fluorinated samples showed a decreasing trend at 100 kPa, 20 kPa, and 10 kPa. For PEEK materials, both 15 min and 60 min samples can increase the flashover voltage. The 15 min sample lifting effect is the best at 10 kPa, and the 60 min sample lifting effect is better at 20 kPa.

3. PEEK and XLPE fluorinated for 15 minutes and 30 minutes are not stable in their anti-aging performance, and 60 minute-fluorinated samples show the best performance in the leakage current test. For XLPE materials, from the electrical aging results, the 30 min sample has the best electrical aging resistance.

The research in this article provides a reference for insulating materials selection for the propulsion system of all-electric aircraft. The main focus of this work is on basic
electrical performance and flashover characteristics under ramp voltage, which shows some interesting findings. However, this is far from enough for evaluating altitude-ready insulating materials to be used in the all-electric aircraft. Breakdown voltage, partial discharge, especially electrical parameters under DC voltage need to be paid attention to. In addition, the investigation of material properties and aging at higher temperatures are also critically needed.

6 ACKNOWLEDGMENT

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