IMPROVEMENT METHOD OF THE ADHESIVE BONDING BETWEEN THE PEI AND CFRP FOR THE ULTRACENTRIFUGE ROTOR

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1. Introduction

The ultracentrifuge is essential equipment in Bio-industry. The equipment is broadly used to separate biomolecules such as proteins, polysaccharides, nuclei acid. An ultracentrifuge that satisfies such a demand should be able to create gravitational forces more than 600,000×g. Recently, the hybrid composite rotor for the ultracentrifuge equipment was successfully designed using the PEI (Polyetherimide) which is the thermoplastic material and CFRP (Carbon Fiber Reinforced Plastic) which is the thermosetting (Epoxy)-based composite with filament winding and adhesive bonding methods as shown in Fig. 1 [1].

Works [2, 3] have clearly showed that bonding thermoplastic material, using acrylic and epoxy adhesives, based upon abrasion and solvent cleaning pre-treatment techniques leads to significantly lower joint strengths, compared with thermosetting material. The main reason for this observation is the lack of sufficient adhesion of the adhesive to the thermoplastic material [4]. Thus, for such bonding applications, the surface free energy of the thermoplastic matrix polymer needs to be increased significantly [2, 4].

This can be achieved by various pre-treatment routes, and two of the appropriate methods are suggested via corona [2] and oxygen-plasma pre-treatment methods [5]. However, it is not easy to use the corona and plasma treatment methods on the developed hybrid ultracentrifuge rotors due to the complex shape of the structure.

Therefore, in this work, the flame treatment method which is the suitable method for the hybrid rotor was used to improve the adhesion bonding strength between the PEI and CFRP. To identify the optimum treatment condition, double lap shear tests were performed with respect to treatment time at the room temperature (25°C). And the contact angles on the PEI surfaces were measured with respect to flame treatment time with the sessile drop method to investigate the effect of the flame treatment method.

2. Fabricating process of the hybrid rotor

Fig. 2 shows the fabricating process of the hybrid rotor for the ultracentrifuge equipment. Firstly, the PEI body was machined and the carbon fibers were wound around PEI body using filament winding method as shown in Fig. 2 (a) and (b). And then, the CFRP tubes were inserted on the hole of the PEI body with adhesive bonding method before the additional machining of the PEI body and CFRP tubes were performed as shown in Fig. 2 (c) and (d). Since, the maximum shear stress was occurred at the adhesive bonding layer between the CFRP and PEI body, the reliable adhesion method should be used for these parts.
3. Stress on the adhesive layer of the hybrid rotor

Lee et al. [1] designed the hybrid rotor using the analytical method to reduce the radial stress on the PEI body. As a result, the compressive stress occurred on the adhesion area between the CFRP and PEI body. However, the maximum shear stress was occurred on the adhesion area between the CFRP and PEI body due to the circumferential force with the rotating speed of 150,000 rpm as shown in Fig. 3. Since the peel stress was not occurred in the adhesion area, the shear stress was dominant factor for the reliability of the adhesive joints. Therefore, in this work, the performance of the flame treatment was investigated using the double lap shear adhesive joints since the double lap shear adhesive joints are the suitable method to measure the adhesion shear strength without a peel stress.

Fig. 3 Stress on the adhesive layer between the CFRP and PEI body.

4. Specimen preparations

4.1 Double lap shear joint

Fig. 4 shows the drawing of the double lap shear adhesive joint to verify the effects of the flame treatment method with respect to treatment time. The mechanical properties of the PEI (Ultem PEI 1000, General Electric, USA) and the CFRP (USN 150, SK chemical, Korea) which are the adherends of the adhesive joints were listed in Table 1 and 2. Since the direction of the shear stress in the hybrid rotor is parallel to the fiber direction, the stacking sequence of [0/90]_{10s} was selected for the adherend of CFRP to implement the real situation. The bisphenol A-type epoxy adhesive (AW106/HV953U, Huntsman, USA) whose material properties were listed in Table 2 was used for the adhesive of the adhesive joints.

### Table 1 Mechanical properties of the CFRP (USN150, SK chemical, Korea)

<table>
<thead>
<tr>
<th>Property</th>
<th>USN 150 [0]</th>
<th>USN150 [0/90]_{10s}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density, $\rho$ (kg/m$^3$)</td>
<td>1550</td>
<td>1550</td>
</tr>
<tr>
<td>Longitudinal modulus, $E_x$ (GPa)</td>
<td>130.0</td>
<td>70.6</td>
</tr>
<tr>
<td>Transverse modulus, $E_y$ (GPa)</td>
<td>10.5</td>
<td>70.6</td>
</tr>
<tr>
<td>Shear modulus, $G_{xy}$ (GPa)</td>
<td>4.4</td>
<td>4.4</td>
</tr>
<tr>
<td>Poisson’s ratio, $\nu_{xy}$</td>
<td>0.28</td>
<td>0.04</td>
</tr>
</tbody>
</table>

### Table 2 Mechanical properties of the PEI (Ultem PEI1000, General Electric, USA) and Epoxy adhesive (AW106/HV953U, Huntsman, USA)

<table>
<thead>
<tr>
<th>Property</th>
<th>PEI</th>
<th>Epoxy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density, $\rho$ (kg/m$^3$)</td>
<td>1270</td>
<td>1110</td>
</tr>
<tr>
<td>Tensile modulus, $E$ (GPa)</td>
<td>3.2</td>
<td>1.5</td>
</tr>
</tbody>
</table>
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<table>
<thead>
<tr>
<th>Poisson’s ratio, ( \nu )</th>
<th>0.37</th>
<th>0.43</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength, ( S ) (MPa)</td>
<td>118.0</td>
<td>40.1</td>
</tr>
</tbody>
</table>

The thicknesses of the PEI and CFRP were 7.0 and 2.6 mm respectively. And the width of the specimen is 20.0 mm. The Teflon block was inserted between the PEI adherends to control the adhesive thickness of 0.2 mm. The left side bond length of the joint was 20.0 mm, while the right side bond length was 30.0 mm to make the joint fail at the left side as shown in Fig. 4.

![Fig. 4 Drawing of the double lap shear adhesive joint (Unit: mm).](image)

### 4.2 Fabricating process

Since an average surface roughness (\( R_a \)) of 2.0 \( \mu \)m for adherends was recommended for the maximum joint strength, the surfaces of the adherends were mechanically abraded with a 80 mesh abrasive sandpaper, which yielded a surface roughness (\( R_a \)) of about 2.0 \( \mu \)m of the CFRP and PEI adherend as a pre-treatment method [6]. And then the surfaces of the CFRP and PEI were cleaned with ethanol to eliminate the debris and contaminations.

![Fig. 5 flame torch for the flame treatment method.](image)

And then, the flame treatment method using propane (C\(_3\)H\(_8\)) gas was used to eliminate surface contamination and to increase the wettability of the PEI surfaces. Fig. 5 shows a flame torch (KT-2906, Kovea, Korea) for flame treatment of the PEI. The nozzle diameter is 12.1 mm. The gas consumption rate was 168 g/h with treatment distance of 150 mm. The surface temperature of the PEI adherend was about 900°C during the flame treatment process was performed. Perfect combustion could be achieved using a gas regulator of the torch during the flame treatment process. The surface treatment was performed on the surfaces of PEI for different treatment times to investigate the effects of the flame treatment on the PEI surfaces.

After flame treatment process, the adhesive joints were bonded with epoxy adhesive and the adhesively bonded joint was cured in an oven at 125°C for 2 hrs. After curing the adhesively bonded joint, adhesive fillets of the joint were removed with a razor and 2000 mesh sandpaper to reduce the variation in the strength values.

### 5. Test method

#### 5.1 Measurement of the contact angles

The contact angles on the PEI surfaces were measured with respect to flame treatment time with the sessile drop method using a drop shape analysis system (DSA100, KRÜSS, Germany) as shown in Fig. 6.
Fig. 6 Measurement equipment for the contact angle on the PEI surfaces.

Distilled water drops were deposited on the hybrid surface-treated aluminum adherend. The contact angles were measured within 10 sec after the probe liquids were deposited on the specimen to minimize deviation in the measured contact angles.

5.2 Tensile test of the double lap shear joint

The lap shear strengths of the double lap shear joints were measured with respect to the flame treatment time with a material testing machine (Instron 5882, USA) at the room temperature of 25°C as shown in Fig. 7. The displacement rate in the tensile test was set at 1.0 mm/min.

From the measured static tensile load capability, \( F \), the average static lap shear strength, \( \tau \), was calculated as follows:

\[
\tau = \frac{F}{2wl}
\]

where \( w \), and \( l \) are the width (20.0 mm) and bond length (20.0 mm) of the joint, respectively. At least five specimens were tested for each type of the CFRP and PEI adhesive joint to check for repeatability.

6. Test results and discussions

6.1 Water contact angle on the PEI surface

Fig. 8 shows the water contact angles on the PEI surface with respect to flame treatment time.

The water contact angles on the PEI surface decreased as the flame treatment time increased to 3 sec, gradually. When the flame treatment time was 5 sec, the water contact angle increased from the flame treatment time of 3 sec as shown in Fig. 8 and 9.
6.2 Tensile test results of the double lap shear adhesive joints

Fig. 10 shows the lap shear strengths of the double lap shear adhesive joints at 25°C with respect to flame treatment time. The lap shear strength of the double lap adhesive joints with flame treatment time of 3 sec was 139% higher than that of the double lap adhesive joints without flame treatment. The lap shear strength of the double lap adhesive joints decreased from the treatment time of 3 sec to 5 sec gradually as shown in Fig. 10. The trend of the lap shear strength was almost inversely same with contact angle test results (Wettability) as shown in Fig. 9 and 10.

From the test results, it might be concluded that the lap shear strength of adhesive joint consist of PEI adhered decreased drastically after certain optimum flame treatment time due to the changes of the wettability on the PEI surface.

Fig. 9 Water contact angle of the PEI surface with respect to flame treatment time.

Fig. 10 Lap shear strengths of the double lap shear adhesive joints with respect to flame treatment time.
Fig. 11 Fracture surfaces of the double lap shear adhesive joints.

Fig. 11 shows the fracture surfaces of the double lap shear adhesive joints. In the case of the without treatment condition, the perfect interfacial failure mode occurred on the PEI surface (Lower adherend) with low lap shear strength as shown in Fig. 10 and 11 (a). However, transient failure mode occurred for all the flame treated adhesive joints with higher lap shear strength as shown in Fig. 11 (b), (c), (d) and (e). Also, the remained area of the adhesive on the PEI surface increased to the flame treatment time of 3 ~ 4 sec as shown in Fig. 11 (a), (b), (c) and (d).

From the test results, it could be concluded that the flame treatment is effective method to increase the wettability and adhesion characteristic for the PEI surface. However, the treatment time should be controlled sensitively to neglect the decrease of the wettability and adhesive characteristic.

7. Conclusion

The optimum surface treatment condition of the flame treatment method found in this study will give a reliable adhesion performance for the hybrid rotor of ultracentrifuge.

The lap shear strength of the double lap shear adhesive joints with treatment time of 3 sec was 139% higher than that of the double lap shear adhesive joints without treatment. However, the lap shear strength of adhesive joint consist of PEI adherend decreased drastically after the optimum flame treatment time of 3 sec due to the changes of the wettability on the PEI surface.

From the test results, it could be concluded that the flame treatment is effective method to increase the wettability and adhesion characteristic for the PEI surface. However, the treatment time should be controlled sensitively to neglect the decrease of the wettability and adhesive characteristic.

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References