Imaging the interphase of carbon fiber composites using transmission electron microscopy: Preparations by focused ion beam, ion beam etching, and ultramicrotomy

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Abstract Three sample preparation techniques, focused ion beam (FIB), ion beam (IB) etching, and ultramicrotomy (UM) were used in comparison to analyze the interphase of carbon fiber/epoxy composites using transmission electron microscopy. An intact interphase with a relatively uniform thickness was obtained by FIB, and detailed chemical analysis of the interphase was investigated by electron energy loss spectroscopy. It shows that the interphase region is 200 nm wide with an increasing oxygen-to-carbon ratio from 10% to 19% and an almost constant nitrogen-to-carbon ratio of about 3%. However, gallium implantation of FIB tends to hinder fine structure analysis of the interphase. For IB etching, the interphase region is observed with transition morphology from amorphous resin to nano-crystalline carbon fiber, but the uneven sample thickness brings difficulty for quantitative chemical analysis. Moreover, UM tends to cause damage and/or deformation on the interphase. These results are meaningful for in-depth understanding on the interphase characteristic of carbon fiber composites.

1. Introduction

The use of carbon fiber composites has substantially increased in the fields of aerospace, transportation, and sports goods due to their excellent properties, such as high specific strength, high specific modulus, and the ability to be tailored for specific applications. At the same time, a great deal of scientific efforts has been focused on the analysis of interfacial properties and the approaches to improve, since it is well recognized that the interphase significantly impacts the final behavior of
composites. At the core of these efforts lies in the need to understand the structure, mechanical, and physico-chemical properties of components, as well as their interactions at the interphase region across multiple length scales starting from nanoscale characterization. Based on this, nanomechanical techniques, such as dynamical modulus mapping, atomic force microscopy, nanoindentation and nanoscratch, and fiber push-in and push-out tests, have been developed to reveal the thickness and mechanical properties of interphase in specific composite systems. However, knowledge of the interphase morphology as well as the structure and chemical properties between carbon fiber and polymer is still lacking at the present time. Detailed studies of microstructure and physico-chemical properties are favorable to understand the interfacial functionality mechanism and the structure–property relationship, which are critical to optimize composite behaviors.

Transmission electron microscopy (TEM) provides the unique combination of analytical techniques, for example, electron energy loss spectroscopy (EELS) and energy dispersive X-ray (EDX), which is suited and able to characterize the structural and chemical information of a sample at the nanometer scale. However, such studies require the sample to be transparent (approximately 100–150 nm) to the electron beam and being much thinner is preferred for EELS analysis. Preparation of such a sample, especially for the interphase of heterogeneous materials, is both a science and a challenge. Focused ion beam (FIB), ion beam (IB) etching, and ultramicrotomy (UM) are three common preparation techniques. FIB uses a finely focused beam of ions to bombard a target so that site-specific milling or cutting can be performed. IB etching is a sputtering process in which energetic neutral atoms or ions from a cathode impinge on a sample wafer, at an angle. UM produces an ultrathin section with the thickness down to approximately 30 nm by creating a micro crack that progressively propagates into a sample.

Since sample preparation techniques are very material-dependent, the selection of a suitable preparation technique is of great significance for TEM analysis. For carbon fiber reinforced resin composites, the challenge arises because of the huge mismatch in properties, such as modulus and hardness, between carbon fiber and resin. This brings great difficulty for preparing a thin TEM sample with an intact interphase. Based on this, herein, FIB, IB etching, and UM techniques were respectively used to prepare a TEM sample of a carbon fiber/epoxy composite. Our specific goals were to (1) identify the capabilities of these preparation methods for TEM analysis of the interphase in the carbon fiber/epoxy composite, (2) reveal the suitability and strength of each method for investigating which characteristic of the interphase, and (3) understand the microstructure, chemical components, chemical bonding states, and thickness of the interphase.

2. Experimental

2.1. Materials

Unidirectional T300-3K-40B carbon fiber (7 μm in diameter, Toray Inc.)/epoxy (5228, Beijing Institute of Aeronautical Materials) prepreg was impregnated by the hot-melt method and cured in autoclave. The cure cycle and metallographic image of the prepared composite are shown in Fig. 1. The composite has a uniform distribution of carbon fibers and a low void content. The tensile modulus of the fibers is 230 GPa (from TORAYCA™ carbon fibers data sheet), and that for the epoxy matrix is determined to be about 3.5 GPa according to GB/T 2567–2008.

2.2. Preparation methods

FIB experiment was performed in an AURIGA 40 (Carl Zeiss, Germany) Dual Beam FIB-scanning electron microscope (SEM) system with a Ga⁺ ion source at 30 kV. The preparation process is illustrated in Fig. 2. Firstly, the cross-section of the composite was identified and targeted as the region of interest (Fig. 2(a)). Secondly, a Pt protective layer was deposited on the surface of the target milling area (see Fig. 2(b)), and then coarse and medium milling was performed (see Fig. 2(c)). 20 nA beam current was used for coarse milling until the sample was left with a 2 μm thickness, and then 4 and 1 nA beam current was for medium milling until a thickness of ~1 μm was reached. Water gas was used for fast material sputtering. Thirdly, the section was detached from the surrounding material and transferred to a TEM half-grid for fine thinning (see Fig. 2(d)). Fourthly, fine thinning was carried out only at the desired areas (two braces in Fig. 2(e)) until they became transparent at 3 kV (SEM mode), using 600 pA and 240 pA.
beam current. The un-thinned part serves as a support frame to avoid bending and shrinkage deformations of the sample. Finally, the amorphous layer on the sample’s surface was removed with a low-energy Ga beam (5 kV, 20 pA).

For IB etching, a Minitom precision cut-off machine (Struers Inc. Copenhagen/Denmark) was used to cut a 1 mm thickness sample from the composite block. Then, the sample was carefully grinded and polished to 10–15 \( \mu \text{m} \) thickness by a combination of an automatic polish-grinding machine (Struers Inc.) and manual operations. Intermittent IB etching, i.e., 15 min milling followed by 10 min rest, was carried out in an RES 101 (BAL-TEC, GER) Ion Mill using a rotating stage and two ion guns with argon to prevent the temperature increase of the sample and its surroundings. The voltage was 6 kV and the ion current reached 2 mA. According to the thinning degree, the milling angle was lowered from 15\(^\circ\) to 5\(^\circ\) as milling progressed.

For UM, a Leica EM TXP fitted with a diamond knife was used to trim down the cutting face of the composite block to a taper, which would provide a suitable cutting area to the knife. The sample after trimming has a smooth and flat surface, as well as sharp edges. This is significant to obtain high-quality ultrathin sections. Ultrathin sectioning was conducted on a Leica EM UC7/FC7 ultramicrotome with an ultra 35\(^\circ\) diamond knife. The sectioning speed was 25 mm/s and the prepared sections were picked up with a C-flat grid from the water trough.

2.3. Characterization

TEM imaging was performed using a JEM-2100F (JEOL) field-emission electron microscope operating at an accelerating voltage of 200 kV.

EELS test was carried out on a Tecnai G2 F20 U-TWIN (FEI Inc.) field-emission electron microscope operating at 200 kV in the scanning transmission electron microscope (STEM) mode, providing an energy resolution of 0.6–0.8 eV. EELS is based on the acquisition of a spectrum of inelastically scattered electrons. Digital images and energy loss spectra were captured using a Gatan imaging filter (GIF Tridiem) with a dispersion of 0.2 eV/pixel and an energy shift of 270 eV. Spectral background was removed by fitting the pre-edge background with a power law function. The beam convergence angle \( \alpha \) was 0.58 mrad. The distance from the projector crossover to the recording plane was 438.5 mm, and that from the crossover to the actual entrance aperture (2 mm in diameter) was 748.74 mm. Obtaining spectra in the STEM mode using a camera length of 500 mm provided a collection semi-angle (\( \beta \)) of 2.34 mrad. With the need of chemical analysis, multiple scattering was removed off from EELS spectra by the Fourier-ratio deconvolution method. The sample thickness can be measured from the low energy electron loss spectrum, since the amount of all inelastic scattering increases with the sample thickness. The thickness (\( t \), nm) normalized to the average mean free path (\( \lambda \), nm) is measured using the log-ration method:

\[
\frac{I}{I_0} = \ln \left( \frac{I}{I_0} \right)
\]

(1)

where \( I \) is the total intensity in the spectrum and \( I_0 \) is the intensity under the zero-loss peak. From the quantitative view, \( \lambda \) needs to be determined according to the following equations:

\[
\lambda = \frac{106FE_0}{E_m \ln(2fE_0/E_m)}
\]

(2)

where \( E_0 \) is the voltage, kV, \( F \) is a relativistic correction factor related with \( E_0 \), and \( E_m \) is the average energy loss, kV, which can be calculated by the average atomic number \( Z \) of a material.
\[ E_m = 7.6Z^{0.36} \]  
\[ F = \frac{1 + E_0/1022}{1 + (E_0/511)^2} \]

Since carbon is the main element in both fiber and resin, here assume \( Z \approx 6 \) for all the three phases (i.e., fiber, interphase, and resin) of the T300/epoxy composite. Accordingly, \( \lambda \) is calculated to be approximately 169 nm. Thus, the thickness of the FIB-produced sample was in the range of 95–118 nm (0.56\( \lambda \)–0.70\( \lambda \)) with no obvious variation. For the IB etching-produced sample, gradual increasing of the thickness from 30 to 186 nm (0.18\( \lambda \)–1.10\( \lambda \)) was observed from the resin to the fiber.\(^{23}\) The thicknesses of carbon fiber and epoxy in the UM-produced sample were respectively about 50 and 12 nm (0.30\( \lambda \) and 0.07\( \lambda \)). It’s worth noting that \( \lambda \) will decrease with increasing content of nitrogen or oxygen, which have higher atomic numbers than carbon. Thus the true thicknesses for the interphase and the resin might be 1%–3% lower.

EDX tests were conducted on an analysis system (type TEAM\(^{\text{TM}}\), from EDAX Inc.) with an energy resolution of 133 eV. The beam spot size of the EDX-line scan was 0.5 nm.

3. Results and discussion

3.1. FIB preparation for composite interphase

Fig. 3(a) presents the TEM image of the FIB-produced T300/epoxy, including the bright epoxy resin, the dark carbon fiber, and the in-between interphase region. The periodical arrangement of lighter and darker double stripes along the fiber axis is seen at the top right corner, which is ascribed to the fiber cortex. The epoxy resin is the amorphous structure. The interphase region is further magnified in Fig. 3(b); however, the detailed nanostructure cannot be analyzed. The main reasons are associated with two aspects. Firstly, the sample is not thin enough, and thus the electron beam transmitted through the sample, which carries the interior structure information of a material, is less. Secondly, there are many black spots in Fig. 3(b), particularly obvious in the region marked by a yellow ellipse, which is the evidence of Ga\(^{+}\) ion implantation. The implantation amount of Ga is investigated by the EDX line-scan by performing through a structural repeating unit, as shown in Fig. 4(a). The obtained Ga X-ray intensity variation versus the position is illustrated in Fig. 4(b). Low Ga intensities locate at about 0–0.3 \( \mu \text{m} \) and 0.9–1.16 \( \mu \text{m} \), and the high intensities are at 0.5–0.7 \( \mu \text{m} \). Two gradual transition regions at probably 0.3–0.5 \( \mu \text{m} \) and 0.7–0.9 \( \mu \text{m} \) are observed. The Ga\(^{+}\) ion implantation capability is different in the three phases of the composite, and the bulk carbon fiber has a better Ga\(^{+}\) resistance due to its crystal structure than the resin matrix and the interphase. Six spots (the red dashed circles in Fig. 4(a)) were selected to calculate the atomic contents of elements, particularly for Ga. As listed in Fig. 4(c), the Ga contents are no more than 0.48, indicating that Ga\(^{+}\) ion implantation is well controlled by Pt-strap prior to FIB-milling. Compared with the light elements contained in the composite, the relatively high atomic number 31 of Ga makes it eye-catching in the image. Therefore, such a small amount of Ga would hinder fine structural analysis of the interphase under a high resolution.

The former result indicates uniformity of the FIB-produced composite, and thus detailed chemical analyses are feasible by EELS, performed at 12 probe-points along 550 nm crossing the interphase region, as shown in Fig. 5(a). The C-K, N-K, and O-K edges, respectively starting at around 287, 402, 540 eV, are clearly identified in the EELS spectra (Fig. 5(b)). A feature at approximately 330 eV or 350 eV is a multiple-scattering resonance (MSR), which represents the energy of the standing wave set up when the excited wave is scattered back from the parent atom’s second nearest neighbors.\(^{30,31}\) The MSR is related with the C–C bond length,\(^{30}\) and the shorter the bond length is, the lower energy is needed to scatter back. Fiber has a high degree of graphite crystallite, which has minimum heteroatoms and aliphatic molecules (plenitude in the interphase and the epoxy resin). Thus, probe-points 1–3 are inferred at the fiber cortex. Other probe-points 4–12 are at the interphase and the resin, while the exact belongings need further analysis.

The process of inner-shell ionization is one of the principal inelastic interactions and the inner-shell ionization edges of elements can be used for chemical bonding analyses of a sample, especially sensitive for light elements. Thus, chemical bonding states were further analyzed from the energy-loss
near-edge structure (ELNES) of C K-, N K-, and O K-edges, as shown in Fig. 5 (c) and (d). Since chemical bonding analysis for carbon fiber/epoxy composites is rarely reported, the spectra discernible features are assigned based on relevant carbon materials and organic polymers. In Fig. 5 (c), the C K-ELNES shapes of probe-points 1–3 are similar to those of graphitic carbon, while those of other probe-points 4–12 agree with those of amorphous carbon. The sharp and clear features (peaks 1) of probe-points 1–3 at (287 ± 0.3) eV and probe-points 10, 11 at 289.3 eV are respectively ascribed to the C=C σ* resonance and the overlap of C=O π* and C–H σ* resonances. In comparison, peaks 1 of other probe-points (4–9 and 12) at (288 ± 0.4) eV become wider, which could be affected by C=C π* and C=O π* resonances together. Peak 2 at (295.0 ± 0.5) eV is a C=C σ* resonance. Based on the bond length correlation, peaks 3 at 299.2 eV for probe-points 1–3 are C–N σ* resonances. Sizing agents of both the T300 and the matrix are both epoxy type, so the bond length of C–O in epoxy is large due to the existence of angle tension, which makes the electron cloud not well overlapped. Thus, peaks 3 of probe-points 4–12, ascribed to the superposition of C–O σ* and C=N σ* resonances, are shifted lower. By contrast with probe-points 4–9 and 12, peaks 3 of probe-points 10, 11 at 298.4 and 298.8 eV are slightly higher, which might be affected by higher contents of nitrogen. The weak features of peaks 4 at (302 ± 1.3) eV are observed for probe-points 4–12, which is associated with the σ* resonance of C=O bonds. In Fig. 5 (d), two features at 402 eV (peak 5) and 410 eV (peak 6) of the N–K edge are the 1s to π* and 1s to σ* anti-bonding orbitals. Peaks 7, 8, and 9 are respectively O=C π*, O–C σ*, and O=C σ* resonances, and only probe-points 10 and 11 observe peaks 9. From above analyses, the low Ga intensity, the position of MSR, and the C K-ELNES shapes confirm that probe-points 1–3 are at the carbon fiber. In addition, the different features, positions, and assignments of peak 1 and peak 3 from the C K-ELNES indicate that the interphase region is located between probe-points 4 and 9, about 200 nm in thickness. Probe-points 10 and 11 are in the epoxy resin.

The element amounts, calculated by the Digital Micrograph software, are dependent on beam energy, convergence semi-angle, collection semi-angle, and the collected EELS spectra. The results are plotted in Fig. 6. Taking account of the semi-quantitative accuracy of this method, only the integer part of percentage is deemed to be valid. There is about 98% carbon in the carbon fiber (probe-points 1–3), and a gradual decrease of carbon content from 88% to 81% is observed in the interphase region (probe-points 4–9). The resin (probe-points 10, 11) has the minimum carbon content of about 70%. The ratio of nitrogen to carbon (N/C) is basically the same of 3% except higher at the epoxy resin (about 8%) due to the existence of amine cure agent. No oxygen is detected in the carbon fiber. The ratio of oxygen to carbon (O/C) shows an increasing trend from probe-point 4 (10%) to 6 (19%), and then basically is constant until a significant jump between probe-points 9 and 10.

3.2. IB etching and UM preparations for interphase

The results of the IB etching-produced carbon fiber/epoxy composite and the high resolution transmission electron microscopy (HRTEM) image of its interphase are shown in Fig. 7. Fig. 7(a) shows the amorphous epoxy resin (light grey), the oriented graphite crystallites of fiber (dark grey), and the intact interphase region (grey) between them. Fig. 7(b) exhibits a gradual variation from nanoscale crystalline to amorphous structure as getting to the matrix. Nanocrystals that consist of a stack of aromatic layers, two or three nanometers in length, are basic structural unit. Our previous work shows

![Image](image-url)
that the O/C is 15% for the resin and 4%-9% for the interphase region in the IB etching-produced sample, which are both lower than the results in the FIB-produced sample. The reason might be ascribed to the thin thicknesses of the resin and the interface regions (lower than 50 nm) that are unable to sustain electron beam irradiation and are easier to be penetrated. Moreover, the sample thickness changes in the three phases, produced by the IB etching method itself. Thus, estimation of the interphase width based on a TEM image of the IB etching sample is not accurate. Related methods and detailed descriptions can be found in our previous work.23 Here, we will not elaborate.

For UM, the sample cutting is conducted along two different directions, normal and parallel to the fiber axis respectively on the composite, as shown in Fig. 8(a) and Fig. 9(a). The cutting in ultramicrotome is always in the vertical direction. When the fibers are lying horizontally, the prepared composite ultrathin section is given in Fig. 8(b). It evidently shows two different structures, which are the stripe structure of the carbon fiber and the homogeneous structure of the epoxy. One of the interphases is reasonably preserved, while on the second there is a gap. During sectioning, the upper surface of the section is under tension, and the lower surface is under compression, similar to a curved cantilever beam in Fig. 8(c). In fact, the cutting force (F) on the section can be decomposed into two directions, i.e., F1 and F2 in Fig. 8(d). The tension at

![Fig. 5 EELS spectra analysis of the interphase region in the energy loss range of C K-, N K-, and O K-edges.](image1)

![Fig. 6 Variation of element contents with different EELS probe-points.](image2)

![Fig. 7](image3)
the upper cutting surface can be partly offset by shear stress $F_2$, but at the lower original surface, high compression tends to cause failure, particularly in the interphase region. For the interphase at the upper cutting surface, a magnified TEM image is shown in Fig. 8(e), in which the resin region is smooth with an amorphous structure and the fiber region reveals aligned fibril textures. However, whether the region in-between the fiber and the resin is the original interphase or not remains unclear, because the fiber is pressed against the epoxy resin by the cutting force. Note that there are many fine lines normal to the fiber axis in Fig. 8(b), which should be attributed to the fine imperfections of the cutting edge.

In Fig. 9(a), the composite block was cut longitudinally to the fiber axis. Fig. 9(b) shows that the carbon fiber is seriously distorted and damaged. As the knife is acted on the section, pressure is applied on the fiber and the resin simultaneously. In this case, the carbon fiber tended to be damaged due to brittleness, and shear stress ($\tau$) concentrated along the interphase region. A sketch of stress on the section is depicted in Fig. 9(c). Therefore, the intact interphase structure cannot be identified from the distorted and fractured carbon fiber.

### 3.3. Comparison of different preparation methods

Three preparation methods used in this paper are compared in analyzing the interphase of the carbon fiber/epoxy composite, as summarized in Table 1. FIB is a suitable and convenient preparation technique for chemical analysis of the interphase.
A relatively uniform thin section area and a small incident angle of ion beam are the benefits for TEM and EELS analysis. However, the Ga\(^+\) ion implantation limits high resolution analysis of fine structures and lattice imaging. IB etching is a feasible method for structure and bonding state analyses of the interphase region, but an uneven thickness of the section brings difficulty for quantitative chemical analyses. Moreover, in order to avoid sample damage and artifact effect of IB etching, extra care and awareness are necessary for operation, including the degree of grinding, the sample cooling, and the milling parameters. For the UM method, distortion and fracture inevitably occur in fine structures, particularly in the interphase region and the carbon fiber of the composite. Quality of the knife edge is vital for acquiring a high-quality ultrathin section; however, the diamond knife can be easily damaged by the hard carbon fibers. Hence, UM is not suitable for investigating the interphase of carbon fiber/epoxy composites in TEM.

Table 1  Comparison of three preparation techniques for interphase analysis in carbon fiber/epoxy composites.

<table>
<thead>
<tr>
<th>No.</th>
<th>Comparison aspects</th>
<th>FIB</th>
<th>IB etching</th>
<th>UM</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Capability for TEM analysis of interphase</td>
<td>A(^x)</td>
<td>A(^x)</td>
<td>NA(^1)</td>
</tr>
<tr>
<td>2</td>
<td>Interphase integrity</td>
<td>Intact</td>
<td>Intact</td>
<td>Deformed or broken</td>
</tr>
<tr>
<td>3</td>
<td>Suitable for interphase:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>(1) structure</td>
<td>NA(^1)</td>
<td>A(^x)</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>(2) chemical component</td>
<td>A(^x)</td>
<td>BA(^3)</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>(3) chemical bonding</td>
<td>A(^x)</td>
<td>A(^x)</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td>(4) width (thickness)</td>
<td>A(^x), directly from chemical analysis</td>
<td>A(^x), by tilting the holder(^19)</td>
<td>–</td>
</tr>
<tr>
<td>4</td>
<td>Drawbacks</td>
<td>Ga(^+) ion implantation</td>
<td>(1) Uneven thickness variation (2) Complex preparation process (3) Low success rate of sample preparation</td>
<td>Expensive diamond knife is easy to damage</td>
</tr>
<tr>
<td>5</td>
<td>Preparation time &amp; efficiency</td>
<td>Fast (2 h)</td>
<td>Time-consuming (generally a few days or weeks)</td>
<td>Simplification &amp; Time efficiency</td>
</tr>
<tr>
<td>6</td>
<td>Preparation cost</td>
<td>Expensive</td>
<td>High</td>
<td>Low</td>
</tr>
</tbody>
</table>

Note: A\(^x\) indicates Applicable; NA\(^1\) indicates Not Applicable; BA\(^3\) indicates Barely Applicable.
4. Conclusions

Three methods, namely FIB, IB etching, and UM, were conducted for TEM sample preparation to study the interphase of a carbon fiber/epoxy composite. The section qualities were analyzed for different preparation techniques and the effects on structure and composition of the interphase were discussed.

(1) FIB can produce intact morphology of the interphase with a relatively uniform thin area, allowing detailed analyses of the chemical compositions and the bonding states. According to the bonding states, a transition interphase area of 200 nm thick is estimated, through which the O/C increases gradually from 10% to 19% and the N/C is almost constant at 3%. Ga⁺ implantation is observed in the composite, and its relatively high atomic number (compared with carbon and oxygen) obscures further fine structure analysis of the interphase.

(2) The IB etching technique shows that the interphase region is a transition area from basically an amorphous structure to a crystalline structure. However, the uneven section thickness, caused by the method itself and quite differing sputter yields of fiber and resin, brings difficulty for fine chemical analysis within the interphase. In order to avoid sample damage and artifact effect of IB etching, utmost care and awareness are necessary for operation during the milling process.

(3) UM tends to cause mechanical damage and/or deformation in the interphase region, which is not suitable for interphase investigation of carbon fiber/epoxy composites.

These results are meaningful for accurate and in-depth understanding on the interphase characteristic of carbon fiber/epoxy composites.

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References


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