Research Article

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Effect of laser irradiation on morphology and dielectric properties of quartz fiber reinforced epoxy resin composite

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Abstract: The quartz fiber-reinforced epoxy resin-based composites (QFRECs) were fabricated by resin transfer molding (RTM), and then the effect of laser ablation on the structure and dielectric properties of the prepared composite was investigated. The FTIR, XRD, and SEM analyses show that the thermal decomposition, pyrolysis, carbonization, graphitization, and ablation occurred on the surface of the epoxy resin under laser irradiation. The in situ produced carbon endows the QFREC with an improved dielectric constant, which increases maximally from 3.3 to 4.5 in the range of 7–17 GHz under the energy intensity of 226 W cm⁻². Compared with short-time irradiation (5 s), long-time irradiation (10 s) exhibits a greater impact on the dielectric constant due to the formation of crystal graphite at prolonged high temperature. Meanwhile, the rough and deep pits are inclined to form on the surface of the long-time irradiative composite. These findings provide guidance for the practical application of QFREC as wave-transmitting materials.

Keywords: quartz/epoxy composite, RTM, laser ablation, graphitization, dielectric constant

1 Introduction

Polymer-based composite has been widely used in the fields of communication and telemetry due to its characteristic merits of process efficiency, excellent dielectric properties, good environmental resistance, and low cost (1–3). The wave permeability, an important parameter of wave-transmitting composite, not only depends on the inherent characteristics of raw materials but also mainly affected by the service environment. In practical use, the wave-transmitting material may be subjected to long-term high-temperature service caused by high-speed air flow, strong electronic confrontation, and laser ablation (4–6). The microstructure and the composition of polymer inevitably change in some extreme cases on account of its poor intrinsic thermal property, which will result in serious deterioration of wave permeability. This severely harmed the stability and reliability of polymer-based composites and may cause unacceptable performance degradation of wave-transmitting materials (6,7). In consideration that the wave permeability is often determined by electromagnetic parameters of materials, the effect of application environments on the functional parameters of polymer-based wave-transmitting composite is necessary to be investigated systematically.

As the wave permeability is directly related to the dielectric parameters (dielectric constant $\varepsilon$ and dielectric loss $\delta$) of the material, the dielectric property is considered as one of the most important criteria for evaluating the wave transmission (8). In general, low $\varepsilon$ and $\delta$ of wave-transmitting material often result in good wave transmission due to weak interfacial polarization, electron polarization, and dipole polarization (9). So, the quartz fiber-reinforced polymer resin composite (QFRPC) has attracted great attention owing to its low $\varepsilon$ and $\tan \delta$ (loss angle tangent), as well as good mechanical and thermal properties (10). However, the polymer matrix can easily decompose into gas volatile and produce carbonization zone under high-temperature ablation process (11–16). Although the wave-transmitting variation of QFRPC under high temperature caused by rapid speed air flow has been investigated in depth and systematically, few researches focused on the effect of laser ablation on its dielectric property. In fact, the laser ablation might be the most probably encountered destruction for composite in the future. Under the laser ablation, the surface temperature of composite in the local region...
abruptly increases and results in oxidation of the organic compound matrix (11–13). The ablated surface can turn into carbon compounds, including the formation of splitting carbon residue (17–19), and simultaneously eject carbon monoxide, carbon dioxide, and water vapor. The produced carbon compounds can form crystalline graphite under continuous laser ablation (20–23). The power intensity and time of laser ablation inevitable determine the graphitization degree and microstructure of ablated surface, which mainly affect the conductivity and electromagnetic wave attenuation capacities of wave-transmitting material (24). However, it is a great challenge to clearly understand the relationship between laser ablation and wave permeability of QFRPC due to the complex ablative mechanism and rigorous conditions.

Herein, quartz/epoxy composite, the most commonly used wave-transmitting material, was first fabricated via resin transfer molding (RTM) (25,26). Then, the laser ablation experiment was carried out on the prepared composite under two conditions of the same laser energy, i.e., the power intensity of 226 W·cm⁻² for 10 s irradiation and the power intensity of 452 W·cm⁻² for 5 s irradiation. The analysis of morphology shows that the epoxy resin undergoes ablation, and in situ formed the graphitized carbon with “sea-island” structure on the ablated surface, resulting in the increased dielectric constant of the material. Compared with the ablation of 452 W·cm⁻² for 5 s, the ablation of 226 W·cm⁻² for 10 s results in higher graphitization degree and dielectric constant.

2 Experimental section

2.1 Materials

JC/T 2244-2014 Quartz fiber cloth woven from C-type Quartz fiber with the average diameter of 7.5 μm, linear density of 190 tex, and density of 2.2 g·cm⁻³ was provided by Hubei Feilihua Quartz glass Co. Ltd. (Jingzhou, China). The poly CC135 epoxy resin with the epoxy equivalent of 0.48–0.54 mol·100 g⁻¹ and W-93 acted as a curing agent were supplied by Guangzhou Jiuying Chemical Materials Co., Ltd. (Guangzhou, China).

2.2 Preparation of the quartz/epoxy composite

Quartz/epoxy composite was prepared by the resin transfer molding (RTM), and according to the shape of the finished product, a certain size of quartz fiber cloth was cut for manual layering. To obtain the total height of the composite of 7 cm, the stacked layers are set about 50. The quartz fiber was put in the mold and the resin of 20 mPa·s was injected. After the resin infiltrates the quartz fiber completely, the composites were cured at 130°C for 24 h. The mass fraction of the fiber in the composites was 65 ± 2 wt%.

2.3 Laser ablation

The ablative carbonization experiment of quartz/epoxy composite was carried out by 2 kW near-infrared commercial solid laser device (homemade instrument, Nd: YAG, wavelength 1,064 nm), which has uniform laser intensity in the 20 mm diameter beam area. To realize full ablative carbonization of the material surface, the laser beam acting on the material surface is 20 mm in diameter, slightly larger than 18 mm in diameter of the material. To obtain the effects of laser parameters on the dielectric constant after ablative carbonization of materials, different laser heat flux and irradiation time were selected in the experiment under the same laser energy applied on the surface of materials, as shown in Table 1.

2.4 Characterization

The thermogravimetric analysis (TGA) was performed on a thermogravimetric analyzer (Pyris1, Perkin-Elmer, USA). The dielectric property of composites was obtained by the
precision impedance analyzer (HDMS-1000). The surface morphologies of the ablated composite were observed by using a field emission scanning electron microscope (SEM) (JSM-6700F) at an accelerating voltage of 5.0 kV. The samples were coated with a thin layer of gold beforehand. Wide angle X-ray diffraction (XRD) measurements were performed on a Y-2000X diffractometer (40 kV, 40 mA) with Cu (wavelength = 0.1542 nm). The recorded region went from 10° to 70° with the scanning speed of 5°·min⁻¹. The ablated composites were prepared with KBr by pressing and tested on a Nicolet 6700 Fourier transform infrared spectroscopy analyzer (Thermo Fisher Scientific). The spectra were obtained in the wave number range of 4,000–700 cm⁻¹ by averaging 16 scans with a resolution of 2 cm⁻¹. The dielectric constant and loss were measured by a high Q cylindrical resonant cavity test set at the frequency range from 7 to 17 GHz. The testing was conducted on a cylindrical specimen with a diameter of 50 mm and a thickness of 3 mm.

3 Results and discussion

3.1 Morphology analysis

The digital photographs of the prepared quartz/epoxy composite before and after laser ablation are shown in Figure 1. The quartz/epoxy sample exhibits a uniform surface with light yellow, which is the characteristic of cured epoxy matrix composite. The black appearance of the samples after laser ablation indicates that serious carbonization occurs on the ablative surface. Closer observation reveals the carbonization layer is rough, bulging, and nondense, and lots of potholes existed in the surface of the ablated samples. This is due to the nondensification of the ablative products formed by laser heat flow, as well as the thermal decomposition and pyrolysis of resin matrix during the process to produce gas, so that the quartz fiber layer is no longer glued between layers. Although the laser intensity in the beam area is uniformity, the temperatures of the sample in center and edge position are actually different due to the heat conduction. In spite of this, nearly uniform morphologies of the samples in SEM images suggest that the difference of temperature can be neglected, which may be attributed to the short irradiation time (5–10 s). Compared with that of the ablation under 452 W·cm⁻² for 5 s, the ablated sample of 226 W·cm⁻² for 10 s exhibits a deeper pit and rougher surface. It indicates that the carbonization of epoxy resin is influenced not only by the energy intensity of the laser but also by the ablation mode. For the case of the same laser energy intensity of 2.26 kJ·cm⁻², the damage of quartz/epoxy composite under long-time ablation is more serious than that of short-time ablation. This may be related to the combination of the low thermal conductivity of the epoxy resin and the high thermal stability of the quartz fiber.

To better understand the morphologic difference of quartz/epoxy composite under the two ablative models, the scanning electron microscopy (SEM) of quartz/epoxy composite surface after laser ablation was carried out and the corresponding results are shown in Figure 2. Lots of little bits and pieces are observed in the ablated surface of quartz/epoxy composite, which is attributed to the ablative products from epoxy resin. By comparing Figure 2a with Figure 2b, it can be seen that the ablative fragments under 226 W·cm⁻² power intensity for 10 s are larger and looser than that of 452 W·cm⁻² for 5 s. This indicates long-time ablative action at low energy is more detrimental to the quartz/epoxy composite than high-power intensity but short-time ablation, which is consistent with the macro-observation from Figure 1. Although the large number of

![Figure 1: Digital photographs of the prepared quartz/epoxy composite before (a) and after laser ablation with 452 W·cm⁻² power intensity for 5 s (b) and 226 W·cm⁻² for 10 s (c).](image-url)
ablative debris appeared in the enlarged images of both samples (Figure 2a', a", b', and b") are attached to the quartz fibers, the fibers are separated from each other. It manifests that the quartz fiber cloth has been obviously broken due to the thermal damage and rapid melting under laser irradiation.

3.2 Structure analysis

The Fourier-transform infrared (FTIR) spectra of the quartz/epoxy composite before and after laser ablation are shown in Figure 3. The presence of numerous absorption peaks in the original composite sample, especially in the fingerprint region, suggests the existence of a large number of organic groups in epoxy resin. However, most of these peaks including C–O–C group (absorption peak at 1,020 cm⁻¹), tertiary alcohol (1,240 cm⁻¹), and benzene ring (1,510 cm⁻¹) completely disappeared after laser ablation. Nevertheless, the absorption peaks at 3,438, 2,920, and 1,630 cm⁻¹ still existed in the spectra of the ablated samples, which are assigned to −OH, −CH₂, and C=C groups. This indicates that most oxygen-containing groups are eliminated from epoxy resin after laser ablation, and the ablation product contains C=C, C–OH, and C–H groups. Compared with that of laser irradiation under 452 W·cm⁻² for 5 s, the composite irradiated under 226 W·cm⁻² for 10 s exhibits strong absorption at 1,410 and 775 cm⁻¹, assigning to stretching vibration and bending vibration outside benzene ring surface of C–H. The difference in spectra between various laser ablative

![Figure 2](image2.png)

**Figure 2:** SEM images of sample under different ablative conditions: (a) 452 W·cm⁻² for 5 s and (b) 226 W·cm⁻² for 10 s.

![Figure 3](image3.png)

**Figure 3:** FTIR spectra of the quartz/epoxy composite before and after laser ablation.
conditions displays that more organic-functional groups existed in the sample of 5 s irradiation compared with that of 10 s irradiation. It implies the surface ablation at high power intensity is more serious than that of low power intensity. This may be explained by the fact that high-power intensity of laser irradiation results in high temperate on the sample surface, which leads to complete decomposition of epoxy resin. Therefore, it may conclude that high-power intensity leads to sufficient ablation on the surface, but the irradiation is relatively shallow. The ablation under low-power intensity is just the opposite and exhibits inadequate decomposition on the surface, but deeper irradiation pit.

To better explore the ablative mechanism of the prepared composite, the thermal analysis of epoxy resin and quartz fiber was carried out under air conditions by using a simultaneous thermal analyzer, and the corresponding TG and DSC curves are shown in Figure 4. Three mass loss stages are observed in the TG curve of epoxy resin. The mass loss of the first stage from room temperature to 310°C is about 2.9%, while no obvious absorption peak exists on the DSC curve within this range. This suggests that the mass loss in the first stage is attributed to the volatilization of annex such as absorbed water and other small molecules in epoxy resin. Rapid mass loss of epoxy resin occur in the second stage. The mass residual rate declines from 97.1% to 27.8% when the temperature rises further to 450°C. Combining with the obvious exothermic peak that appeared in the DSC curve at 410°C, the mass loss at this stage is mainly ascribed to the thermal degradation and decomposition. In this stage, the chemical bond of epoxy resin breaks gradually as the temperature increases, forming the volatile matter including H2O (gaseous), CO, and CO2. The third stage from 450°C to 660°C exhibits the moderate mass loss rate, and the final mass residual is only 1%. Meanwhile, the medium exothermic peak also appears on the curve of DSC. This may be attributed to the further decomposition of material with high chemical bonds and oxidation of the residual carbon. Compared with the second stage, the mass loss rate of this stage is relatively low. This indicates that the serious ablation and maximum mass loss of epoxy resin occurs near 400°C. Unlike epoxy resin, the quartz fiber shows excellent thermal stability. Only 3% mass loss is observed below 300°C, which may be attributed to the volatilization of absorbed water. Thereafter, almost no mass loss has been detected until 1,000°C. This is because the decomposition temperatures of SiO2, the main ingredient of quartz fiber, exceed 2,000°C. Combining with the small exothermic and endothermic fluctuations in the DSC curve, it implies that the quartz fiber has hardly changed during the laser ablation. In conclusion, the laser ablation mainly affects the composition of epoxy resin rather than quartz fiber, although the quartz fiber may rapidly melt during the laser irradiation.

X-ray diffraction (XRD) is a universal method to test the microcrystalline structure of substances, such as grain size and crystallization degree. The XRD diffraction pattern of quartz/epoxy composite before and after laser irradiation is shown in Figure 5. The obvious difference exists in the XRD diffraction pattern between the initial composite and the ablated samples. Two sharp diffraction peaks at 2θ = 18° and 21° can be observed in the initial composite. However, these peaks disappeared from the diffraction spectra of the ablated samples, and some new peaks appeared at 2θ = 26.6° and 44.6°, assigning to graphite (002) (G (002)) and graphite (100) (G (100)) lattice plane (XRD PDF standard card No. 75-1621). This indicates

Figure 4: TG and DSC curves of epoxy resin (a) and quartz fiber (b).
that the carbonization reaction occurred in the prepared composites under laser irradiation. The relatively wide diffraction peak of G(002) lattice plane under the condition of 5 s irradiation exhibits the characteristics of a typical bread peak and "disorderly layer structure" microcrystal. However, the corresponding peak under the condition of 10 s irradiation is relatively narrow, showing a higher degree of crystallization. The degree of graphitization (G) is often used to describe the crystallization degree of carbon materials. The corresponding expression is shown in Eq. 1:

\[
G = \frac{0.344 - d_{002}}{0.344 - 0.3354}
\]

where \(d_{002}\) (nm) is the layer spacing calculated by the main characteristic peak of G(002) lattice plane in the XRD spectrum.

By means of the XRD spectrum analysis software JADE6.0, the crystal plane diffraction peak spacing under the laser irradiation of 5 s and irradiation of 10 s is 0.3401 and 0.33918 nm, respectively, and the corresponding graphitization degrees calculated from the formula is 45.3% and 56.0%. It suggests that under the same laser energy intensity, long-time irradiation tends to form the high graphitization degree in the quartz/epoxy composite.

The epoxy resin in quartz/epoxy composite is first decomposed to produce carbon materials under high temperature caused by laser irradiation. With the ablation time prolonging, the produced carbon material with the amorphous structure gradually turns into crystalline graphite. Because the dielectric properties and electrical conductivity of carbon materials are determined by their microstructure, the wave permeability of quartz/epoxy composite therefore mainly depends on laser irradiation conditions.

In addition, the peak appeared at \(2\theta = 62.5^\circ\) in composite after 5 s irradiation is assigned to SiC (110) (SiC (110)) (XRD PDF standard card No. 73-1749), which indicates the carbon can react with quartz fiber to form SiC crystal under the laser irradiation condition. Compared with that of the 5 s laser irradiation, a relatively narrow and intensive peak appeared at \(2\theta = 35.6^\circ\) in the composite after 10 s irradiation is assigned to SiC (002) lattice plane (SiC (002)). This further suggests that the perfect graphite and SiC crystals are formatted at high temperature for a long time.

### 3.3 Dielectric performance analysis

The dielectric constants of quartz/epoxy composites before and after laser ablation are shown in Figure 6. The initial quartz/epoxy composite exhibits a relatively low and stable dielectric constant of 3.3–3.4 in the range of 7–17 GHz, thanks to the excellent performance of quartz fiber and epoxy resin. However, the dielectric constant of the ablated composites increases obviously and shows some fluctuations.

![Figure 5: The surface XRD spectra of quartz/epoxy composite.](image)

![Figure 6: The dielectric constant of the quartz/epoxy composites before and after ablation.](image)
The average dielectric constants of both ablated composites are 4.0–4.5, which is about 21–36% higher than that of the initial composite. The improvement in the dielectric constant is attributed to the formation of highly conductive carbon/graphite materials after laser ablation. Compared with laser irradiation of 5 s, the dielectric constant under 10 s irradiation increases greatly. The maximum dielectric constant of the latter reaches 4.87 at 16.6 GHz, almost 20% increasing than that of the former. This phenomenon can be explained by the difference in graphitization degree between ablated composites. Because the electric conductivity of crystal graphite is superior to that of amorphous carbon, the quartz/epoxy composite ablated for 10 s displays a higher dielectric constant due to its relatively complete graphitization compared with that of 5 s irradiation.

Overall, the quartz/epoxy composite is suitable to be used as a wave-transmitting material on account of its low and invariable dielectric constant. However, the surface structure and composition of the composite change significantly under the laser irradiation with high energy, resulting in the obvious improvement of the dielectric constant. In the case of constant energy intensity, amorphous carbonation is liable to form on the surface of ablated composite with short-time laser irradiation, while crystal graphite structure is easily formed under long-time laser irradiation, which is more harmful to wave-transmitting material.

4 Conclusion

Although low and invariable dielectric performance endows quartz/epoxy composite with extensive application in the fields of communication and telemetry, obvious improvement in dielectric performance was observed in laser ablated composite due to the existence of thermal decomposition, pyrolysis, carbonization, graphitization, and ablation on the surface of epoxy resin. Rougher and deeper pits are easier to form on the surface of composite under long-time irradiation, rather than short-time irradiation. In addition, short-time laser irradiation tends to produce amorphous carbonation on the surface of ablated composite, while crystal graphite structure is easily formed under long-time laser irradiation, which is more harmful to wave-transmitting material due to its high electrical conductivity and dielectric constant. Under the 226 W cm⁻² energy intensity of the laser irradiation, the dielectric constant of the quartz/epoxy composite ablated for 5 and 10 s increased from 3.3 to 4.0 and 4.5, respectively.

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