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# Preparation and characterization of the modulus intermediate layer in carbon fiber/epoxy composites by depositing sepiolites

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**ABSTRACT:** An important aspect in development of multi-scale reinforced composites is their mass production which can be easily realized. In this article, the sepiolites (Si12O30Mg8(OH)4(OH2)4·8H2O) are directly deposited onto the surface of JH-T800 carbon fibers for the first time with no need for removal of the commercial sizing agent. The sepiolites adhering to the carbon fibers are uniformly distributed with random orientation, and participated in the formation of high modulus intermediate layer encompassing the carbon fiber. After the deposition of sepiolites, the interfacial shear strengths (IFSS) of the carbon fiber/epoxy composites are significantly improved as shown in single-fiber composite fragmentation tests. Compared to the commercial carbon fiber composites, the sepiolite-deposited fiber composites also exhibit obvious improvement in the interlaminar shear strength and flexural strength. As a new kind of multi-scale reinforcement with industrial application value, the sepiolite-deposited carbon fibers can further raise the level of mechanical properties of the existing carbon fiber reinforced composites. © 2016 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 43955.

**KEYWORDS:** composites; fibers; mechanical properties; resins; surfaces and interfaces

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## INTRODUCTION

Inorganic clay is a kind of effective reinforcing nanofillers to improve the properties of neat polymers.[1] However, most researches on inorganic clay were mainly focused on the two-dimensional (2D) clay materials, and the one-dimensional (1D) nano-scale clay was rarely concerned. Sepiolite (Si12O30Mg8(OH)4(OH2)4·8H2O) is a kind of 1D nano-scale clay with abundant silanol-based chemical bonds on the surface. Recently, sepiolite, as a special needle-like clay species, has gained increasing attention because it is believed that this needle-like nanofiller can be more easily dispersed in polymeric matrices due to its relatively low external specific contact surface area compared with platelet-like clays of the same aspect ratio[2] and it exhibits extraordinary performance on the mechanical properties, thermal stability, flame retardancy, and barrier properties derived from its special structure.[2–5] The isolated sepiolite is in the form of fiber, about 10–12 nm thick and 10–30 nm wide, and its aspect ratio is about 200–300.[6,7] It is a naturally fibrous phyllosilicate with a typical length of 100–5,000 nm.

Due to these distinctive structures, sepiolite can potentially be well dispersed in polymer matrix and thus improve the mechanical and thermal properties of these composites. Thus, sepiolite has been used to improve various polymer composites, such as polypropylene,[2,6] Nylon-6,[3] epoxy resin,[4,7] poly(ethyl methacrylate), poly(2-hydroxyethyl methacrylate),[8] polyester,[9] polyurethane,[10] poly(hydroxyethyl acrylate),[11] chitosan,[12] poly(dimethylsiloxane),[13] and poly(sodium acrylate).[14] But so far, no research demonstrates that by adopting sepiolite, improvements can be achieved in multiscale composites.

Multiscale composites, where nanoscale materials are topologically combined with microscale reinforcements, have attracted a considerable amount of attention from composite researchers. It is well known that one of strategies for forming multiscale composites is attachment of nanofiller onto the fiber surface. And recent research demonstrates that significant improvements can be achieved in these multiscale composites e.g., interfacial shear strength (IFSS),[15–18] interlaminar fracture toughness,[19,20] and fatigue resistance.[21] There are different techniques to directly place nanofiller on fiber surface given in the literature so far: growing them directly on the surface of fibers,[22,23] electrophoretic deposition on fiber surface,[24] and sizing the fiber surface with carbon-based nano-reinforcement coating.[25,26] Note that

direct growth of nanofiller on fibers by means of chemical vapor deposition need complex chemical treatments. What's more, it is believed that growth of nanoscale materials on fibers could introduce damage to the fiber and decrease the fiber tensile property.

For the purpose of improvement in interfacial property of multiscale composites through simple process, the nanofiller-sizing method have been used in glass fiber composites, while the spraying technology of the depositing nanofiller is adopted for carbon fiber composites. For example, Ashish Warrier reported that the presence of carbon nanotubes (CNTs) in the sizing resulted in an increased resistance of crack initiation fracture toughness by +10%.[27] Kwon realized significant increase in the interlaminar fracture toughness of carbon fiber/vinylester composite laminate by means of spraying CNTs.[19,20] These results reveal that the deposited-nanofiller technology is a promising method for strengthening interfacial bonding between matrix and fiber, helping improve composite mechanical properties.

In this article, we delivered sepiolite to the surface of carbon fiber by immersing the fiber bundles in sepiolite-containing aqueous suspension, which takes advantages of the aforementioned sizing and spraying methods. Without any chemical treatment, sepiolite was directly used for the preparation of multiscale composites and realized sepiolite-depositing on fiber surface. The composite samples were characterized by scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FTIR), and mechanical measurement test. The results suggest that our present work provided a very good control on the preparation of multiscale composites.

## EXPERIMENTAL

### Materials

JH-T800 carbon fiber rovings were produced by Jilin Petrochemical Company, China National Petroleum Corporation. The average diameter of the carbon fibers was 5 μm and the sizing content of the carbon fibers was about 1 wt %. The sepiolites were obtained from Beijing Sinmaya Chemicals Co. Ltd., China.

The epoxy resin, 4,5-epoxyclyclohexyl-1,2-diglycidyl diformate (TDE85, produced by Tianjin Jindong chemical plant, China) and the curing agent, diethylene toluene diamine (DETDA, produced by Changzhou Sunlight Pharmaceutical Co. Ltd., China) were used as matrix.

### Preparation of Sepiolite-Coated Fibers

Firstly, the sepiolite particles were washed using de-ionized water to get rid of soluble impurity. Then, the sepiolites were dispersed into de-ionized water by using an ultrasonic washer operating at 100 W and 60 min elapsed time to obtain the stable suspension with a concentration of 0.05 wt %. The suspension can remain stable for 2 h. Then, the carbon fiber rovings were pulled through the depositing system comprising a suspension bath and a set of rollers. The rollers can make the carbon fibers achieve sufficient filament spreading. The carbon fiber rovings, consisting of 6k monofilaments were successively impregnated after passing through the bath containing the sepiolite suspension with a speed of 0.1 m/min. This pulling speed of carbon fiber rovings was slow enough to attain good depositing of the sepiolites on the surface of carbon fibers. After impregnation, the carbon fiber rovings were pulled through an oven to remove moisture. After drying at

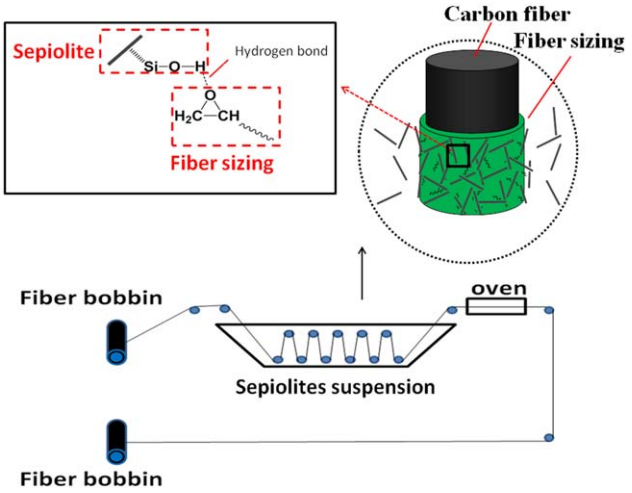


**Figure 1.** The fabrication process of the sepiolite-coated carbon fibers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

120 °C for 15 min, the sepiolite-coated fibers were stored in a constant temperature oven at temperature 80 °C for 6 h prior to use. The depositing amount of sepiolites on the surface of carbon fibers can be controlled by pulling speed of continuous carbon fiber rovings. Figure 1 shows the fabrication process of the sepiolite-coated carbon fibers. This route of depositing sepiolites did not damage the structure and properties of carbon fibers and the whole process was continuous and easy to implement.

### Preparation of Single-Fiber Composite Samples

The sepiolite reinforced epoxy samples were prepared by mixing the epoxy resin with curing agent in the weight ratio of 100 to 40 at room temperature for 2 h, degassing the mixture under vacuum conditions, and curing in a steel mold after a thermal cycle (150 °C/2 h + 200 °C/3 h). For preparation of single-fiber composite samples, the single carbon fibers were carefully separated from the deposited-sepiolite roving. Then, the single fiber was positioned in the center of a mold with a dog bone-shaped cavity. The two ends of the fiber extended over the mold and were stuck to a piece of plastic with 1.2 g, to hold the fiber straight in the sample. Subsequently, the epoxy resin was cast into the mold, covering the fiber completely. Curing temperature and procedure were subjected to the same thermal curing schedule as that of epoxy matrix in vacuum oven, avoiding any obvious voids in the sample. After the specimen had cooled, they were removed from the mold. The surfaces of the cured specimen were polished for facilitating the observation of the single fiber.

### Characterization

The microstructure of the sepiolites and the surface of sepiolite-coated carbon fibers were examined by using transmission electron microscope (TEM, JEM-3100, Japan) and scanning electron microscope (SEM, Zeiss Supra55, Germany), respectively. The content and distribution of Si in the sepiolites were determined by energy dispersive spectroscopy (EDS, Zeiss Supra55, Germany). The mechanical properties of epoxy resin with different content of sepiolites were characterized by three-point bending tests using universal testing machine (Instron1211, UK).

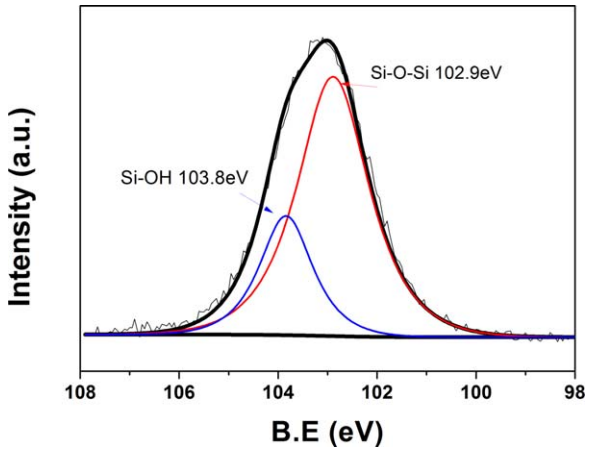


**Figure 2.** X-ray photoelectron spectroscopy (XPS) deconvolution spectra of Si 2p peak for sepiolites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The specimen was fixed to a micro-tension apparatus equipped with a 1 kN capacity load cell. The measurement under a tensile load was taken at a crosshead speed of 0.76 μm/min. During the testing, entire single fiber fragmentation was monitored by polarized light microscope and the number of fiber fragment was counted within the gauge length of 20 mm. After the saturation point was reached, i.e., the number of fiber fragment stopped increasing, the state of interfacial bonding was evaluated.

By assuming the shear stress, interface shear strength ($\tau_{IFSS}$), can be estimated by the follow equations:

$$\tau_{IFSS} = \sigma_f d_{f/}(2l_c) \quad (1)$$

$$l_c = 4l/3 \quad (2)$$

where $d_f$ is the fiber diameter and $\sigma_f$ is the fiber strength at the critical fragment length ($l_c$), which can be obtained from the mean fiber fragment length (l) at crack saturation; $\sigma_f$ can be calculated based on the following eq. (3):

$$\sigma_f = \sigma_o(l_o/l_c)^{1/\beta} \quad (3)$$

where $l_o$ is initial length of the single carbon fiber (25 mm in this study); $\sigma_o$ is the fiber tensile stress; and β is Weibull shape parameter. Therefore, for two fibers with equal strength, strength distribution, and diameter, the fiber with the shorter critical length will present the higher IFSS.

## RESULTS AND DISCUSSION

We delivered sepiolites to the surface of carbon fiber by immersing the fiber bundles in sepiolite-containing aqueous suspension, which takes advantages of the sizing[27] and spraying methods.[19,20] Like the sizing and the spraying methods, our route of depositing sepiolites is undamaged for fiber and the process is not complex. Moreover, this method does not need to remove industrial sizing and the deposition process can be used for various types of carbon fiber reinforcements, such as yarn, fabric, and preform.

### Surface Characteristics of Sepiolite-Deposited Carbon Fibers

The deconvolutions of the Si 2p peaks for sepiolites are shown in Figure 2. Two peaks are found for Si 2p sepiolites, attributed to Si–OH bonds and Si–O–Si groups. It can be seen that there are some active groups including Si–OH on the sepiolites.

The dispersion of sepiolites in the form of needle-like fibers has been achieved by ultrasonic processing without any chemical treatment. From Figure 3(a), it can be seen that the sepiolites have the lengths between 1 and 5 μm with the diameters about

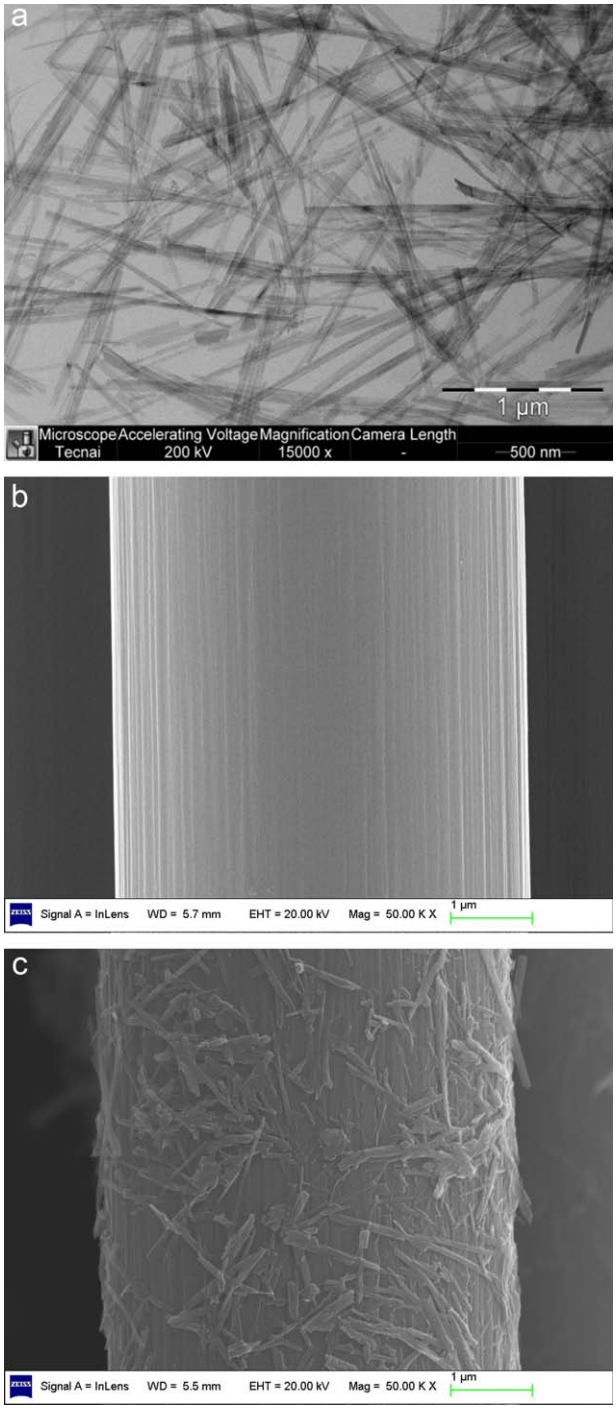


**Figure 3.** TEM image of the sepiolites (a) and SEM images of as-received JH-T800 carbon fiber (b) and sepiolite-deposited JH-T800 carbon fiber (c). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

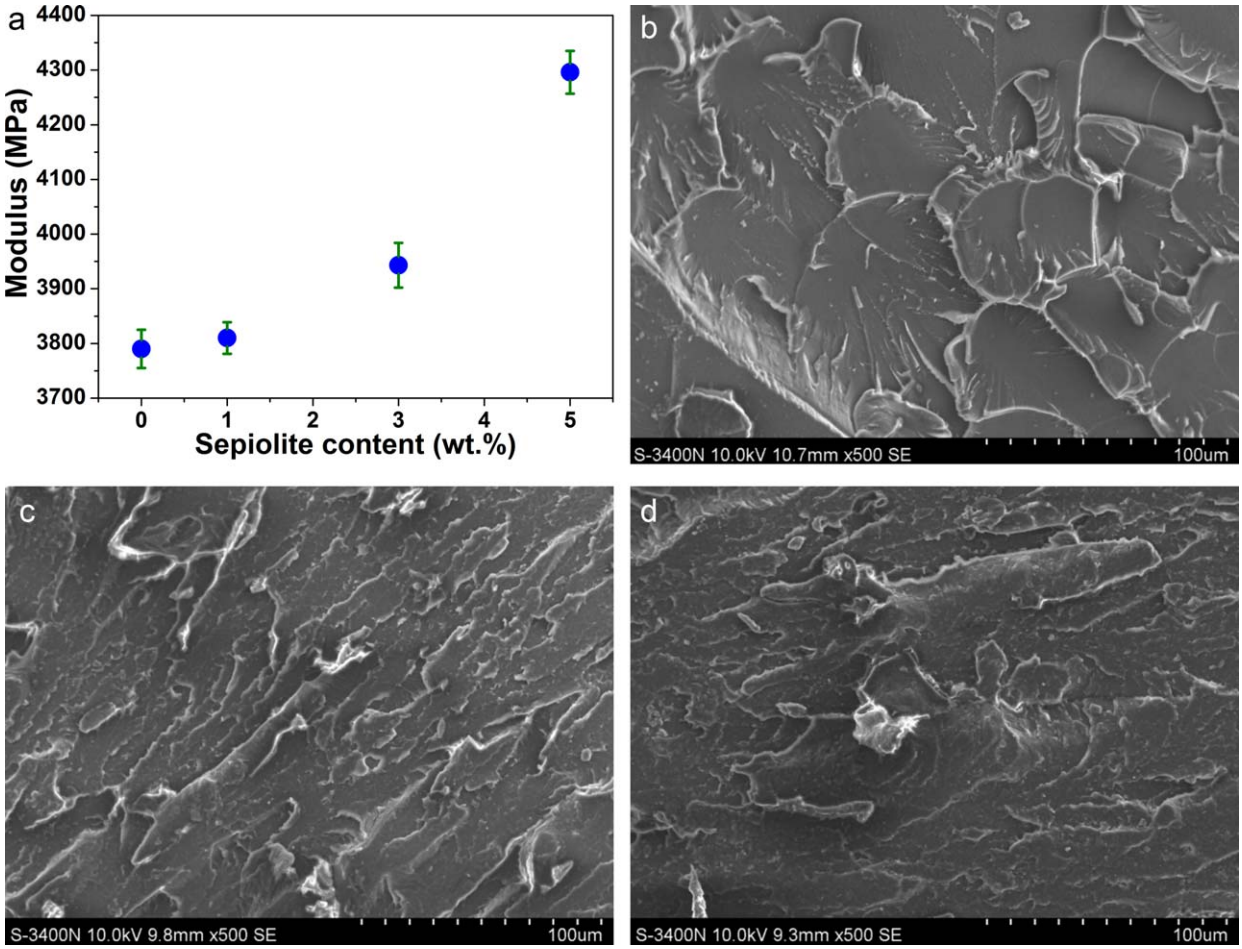


**Figure 4.** The flexural modulus of sepiolite reinforced epoxy resin (a) and SEM images of fractured surface from sepiolite reinforced epoxy resins: (b) pure epoxy matrix; (c) 1 wt % sepiolites; (d) 3 wt % sepiolites; and (e) 5 wt % sepiolites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

20–100 nm. The ultrasonic treatment can efficiently break down the silicate aggregation and separate small fibrous bundles from one another. Almost-isolated state of sepiolites is in favor of homogeneous deposition on the surface of individual carbon fibers.

The surface morphologies of carbon fibers before and after sepiolite deposition process are shown in Figure 3(b,c). The raw JH-T800 carbon fiber shows a smooth surface, with some longitudinal grooves [Figure 3(b)]. The typical SEM image of the sepiolite-deposited carbon fiber is shown in Figure 3(c), which identifies the presence of the sepiolite on the surface of carbon fiber with random orientation and uniform distribution.

### The Modulus Improvement of Sepiolite Reinforced Epoxy Matrix

Figure 4(a) shows the flexural modulus of sepiolite-filled epoxy resin. The stiffness of the sepiolite is much higher than that of the epoxy matrix. In consequence, an increase in flexural modulus of epoxy matrix was realized with raising sepiolite content. It can be seen that the flexural modulus of epoxy matrix were increased with the addition of the sepiolites from 1 to 5 wt %.

Figure 4(b∼e) shows the fracture surface of epoxy resins. The large-scale smooth regions can be observed in the fracture surface of pure epoxy matrix, indicating a brittle fracture mode of crack propagation. Micro-size roughness, as shown in the fracture surfaces of sepiolite reinforced epoxy resins, increases the absolute fracture surfaces which can dissipate more external energy and provide the high resistance to fracture.

### Effect of Fiber Sizing on Sepiolite-Deposition State

Since the sizing of the JH-T800 is epoxy type and sepiolite might react with epoxy, it is supposed that OH–sepiolite might react with the active groups in the sizings. To confirm this hypothesis, FTIR of the sizing and the mixture of the sepiolite and the sizing were determined, as presented in Figure 5. For the sizing without sepiolite [Figure 5(a)], the epoxy groups (around 905 $cm^{-1}$) can be found and the curves of FTIR change little after 150 °C treatment. For the sizing containing sepiolite [Figure 5(b)], the epoxy groups decrease after 150 °C treatment, indicating chemical bonding between the epoxy in the sizing and hydroxyl groups on the sepiolite. The covalent bonding between the sepiolite and the sizing can provide strong adhering force of the sepiolite on fiber surface and can reduce the amount of sepiolite dispersing in matrix, which drop from

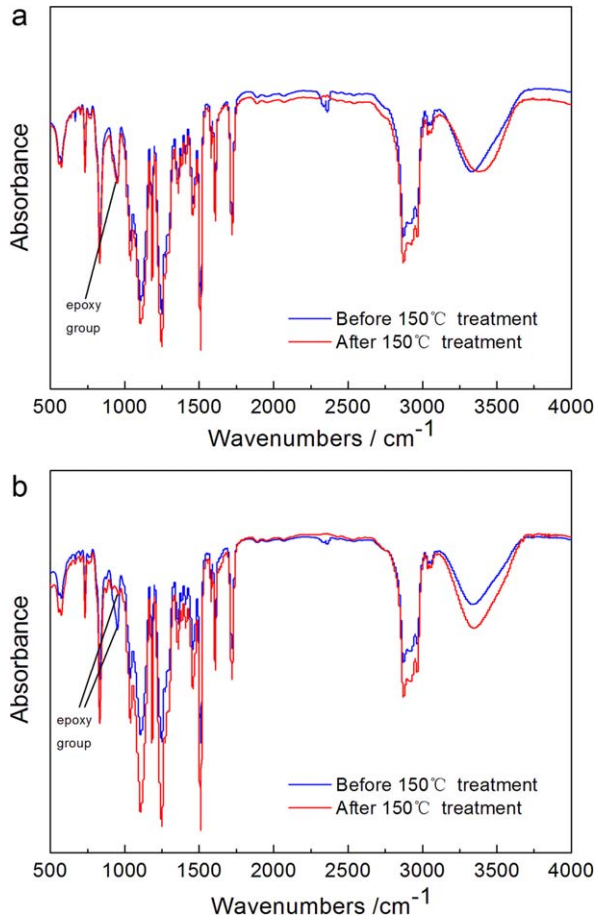


**Figure 5.** FTIR spectrum of (a) sizing of JH-T800, (b) the mixture of the JH-T800 sizing and sepiolites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

fiber during fabrication of the composite. Consequently, the compatibility of the deposited-sepiolite with the properties of the fiber sizing should be regarded as a critical factor for preparing the multiscale reinforcement.

**Interfacial Bonding of Single-Fiber Composite**

The IFSS of the carbon fiber/epoxy composites with and without deposited sepiolite were tested, and the measured data are given in Figure 6. As shown in Figure 6(a), the deposition of the sepiolites gives rise to a significant increase of the IFSS for the JH-T800 carbon fiber composites. At the same time, the composite specimens with the sepiolite-deposited JH-T800 fibers display shorter critical fragment lengths than those with the raw carbon fibers [Figure 6(b)]. This phenomenon must result from the sepiolite incorporation into the interphase, improving the modulus and strength of interphase and resulting in improved load transfer between carbon fiber and epoxy resin matrix. The longer critical fragment is an indication of interfacial debonding, which is associated with low fiber–matrix adhesion. The shorter critical fragment results from high shear stresses. This is a consequence of a strong interface. These results are consistent with those of the IFSS measurements, demonstrating that the sepiolite-deposition process significantly improved carbon fiber/epoxy interfacial adhesion.

These results benefit from the deposited sepiolites which can diffuse into the interphase, increase the modulus and strength of interphase layer and improve load transfer between carbon fibers and epoxy resin matrix.

Figure 6(c,d) displays the photoelastic effects of the single fiber fragments in composites. The results of birefringence at fragment regions are consistent with the measurements of IFSS and the critical fragment length. Photoelastic observations of the raw fibers near the fiber breaks show a thin, flat region of

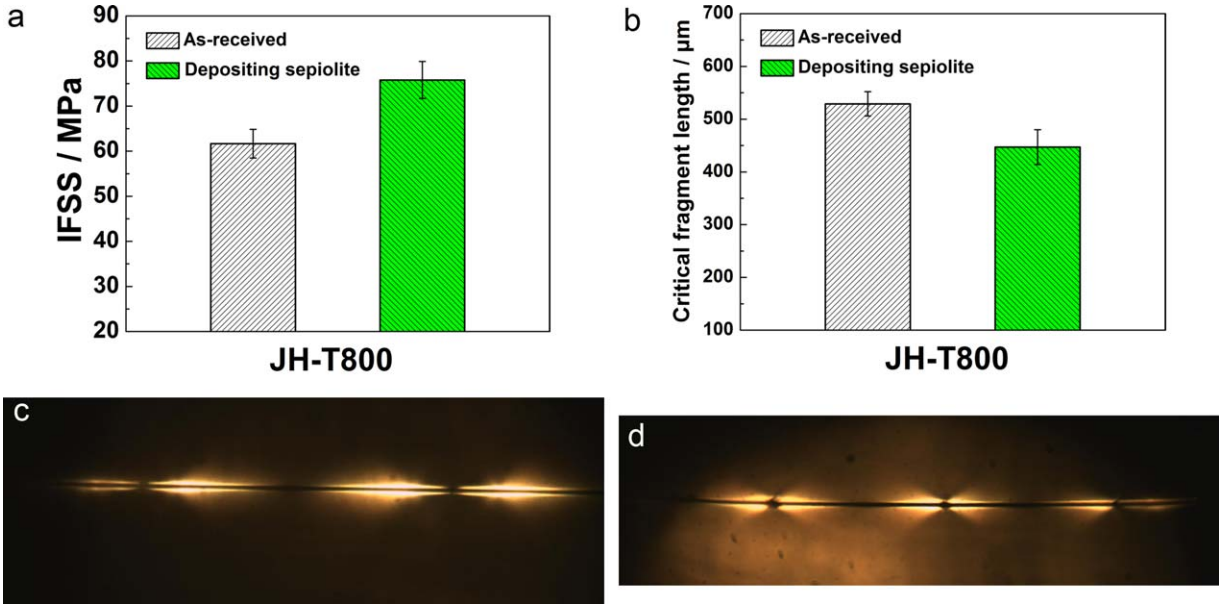


**Figure 6.** The effects of the deposited sepiolites on the interfacial properties of the single fiber composites: (a) interfacial shear strength; (b) critical fragment length; the birefringence effects of fragmented specimens at 4.0% tensile strain: (c) raw JH-T800, and (d) JH-T800 with deposited sepiolites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

birefringence followed by a bulge of birefringence [Figure 6(c)]. In Figure 6(d), the sepiolite-deposited fiber breakages create obvious matrix cracks between adjacent fragment ends, which are perpendicular to the fiber and produced bright ellipsoidal regions because of high shear stresses at the fiber ends. This is a consequence of a strong interface bonding.

Interlaminar shear strength (ILSS) and flexural strength are important mechanical properties for composite. To better understand the relationship between the sepiolites surrounding the fiber and the mechanical properties, three-point short beam shear method and bending test were used to evaluate the interlaminar shear strength and flexural strength of the composites. In Figure 7, the ILSS and flexural strength values of the composites are presented. It is readily observed that both the ILSS and flexural strength of the sepiolites decorated fiber composites are higher compared with that of the commercial fiber composites. These results are probably due to the homogenously dispersed sepiolites in the interfacial region of the composite, which can serve as a supplementary reinforcement to the interface and further reduce the interlaminar stress concentration, enhance the strength and toughness of interfacial regions surrounding the fiber, and then finally result in improving the interlaminar shear strength and flexural strength.

**intermediate Layer in Carbon Fiber/Epoxy Composites**

Figure 8(a) shows the microstructure of the sepiolite-deposited JH-T800 carbon fiber/epoxy composites perpendicular to fiber direction. It can be seen that the sepiolite-reinforced epoxy intermediate layer encompassing the carbon fiber has been formed. Because there is abundant silanol-based chemical bond on the surface of sepiolites, the EDS analysis is carried out to understand the distribution of sepiolites. Figure 8(c) is the results of EDS analysis for Si element distribution corresponding to the area in Figure 8(b). The enrichment region of the Si element is observed around the carbon fiber, indicating the existence of sepiolite-reinforced epoxy intermediate layer.

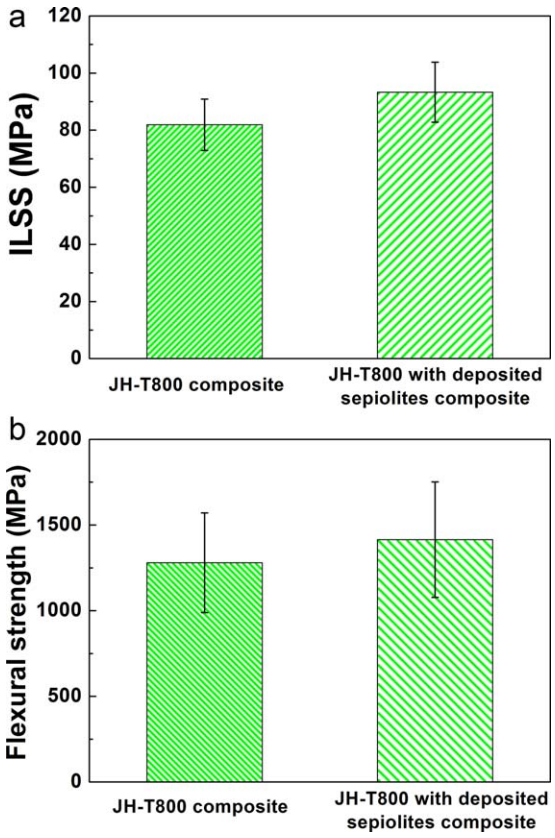


**Figure 7.** Interlaminar shear strength (ILSS) and flexural strength of unidirectional carbon fiber/epoxy composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

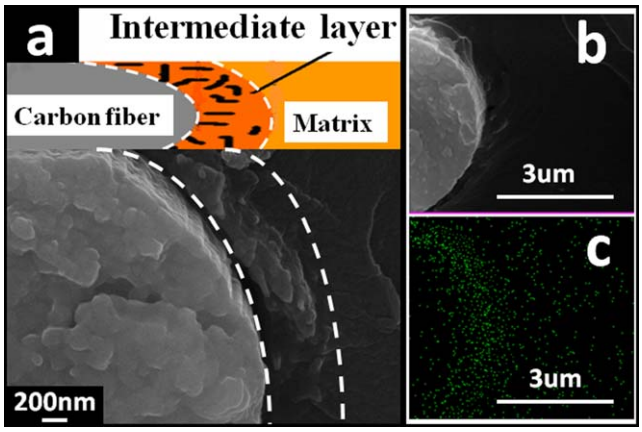


**Figure 8.** SEM images of sepiolite reinforced intermediate layer (a) and the EDS mapping of silicon distribution (b, c) in carbon fiber/epoxy composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

## CONCLUSIONS

A facile effective process was used to uniformly deposit commercially available sepiolites on the surface of sized JH-T800 carbon fibers for the first time. This method is undamaged for fiber and the process is not complex. Moreover, this method does not need to remove industrial sizing and the deposition process can be used for various types of carbon fiber reinforcements, such as yarn, fabric, and preform. The sepiolites on the surface of carbon fiber can participate in the formation of high modulus intermediate layer encompassing the carbon fiber. According to the single fiber-composite fragmentation tests, the deposition of sepiolites resulted in the improved carbon fiber/epoxy matrix interfacial bonding. The sepiolite-deposited carbon fiber composites also exhibited a tendency to higher values after treatment of the fibers in the interlaminar shear strength and flexural strength compared to the commercial carbon fiber reinforced samples. The deposition of commercial sepiolites is a promising industrial approach to produce multi-scale reinforcement which can further improve mechanical properties of carbon fiber reinforced composites.

## ACKNOWLEDGMENTS

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