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Cite this article as:

Chen Bin, Ni Bao-Jia, Fu Meng-Xiang, Zhong Hang, Jiang Wei-Feng, Liu Si-Yuan, Zhang He-Xin, Yoon Keun-Byoung. Effect of Molybdenum Disulfide Exfoliation Conditions on the Mechanical Properties of Epoxy Nanocomposites[J]. *Chinese J. Polym. Sci.*, 2019, 37(7): 687-692. doi: 10.1007/s10118-019-2239-7

View online: <https://doi.org/10.1007/s10118-019-2239-7>

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Effect of Molybdenum Disulfide Exfoliation Conditions on the Mechanical Properties of Epoxy Nanocomposites

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Abstract In this work, the MoS₂ fillers were prepared through chemical exfoliation method and used as fillers to fabricate epoxy (EP)/MoS₂ nanocomposites. The effects of molybdenum disulfide (MoS₂) intercalation conditions on the properties of EP/MoS₂ nanocomposites were investigated. As the intercalation time was prolonged, the surface of MoS₂ exhibited a totally crumpled structure and more functional groups formed. Because of the higher functional group concentration, the interfacial adhesion force between EP and MoS₂ was enhanced. With the addition of 1.0 wt% exfoliated MoS₂ fillers, the tensile strength and tensile modulus of EP were even improved ~500% and ~6800%, respectively. Therefore, this work provides a facile way to produce high-performance EP nanocomposites.

Keywords Epoxy; Molybdenum disulfide; Nanocomposite; Mechanical properties

Citation: Chen, B.; Ni, B. J.; Fu, M. X.; Zhong, H.; Jiang, W. F.; Liu, S. Y.; Zhang, H. X.; Yoon, K. B. Effect of molybdenum disulfide exfoliation conditions on the mechanical properties of epoxy nanocomposites. *Chinese J. Polym. Sci.* 2019, 37, 687–692.

INTRODUCTION

Nanocomposites based on MoS₂ have received a tremendous amount of attention during the past few years.^[1–5] Much of the excitement is due to the interesting set of properties that MoS₂ can exhibit, including extraordinarily high breaking strength (~23 GPa) and Young's modulus (~300 GPa), which are greater than those of chemically reduced graphene oxide.^[6,7] Derived from its excellent mechanical properties, the MoS₂ fillers may hold considerable potential as polymer reinforcement nanofiller.

Thermoset polymer comprises of a broad range of polymers with a large number of industrially relevant applications, including coatings, encapsulants, and matrices for fiber reinforced composites.^[8–13] Among the thermoset polymers, EP resin is one of the most important materials.^[14–18] Thus, the idea of combining MoS₂ with thermosets is very appealing from several perspectives. A few recent studies have reported the dispersion of MoS₂ in thermoset and their resulting physical and mechanical properties. In general, the mechanical and physical properties were found to depend on the dispersion degree of MoS₂.^[19–23] Control of the MoS₂ dispersion can be facilitated by manipulating the polymer-MoS₂

interface.^[24,25] Hu and Gui *et al.*^[26] reported a MoS₂-carbon nanotube reinforced EP composite. With the introduction of 2 wt% MoS₂-carbon nanotube, the organic volatiles and carbon monoxide were suppressed, while the mechanical properties were improved. Koratkar *et al.*^[27] also found that the addition of exfoliated MoS₂ could enhance the mechanical properties of EP resin. In our previous report, a melamine-functionalized molybdenum disulfide (M-MoS₂) was prepared and used as fillers to fabricate epoxy (EP)/MoS₂ nanocomposites.^[28] Upon the mere addition of 0.8 wt% M-MoS₂, the tensile strength and modulus of EP/M-MoS₂ nanocomposites showed 4.5 and 4.0 times increase over the neat epoxy. During the research, we found that the mechanical properties of EP/MoS₂ nanocomposites were severely affected by the intercalation conditions of MoS₂. Therefore, in this research, the effects of MoS₂ intercalation time on the properties of EP/MoS₂ nanocomposites were investigated and discussed.

EXPERIMENTAL

Materials

Molybdenum disulfide (MoS₂, ~6 μm), *n*-butyllithium (2.5 mol·L⁻¹ in hexane), polypropyleneglycol diglycidyl ether (PPGDGE, *M_n* ≈ 640) and polyoxypropylene diamine (D230, *M_n* ≈ 640) were purchased from Sigma-Aldrich and used as received. EP resin (EPON 828) was obtained from Shell.

Preparation of Organophilic MoS₂

MoS₂ was first exfoliated according to a literature method.^[29]

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Received December 25, 2018; Accepted February 12, 2019; Published online March 26, 2019

G-MoS₂: 5 g of MoS₂ was placed in an autoclave, and 20 mL of *n*-butyllithium (2.5 mol·L⁻¹ in hexanes) was added. The autoclave was heated at 90 °C for 12 h under stirring. After that, the product was filtered and washed with anhydrous hexane (5 × 100 mL). The resultant lithium intercalated MoS₂ was vacuum-dried and immersed in 1000 mL H₂O under ultrasonication for 4 h to produce a colloidal suspension of exfoliated MoS₂. The suspension was neutralized with 1 mol·L⁻¹ HCl, and the products were washed with distilled water (3 × 1 L) and then washed with methanol. Subsequently, G-MoS₂ was obtained by freeze-drying. **E-MoS₂:** E-MoS₂ was prepared in the same method as G-MoS₂ except that the intercalation time was extended from 12 h to 24 h.

Preparation of EP/MoS₂ Nanocomposites

The desired amount of MoS₂, G-MoS₂, or E-MoS₂ was dispersed in appropriate amount of acetone and then stirred with a magnetic stirrer at room temperature for 30 min, followed by sonication for 60 min. After that the mixture was mixed with desired amount of EP (*m*(EPON828)/*m*(PPGDGE) = 60/40) and sonicated for another 60 min at 50 °C. Acetone was then removed by vacuum distillation under stirring with a magnetic stirrer at 80 °C. When the mixture was cooled down to 50 °C, a stoichiometric amount of curing agent (D230) corresponding to 100% of EP resin content was added and stirred for some time. The resulting mixture was then outgassed in a vacuum oven at 60 °C for a short period of time and then cast into a Teflon mold with special size. The sample was cured at 75 °C for 2 h and post-cured at 120 °C for 8 h.

Characterization of EP/MoS₂ Nanocomposites

X-ray diffraction (XRD) patterns were obtained on a Germany BRUKER D8 ADVANCE diffractometer with Cu K α X-ray radiation. The scanning range was 2°–50° with a scanning speed of 1 (°)·min⁻¹. The specimens for transmission electron microscopy were microtomed with a diamond knife using a Leica Ultramicrotome at liquid N₂ atmosphere. TEM images were obtained from a Japan JEOL JEM-1011 microscope operated at 200 kV in bright field mode. Differential scanning calorimetry (DSC) was conducted on Perkin-Elmer Diamond thermal analyzer. The samples were first heated from room temperature to 50 °C under N₂ atmosphere at a heating rate of 10 °C·min⁻¹, then cooled to -50 °C, and finally reheated to 50 °C at 10 °C·min⁻¹. Tensile properties were performed on a USA INSTRON 5869 electronic testing instrument according to ASTM D638 at a cross-head speed of 5 mm·min⁻¹ at room temperature. The dumbbell-like specimens (20 mm × 4 mm × 2 mm) were cut from the above cured sample casted on Teflon mold. Each tensile value reported is the average of 5 tests. Optical microscope photos were obtained from an optical microscope (ANA-006, Leitz, Germany) and recorded using a charge-coupled device camera.

RESULTS AND DISCUSSION

MoS₂ was exfoliated according to our previous research. To confirm the successful exfoliation of MoS₂, SEM and XRD

analyses were conducted. Fig. 1(a) shows the morphology of MoS₂ before exfoliation. The MoS₂ fillers clearly contained a large number of stacked MoS₂ sheets with a relatively smooth surface. As shown in Figs. 1(b) and 1(c), after exfoliation, the sheet structure of MoS₂ remained and the stacked MoS₂ layers were exfoliated. With regards to 12 h intercalation time, the surface of MoS₂ slightly wrinkled. However, when the intercalation time extended to 24 h, the surface of MoS₂ exhibited a totally crumpled structure. This phenomenon could correspond to the exfoliated MoS₂ layers in suspension being hydroxylated, as indicated by their negative charge.^[30] Thus, prolonging the reaction time led to more functional groups formed.

Fig. 2 presents the XRD patterns for MoS₂, G-MoS₂, and E-MoS₂. The commercial MoS₂ showed a sharp diffraction peak (002) at 2 θ = 14.3°, corresponding to a *d*-spacing of 0.6 nm. After exfoliation, the peak at 14.3° almost disap-

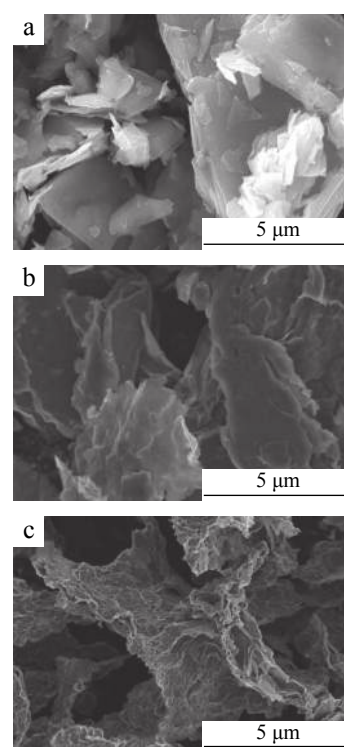


Fig. 1 SEM images of (a) MoS₂, (b) G-MoS₂, and (c) E-MoS₂

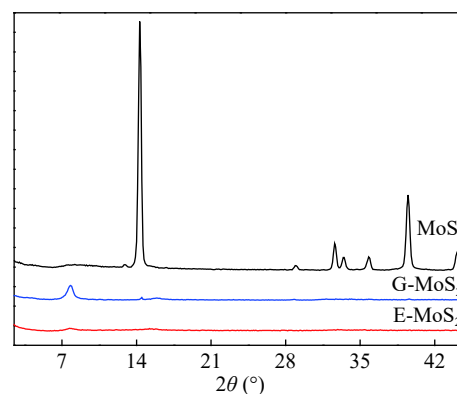


Fig. 2 XRD patterns of (a) MoS₂, (b) G-MoS₂, and (c) E-MoS₂

peared. With regards to 12 h intercalation time, a very weak diffraction peak is observed at $2\theta = 8.2^\circ$ (corresponding to d -spacings of 1.0 nm), which indicates the existence of a small number of loosely stacked MoS_2 layers. When the intercalation time extended to 24 h, almost no diffraction peaks could be found on the XRD pattern. The weak diffraction peak also indicates the crystallinity of G- MoS_2 and E- MoS_2 were very low.

Fig. 3 shows the FTIR spectra of MoS_2 , G- MoS_2 , and E- MoS_2 . No characteristic peaks appear in the spectrum of un-exfoliated MoS_2 , whereas several sharp peaks are observed for the G- MoS_2 and E- MoS_2 samples. The FTIR spectrum of E- MoS_2 is very similar to that of G- MoS_2 . The peak at 1632 cm^{-1} due to the adsorbed water and O—H stretching broad band at $3000\text{--}3700\text{ cm}^{-1}$ can be observed.^[31,32]

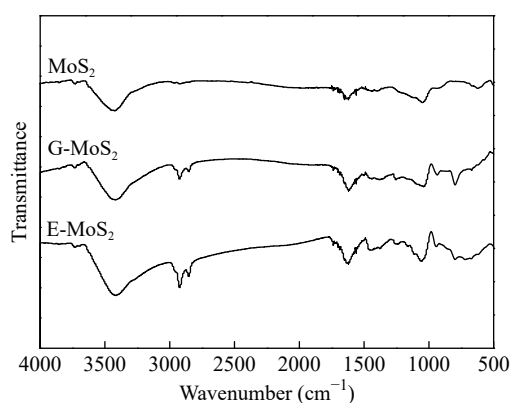


Fig. 3 FTIR spectra of (a) MoS_2 , (b) G- MoS_2 , and (c) E- MoS_2

The synthetic procedure used for EP/ MoS_2 nanocomposites preparation consisted of two stages: the first includes the dispersion of MoS_2 fillers in the epoxy solution, and the second stage includes the curing of MoS_2 -modified epoxy resin. The dispersion of G- MoS_2 and E- MoS_2 in the EP matrix was investigated by optical microscopy and TEM (Figs. 4 and 5), which suggested that the G- MoS_2 and E- MoS_2 fillers were well dispersed in the EP matrix. The resulting EP/G- MoS_2 and EP/E- MoS_2 composites contained two phases. The EP region was the continuous phase and the discontinuous phase was the G- MoS_2 or E- MoS_2 fillers.

The thermal properties of EP, EP/ MoS_2 , EP/G- MoS_2 , and EP/E- MoS_2 were characterized by DSC. As shown in Fig. 6, the glass transition temperature (T_g) of the neat EP was 9.0°C . After introduction of 1.0 wt% G- MoS_2 and E- MoS_2 fillers, T_g of the EP composites rose up to 18.5 and 22.3°C , respectively. The increment of T_g could be attributed to the reduction of EP matrix chain mobility in the presence of G- MoS_2 and E- MoS_2 . During the polymerization process, the functional groups of exfoliated MoS_2 (G- MoS_2 and E- MoS_2) could react with the epoxy group of EP and a strong interface was thus produced. The strong interface restricted the motion of EP chains and thus gave rise to the increase in T_g . In order to prove the existence of reaction between functional groups and EP, EP with 1.0 wt% un-exfoliated MoS_2 nanocomposite was prepared following the same method. It was found that T_g of the EP composite obtained was slightly

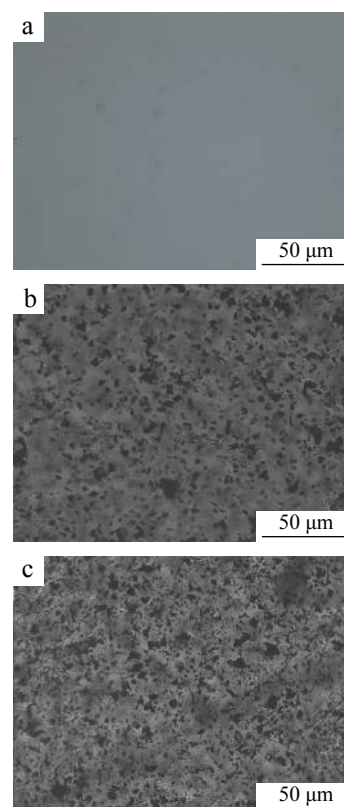


Fig. 4 Optical micrographs of (a) EP, (b) EP/1.0 wt% G- MoS_2 , and (c) EP/1.0 wt% E- MoS_2 nanocomposites

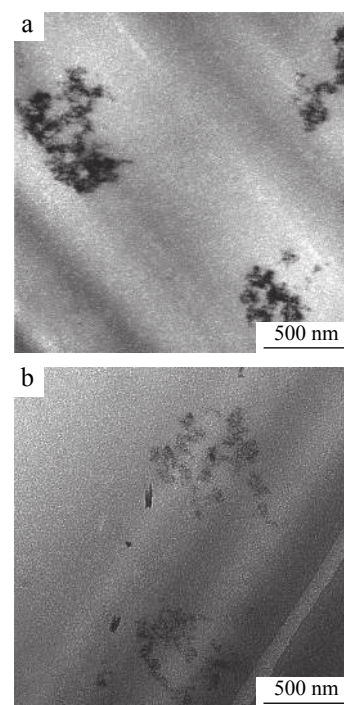


Fig. 5 TEM images of (a) EP/1.0 wt% G- MoS_2 and (b) EP/1.0 wt% E- MoS_2 nanocomposites

reduced with the introduction of MoS_2 . This was probably caused by two reasons: (i) agglomeration when neat MoS_2 fillers added, and (ii) reduction of the EP matrix's crosslinking density due to the barrier effect of MoS_2 .

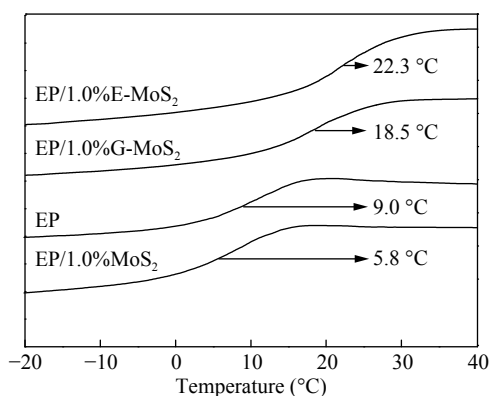


Fig. 6 DSC curves of EP as well as EP with 1.0 wt% MoS₂, G-MoS₂, and E-MoS₂ nanocomposites

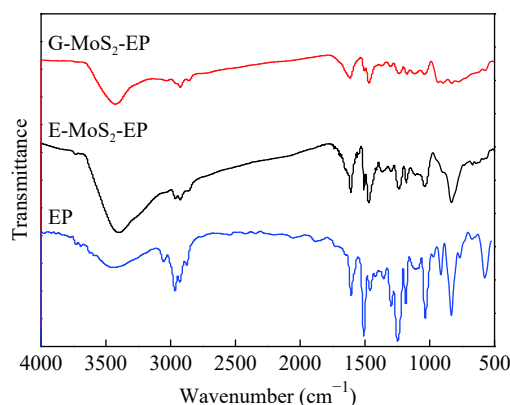


Fig. 7 FTIR spectra of EP, G-MoS₂-EP, and E-MoS₂-EP

In order to further prove the existence of reaction between functional groups and EP, the exfoliated MoS₂ and EP were reacted at 120 $^{\circ}\text{C}$ with xylene as the solvent. After reaction, EP was removed through soxhlet with xylene. The resultant G-MoS₂ and E-MoS₂ were characterized by FTIR. As shown in Fig. 7, the G-MoS₂-EP and E-MoS₂-EP samples displayed the characteristic peaks of EP. Therefore, it could be well proved that there was reaction between the functional groups of exfoliated MoS₂ and EP. Compared to that of G-MoS₂-EP, the characteristic peaks of EP in E-MoS₂-EP sample were more intense. This could be due to the more functional groups formed during the longer intercalation time. The peaks appeared at around 800 cm^{-1} due to symmetric C—O—C (epoxy group) stretch. G-MoS₂ had more

polar groups to initiate ring-opening reaction of epoxy groups, which resulted in the disappearance of the characteristic peaks of epoxy group ($\sim 800\text{ cm}^{-1}$) and the enhancement of O—H stretching peaks at 3400 cm^{-1} . Thus, we believe that the interfacial adhesion force between EP and E-MoS₂ could be higher than that between EP and G-MoS₂.

The influence of G-MoS₂ and E-MoS₂ on the mechanical properties of the EP nanocomposite was studied by using a universal testing machine (UTM) and the results are summarized in Fig. 8 and Table 1. Clearly, the tensile strength and modulus of EP were significantly enhanced with the introduction of exfoliated MoS₂ fillers, even at very low content. The tensile modulus of EP/G-MoS₂ nanocomposites increased from 2.5 MPa to 29.1 MPa (approximately a 1000% increase over neat EP), and the tensile strength increased

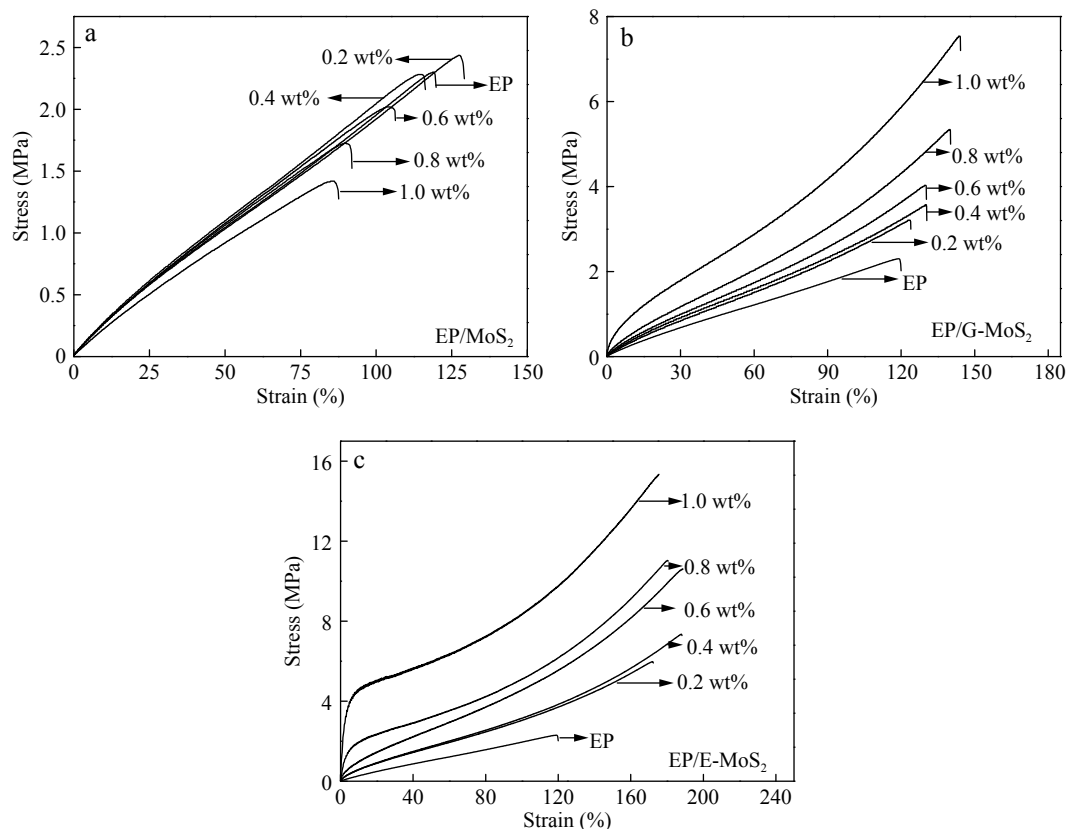
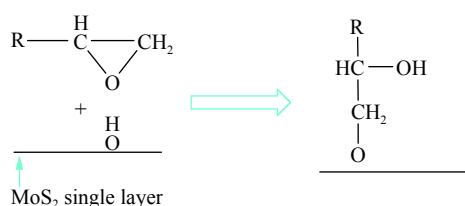


Fig. 8 Strain-stress curves of (a) EP/MoS₂, (b) EP/G-MoS₂, and (c) EP/E-MoS₂ nanocomposites

Table 1 Mechanical properties of EP, EP/G-MoS₂, and EP/E-MoS₂ nanocomposites

	Filler content (wt%)	Tensile strength (MPa)	Tensile modulus (MPa)	Elongation at break (%)
EP	0	2.3 ± 0.1	2.5 ± 0.1	123.3 ± 5.8
EP/G-MoS ₂	0.2	3.2 ± 0.2	3.2 ± 0.2	124.6 ± 5.1
	0.4	3.4 ± 0.2	3.8 ± 0.5	125.0 ± 7.1
	0.6	4.1 ± 0.3	5.1 ± 1.1	126.0 ± 5.5
	0.8	5.1 ± 0.2	7.0 ± 0.4	136.7 ± 5.8
	1.0	7.3 ± 1.0	29.1 ± 15.4	136.0 ± 11.4
EP/E-MoS ₂	0.2	6.5 ± 0.7	14.4 ± 5.0	176.0 ± 11.4
	0.4	6.8 ± 0.6	16.8 ± 4.7	180.0 ± 10.0
	0.6	9.1 ± 1.3	19.8 ± 1.6	186.7 ± 5.8
	0.8	10.9 ± 1.2	77.8 ± 41.1	183.3 ± 25.1
	1.0	14.9 ± 0.7	174.8 ± 19.4	178.0 ± 4.5

from 2.3 MPa to 7.3 MPa (approximately a 300% increase over neat EP) when 1.0 wt% G-MoS₂ was loaded. Incorporation of the same amount of E-MoS₂ fillers could even improve the tensile strength and tensile modulus of EP by ~500% and ~6800%, respectively. The dramatic reinforcement in mechanical property is surely due to the presence of more functional groups that led to stronger interfacial adhesion force between EP and E-MoS₂ fillers (Scheme 1). Moreover, elongation at break was also increased by ~40%. Therefore, prolonging the intercalation time of MoS₂ can greatly improve the efficiency of exfoliated MoS₂ for enhancing EP mechanical properties.

**Scheme 1** Reaction between EP and E-MoS₂

CONCLUSIONS

In summary, the MoS₂ fillers were successfully exfoliated through chemical exfoliation method. As the intercalation time extended, the surface of exfoliated MoS₂ gradually changed from smooth to crumpled. The crumpled surface could be caused by the more functional groups formed during the longer time intercalation. Because of the higher functional group concentration, the interfacial adhesion force between EP and MoS₂ was enhanced. Therefore, prolonging the intercalation time of MoS₂ can greatly improve the efficiency of exfoliated MoS₂ for enhancing EP mechanical properties.

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