Growth of MoS$_2$ Nanotubes Templated by Halloysite Nanotubes for the Reduction of Friction in Oil

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ABSTRACT: One-dimensional MoS$_2$ nanotubes with the specific surface area of 89.34 m$^2$/g and the average pore size of 2.52 nm were successfully synthesized by the thermolysit approach assisted by halloysite nanotubes. The tribological properties of MoS$_2$ nanotubes with good dispersion in oil were tested with a four-ball wear tester. The tribological testing results indicated that the average friction coefficient and the average wear scar diameter of the 0.08 wt % MoS$_2$-based oil at 25 °C decreased about 39.2 and 35.0%, respectively, compared to those of the 150 SN base oil, indicating that the as-prepared MoS$_2$ nanotubes as a lubricating additive can enhance the tribological performances. Finally, the lubrication mechanism of MoS$_2$ nanotubes was put forward.

1. INTRODUCTION

One-dimensional nanostructured materials, including nanowires, nanorods, nanobelts, and nanotubes, have attracted great interest in research because of the discovery of graphitic carbon nanotubes. The MoS$_2$, with a layered structure can be rolled up to form nanotubes because of the stacked S-Mo-S monolayers. MoS$_2$ nanotubes are an outstanding lubricating additive because of the excellent lubrication performances. Maharaj et al. investigated the influences of MoS$_2$ and WS$_2$ nanotubes on friction and wear decrease on the nanoscale in a dry and low-viscosity liquid environment. The results indicated that MoS$_2$ and WS$_2$ nanotubes could contribute to enhance lubrication performance because of the reduction of contact surface and the exfoliation of outer layers. Jelenc et al. measured the friction on a single MoS$_2$ nanotube. The results showed that the average friction value of a MoS$_2$ nanotube in ultrahigh vacuum was lower than that in air. Remskar et al. added the MoS$_2$ nanotubes to poly(vinylidene fluoride) to adjust the friction properties. The testing results showed that the friction coefficient (COF) value had a more substantial decline. Kogovšek et al. discussed the influences of the roughness of contact surface on the tribological performances of MoS$_2$ nanotubes in polyalphaolefin (PAO) oil. It was found that the friction of the oil with MoS$_2$ nanotubes was 40–65% less than that of the base oil owing to the different contact conditions used. However, the dispersion ability of MoS$_2$ nanotubes in PAO oil was unknown in this study. As is well known, the dispersibility of nanomaterials in oil has a key influence on their friction performance, directly determining the tribological improvement of oil. Compared to other nanostructures, nanotube structure has a natural advantage in dispersion because of its hollow structure being conductive to suspension in a liquid; thus, it is one of the best candidates for nanostructure. Therefore, it is valuable to prepare a type of the nanostructured MoS$_2$ nanotube with a specific structure, which has a good dispersion in the base oil.

So far, many synthetic methods have been explored to fabricate MoS$_2$ nanotubes, for example, solvothermal, hydrothermal, high-temperature synthesis, and thermal evaporation methods. Numerous studies have shown that MoS$_2$ nanotubes have super properties in the application of nanoscale sensors, optoelectronics, electrochemistry, catalysts, and lubrication.

Halloysite nanotubes (HNTs), as a natural aluminoisilicate (Al$_2$Si$_2$O$_5$(OH)$_4$·nH$_2$O), are hollow nanotubes with high aspect ratios in the submicron range. Usually, HNTs have a length of 50–1500 nm, an inner diameter of 10–30 nm, and an outer diameter of 40–100 nm. HNTs have been a promising candidate template for nanomaterials. Zhang et al. successfully fabricated the polyvinyl alcohol nanotubes with a diameter of 40–60 nm using HNTs as templates. Abdullah et al. prepared silver nanorods in the HNTs’ cavity by thermal decomposition, which was absorbed from an aqueous solution into HNTs by vacuum circulation. Our research group ever successfully constructed the carbon nanotubes through the PVA modification of the inner surface in a hollow nanotube of HNTs as a template.

In this article, the MoS$_2$ nanotubes were successfully synthesized using HNTs as a template via a thermolysit method. Many characterizations were done to investigate the morphology of the as-prepared MoS$_2$ nanotubes. The formation process of the MoS$_2$ nanotubes was put forward. Finally, the tribological performances of the MoS$_2$ nanotubes in oil were completely investigated and the lubrication mechanism was put forward.

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Figure 1. (a) XRD pattern, (b) Raman spectrum, (c) SEM, and (d) TEM and SAED images (inset) of the MoS$_2$ nanotubes.

Figure 2. (a) Thermogravimetric analysis (TGA), (b) nitrogen physisorption isotherms and pore diameter distribution, (c) Mo 3d XPS spectrum, and (d) S 2p XPS spectrum of the MoS$_2$ nanotubes.
Figure 3. UV–vis spectra, dispersion pictures, and corresponding absorption spectra at 235 nm (inset) of the 0.02 wt % MoS$_2$-based oil (base oil as the solvent) with the changes of time.

Figure 4. (a) Average COFs and AWSDs and (b) average oil temperatures of the MoS$_2$-based oil as a function of content at 25 and 75 °C. (c) COFs and (d) temperatures of the 150 SN base oil and the 0.08 wt % MoS$_2$-based oil as a function of testing time at 25 °C. (e) COFs and (f) temperatures of the 150 SN base oil and the 0.08 wt % MoS$_2$-based oil as a function of testing time at 75 °C.
2. RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction (XRD) pattern, Raman spectrum, scanning electron microscopy (SEM) image, transmission electron microscopy (TEM) image, and selected area electron diffraction (SAED) image (inset) of MoS2 nanotubes. In Figure 1a, the crystalline structure of the as-synthesized MoS2 nanotubes is analyzed by XRD and the four peaks of MoS2 correspond to the crystalline planes of the primitive MoS2 (JCPDS No. 37-1492). Particularly, the characteristic peaks at 2θ = 14.1, 32.9, 39.4, and 58.6° correspond to the (002), (100), (103), and (110) crystalline planes of MoS2 respectively. In Figure 1b, the double peaks of MoS2 at around 380 and 404 cm⁻¹ are found in the spectra of the as-synthesized MoS2 nanotubes within the information from 250 to 550 cm⁻¹, which individually are equivalent to E₂g¹ and A₁g modes. In addition, the frequency difference between E₂g¹ and A₁g peaks for the as-prepared MoS2 sample is 23 cm⁻¹, indicating that the layers of the MoS2 nanotube wall are few and the tube wall is ultrathin. Figure 1c,d shows that the appearance of the as-prepared MoS2 is the nanotubular structure. In the selected area electron diffraction image (the illustration of Figure 1d), four diffraction rings with lattice characteristics also correspond to the (002), (100), (103), and (110) planes, which is consistent with the results of XRD.

Figure 2a shows the thermal decomposition pattern of MoS2 nanotubes under the oxygen flow. The mass of MoS2 nanotubes after heating is calculated to be around 70.0% according to MoO3 mass, in line with the data changes of MoS2 to MoO3, after heating is calculated to be around 70.0% according to the content further increases to 0.1 wt %, the COF of the MoS2-based oil increases 39.2% compared to the 150 SN base oil. When the content increases from 0.02 to 0.08 wt %, the result indicates that the MoS2 nanotubes in oil could be grated into the furrow by the flowing oil and then peel off and settle in the ditch and become a layer of tribofilm. Distinctly, the base oil with 0.08 wt % MoS2 nanotubes at 25 °C shows lower COF value and the average COF decreases 39.2% compared to the 150 SN base oil. When the content further increases to 0.1 wt %, the COF of the MoS2-based oil improves conversely. It could be due to the accumulation of MoS2 nanotubes. Furthermore, the AWSDs of the MoS2-based oil at 25 and 75 °C show the trends similar to those of the average COF. The AWSD of the MoS2-based oil at 25 °C with 0.08 wt % is about 35.0% less than that of the base oil. In addition, at 75 °C, the COF and WSD of the 0.08 wt % MoS2-based oil are 52.1 and 24.7% lower than those of the base oil, respectively. For antifriction and antioxidation performances, either at 25 or 75 °C, the 0.08 wt % MoS2-based oil shows the best tribological performance in all tested samples and the average COF decreases 39.2% compared to the 150 SN base oil. The AWSD of the MoS2-based oil at 25 °C with 0.08 wt % is about 35.0% less than that of the base oil. Distinctly, the base oil with 0.08 wt % MoS2 nanotubes at 25 °C shows lower COF value and the average COF decreases 39.2% compared to the 150 SN base oil. When the content further increases to 0.1 wt %, the COF of the MoS2-based oil improves conversely. It could be due to the accumulation of MoS2 nanotubes. Furthermore, the AWSDs of the MoS2-based oil at 25 and 75 °C show the trends similar to those of the average COF. The AWSD of the MoS2-based oil at 25 °C with 0.08 wt % is about 35.0% less than that of the base oil. In addition, at 75 °C, the COF and WSD of the 0.08 wt % MoS2-based oil are 52.1 and 24.7% lower than those of the base oil, respectively. For antifriction and antioxidation performances, either at 25 or 75 °C, the 0.08 wt % MoS2-based oil shows the best tribological performance in all tested samples and the average COF decreases 39.2% compared to the 150 SN base oil. The AWSD of the MoS2-based oil at 25 °C with 0.08 wt % is about 35.0% less than that of the base oil. Distinctly, the base oil with 0.08 wt % MoS2 nanotubes at 25 °C shows lower COF value and the average COF decreases 39.2% compared to the 150 SN base oil. When the content further increases to 0.1 wt %, the COF of the MoS2-based oil improves conversely. It could be due to the accumulation of MoS2 nanotubes. Furthermore, the AWSDs of the MoS2-based oil at 25 and 75 °C show the trends similar to those of the average COF. The AWSD of the MoS2-based oil at 25 °C with 0.08 wt % is about 35.0% less than that of the base oil. In addition, at 75 °C, the COF and WSD of the 0.08 wt % MoS2-based oil are 52.1 and 24.7% lower than those of the base oil, respectively. For antifriction and antioxidation performances, either at 25 or 75 °C, the 0.08 wt % MoS2-based oil shows the best tribological performance in all tested samples and the average COF decreases 39.2% compared to the 150 SN base oil. The AWSD of the MoS2-based oil at 25 °C with 0.08 wt % is about 35.0% less than that of the base oil. Distinctly, the base oil with 0.08 wt % MoS2 nanotubes at 25 °C shows lower COF value and the average COF decreases 39.2% compared to the 150 SN base oil. When the content further increases to 0.1 wt %, the COF of the MoS2-based oil improves conversely. It could be due to the accumulation of MoS2 nanotubes. Furthermore, the AWSDs of the MoS2-based oil at 25 and 75 °C show the trends similar to those of the average COF. The AWSD of the MoS2-based oil at 25 °C with 0.08 wt % is about 35.0% less than that of the base oil. In addition, at 75 °C, the COF and WSD of the 0.08 wt % MoS2-based oil are 52.1 and 24.7% lower than those of the base oil, respectively. For antifriction and antioxidation performances, either at 25 or 75 °C, the 0.08 wt % MoS2-based oil shows the best tribological performance in all tested samples and the average COF decreases 39.2% compared to the 150 SN base oil. The AWSD of the MoS2-based oil at 25 °C with 0.08 wt % is about 35.0% less than that of the base oil. Distinctly, the base oil with 0.08 wt % MoS2 nanotubes at 25 °C shows lower COF value and the average COF decreases 39.2% compared to the 150 SN base oil. When the content further increases to 0.1 wt %, the COF of the MoS2-based oil improves conversely. It could be due to the accumulation of MoS2 nanotubes.
heat from the friction surface to decrease the oil temperature. As shown in Figure 4e,f, the COF at 75 °C of the MoS2-based oil with 0.08 wt % is far below that of the base oil and the oil temperatures of the base oil and the MoS2-based oil maintain unanimity, showing that the MoS2 nanotubes have no effect at a higher testing oil temperature.

Figure 5a,b displays the changes of average COF and AWSDs of the base oil and the 0.08 wt % MoS2-based oil at an applied load (50−250 N) and a rotating speed (200−1200 rpm) in 2 h testing time. In Figure 5a, the average COFs of the base oil and the MoS2-based oil decrease with the increase of applied loads and the average COFs of the MoS2-based oil under different applied loads are all lower than those of the base oil. In detail, the average COF of the MoS2-based oil under 250 N of applied load is about 18.8% lower than that of the base oil under 250 N of applied load and is about 38.4% lower than that of the MoS2-based oil under 50 N of applied load. The results demonstrate that the MoS2 nanotubes more effectively reduce the COF of the base oil at high applied loads. In contrast, the AWSDs of the base oil and the 0.08 wt % MoS2-based oil increase with the increase of the applied load and the AWSDs of the MoS2-based oil are always less than those of the base oil. For instance, the AWSDs of MoS2-based oil under 50 N are about 34.1% lower than those of the base oil under 50 N and about 59.2% lower than those of the MoS2-based oil under 250 N. It could be because the MoS2 nanotubes can smoothly slide into the friction surface of balls to form a protective film.31,32 In Figure 5b, the average COFs and AWSDs of the base oil and the 0.08 wt % MoS2-based oil increase with the spinning speeds increasing from 200 to 1200 rpm at 100 N of applied load in 2 h testing time. The average COFs and AWSDs of the MoS2-based oil are less than those of the base oil at 1200 rpm of speed and about 43.4 and 44.4% less than those of the MoS2-based oil at 200 rpm of speed, respectively. The results demonstrate that MoS2 nanotubes have a better antifriction property at a higher applied load and a higher rotating speed, which is because the MoS2 nanotubes are more easily exfoliated into nanosheets, meaning the larger the spreading area of the MoS2 lubricating film, the lower the friction coefficient.3

To distinctly determine the tribological behavior mechanism, the wear surfaces were inspected by a three-dimensional (3D) laser scanning micrograph. Figure 6a,b depicts the 3D morphologies of the wear scars tested by the 150 SN base oil and the 0.08 wt % MoS2-based oil. After a testing time of 6 h, the wear scars of the steel ball surfaces are seriously worn and display pretty deep furrows and rough traces along the traveling direction. The wear surface of the MoS2-based oil is less than that of the base oil owing to the formation of a film. To detect the film, the Raman spectra of wear surfaces were further recorded (Figure 6c,d). The results confirm that the two characteristic peaks at about 380 and 404 cm−1 appear on the wear surface lubricated by the MoS2-based oil. It demonstrates that MoS2 nanotubes in oil can smoothly slide with oil into the interface to prevent the direct contact and avoid the damage to steel balls.33 Based on the above testing results, the schematic description of the lubrication mechanisms of MoS2 nanotubes in the base oil is proposed and depicted in Figure 7. In detail, MoS2 nanotubes are easier to enter into the contact surface because of the thinner wall and the smaller size and then MoS2 nanotubes are rolling, sliding, and dragging.19 Furthermore, MoS2 nanotubes can form the tribofilm through the direct exfoliation of the outer layers of nanotubes or the indirect exfoliation after rolling, sliding, and dragging to decrease friction and wear scar. The formation of the tribofilm means that MoS2 nanotubes can improve the tribological properties.
3. CONCLUSIONS

MoS2 nanotubes were prepared via the thermolytical approach using HNTs as templates, and the tribological performance of MoS2 nanotubes was further investigated by a multifunctional friction machine. The influences of the content of MoS2 nanotubes, testing time, initial oil temperature, rotating speed, and applied load, and applied load of the base oil on the tribological performances were comprehensively inspected. The results showed that the tribological values of the MoS2-based oil with different contents are less than those of the base oil; especially, the 0.08 wt % MoS2-based oil possessed the lowest average COF and AWSD values and the average COF and AWSD of the 0.08 wt % MoS2-based oil at 25 °C decreased about 39.2 and 35.0% compared to those of the 150 SN base oil. Furthermore, the tribological values of all samples under the same conditions at 75 °C were lower than those at 25 °C. In addition, the COF was decreased and the AWSD was increased with the increasing applied load, while the COF and the AWSD were reduced with the increasing spinning speed. These results clearly illustrate that the MoS2 nanotubes with the thinner wall and the smaller size as lubricating additives used in the base oil showed excellent tribological properties.

4. EXPERIMENTAL SECTION

4.1. Materials and Methods. In our previous work,34 the MoS2 nanotubes were prepared by a thermolysyal approach using HNTs as templates. First, HNTs were dried in a 100 °C oven for 24 h, which was purchased from Yangzhou Xigema New Material Co. Ltd. Then, 2.0 g of HNTs and 1.9 g of (NH4)6MoO24·4H2O were ground into a uniform powder and transferred into a 10 mL crucible. The mixture was kept at 300 °C for 4 h in air to obtain intermediates. Afterward, 0.4 g of HNTs and MoO3, and 0.3 g of (NH4)2CS were kept at 600 °C under N2. Finally, the samples were washed by hydrofluoric acid (40%) and hydrochloric acid (36–38%) to remove HNT templates, washed by deionized water and anhydrous ethanol at least three times, and dried in an oven at 60 °C for 24 h, and were denoted MoS2.

4.2. Testing of Tribological Properties. For testing the tribological properties of the MoS2 nanotubes, the MoS2-based oils with different MoS2 contents, including 0.02, 0.04, 0.06, 0.08, and 0.10 wt %, were added to the 150 SN base oil (exterior: transparent and clear; viscosity: 32) by ultrasonic dispersion, and were denoted MoS2-based oils.

The tribological experiment was carried out on a multifunctional friction test machine (Jinan Chenda Ltd. Co., China). The steel balls (GCr15) were fully immersed in a lubricant, and the tribological properties, including content, initial oil temperature, rotating speed, and applied load, were inspected. Each experiment was repeated at least three times under the same conditions. The WSD was gauged by an optical microscope. The wear scars of steel balls were cleaned with ethanol to remove the base oil and then detected through a Raman spectrometer and a 3D laser scanning microscope. The formation of MoS2 nanotubes by HNTs as templates and the testing of tribological properties are shown in Figure 8.

4.3. Characterization. XRD analysis was carried out by powder X-ray diffraction (D8 advance, Bruker AXS, Germany). The Raman spectrum was recorded by a Raman spectrometer (inVia, Renishaw, U.K.). The SEM image was observed by a field emission scanning electron microscope (S-4800 II, Rili, Japan). The TEM image was observed by a transmission electron microscope (Tecnai 12, Philips, Netherlands). TGA analysis was carried out by a thermogravimetric analyzer (Pyris 1 TGA, PerkinElmer). The Brunauer–Emmett–Teller specific surface areas were analyzed by specific surface analysis and a pore size analyzer (Autosorb IQ3, Quantachrome Instruments). XPS analyzes were carried out by an X-ray photoelectron spectrometer (ESCALAB 250Xi, Thermo Scientific). The UV–vis spectra were recorded by a spectrophotometer (Cary 5000, Varian). The wear scar micrographs were observed by a 3D laser scanning microscope (LSM 700, Carl Zeiss, Germany).

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