



# Tribological properties of MoS<sub>2</sub> nano-balls as filler in polyoxymethylene-based composite layer of three-layer self-lubrication bearing materials

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## ABSTRACT

POM/MoS<sub>2</sub> nano-balls composite was prepared by adding MoS<sub>2</sub> nano-balls synthesized from Na<sub>2</sub>MoO<sub>4</sub> and CH<sub>3</sub>CSNH<sub>2</sub> into polyoxymethylene (POM). The comparative POM-based composite blended with micro-MoS<sub>2</sub> particles was also prepared. The obtained POM/MoS<sub>2</sub> composites were used as the polymeric layer in the three-layer self-lubrication materials. The results of tribological tests showed that the POM with MoS<sub>2</sub> nano-balls presented better tribological properties than that with micro-MoS<sub>2</sub>. When the content of MoS<sub>2</sub> nano-balls was not more than 1.0 wt%, the POM/MoS<sub>2</sub> nano-balls samples presented lower friction coefficients and smaller wear volumes. However, higher contents of MoS<sub>2</sub> nano-balls than 1.0 wt% were very disadvantageous to the tribological performances. DSC results showed the excessive MoS<sub>2</sub> nano-balls affected the POM crystallinity, and accordingly, the self-lubricating capabilities of these samples were influenced as well. SEM micrographs for wear scars confirmed that the worn manner of the POM sample was changed when the content of MoS<sub>2</sub> nano-balls was increased. XPS analysis showed that MoS<sub>2</sub> nano-balls was transferred to the mated friction surface, on which Mo(IV) was oxidized into Mo(VI) via tribochemical reaction. TEM micrographs of worn debris proposed a wear manner concerning the exfoliation of nano-sheets from MoS<sub>2</sub> nano-balls. The reason for the stable self-lubrication properties of POM/MoS<sub>2</sub> nano-balls composite was ascribed to the forming-destroying of debris clusters in a long-time sliding process.

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## 1. Introduction

Molybdenum disulfide (MoS<sub>2</sub>) is one of typical layered compounds, which is particularly important for solid lubrication or as an additive for lubricating oils and greases. The laminar structure of MoS<sub>2</sub> is composed of strong S–Mo–S covalent bonds inside layers and the weak van der Waals force between layers. The easy sliding between MoS<sub>2</sub> layers is generally regarded as a significant feature for its excellent lubricity. It has been well known that nanosized MS<sub>2</sub> (M = W, Mo) usually has better tribological properties either in friction reduction or wear resistance than microsized MS<sub>2</sub> [1–12]. However, layered MoS<sub>2</sub> nanoparticles are easily oxidized in oxygen-rich and temperature-high environments, because the rim atoms of 2H-MoS<sub>2</sub> platelets with dangling bonds have high chemical activity. Luckily, some researches discovered MS<sub>2</sub> with closed structures such as fullerene-like nano-MS<sub>2</sub> (M = W, Mo) had not only good chemical stability but also outstanding lubrication properties

[6–12]. Several methods have been reported to prepare fullerene-like nano-MS<sub>2</sub> such as gas–solid reaction and hydrothermal method [13–15]. Recently, we reported that ball-like amorphous nano-MoS<sub>x</sub> could be prepared conveniently by a very simple method—the quick homogenous precipitation method [16]. Calcining treatment of the resultant amorphous MoS<sub>x</sub> under hydrogen flow led to ball-like MoS<sub>2</sub> nanoparticles (MoS<sub>2</sub> nano-balls) with close layered structure.

Polyoxymethylene or polyacetal (POM), an excellent engineering plastic, can be a substitute for metals in many occasions because of its excellent mechanical properties and self-lubricating characteristics [17]. For example, POM has been successfully used as the polymeric layer in the three-layer (polymer/copper-powder/steel) self-lubrication bearing materials [18]. However, the capability for friction reduction and wear resistance of POM are not very satisfactory especially in extreme conditions involving high temperatures and high loads. To enlarge its range of application, both inorganic and organic additives were selected to modify POM, such as PTFE, LLDPE, graphite, MoS<sub>2</sub> and so on [19–22]. This paper reports our recent studies on the tribological behaviors of MoS<sub>2</sub> nano-balls as filler in POM layer of the three-layer bearing material. The lubrication mechanisms involved are also discussed.

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## 2. Experimental

It is very significant to discover the relationship between the properties of POM-based materials and the morphology and size of  $\text{MoS}_2$  particles, thereby four  $\text{MoS}_2$  samples with different morphologies and sizes were selected as additives in the work. The mixture of  $\text{MoS}_2$  particles and POM had to be processed at  $\sim 185^\circ\text{C}$ . During heating, it was found that the POM blended with  $\text{MoS}_2$  nano-slices with size of  $\sim 30\text{ nm}$  or  $\sim 50\text{ nm}$  represented a terrible degradation at  $\sim 150^\circ\text{C}$ . Due to thermal degradation of POM it leads to the occurrence of poisonous formaldehyde,  $\text{MoS}_2$  nano-slices with  $\sim 30\text{ nm}$  and  $\sim 50\text{ nm}$  cannot be used as additives in POM. This is an interest phenomenon, and further studies are needed to clarify the degradation mechanisms. As a result, the two POM samples with commercial  $\text{MoS}_2$  microparticles and  $\text{MoS}_2$  nano-balls were only studied.

Ball-like amorphous molybdenum sulfide nanoparticles were prepared from  $\text{Na}_2\text{MoO}_4$ , and  $\text{CH}_3\text{CSNH}_2$  by a quick homogeneous precipitation method reported in the literature [16]. The reaction solution was obtained by dissolving  $1.0\text{ g}$   $\text{Na}_2\text{MoO}_4$  and  $2.0\text{ g}$   $\text{CH}_3\text{CSNH}_2$  in  $100\text{ ml}$  distilled water. The obtained solution was heated and then added with  $10\text{ ml}$  of alcohol. When the solution temperature reached  $82^\circ\text{C}$ ,  $23\text{ ml}$  hydrochloric acid was rapidly dumped into the reaction system. The resultant precipitation was washed with the distilled water and dried at  $120^\circ\text{C}$  for  $6\text{ h}$ . Calcination treatment of the as-synthesized ball-like amorphous molybdenum sulfide nanoparticles was carried out in a tube furnace at  $780^\circ\text{C}$  for  $50\text{ min}$  under a hydrogen flow. In the end, the obtained nanoparticles were characterized respectively using a Rigaku model D/Max- $\gamma\text{B}$  X-ray diffractometer with  $\text{Cu K}\alpha$  radiation, a Hitachi model H-800 transmission electron microscopy (TEM), and a JEOL model JEM-2010 high-resolution transmission electron microscopy (HRTEM).

The so prepared  $\text{MoS}_2$  nano-balls were blended with POM (M90, purchased from Yuntianhua Group, China) at  $185^\circ\text{C}$  on a two-roller milling apparatus (SK-160B, Shanghai Rubber Machinery Works, China). The two-roller mill is used for plasticization and mixing. Polymeric particles and additives are fed between two counter rotating heated rollers, after that the plastics and additives can attain the desired blending effect. The obtained blend was characterized on a Shimadzu Model DSC-60 differential scanning calorimetric (DSC). The blend was then made into some square samples with a  $34\text{ mm}$  side length on the surface of steel/copper-powder composite materials by a general hot-rolling method at  $190^\circ\text{C}$ . The whole preparation process was shown in Fig. 1. Using the same treatment, the comparative pure POM sample and POM samples filled with commercial micro-sized  $\text{MoS}_2$  (micro- $\text{MoS}_2$ ) with an average size of less than  $30\text{ }\mu\text{m}$  (provided by Anhui Institute of Metallurgy, China) were prepared as well. The tribological behavior of the obtained square samples was investigated on a MQ-

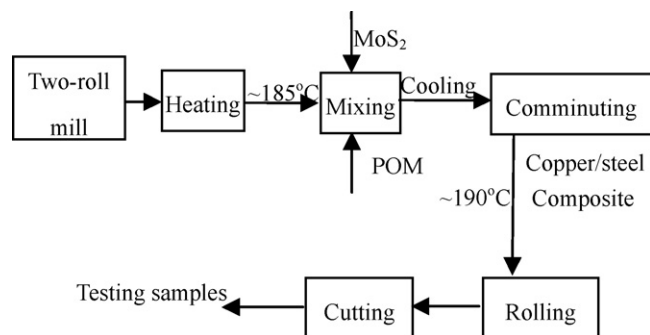


Fig. 1. Preparation process of the three-layer self-lubrication materials.

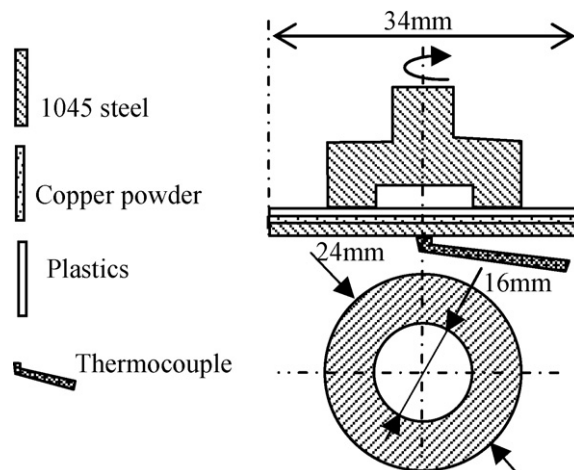


Fig. 2. Schematic illustration of end-face friction configuration.

800 end-face tribometer through rubbing against ASTM 1045 steel under dry friction. The wear rate was determined by the worn polymeric volume. The selected frictional configuration was shown in Fig. 2. The tribological test for average friction coefficient and wear volume was repeated for three times. The back-face temperature of the three-layer materials was measured and used to represent the friction temperature between the rubbing pair indirectly, as shown in Fig. 2.

Micrographs of wear surfaces were obtained using a FEI model Sirion 200 field emission scanning electron microscope. Elements of the counterpart steel surface were investigated on a VG model Escalab 250 X-ray photoelectron spectroscopy (XPS). TEM image of worn debris with nano-balls was obtained using a Hitachi model H-800 transmission electron microscope.

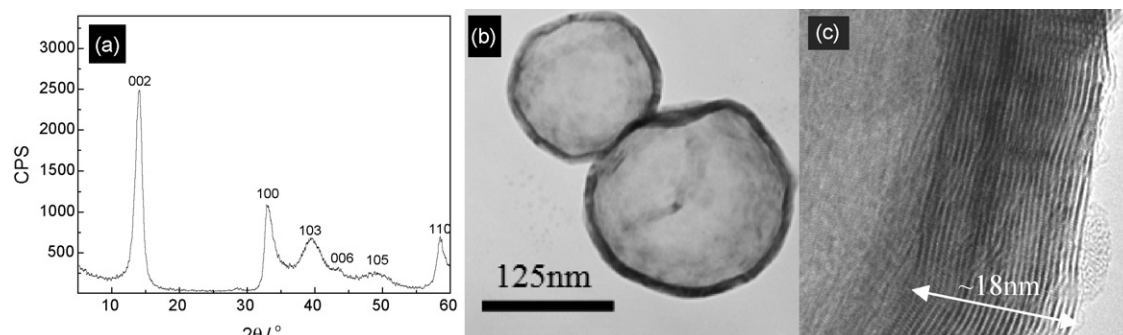


Fig. 3. Results of XRD (a), TEM (b) and HRTEM (c) for the obtained  $\text{MoS}_2$  nanoparticles.

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