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铅纳米微粒用作油性润滑的摩擦学性能研究*

赵彦保[a,b]， 张治军[a**]， 吴志申[a]， 党鸿辛[a,b]
（a. 河南大学特种功能材料实验室，开封 475002；
b. 中国科学院兰州化学物理研究所 固体润滑国家重点实验室，兰州 730000）

摘 要： 在石蜡油-聚乙二醇的混合溶剂中，通过液相分散法成功地制备出了铅纳米微粒. 其中，石蜡油是反应介质，聚乙二醇是抗氧化剂. 同时，对铅纳米微粒的形貌和结构进行了透射电镜（TEM）和 X 光衍射（XRD）表征. 结果表明，铅纳米微粒呈球形，平均粒径为 70 nm，具有与本体铅相同的晶体结构. 另外，在四球试验机上表征了铅纳米微粒作为润滑油添加剂的摩擦学性能. 摩擦试验表明，铅纳米微粒具有良好的减摩抗磨性能，并能够显著改善基础油的承载能力. 磨斑表面分析表明，铅纳米微粒的抗磨减摩机制不是形成金属沉积膜，可能是在摩擦接触面形成滑动-轴承系.

关键词： 铅纳米微粒；油性添加剂；结构表征；摩擦学性能
中图分类号：O613.51　　**文献标识码：**A

Tribological Properties of Pb Nanoparticle in Oil*

Zhao Yanbao[a,b]， Zhang Zhijun[a**]， Wu Zhishen[a]， Dang Hongxin[a,b]
（*a. Laboratory of Special Functional Materials*，*Henan University*，*Kaifeng* 475002；
b. State Key Laboratory of Solid Lubrication，*Lanzhou Institute of Chemical Physics*，*Chinese Academy of Sciences*，*Lanzhou* 730000）

Abstract The solution dispersion method has been successfully used to prepare Pb nanoparticles in a paraffin oil-polyglycol mixed solution by directly dispersing melted Pb granules. In this solution system，paraffin oil was used as reacting media and polyglycol served as an antioxidant to protect Pb nanoparticles from oxidizing. The size and structure of the prepared Pb nanoparticles were characterized by means of the transmission electron microscopy（TEM）and powder X-ray diffraction（XRD）. Their tribological behavior was evaluated with a four-ball tester. The TEM and XRD investigations reveal that the prepared Pb nanoparticles，with the average particle diameter of 70 nm，appear to be of close spherical shape and possess the same crystal structure as the bulk Pb. The tribological results show that the Pb nanoparticles as an oil additive exhibit good friction-reduction and antiwear properties at different additive concentration and applied load. Meanwhile，they can also strikingly improve the load-carrying capacity of the base oil. The rubbed surface was also investigated by the scanning electron microscope（SEM）and energy dispersive spectroscopy（EDS）. However，there was not presence of Pb element on the worn surfaces，which indicated that no chemical reaction occurred between the Pb nanoparticles and the rubbing surfaces. In addition，the tribological mechanism of the formation of the sliding-bearing system was also proposed.

Keywords Pb nanoparticles，Oil additive，Structure characterization，Tribological property

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** Corresponding author，Tel：0378-2192330，E-mail：zhaoyb902@henu.edu.cn
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1 Introduction

In recent years, the application of inorganic nanoparticles in lubricating additives has been receiving considerable attention[1-3]. Studies show that inorganic nanoparticles as additives in oil have following features: rolling friction, serving as spacer, which eliminate metal to metal contact between the asperities of the two mating metal surfaces, and forming lubrication film[4,5]. All of them, metal nanoparticles, are expected to be potential additives of lubricating oil in the future[6,7].

Today, many methods have been used for the preparation of metal nanoparticles, such as metal vapor deposition[8], chemical reduction[9] and thermal decomposition[10]. Generally, all these methods require complicated steps and use of toxic and highly sensitive precursors. In this study, the main goal is to prepare Pb nanoparticles by simple solution dispersion and evaluate their tribological properties as additives in lubricating oil.

2 Experiment

2.1 Preparation of Pb nanoparticles

Pb nanoparticles were prepared with the method similar to references[3,11]. In a typical synthesis, 1 g commercial Pb granules were added to paraffin oil of 30 mL and polyglycol of 5 mL in a flask, and the system was stirred at above 340℃ for at least 10 h. Then, the supernatant was transferred to another flask and cooled to room temperature. Lastly, the supernatant was centrifuged and washed to get the product. In this process, polyglycol served as both antioxidant and solvent.

2.2 Characterization

Transmission electron microscopy (TEM) images were obtained with a JEOL JEM 2010EX/S microscope. The sample was dispersed in chloroform with an ultrasonic vibrator, and then a drop of chloroform was placed on a carbon-coated copper grid and evaporated under ambient atmosphere. Powder X-ray diffraction measurements (XRD) were carried out using X′Pert Philips diffractometer, operating at 40 kV and 40 mA.

2.3 Tribological properties of Pb nanoparticle as an additive in oil

The antiwear and friction reduction properties of Pb nanoparticle as an additive in oil were evaluated with a four-ball tester under the following conditions: 1450 r/min, 30 min, and room temperature. The balls (diameter 12.7 mm) used in the test were made of GCr15 with an *HRc* of 59 ~ 61. The base oil was chemically pure paraffin oil with the boiling point at above 300℃. All additives were dispersed in paraffin oil with an ultrasonic vibrator before carrying out the tests. The scanning electron microscope (SEM) images were collected on a JSM 5600 microscope equipped with energy dispersive spectroscopy (EDS).

3 Results and discussion

3.1 Morphology and structure

TEM examination (Fig. 1) has shown that Pb particles appear to be of spherical shape and have wide size distribution. The average particle diameter is in the range of 70 nm. The large size distribution is naturally caused by the broad size distribution of the liquid Pb droplets formed in solvent by stirring effect.

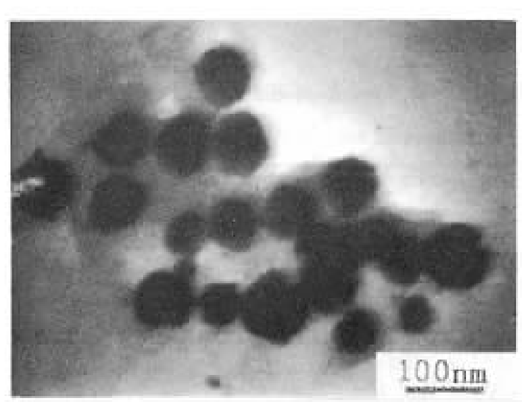

Fig. 1 TEM images of Pb nanoparticles

The crystalline structures of Pb nanoparticles are investigated by XRD (Fig. 2). In the XRD pattern, there are five identified peaks, which are assigned to scattering from the 111, 200, 220, 311 and 222 crystal planes, respectively, of cubic phase of Pb (JCPDS 4-0686). The lattice parameter for the unit cell of Pb sample is calculated as $a = 4.929$ in good agreement with the known lattice parameter for bulk Pb ($a =$

4.930). The presence of Pb identified peaks shows that Pb nanoparticles have good crystallinity.

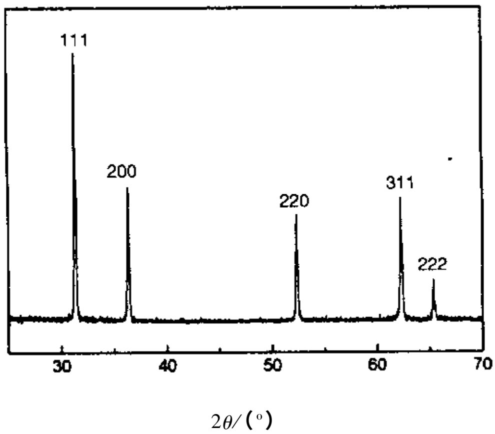

Fig. 2　XRD patterns for the prepared Pb nanoparticles

3.2　Antiwear and friction reduction properties

Fig. 3 gives the relationship between the concentration of Pb nanoparticles in oil and the diameter of wear scar (DWS). When the concentration is zero, the y-coordinate stands for DWS of paraffin oil and is about 0.72 mm. It can be seen that Pb nanoparticle presents the excellent anti-wear ability 0.06% ~ 1.0%. At an additive concentration of 0.06%, the DWS is reduced from 0.72 to 0.61 mm. With an increase in additive concentration, the diameter of wear scar becomes smaller. When 1% of Pb nanoparticle is added into oil, the DWS is only 75% of that of paraffin oil. These results indicate that Pb nanoparticle has a wide concentration range and can improve efficiently the antiwear property of paraffin oil.

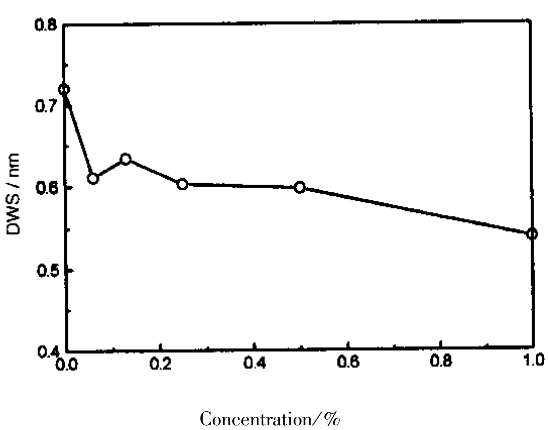

Fig. 3　The diameter of wear scar as a function of Pb nanoparticle concentration

The diameter of wear scar as a function of load is given in Fig. 4. For the paraffin oil, the DWS is 0.72 mm at a load of 300 N, and the system-scuffing load is below 400 N. It can be seen that Pb nanoparticles have excellent anti-wear properties under the different loads. At the load of 300 N, the DWS of paraffin oil containing 0.5% Pb nanoparticles is 0.59 mm, which is large below that of paraffin oil. With the increase in load applied, the DWS of paraffin oil containing 0.5% Pb nanoparticle is being larger, but the friction system could be lubricated effectively even at 600 N. At a load of 600 N, the DWS of paraffin oil containing Pb nanoparticles is only 0.74 mm, which is close to that of paraffin oil at 300 N. These indicate that the Pb nanoparticles have the excellent antiwear property at high load.

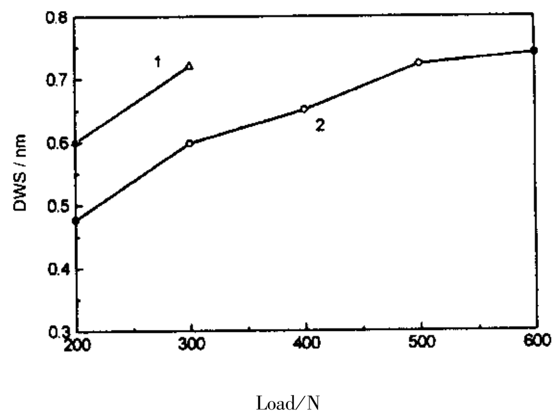

Fig. 4　Relationship between diameter of wear scar and load applied
1. Paraffin oil; 2. 0.5% Pb nanoparticles

The variation of friction coefficient with additive concentration is given in Fig. 5. The friction coefficient

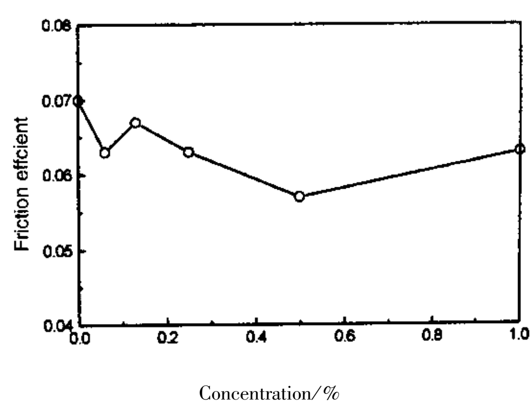

Fig. 5　Variation of friction coefficient with additive concentration of Pb nanoparticles

of paraffin oil is about 0.07. As compared with paraffin oil, the friction coefficient of the oil containing Pb nanoparticles is smaller under the same condition. This means that Pb nanoparticles are a good friction reduction additive. When the concentration of Pb nanoparticles is about 0.5%, the best friction reduction properties are obtained, which indicates that the additive concentration in oil must be controlled in an optimum range. When the concentration increases, the friction coefficient is a little higher, and perhaps the aggregation of Pb nanoparticles occurs.

In order to study the tribological mechanism of Pb nanoparticles, we observe the wear scar morphology through SEM observation (shown in Fig.6). It can be seen that the wear scar surface is relatively smooth,

Fig.6 SEM image of the rubbing surface lubricated by 0.5% Pb nanoparticles

and the rubbing traces are shallow. However, the energy dispersive spectroscopy (EDS) could not detect the presence of Pb element on the rubbing surface, showing that no chemical reaction occurs between Pb nanoparticles and the rubbing surfaces. The results also imply the sliding-bearing effect of the Pb nanoparticles. As discussed above, we suppose that the good tribological performance of Pb nanoparticles might be attributed to the formation of the sliding-bearing system.

4 Conclusions

Pb nanoparticle can be prepared by solution dispersion starting from bulk metal. The nanoparticles have good nano-crystallites and exhibit excellent anti-wear and friction reduction properties. The tribological mechanism of Pb nanoparticles might be contributed to the formation of sliding-bearing system.

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