



Tribological behavior of polyimide/epoxy resin-polytetrafluoroethylene bonded solid lubricant coatings filled with in situ-synthesized silver nanoparticles

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ABSTRACT

Polyimide/epoxy resin-polytetrafluoroethylene (denoted as PI/EP-PTFE) bonded solid lubricant coatings filled with silver nanoparticles synthesized in situ were prepared. The phase composition and grain size of the Ag nanoparticles were analyzed by X-ray diffraction and field-emission scanning electron microscopy, respectively. A micro-hardness tester was employed to determine the micro-hardness of the as-fabricated PI/EP-PTFE lubricant coatings, and a ring-on-block test rig was selected to evaluate the tribological behavior of the lubricant coatings under dry friction and RP-3 aviation kerosene lubrication conditions. The worn surfaces of the lubricant coatings were observed with a scanning electron microscope. Results indicate that the silver citrate precursor was decomposed to generate Ag nanoparticles during the curing process of PI/EP-PTFE lubricant coatings. Resultant in situ-synthesized Ag nanoparticles with a size of about 100 nm were homogeneously dispersed in the lubricant coatings, thereby significantly increasing the micro-hardness, reducing the friction coefficient and enhancing the wear life of the lubricant coatings. Particularly, the lubricant coating filled with 5% Ag nanoparticles exhibited the lowest friction coefficient and the longest wear life. Moreover, due to the excellent lubrication and cooling effect of RP-3 aviation kerosene, the lubricant coating achieved better friction-reducing and anti-wear abilities under RP-3 aviation kerosene lubrication than that under dry friction.

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1. Introduction

Bonded solid lubricant coatings are widely used as a type of solid lubricating materials to mitigate wear, adhesion and scuffing of mechanical parts [1–7]. This is because they consist of solid lubricant, adhesive resin and functional filler and usually exhibit excellent friction-reducing and anti-wear performance. Particularly, the incorporation of various functional fillers can often decrease the internal stress and porosity of bonded solid lubricant coatings, thereby improving the mechanical and tribological properties of the coatings [8,9].

Traditional functional fillers for lubricant coatings include metal like Ag, transition metal oxides such as Sb₂O₃ and PbO, as well as rare earth fluorides such as LaF₃ and CeF₃ [10–12]. These particu-

late fillers, however, usually have a relatively large grain size and poor dispersibility in the lubricant coatings, which is unfavorable for enhancing the mechanical properties of the lubricant coatings. This drawback, fortunately, could be eliminated by applying nanotechnology to decrease the size of the particulates to nanolevel. Previous researches demonstrate that Fe₃O₄, SiC, ZrO₂, ZnO, TiO₂, SiO₂, Al₂O₃, LaF₃ nanoparticles and carbon nanotube can significantly promote the densification of lubricant coatings and improve their load-carrying capacity and tribological properties [4,13–21]. In these works, nanoparticles are prepared and dispersed into lubricant paints, and then lubricant coatings are prepared. However, such nanoparticles are easy to agglomerate because of their high surface energy leading to less efficient dispersion and hence much lower enhancement in performance than expected. In order to improve the stability and homogeneity of the nanoparticles dispersed in lubricant coating, organic complexes with active functional group are commonly used to modify the nanoparticles to avoid their agglomeration. Such surface modified nanoparticles can be dispersed into the paints well and the tribological properties

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of the lubricant coatings would be further advanced [15–17]. However, it is obvious that organic modifiers can not bond to the binder, which will weaken the bonding strength between nanoparticles and binder resin. This is harmful to further improve the tribological properties of the lubricant coatings.

In the field of chemical preparation of nanomaterials, much more investigations have been focused on the technique of precursor thermal decomposition. By this method to control the decomposition of unstable metal organic complexes, various monodisperse nanoparticles have been prepared [22–26]. For example, CdS, ZnS, ZnSe and CdSe nanoparticles have been prepared through the thermal decomposition of dithio- or diselenocarbamates of cadmium or zinc [27,28]. Moreover, it is well known that metallic silver has excellent lubricating property as a soft metal, and also the incorporation of Ag nanoparticles can effectively advance the load-carrying capacity and tribological properties of the lubricant materials [29–34]. Enlightened by the preparation method of nanoparticles with precursors, and combining the advantages of Ag nanoparticles as filler, silver citrate as silver precursor instead of silver nanoparticles was incorporated into polyimide/epoxy resin-PTFE bonded solid lubricant coating homogeneously. At high curing temperature, silver citrate would be decomposed, and uniform and stable Ag nanoparticles could be synthesized in situ. It is anticipated that the tribological properties of the lubricant coating can be improved efficiently through the incorporation of such Ag nanoparticles. As the lubricant coating usually works under dry friction or lubricating oil conditions, in this work, the tribological behaviors of the bonded PTFE lubricant coatings filled with Ag nanoparticles synthesized in situ under dry friction and RP-3 aviation kerosene lubrication conditions at different applied load and sliding speed were investigated, respectively, and the corresponding wear mechanism was discussed.

2. Experimental

2.1. Materials

Polyamic acid and epoxy resin (commercial code: AG-80) were commercially obtained from Beijing Sino-Rich Company Ltd (Beijing, China, percent content: 33) and Shanghai Research Institute of Synthetic Resins (Shanghai, China), respectively. PTFE with an average diameter of less than $5\ \mu\text{m}$ was purchased from Shandong Huafu Fluoro-Chemical Company Ltd (Jinan, China). Silver citrate was synthesized in deionized water via an ion exchange method in our laboratory. *N*-methylpyrrolidone/*N,N*-dimethylformamide mixed at a volume ratio of 1:1 was used as the dispersion medium for fabricating title lubricant coating. AISI-1045 steel block ($12.35\ \text{mm} \times 12.35\ \text{mm} \times 19.00\ \text{mm}$, HRC 28–32, $E = 210 \times 10^3\ \text{MPa}$) was used as the substrate, and AISI-52100 bearing steel ring ($\Phi 49.24\ \text{mm}$, HRC 58–61, $R_a = 0.02\ \mu\text{m}$, $E = 208 \times 10^3\ \text{MPa}$) was used as the counterpart for assembling the frictional pair.

2.2. Preparation of PI/EP-PTFE lubricant coatings

Briefly, 18.6 g of polyamic acid and 1.6 g of epoxy resin were homogeneously dispersed in 15 ml of dispersion medium under mechanical stirring and ultrasonic vibration. In the meantime, 8.0 g of PTFE and 1.3 g of silver citrate were dispersed in 25 ml of the dispersion medium under 48 h of mechanical ball milling. The resultant PTFE-silver citrate dispersion was then added into the dispersion of the resin binder while a proper amount of the dispersion medium was supplemented to adjust the solid content of the final mixed dispersion as about 20%. Prior to preparation, the AISI-1045 steel substrate was sandblast ($R_a = 2.00 \pm 0.20\ \mu\text{m}$) and

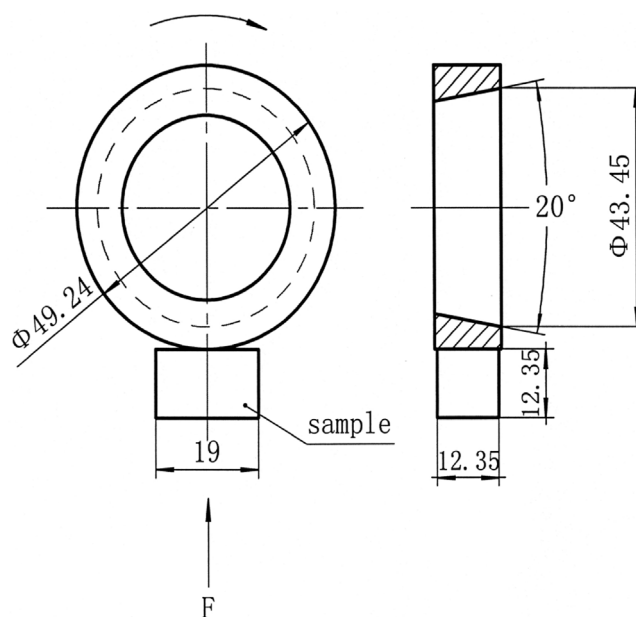


Fig. 1. Schematic contact configuration of MHK-500A ring-on-block test rig (unit: mm).

ultrasonically cleaned with acetone in order to ensure good adhesion of the sprayed lubricant coatings. A spray gun working with 0.2 MPa of nitrogen gas was adopted to spray the mixed dispersion onto the roughened and cleaned surface of the substrate. The as-sprayed mixed dispersion was sequentially cured at $150\ ^\circ\text{C}$ for 0.5 h and at $190\ ^\circ\text{C}$ for 1 h to eliminate the organic solvents and to allow the decomposition of silver citrate, thereby affording PI/EP-PTFE lubricant coating filled with 5% Ag nanoparticles (weight percentage). The percentage of Ag nanoparticles in the lubricant coating was theoretically calculated from the additive amount of silver citrate assuming that silver citrate can be decomposed completely. In order to discuss the effect of different content of Ag nanoparticles on the tribological properties of the lubricant coating, a series of lubricant coatings filled with 0%, 1%, 3%, 5%, 7% and 9% Ag nanoparticles were fabricated with the adjustment of dosages of silver citrate. The thickness of the lubricant coatings, measured with a MINITEST 1100 microprocessor coating thickness gauge, was 35–40 μm .

2.3. Friction and wear test

The friction and wear behaviors of the as-fabricated lubricant coatings under dry friction and RP-3 aviation kerosene lubrication conditions were evaluated with an MHK-500A ring-on-block test rig (Jinan Testing Machine Factory, Jinan, China). Fig. 1 shows the contact schematic of the frictional couple. The AISI-52100 steel ring (abraded with 900 grade water proof abrasive paper and cleaned with acetone) was driven to rotate against the lubricant coatings under dry friction in the linear speed range of 1.28–3.20 m/s and applied load range of 200–400 N. The sliding tests under the lubrication of RP-3 aviation kerosene were run in the same linear speed range but in extended applied load range of 200–1000 N. A strain-gauge bridge connected to the specimen holder was utilized to record the friction forces of the specimens. The wear scar depth (corresponding to the sliding-induced reduction in coating thickness) of the lubricant coatings was measured with a surface roughness measuring instrument and a surface mapping microscope, respectively. The wear life is described as the quotient of the sliding distance (sliding speed \times sliding time) divided by the wear scar depth (unit: $\text{m}/\mu\text{m}$). All the sliding tests were conducted at an ambient temperature of 20–30 $^\circ\text{C}$ and a relative humidity

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