Surface & Coatings Technology 204 (2010) 1330–1338

Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Tribology of UHMWPE film on air-plasma treated tool steel and the effect of PFPE overcoat

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ARTICLE INFO

Article history:
Received 14 April 2009
Accepted in revised form 3 September 2009
Available online 18 September 2009

Keywords:
Thin films
Air-plasma
Friction
Wear
UHMWPE
PFPE

ABSTRACT

The effects of air-plasma treatment, film thickness, normal load and sliding speeds on the tribological properties of a thin film of Ultra-High Molecular Weight Polyethylene (UHMWPE) coated onto a tool steel substrate sliding against a Φ4 mm silicon nitride ball was investigated. Wear tests are carried out on a ball-on-disk tribometer. Air-plasma treatment has enhanced the adhesion of the polymer film to the steel substrate which led to an increased wear life (>100,000 cycles) and low coefficient of friction (~0.14) of the thin film. A film of optimum thickness of 16.3 ± 2 μm shows the maximum wear resistance. The effect of varying loads (0.3, 1, 2 and 4 N) and speeds (200, 400, 600, 1000 and 2000 rpm) on wear life and coefficient of friction were also studied. The dual-film (UHMWPE/PFPE) on the air-plasma pre-treated tool steel surface further increased the wear life (>200,000 cycles) at a load of 4 N and a rotational speed of 1000 rpm.

1. Introduction

Steel is one of the extensively used materials in engineering applications such as bearings and gears where the tribological aspects of the contacting surfaces play very important roles in determining the life of the component and the frictional energy losses. The ever-increasing demand for improving the wear life and decreasing the coefficient of friction in order to facilitate energy conservation in mechanical systems has motivated many researchers to investigate the role of polymer coatings in these tribological applications. Recent research has shown that polymer thin films have excellent tribological properties when coated onto various metallic substrates such as steel and aluminium [1–5]. These polymer coatings, if used effectively, may result in reducing the overall consumption of lubricants. Excellent wear resistance of the film will also allow the use of lubricants with low harmful additive contents or even bio-lubricants (such as soya bean oil), leading to an environmental-friendly lubrication technology. Lubricant additives such as phosphates and sulphates are used as boundary lubricants, which pose major health hazard. In the presence of a thin layer of a highly wear resistant polymer on a metallic substrate, there may not be any need of a boundary layer forming lubricant additive.

Ultra-High Molecular Weight Polyethylene (UHMWPE) is a unique polymer which has exceptional tribological properties [6]. In its bulk form, UHMWPE is highly wear resistant compared to many other polymers such as polyetheretherketone (PEEK), polyethylene (PE), polystyrene (PS) etc. [7]. In a previous work, UHMWPE was coated successfully onto Al substrate to improve the wear life of this metal [5]. Research on the tribological properties of UHMWPE films on bare Si surface and suitably modified Si surface has shown that, UHMWPE is an excellent candidate material as thin film coating because of its very high wear resistance coupled with low coefficient of friction against metallic and ceramic materials [8,9]. However, not much research has been carried out to tap the potential of the UHMWPE coatings on substrates such as steel for tribological applications in mechanical sliding components such as bearings and gears, which is the focus of the current study.

The concept of surface cleaning plays a very important role in the adhesion of thin films onto a substrate. Plasma treatment is an effective surface adhesion enhancement technique to reduce processing cost, time and environmental pollution problems [10]. Studies have shown that plasma cleaning is one of the most effective methods of pre-treatment for metals prior to the thin film deposition which improves the adhesion between the film and the substrates [11–13]. The advantages of air-plasma treatment are: it eliminates the use of harmful elements such as sulphur and phosphorous which most of the other pre-treatment procedures implement, it is simple, cost effective and can easily be adapted to industrial applications.

Polymer film thickness is usually measured by using non-contact laser profilometer [8], sectioning/SEM [9], AFM [14]. In this study a new technique of Focussed Ion Beam (FIB) milling is implemented for measuring the film thickness deposited onto the steel substrates. Conventionally, FIB is used for failure analysis, defect characterization, design modification. However, in the present study, FIB is used to cut the film in the direction perpendicular to the film surface until a certain depth into the substrate and the thickness is measured through the cross-sectional analysis of the cut obtained through this milling. The thicknesses of the UHMWPE films obtained using FIB

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doi:10.1016/j.surfcoat.2009.09.011

milling in the present study are in good agreement with the earlier obtained thicknesses for the UHMWPE film [9].

In the present study, a thin film of UHMWPE is coated onto DF3 tool steel substrate using a very cost effective dip-coating process [8,9,15,16]. Prior to film deposition, the steel substrate was cleaned using air-plasma treatment. Scratch tests were conducted to evaluate the adhesion strength of the film to the steel substrate. The coated substrate was slid against a Φ4 mm silicon nitride ball under dry conditions to investigate the tribological performance. Varying film thicknesses were obtained by using different wt% concentrations of UHMWPE in the polymer solution and the film thickness was measured by the technique of FIB milling. Wear tests were conducted to ascertain the optimum film thickness for the best tribological performance. Effects of different sliding speeds and normal loads on the coefficient of friction and wear life were also investigated. UHMWPE wear track morphology was studied in an effort to deduce the wear mechanisms. After a thorough investigation of the tribological properties of the UHMWPE film, an ultra-thin layer of PFPE was overcoated onto UHMWPE film to further improve the tribological performance of the UHMWPE film. The reasons for the selection of PFPE being, its chemical and thermal stabilities, low vapour pressure, low surface tension and good lubricity that are all essential for better tribological performances [8]. The wear life of the dual-layer film is investigated at a normal load of 4 N and at rotational speeds of 1000 and 2000 rpm, respectively, and the results are compared with those of pristine UHMWPE film.

2. Experimental procedures

2.1. Materials and chemicals

Specimens were prepared on DF3 Tool steel (C—0.95%, Mn—1.1%, Cr—0.6%, W—0.6%, V—0.1% and the rest Fe) coupons of 25 mm × 25 mm × 5 mm dimensions with an average hardness value of 60 HRC. The steel specimens were grounded flat and polished to an average surface roughness of ~108 nm (measured using AFM over a scan size of 40 μm × 40 μm). UHMWPE polymer powder (Grade: GUR X 143) used for coating the specimens was supplied by Ticona Engineering Polymers, Germany, and was procured from a local Singapore supplier (Melt index MFR 190/15 = 1.8 ± 0.5 G/10 min; Bulk density = 0.33 ± 0.03 g/cm^3; Average particle size = 20 ± 5 μm). Decahydronapthalene (decalin) was used as a solvent to dissolve the polymer powder prior to dip-coating. PFPE used was Z-dol 4000 (obtained from Solvay Solexis, Singapore) which was dissolved in H-Galden ZV60 (obtained from Ausimont INC). Chemical formulae of Z-dol and H-Galden ZV60 are $HOCH_2CF_2O$-$(CF_2CF_2O)_p$-$(CF_2O)_q$-CF_2CH_2OH and HCF_2O-$(CF_2O)_p$-$(CF_2CF_2O)_q$-CF_2H, respectively, where the ratio p/q is 2/3.

2.2. Pre-treatment procedure

The pre-treatment procedure included the following steps. Steel samples were polished and cleaned with distilled water and acetone successively in an ultrasonic bath prior to drying using nitrogen gas. The samples were then air-plasma treated using Harrick Plasma Cleaner/Steriliser. The sample surface was exposed to plasma under vacuum for approximately 5 min using a RF power of 30 W. Care was taken not to expose the surface to any further contamination and was immediately processed for the dip coating of UHMWPE films.

2.3. Coating procedure

UHMWPE polymer in powder form was dissolved in decalin by heating the solution to 150 °C for 30 min followed by another heating sequence to 250 °C for 30 min. Magnetic stirrers were used for uniform distribution of heat in the solution and for speeding up the dissolution process. Coating was carried out once the solution turned from white colour to transparent indicating a complete dissolution. The specimens were dip-coated using a custom-built dip-coating machine which could submerge and withdraw the sample at a speed of 2.1 mm/s. The samples were held in the polymer-decalin solution for 30 s in submerged condition prior to withdrawal. The coated samples were dried in air for 60 s and then post-heat-treated in a hot oven at 120 °C for about 20 h. After the post-heat-treatment the samples were cooled slowly to room temperature in the oven and stored carefully in a desiccator before proceeding to tribological testing.

As for the dual-layer of UHMWPE/PFPE, the UHMWPE coated samples were dipped using the same dip-coating machine into a PFPE solution (0.2 wt% PFPE in H-Galden ZV solvent). No additional heat-treatment was conducted after PFPE coating on the film.

2.4. Surface characterization and analysis

VCA Optima Contact Angle System was used for the measurement of contact angles with de-ionised water. A water droplet of 0.5 μl was used for the contact angle measurement. A total of five independent measurements were performed randomly at different locations on the samples and an average value was taken for every sample. The measurement error was within ± 3°.

Bio-Rad FTIR model 400 spectrophotometer was used to obtain FTIR spectra for UHMWPE film in air, using transmission mode. The spectra were collected by accumulating 32 scans at a resolution of 4 cm^{-1} from at least five to six replicate points and they were found to be identical at all points of measurement. Bare DF3 tool steel was used for background scan.

Atomic force microscope (Dimension 3000 AFM, Digital Instruments, USA) was used to study the surface topography of the polymer films. A silicon tip was used for scanning and images were collected in air, in the tapping mode.

2.5. Thickness measurement

The film thicknesses were measured using the Focussed Ion Beam milling technique. FIB-Quanta 200 3D-Dual Beam from FEI Company, USA, was used for this purpose. Square patterns of size 50 μm × 50 μm with a depth of 40 μm were cut in the coated sample using an ion beam with a current of 7 nA under vacuum. The walls of the cut were viewed under Scanning Electron Microscopy (SEM) in a tilted position at 52° to clearly see the cross-section of the film, the substrate and the interface, and, the thickness of the polymer film was measured. Three readings for every sample were recorded and an average value was calculated. The actual thickness of the film was calculated from the reading obtained in the tilted position by using the following equation: Thickness = (Reading) × sin52°, as the stage in the FIB-Quanta 3D machine is inclined at 52° to the horizontal to the line of view.

2.6. Scratch test

Scratch tests were carried out on a custom-built scratch tester by a conical diamond tip of radius 2 μm [17]. The length of the scratch and the traverse velocity of the tip were kept constant for every scratch as 10 mm and 0.1 mm/s, respectively. Normal load was also kept constant for each scratch. However, the normal load was varied from 1 g to 15 g with an increment of 1 g for every successive scratch. After the test, the scratches were characterized using FESEM/EDS technique to ascertain the critical load, defined as the load at which the polymer film showed signs of failure which was identified by the peeling-off or ploughing mechanism and the appearance of Fe peaks (because of exposed substrate) in the EDS spectrum. Before observing the scratches under FESEM (Field Emission SEM), gold coating was performed on the films at 10 mA for 40 s by using a JEOL, JFC-1200 Fine Coater.