January 2008 Volume 4 Issue 2

Materials Science

Trade Science Inc.

An Indian Journal

Full Paper

MSAIJ, 4(2), 2008 [111-115]

Atomic force microscopy as a tool for comparing lubrication behavior of lubricants

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Received: 5th August, 2007 ; Accepted: 10th August, 2007

ABSTRACT

Choosing and optimizing the additives for formulation of base oils has some trial and error aspects which needs time and cost consuming experiments. Among the various types of additives, friction modifiers are used in the formulation of a wide range of industrial lubricants. In this paper the results of using contact mode Atomic Force Microscope for studying the nanotribological behavior of mica in the absence and presence of lubricants are presented and compared with the results of macro scale evaluations by FZG test. A Polyol Ester (POE) base oil and a formulation of it with a combination of a friction modifier and an antiwear agent have been used as lubricants.

KEYWORDS

Nanotribology;
Atomic force microscope;
Polyol ester;
Friction modifier;
Friction force;
Lubricant;
Force-distance curve;
Mica.

INTRODUCTION

Choosing and optimizing the additives for formulation of base oils has some trial and error aspects and needs time and cost consuming experiments. Among the various types of additives, friction modifiers are used in the formulation of a wide range of industrial lubricants. These additives, which are also called boundary lubrication additives, are polar molecules that are able to grip solid surfaces (or be adsorbed) and contain reactive functional groups with low ionization potential or high polarizability. Boundary lubrication properties are also dependent upon the molecular conformation and lubricant spreading. The Atomic Force Microscope (AFM) can be used for studying the nanoscopic behavior of lubricants and assessing the value of potential lubricant boundary layer additives. AFM experiments show that lubricants with polar (reactive) end group dramatically increase the load that a liquid film can support before solid-solid contact and these exhibit long durability[1]. The lubricant produce protective boundary layers, which are physically robust enough to survive prolonged, service conditions but slippery enough to maintain low coefficient of friction[2]. During AFM studies, the normal and friction forces acting as the tip of the AFM slides over the surface sample are measured.

EXPERIMENTAL

A SPM Solver P47H-PRO (NT-MDT Co.) was used to measure surface topography and friction force in nano-scales in air and liquid. Golden silicon cantilever with rectangular tip was used in the contact mode experiments. Some specifications, which are reported for the probe, are shown in TABLE 1.

The value of normal bending force constant was assumed 0.1N/m that was specified as typical value by tip manufacturer[3].

The mica surface was used as surface sample because its molecularly smoothness minimizes the topographic effects on friction measurements.

The oil lubricants used in this study were a POE base oil and a formulation of it with a combination of a friction modifier and an antiwear additive.

The topography and friction force measurements were performed by using liquid head, so that the entire cantilever and surface were immersed in liquid. The scan area, the number of points along a line and the scan velocity were 130nm×130nm, 128 and 4.6 Hz respectively.

The normal force between tip and sample was estimated from cantilever deflection (nA) curve plotted against Z-displacement of the cantilever and converting this curve to force-distance curve. The conversion factor for converting nA unit to nm was obtained from the slop of the linear portion of the deflection-distance curve. There was also one conversion needed for the x- axis values. The change in piezo height, which has been used for the distance between the tip and the sample, was corrected for the deflection of the cantilever by subtracting the cantilever deflection from the piezo height.

When the cantilever is far from the surface, the interaction forces are virtually zero. This offset that may be due to the initial settings of the equipment or thermal drift[4], was subtracted from all the deflection data in order to calculate the true interaction force.

The conversion factor for nA unit to nm and also normal force constant were used without further calibration.

Friction measurement experiments were performed at different set points so that after scanning each 10 lines of scan area at a definite set point, the amount of set point was changed and by this way, there was 1280 data for both normal and lateral forces at each set point (the topography and lateral imaging was done simultaneously).

TABLE 1: Some specifications, which are reported for the AFM probe[3]

Cantilever Length, μm		250±5
Cantilever Width, μm		35 ± 3
Cantilever thickness, μm	Min	0.7
	Typical	1.0
	Max	1.3
Resonant frequency, KHz	Min	14
	Typical	20
	Max	28
Force constant (N/m)	Min	0.03
	Typical	0.1
	Max	0.2
Chip thickness(μm)		0.4
Reflective side		Au
Aspect ratio		3:1
Cone Angle		<=22°
Curvature radius of a tip(nm)		Typical 10

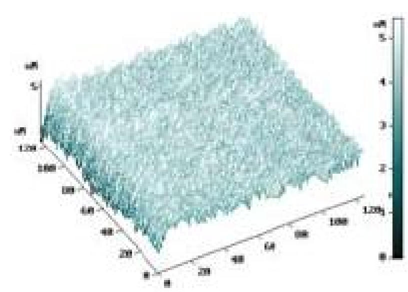

Figure 1: The 3-D topography image of mica without lubricant and at relative humidity less than 5 percent (RMS=0.743)

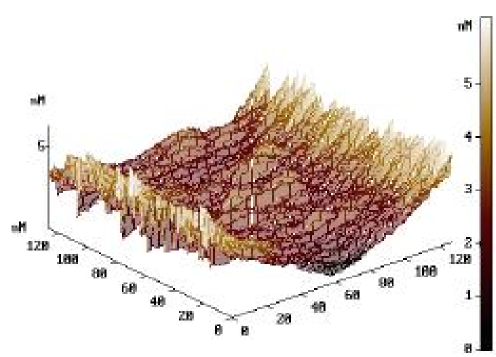

Figure 2: The 3-D topography image of mica at presence of polyol ester base oil as lubricant (RMS=1.197)

During friction measurements, the friction signal were obtained from both the forward and backward scans. Topography-induced effects, that are independent of scanning direction, were eliminated by subtracting the friction data of the backward scan from that of the forward scan, leaving only material-induced effects[5].

RESULTS AND DISCUSSION

The three dimensional topography images of mica surfaces without lubricant and at presence of polyol ester base oil as lubricant,

are shown in figures 1 and 2. During the experiments with mica without lubricant, the relative humidity was kept below 5 percent to lower the coverage of mica surface with water[6].

The roughness of surfaces was expressed by RMS height. It should be mentioned that the RMS height of the surface is used extensively in tribology because of simplicity in calculation and its physical meaning as a reference height scale for a rough surface. It is defined as: $RMS^2=s^2+z_m^2$, Where s is the standard deviation and z_m is the mean value of the surface height signals[7]. The mica surfaces were similarly smooth.

The three-dimensional lateral force images of mica without lubricant and in the presence of POE base oil and a formulated POE base oil as lubricant, are shown in figures 3-5 respectively.

According to results obtained, at set point=0, using both lubricants (base oil and formulated base oil) similarly reduce the friction signals to about 50 times smaller than the values for unlubricated mica. Therefore the performance of two oils should be compared at increased loads. To increase the load, the set point of the AFM apparatus should be decreased and it was necessary to know the amount of the normal force between tip and sample at each set point, means plotting the force-distance curves for both lubricants (base oil

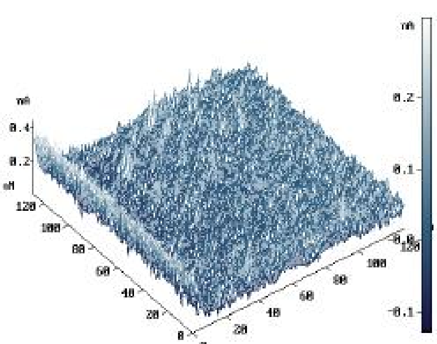

Figure 3: The 3-D lateral force image of mica at relative humidity less than 5 percent (set point =0)

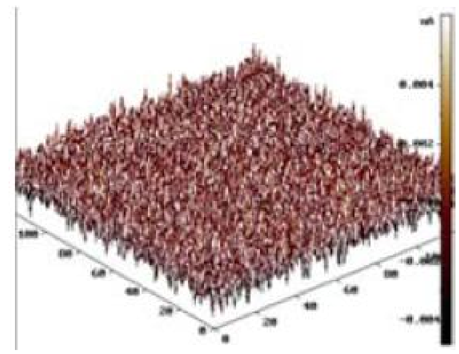

Figure 4: The 3-D lateral force image of mica at presence of POE base oil as lubricant (set point =0)

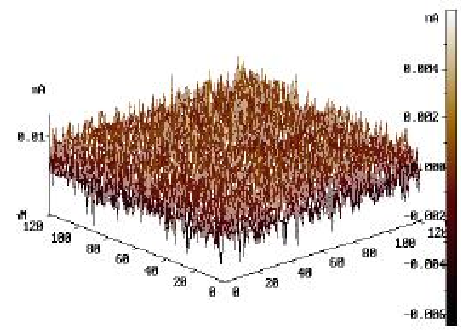

Figure 5: The 3-D lateral force image of mica at presence of a formulation of polyol ester base oil with friction modifiers as lubricant (Set point =0)

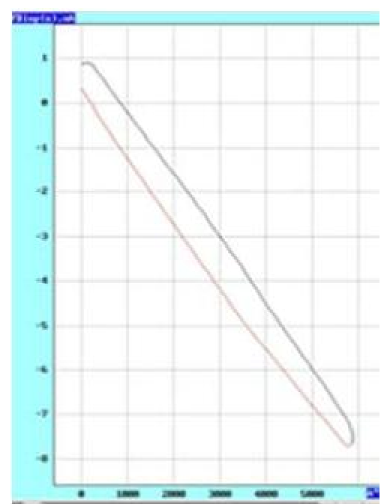

Figure 6: The deflection–distance curve for mica at ambient temperature and pressure and relative humidity less than 5 percent

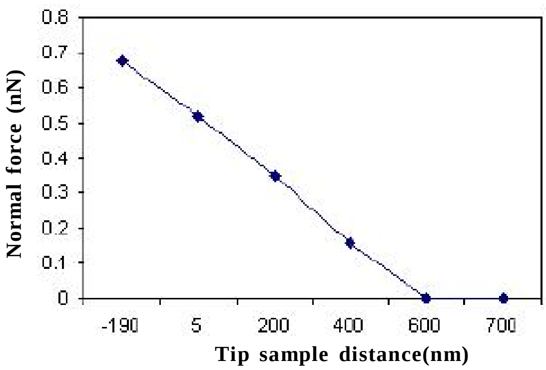

Figure 7: The force-distance curve obtained for the mica at presence of polyol ester base oil as lubricant

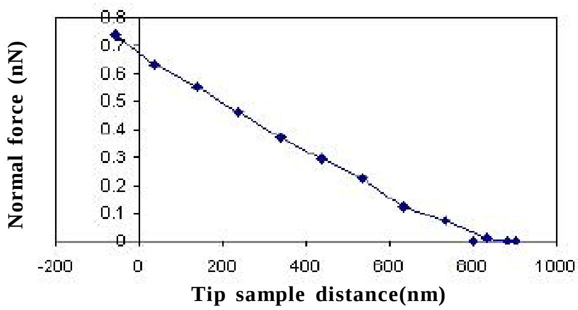

Figure 8: The force-distance curve obtained for the mica at presence of a formulation of polyol ester base oil with friction modifiers as lubricant

and formulated base oil).

The deflection-distance curve for mica which the slop of its linear portion was used for determining the conversion factor of nA to nm is shown in figure 6. This curve was plotted at ambient temperature and pressure and relative humidity less than 5 percent.

The force-distance curves plotted for both POE base oil and formulated POE base oil are shown at figures 7 and 8 respectively.

From the curves, it is obvious that in the case of formulated oil, the value of the force between the mica surface and the AFM tip is larger than the same value for the base oil at the equal distance.

It means that even at same distance between tip and the surface, the normal force effective in friction measurements is larger for the formulated oil.

By plotting the force-distance curves for both lubricants, it was possible to compare the results of the friction force measurements at higher definite loads. Lateral force imaging were performed so that after scanning each 10 lines of scan area at a definite set point, the amount of set point was changed and by this way, there was 1280(128*10) data for each set point.

According to results of friction measurements for the POE base oil (RMS value of mica sample=3.15), up to the load about 0.51nN, the friction signals are too small to enable us to make any quantitative conclusions about the dependence of the friction force on the applied load and above this load there is a very large increase in the friction signal while in the case of formulated oil (RMS value of mica sample=3.55); there was the same situation up to the load about 0.88 nN. The load was not further increased because of the possibility of the tip damaging. Giving rise to an increased friction signal, it should be noted that the effective spring constant for the torsional bending of the cantilever is high (about 16.6N/m) which decreases the force resolution.

The wear characteristics of both lubricants were also evaluated by FZG gear oil rig (DIN 51534). The damage load stage for polyol ester base oil and formulated oil were 7 and greater than 12 respectively.

SUMMARY

In this study the ability of friction modifier additives in improving the lubrication behavior of POE base oils was investigated by AFM.

According to results obtained using both lubricants (base oil and formulated base oil) reduce the friction signals of lateral force microscopy and the addition of friction modifiers increases the lubrication ability. The observation is in agreement with the result of the FZG gear oil rig (DIN51534) evaluation.

ACKNOWLEDGMENTS

We are grateful to the Research Institute of Petroleum Industry (RIPI) for financial support, which enabled this work to be undertaken. We would also like to thank Dr. Alexander Alexeev and Dr. Rachel Owen, for helpful guidance.

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