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Influence of surface roughness and running-in on the lubrication of steel surfaces with oil containing MoS_2 nanotubes in all lubrication regimes

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ABSTRACT

Despite several studies that have confirmed the beneficial effect of MoS_2 and WS_2 nanoparticle-assisted lubrication, an understanding of how the nanoparticles behave in different, even very common contact conditions, such as roughness, is still missing. As a result we have focused on a comparison of the lubrication behaviour of MoS_2 nanotubes mixed with PAO oil using steel surfaces with different roughnesses. Moreover, we have investigated the MoS_2-nanotubes-assisted lubrication of steel/steel contacts in all lubrication regimes and also the effect of the running-in of these contacts. It was realized that the friction with the nanotubes-containing oil was 40–65% lower compared to the base oil, depending on the different contact conditions used. Furthermore, we showed that by using MoS_2 nanotubes in the oil the friction is the same for rough and smooth steel surfaces, meaning that the nanotubes completely govern the lubrication behaviour in self-mated steel contacts in the boundary- and mixed-lubrication regimes, irrespective of the surface roughness or the running-in.

1. Introduction

It is generally accepted that MoS_2 and WS_2 exhibit excellent performance when used as solid lubricants—even surpassing the performance of graphite. Through their layered structure with strong-intralayer and weak-interlayer bonds, these materials offer a low-shear resistance to an external shear stress. Namely, by allowing slippage between the layers, the resistance to tangential movement is decreased, which results in a lower friction [1–4]. Moreover, MoS_2 and WS_2 can also form nanoparticles like fullerene-like nanoparticles and nanotubes [5–7]. Due to their small size, there is a real possibility of making stable suspensions from these nanoparticles in various liquids—for example, by mixing them with oil and thus combining the benefits of solid and liquid lubricants. Even though the technology for ensuring the long-term stability of such suspensions is still not fully developed, it has nevertheless been proven that MoS_2 and WS_2 nanoparticles perform very well in lubricated contacts, significantly reducing both the friction and the wear [8–21].

Several mechanisms and effects were proposed in an attempt to explain the lubricious behaviour of the nanoparticles in oils: rolling friction; nanoparticles acting as spacers, preventing direct contact between the asperities; and their delamination and exfoliation, especially at high contact loads [8–10,20,22]. In spite of several studies and suggestions to explain their performance, it is still not fully clear what the actual, prevalent behaviour of the nanoparticles in a contact is, especially because it may vary with different nanoparticles and contact conditions. Consequently, an understanding of how nanoparticles will perform under various contact conditions and how to tailor any tribological system in which nanoparticles could be applied is still missing.

MoS_2 nanotubes have only recently been synthesized in sufficient quantities for tribological studies [23], after which we showed for the first time how they very effectively reduce the friction in the boundary-lubrication of steel contacts based on the formation of different types of tribofilm [20]. We also found in another study that MoS_2 nanotubes are able to reduce the friction even on relatively inert surfaces, such as diamond-like carbon (DLC) [21]. This means that MoS_2 nanotubes perform very effectively on different types of surfaces, obviously predominantly by physical, instead of chemical principles [21]. The surface roughness thus appears to be a very important parameter for nanoparticles-assisted lubrication, since physically based concepts have to be considered. It is therefore surprising that, in spite of many studies investigating the tribological behaviour of nanoparticles in oils, the effect of surface roughness was not investigated in great detail so far.

Accordingly, in this study we have focused on a comparison of the lubrication behaviour of MoS_2 nanotubes mixed with

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polyalphaolephin (PAO) oil in the lubrication of steel/steel contacts of different roughnesses. We have investigated the behaviour of MoS_2-nanotubes-assisted lubrication in all lubrication regimes by also considering the effect of the running-in of these contacts. The results were compared with the pure base oil and it was realized that the friction with the nanotubes-containing oil was 40–65% lower compared to the base oil, depending on the different contact conditions used. Furthermore, by using MoS_2 nanotubes in the oil the friction was the same for the rough and smooth steel surfaces, meaning that the nanotubes completely govern the lubrication behaviour in self-mated steel contacts in the boundary- and mixed-lubrication regimes, irrespective of the surface roughness in the studied range.

2. Experimental details

2.1. MoS_2 nanotubes

MoS_2 nanotubes with typical diameters below 100 nm and lengths up to 20 μm were synthesized by the sulphurization of $Mo_6S_4I_6$ nanowires at 1073 K in a reactive gas composed of 98 vol% Ar, 1 vol% of H_2S and 1 vol% of H_2 for 1 h [24]. During the sulphurization the iodine was completely removed from the starting material and substituted by sulphur. X-ray powder diffraction and X-ray energy-dispersive analyses of the end product revealed an iodine-free MoS_2 compound. The pristine MoS_2 nanotubes kept the original, hedgehog self-assembly of the starting material (Fig. 1(a)), which can be easily dispersed in polar media using ultrasound. High-resolution studies revealed some dome-terminated ends of the tubes, Fig. 1(b). The walls of the tubes are less than 10 nm thick. A relatively high concentration of structural defects is present in the form of sub-cylinders or parts of separated lamellas that are visible inside the nanotubes. It is reasonable to assume that these defects can influence the mechanical properties of the nanotubes, which can, as a consequence, be more easily exfoliated under shear stress.

2.2. Tribological tests

The friction experiments, in which self-mated steel/steel contacts were used, were performed at room temperature using an MTM, i.e., a mini-traction machine (PCS Instruments, UK) with a ball-on-disc configuration, using a 19.05-mm (3/4 in) diameter ball and a 46-mm diameter disc. The ball is loaded against the disc and the ball and disc are driven independently to create the desired slide-to-roll ratio (SRR). The disc was fully immersed in a lubricant and all of the friction experiments were performed at a normal load of 35 N, which corresponds to a maximum Hertzian contact pressure of 1 GPa (mean 0.7 GPa).

For each sample a Stribeck curve (referred to as the 'initial Stribeck') was performed initially with the mean contact velocity (also known also as the entrainment velocity) decreasing from 3.2 m/s to 0.002 m/s (i.e., the transition from the hydrodynamic to the boundary-lubrication regime) and with an SRR of 50%. After the initial Stribeck curve a long-duration (2 h) test was performed at a constant mean contact velocity of 0.05 m/s. At the end of this long-term test another Stribeck curve (referred to as the 'final Stribeck') was performed under the same conditions as those employed initially. Since the applied force and the oil viscosity were kept constant, all the Stribeck curves can be given as the coefficient of friction as a function of the mean contact velocity. The Stribeck curves (for each combination of oil and material pair) were acquired several times and representative measurements are presented in the diagrams.

The mini-traction machine was equipped with an interferometry system to measure sub-micron additive films on the specimens as they form during the test. An optical interferometry measurement is made ex-situ, immediately after stopping the tribological test. To perform the measurement the ball is loaded against the glass disc and an image of the complete contact area is taken with a high-resolution RGB camera. A film-thickness map can be made, which allows film-thickness measurements to be taken of any surface films with a thickness up to 200 nm. In our experiments the balls were imaged after each Stribeck test.

2.3. Material and lubricants

The samples for the experiments, i.e., the balls and the discs, were made from AISI 52100/DIN 100Cr6 steel, both with a hardness of 760 $HV_{0.1}$, measured with a micro-hardness tester (Leitz Miniload, Wild Leitz GmbH, Wetzlar, Germany).

The arithmetical mean surface roughness, R_a, and the root-mean-square surface roughness, R_q, were measured with a stylus-tip profilometer (T8000, Hommelwerke GmbH, Schwenningen, Germany) and an AFM (CP-II, Veeco, New York, USA). The R_a and R_q of the balls were 0.010 ± 0.0005 μm and 0.012 ± 0.0005 μm, respectively. The experiments were performed with two sets of steel discs that differed in terms of the surface roughness. The R_a and R_q for the smoother discs were 0.006 ± 0.0005 μm and 0.008 ± 0.0005 μm, respectively. For the rougher discs the R_a value was 0.040 ± 0.001 μm and the R_q was 0.087 ± 0.01 μm.

The experiments were performed using polyalphaolephin (PAO) oil. According to the supplier (Neste Oil, Espoo, Finland) its kinematic viscosity was 29.0–30.5 mm^2/s at 40 °C and 5.7–6.0 mm^2/s at 100 °C. Some of the experiments were performed using the base PAO oil without any additives (denoted as 'PAO'), while the other experiments were conducted using the same PAO base oil containing 2 wt% MoS_2 nanotubes (denoted as 'PAO + NT'). The suspension of oil and nanotubes was thoroughly

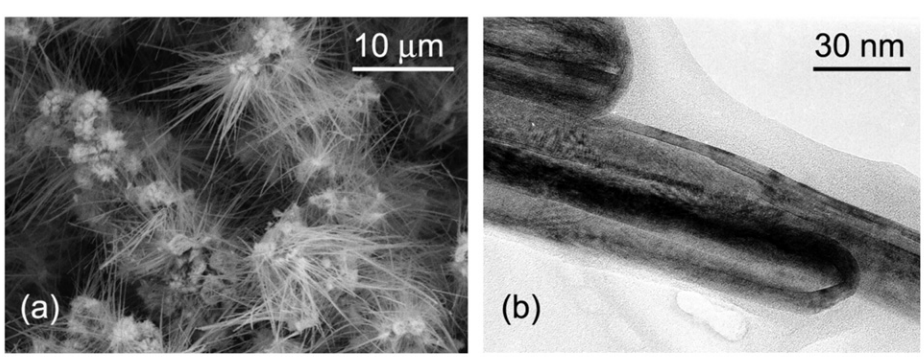

Fig. 1. The MoS_2 nanotubes: (a) SEM micrograph of hedgehog-like self-assemblies and (b) HRTEM micrograph of dome-terminated nanotubes.