Solid Lubrication on Hard Metal Specimens with Micropits Under Normal and Elevated Temperatures

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Abstract
Solid lubrication in tribological applications was studied on hard metal specimens with micropits fabricated using metal injection molding (MIM). This study investigated synergy effects of paraffin wax mixed with 5 wt% MoS₂ on the lubricating potential both under normal and elevated temperatures. Pin-on-plate sliding tests were performed on a CSM tribometer in which WC–Co pins oscillated against microstructured and flat reference WC–Co specimens under 10 N applied normal load. Surface morphology characterization of test specimens was carried out before and after tribological tests using scanning electron microscopy. Solid paraffin wax displayed enhanced lubrication compared to solid paraffin wax mixed with 5 wt% MoS₂ on micropit specimens under normal temperature. On the contrary, under heating conditions, solid paraffin mixed with 5 wt% MoS₂ significantly reduced the dynamic coefficient of friction (COF) values for both flat and micropit specimens. The results showed that the micropits in textured specimens can be used as a reservoir for the lubricant that can significantly reduce friction compared to flat reference specimens.

Graphical Abstract

Keywords MoS₂ · Paraffin · Cemented tungsten carbide · Dynamic coefficient of friction
1 Introduction

Solid lubrication is important for reducing friction and wear between rubbing surfaces in various tribological systems [1]. Solid lubrication can be utilized effectively under extreme conditions of temperature, contact pressure, vacuum, and radiation, where liquid lubricants and greases are ineffective [2]. Solid lubricants are typically applied as a thin film between contact surfaces that can serve as a thin mediating layer between surfaces preventing direct surface contact and damage [3]. Recently surface texturing and patterning have been studied with solid lubricants for enhanced performance [4] with materials ranging from soft polymers to hard metals. In addition, lamellar crystalline materials such as MoS₂ have been studied in combination with microscale surface patterning for enhanced tribological performance [5].

In this paper, solid lubrication was studied in a combination of sample surface texturing with multicomponent lubricants. Surface texturing as micropits provides lubrication storage capacity. Paraffin wax was combined with MoS₂ to study the synergistic effects of layered lubrication additives with surface texturing under solid and liquid conditions in the elevated temperature. Materials with characteristic lamellar crystalline structure having long-range van der Waals forces are typically used in solid lubrication. The layers easily slip or shear over one another in a direction parallel to the sliding motion under stress [6]. MoS₂ is a typical example of such lamellar lubricants that have attracted interest over the years [7].

MoS₂ is a black crystalline material [8] with hexagonal close packing arrangement of atoms similar to graphite [9]. In contrast to graphite, MoS₂ possesses a six-fold symmetry with each unit cell within the crystal lattice consisting of two MoS₂ molecules. The planar structure consists of a sheet of molybdenum periodically sandwiched between two sheets of sulfur [10]. The spatial orientation of atoms in the lattice is such that each molybdenum atom is coordinated by a trigonal prism of six equidistant sulfur atoms at bond lengths of about 1.54 Å from molybdenum. Each sulfur atom is also coordinated to three molybdenum atoms [11, 12]. Atoms within each sheet are bonded together by strong covalent bonds. The bonds between the molybdenum sheets and adjacent sulfur sheets are strongly covalent. On the contrary, adjacent sulfur sheets are bound by weak van der Waals forces at bond lengths of about 3.08 Å. Thus, easy shearing occurs between the sulfur sheets and is followed by low kinetic friction coefficient [13, 14].

In previous studies [6, 15], MoS₂ has been used as an additive to liquid paraffin with enhanced tribological properties. MoS₂ has also been utilized in a study [16] with paraffin grease in boundary lubrication with reportedly enhanced coefficient of friction (COF) values. The lubricity in base oils is reported to be dependent on the morphology and size of the particles as well their additive concentrations [17]. However, little has been reported on the tribological performance of MoS₂ admixed with solid paraffin wax. The present study compares the lubricity of solid paraffin to solid paraffin mixed with MoS₂ on microstructured hard metal specimens under both room temperature and heating conditions. Pin-on-plate kinetic friction tests were carried out on a CSM tribometer under 10 N normal load. Surfaces of the test specimens were characterized using the scanning electron microscope (SEM).

2 Experimental Section

2.1 Sample Preparation

A computer-controlled laboratory-scale microworking robot (Mitsubishi rp-lab robot 1, JP) was used to create a mold insert from rectangular-shaped (64 mm × 12 mm) Ni foils (99.99%, Good Fellow Cambridge Limited, UK) with average thickness of 0.25 mm. Highly ordered micro-cavities of varying sizes and densities were patterned on the Ni foils. Texturing was carried out with a tungsten carbide needle (Fodesco Ltd., Finland) having a tip diameter of 200 μm. Separation between the high- and low-density micro-cavities in a square lattice pattern was 200 μm and 400 μm, respectively (Figs. 1 and 2).

17-4PH stainless steel (PolyMIM GmbH, DE) specimens with ordered high- and low-density micropillars were fabricated using the textured mold inserts in a metal injection molding (MIM) process. Compounding of the feedstock was carried out using a laboratory-scale HAAKE Minijet II (Thermo Fisher, Karlsruhe, DE) injection molding microcompounder. The feedstock was injected into the mold cavity having a cylinder temperature of 191 °C, heating tool temperature of 60 °C, injection pressure of 750 bar, and injection time of 10 s. The green parts (with polymer binder) were debinded overnight in a water bath at a constant temperature of 60 °C. The samples were dried in an oven for 2 h at a constant temperature of 100 °C. The brown parts (without polymer binder) were sintered in a high-temperature furnace (HTK 8 MO/16-1G, Carbolite/Gero, DE) in a hydrogen atmosphere. The sintering cycle as displayed in Fig. 3 was from room temperature to 200 °C at a heating rate of 180 °C/h for 1 h and holding for 2 h, then heating from 200 to 600 °C at 180 °C/h for 2 h 13 min in 2 h holding time, then heating from 600 to 1350 °C at 300 °C/h for 2 h 30 min in 3 h holding followed by cooling from 1350 to 80 °C for 1 h 25 min at 900 °C/h and holding for 15 min and finally cooling from 80 °C to room temperature.
Fig. 1  Fabrication of microstructures on WC–Co hard metal specimens by micro-robot and metal injection molding techniques

Fig. 2  SEM images of metal injection molded WC–Co specimens; a 200 μm, high-density, b 400 μm low-density and c flat reference specimens

Fig. 3  Schematic diagram for sintering of; a 17-4PH and b WC–Co specimens
Micropits (geometric parameters shown in Table 1) with the exact negative of the micropillars were created on cemented tungsten carbide specimens (WC–Co, WC0.8Co13.5, Z360, PolyMIM GmbH, DE) using the as-sintered 17-4PH specimens as mold inserts. The standard deviation of all measured dimensions was below ± 3 µm as all structures were manufactured using the same high precision micro-robot device with the same needle. The average density and Vicker hardness of the WC–Co feedstock used are 14 g/cm³ and 1440 HV10, respectively.

The WC–Co green parts were subjected to similar solvent debinding and drying process as the 17-4PH specimens. After debinding, the obtained brown parts were sintered in a nitrogen atmosphere. The sintering cycle was as follows: heating from room temperature to 100 °C at 100 °C/h for 48 min, heating from 100 to 450 °C at 120 °C/h for 2 h 55 min in 1 h holding time, heating from 450 to 600 °C at 120 °C/h for 1 h 15 min in 2 h holding time, heating from 600 to 1000 °C at 120 °C/h for 3 h 20 min and holding for 1 min, heating from 1000 to 1150 °C at 300 °C/h for 30 min and holding for 40 min, heating from 1150 to 1250 °C at 300 °C/h for 20 min and holding for 40 min, heating from 1250 to 1369 °C at 180 °C/h for 40 min and holding for 2 h followed by cooling from 1369 to 300 °C at 900 °C/h for 1 h 11 min and holding for 1 min, cooling from 300 to 80 °C at 900 °C/h for 15 min and holding for 15 min, and finally cooling from 80 °C to room temperature.

### 2.2 Characterization

Surface morphology analysis of specimens before and after the tribological tests was carried out using the field emission scanning electron microscope (FE-SEM, Hitachi S-4800, JP). Elemental identification was performed using the electron dispersive spectroscopy (EDS). EDS analysis confirmed tungsten, carbide, and cobalt as the main constituents of the WC–Co hard metal specimens in two distinct phases: WC phase and Co phase of cemented tungsten carbides as shown in Fig. 4.

### 2.3 Tribological Tests

Friction and wear tests were carried out on a computer-assisted CSM tribometer for real-time measurements. The tribopairs consisted of WC–Co pins, which oscillated against both flat and microstructured WC–Co specimens. Tests were run under a constant applied normal load of 10 N, a linear acquisition rate of 100 Hz at 100 m sliding distance, sliding speed of 5 mm/s, and an half amplitude of 15.0 mm. Humidity and temperature varied between the range of 35% ± 10% and 25 °C ± 2 °C, respectively, at a standard atmospheric pressure. All measurements were performed under lubricating conditions with two lubricants; solid paraffin and a mixture of solid paraffin and 5 wt% MoS₂. The lubricants were applied manually by first melting a mixture of the weighted amounts of the lubricants. The molten lubricant was then applied onto the surface with the aid of a spatula. The applied lubricant was allowed to solidify on the surface. The solid lubricant on the surface was then exfoliated gently with the flat end of a spatula leaving a thin film on the surface. The solid lubricant was then added to the surface with the aid of a spatula.

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### Table 1 Geometrical parameters of micropits on WC–Co specimen after sintering

| Distance between micropit (µm) | Top diameter (µm) | Bottom diameter (µm) | Depth (µm) |
|--------------------------------|------------------|---------------------|-----------|
| 200                            | 143              | 126                 | 42        |
| 400                            | 141              | 123                 | 52        |

![SEM image with the corresponding EDS spectra of selected spots on WC–Co specimens with WC phase (spot 1) and Co phase (spot 2)](image-url)
3 Results and Discussion

3.1 Effect of MoS2 on Paraffin Lubrication

3.1.1 COF Values Without Heating (Solid Lubrication)

The influence of MoS2 on the lubrication properties of solid paraffin was tested by the addition of 5 wt% MoS2 into solid paraffin, and the results were compared to paraffin in terms of COF values on both flat and textured WC–Co specimens. The measured COF curves in Fig. 5a and b show a lower COF value for solid paraffin lubrication. This is particularly visible with the microstructured specimens that display almost overlapping curves at a COF level of approximately 0.105. A steady increase in the COF values was observed for the flat specimens with paraffin in Fig. 5a similar to all COF curves in Fig. 5b that may result from a reduced lubricant amount between the tip and surface as the distance was increased. Therefore, under normal ambient conditions, solid paraffin functioned better as a lubricant compared to a mixture of solid paraffin and 5 wt% MoS2.

3.1.2 COF Values with Heating (Liquid Lubrication)

Figure 6a and b shows the COF curves in which a heating system was applied to increase the temperature to 55 °C. This kept the lubricants in a molten, liquid form (in the case of solid paraffin) or in the colloidal form (in the case of...
paraffin mixed with 5 wt% MoS2). All measurements were carried out under similar test conditions with an applied normal load of 10 N. A general decrease in the COF values was observed for all test specimens with MoS2 in Fig. 6b as compared to the pure paraffin lubrication in Fig. 6a. On the contrary to solid lubrication in Fig. 5b, a reduction of COF values was observed for the mixed lubricant with paraffin and 5 wt% MoS2. The steady-state COF values for all test matrices were well below 0.100 as displayed in Fig. 6b. The flat reference specimen displayed the lowest COF value at about 0.05.

These results are in good agreement with our previous study [5] in which up to 50 wt% of MoS2 was mixed with a liquid Superlube lubricant. In the case of solid paraffin with heating as presented in Fig. 6a, the COF values were all well above 0.10 with the flat reference having the lowest COF value at about 0.11. This clearly shows the benefit of adding MoS2 in the liquid lubrication, whereas no benefit was observed with solid lubrication.

### 3.2 Effect of Texture on Multicomponent Lubrication with Surface Cleaning

The multicomponent lubrication was also tested on hard metal specimens with chemical treatment of the specimen surfaces after the application of the lubricant to remove the excess amount of lubricant from the surface. The specimen surface cleaning was carried out using a lint-free cloth n-heptane wrapped to spatula that was dipped into n-heptane and the dampened cloth was gently stroked ten times on the specimen surface to remove all films of lubricants. Thus, the only remaining lubricant was located within the microcavities. In the case of the flat specimens, the lubricants were completely removed from the surface, and the results resemble dry sliding conditions. Comparative reciprocating sliding friction measurements were then run on the specimens under both heating and ambient conditions.

#### 3.2.1 COF Values Without Heating (Solid Lubrication)

Figure 7 shows the COF curves of both flat and textured hard metal specimens with solid paraffin (A) and solid paraffin mixed with MoS2 (B) at room temperature after the n-heptane surface cleaning. A general increase in the COF values was observed for all test specimens compared to uncleaned samples, especially for the flat specimens with the highest COF values of approximately 0.3–0.4 in both cases. This was caused by the removal of excess lubricant from the specimen surface by n-heptane. The results also show clearly the positive effect of solid lubrication with microstructured surfaces on as demonstrated by the low COF values recorded for both high- and low-density micropit specimens with both lubricants. The COF value of 0.22 was the same for the high-density specimens under both lubricants. However, a distinct difference existed for the low-density specimens as the COF value with solid paraffin only was lower at 0.12 compared to the value of 0.21 for the paraffin with MoS2. This may result from the high viscosity of the mixed lubricant since the surface roughness of the specimens was rather similar for the low-density specimens. These results clearly show the effectiveness of microstructured specimens for solid lubricants.

#### 3.2.2 COF Values with Heating (Liquid Lubrication)

Figure 8 presents the COF curves obtained from the reciprocating sliding friction measurements on the specimens under heating conditions with solid paraffin (a) and the solid paraffin mixed with MoS2 (b) after cleaning the surface with

![Fig. 7 Average COF curves from three measurements with their standard deviations at 10 N normal load after heptane cleaning of the specimen surfaces with solid paraffin (a) and solid paraffin mixed with 5 wt% MoS2 (b) measured at room temperature](image)
n-heptane. It is evident from Fig. 8a and b that the microstructured specimens had significantly lower COF values compared to the flat specimens. The high- and low-density specimens showed almost perfectly overlapping curves at the same COF value of 0.11 both for paraffin (a) and for paraffin mixed with MoS$_2$ (b). This can be attributed to the meniscus formation of the molten lubricant at the tip of the pin during the oscillatory sliding contact. Thus, the influence of the surface topography of the specimens was suppressed with no clear difference in the recorded COF values. However, the observed low COF values for the microstructured specimens clearly demonstrate the lubrication retention potential of such micropits independent of the chemical specimen surface cleaning.

### 3.3 SEM Analysis of the Tribologically Worn Surfaces

The nature and mechanism of wear occurring on the specimen surfaces were examined using SEM imaging. Reciprocating sliding tests were carried out in a WC–Co/WC–Co contact pair under a normal load of 10 N for all measurements. The surfaces of the test specimens were cleaned with acetone followed by a 2 nm gold spatter coating of the surfaces before the SEM imaging.

Figure 9 shows SEM images of test specimens using solid paraffin (a–c) and solid paraffin mixed with MoS$_2$ (d–f) without heating. As expected from the low COF values, there was almost no observable wear on the specimen surfaces except the mild polishing along the narrow wear track in Fig. 9c and f. One can observe entrapped MoS$_2$ platelets within the micropits as in the SEM images presented in Fig. 9d and e. It was also evident that the micropits were almost completely filled up by the solid lubricant confirming the lubricant reservoir ability of the micropits. Under the heating conditions, without heptane polishing, no clear wearing mechanism were observed on the specimen surfaces.

Representative SEM images for worn WC–Co specimens after reciprocating sliding tests with heptane treatment of the specimen surfaces under heating condition are presented in Fig. 10, i.e., with liquefied paraffin in figure (a–c) and with paraffin mixed with MoS$_2$ in (d–e). Here, a narrow wear track is observed on Fig. 10c and f. This is also supported by the measured higher COF values of these samples presented in Fig. 8a and b as the surface cleaning with heptane removed the lubricant film completely from the surface, especially on the flat reference specimens. However, no visible wear track was observed from the microstructured specimens.

MoS$_2$ has been reported in a previous study [15] as a pressure carrying additive with enhanced load-carrying capacity for liquid paraffin. This may be the reason for the overall low COF values recorded in Figs. 6b and 8a and b in which solid paraffin mixed with MoS$_2$ was used as lubricant with heating. On the contrary, the mixture of solid paraffin and 5 wt% MoS$_2$ in the solid form was highly viscous. This can account for the steady rise in the COF values for all test specimens as shown in Fig. 6b. It was clearly observed that the measured COF curves under heating conditions in Fig. 6a and b were highly stable. This can be attributed to the formation of a meniscus of the liquid form lubricant between the oscillating pin and the specimen surfaces, thus maintaining a lubrication on the surface throughout the measurement. It can also be concluded that the micro-cavities fabricated on WC–Co specimens effectively functioned with solid paraffin as reservoirs only under ambient conditions.
Fig. 9 SEM images of WC–Co specimens after tribological tests at 10 N applied normal load with heptane treatment at room temperature (solid lubrication) for paraffin (a–c) and paraffin mixed with MoS₂ (d–f). 200 μm high-density micropits (a, d), 400 μm low-density micropits (b, e) and flat reference (c, f) specimens.

Fig. 10 SEM images of WC–Co specimens after tribological tests at 10 N applied normal load with heptane treatment and heating at 55 °C (liquid lubrication) for paraffin (a–c) and paraffin mixed with MoS₂ (d–f). 200 μm high-density micropits (a, d), 400 μm low-density micropits (b, e) and flat reference (c, f) specimens.
4 Conclusions

The effect of 5 wt% MoS2 on the lubricating properties of solid paraffin wax was investigated on microstructured MIM WC–Co specimens containing high- and low-density micropits. At room temperature, the lowest COF value for both high- and low-density micropit specimens was observed with solid paraffin as lubricant. On the contrary, the lowest COF values for all test specimens under heating conditions were recorded with paraffin mixed with 5 wt% MoS2 with the flat reference specimens having the lowest value. However, the microstructured specimens clearly outperformed the flat specimens when the excess lubricant was cleaned with n-heptane. Thus, it can be concluded that a good synergy exists between solid paraffin and 5 wt% MoS2 when the lubricant is in the colloidal form rather than in the solid form. Under the used test conditions, all test specimens were highly stable and no significant wear of the surfaces was observed except a slight surface smoothing and polishing of the flat reference specimens. These results can find applications, e.g., in mechanical components operating at high temperatures in which solid paraffin mixed with 5 wt% MoS2 can be used to reduce COF values.

Author Contributions CKD involved in investigation and writing—original draft. KM performed conceptualization, funding acquisition, project administration, and resources. MS participated in conceptualization, funding acquisition, project administration, supervision, and writing—review and editing. JJS carried out conceptualization, project administration, supervision, and writing—review and editing.

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Data Availability All data reported in a graphical format can be requested from the corresponding author.

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Consent to Participate We declare that this work was approved by all participants.

Consent for Publication We hereby confirm that this manuscript is our original work and has not been published nor has it been submitted simultaneously elsewhere. We further confirm that all authors have checked the manuscript and have agreed to the submission.

Ethical Approval Not applicable.

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