



University of **HUDDERSFIELD**

University of Huddersfield Repository

Kubiak, Krzysztof, Mathia, T. G. and Bigerelle, M.

Influence of roughness on ZDDP tribofilm formation in boundary lubricated fretting

Original Citation

Kubiak, Krzysztof, Mathia, T. G. and Bigerelle, M. (2012) Influence of roughness on ZDDP tribofilm formation in boundary lubricated fretting. *Tribology – Materials, Surfaces & Interfaces*, 6 (4). pp. 182-188. ISSN 1751-5831

This version is available at <http://eprints.hud.ac.uk/id/eprint/21596/>

The University Repository is a digital collection of the research output of the University, available on Open Access. Copyright and Moral Rights for the items on this site are retained by the individual author and/or other copyright owners. Users may access full items free of charge; copies of full text items generally can be reproduced, displayed or performed and given to third parties in any format or medium for personal research or study, educational or not-for-profit purposes without prior permission or charge, provided:

- The authors, title and full bibliographic details is credited in any copy;
- A hyperlink and/or URL is included for the original metadata page; and
- The content is not changed in any way.

For more information, including our policy and submission procedure, please contact the Repository Team at: E.mailbox@hud.ac.uk.

<http://eprints.hud.ac.uk/>

Influence of roughness on ZDDP tribofilm formation in boundary lubricated fretting

K.J. Kubiak^{*1}, T.G. Mathia², M. Bigerelle³

¹*ETSI - Institute of Engineering Thermofluids Surfaces and Interfaces, School of Mechanical Engineering - University of Leeds, Leeds LS2 9JT, United Kingdom*

²*LTDS - Laboratoire de Tribologie et Dynamique des Systèmes, CNRS UMR 5513, Ecole Centrale de Lyon, 36 Avenue Guy de Collongue, 69134 Ecully, France*

³*Université de Valenciennes et du Hainaut-Cambrésis, Laboratoire TEMPO, EA4542, Campus Mont Houy 59313 Valenciennes Cedex 9, France*

**Corresponding author: krzysztof@kubiak.co.uk*

Abstract

Influence of initial surface topography on tribofilm formation in ZDDP lubricated contact was analysed. A small displacement fretting tests with sinusoidal motion were carried out in classical sphere/plane configuration. A range of surfaces with different initial roughness were prepared by milling and grinding processes. Tests were carried out using variable displacement method where amplitude of imposed displacement was gradually increased after every 1000 cycles from 2 to 30 μm . The surfaces after tribological tests were measured by interferometric profiler. Main findings confirm that initial roughness has a significant influence on antiwear tribofilm formation in boundary lubricated contact. Tribofilm forms faster and require less energy to activate in case of rough surface obtained by milling process than in case of smooth grinded surface. However, in contact lubricated by oil with ZDDP additive a significant transfer of material occurred from plane to sphere specimen.

Keywords: Zinc Dithiophosphate, ZDDP, Topography, Surface Roughness, Boundary lubrication, Antiwear, Fretting.

Nomenclature

δ - displacement (μm),
 δ^* - displacement amplitude (μm),
 $\Delta\delta^*$ - incremental step of displacement amplitude (μm),
 f - displacement frequency (Hz),
 P - normal force (N),
 Q - tangential force (N),
 Q^* - tangential force amplitude (N),
 p_o - maximum Hertzian contact pressure (MPa),
 a - Hertzian contact half-length (μm),
 N - number of cycles,
 ΔN - number of cycles between the incremental step of displacement,
 S_q - 3D surface root mean square (RMS) roughness (μm),
 S_a - 3D surface average roughness (μm),
 μ - coefficient of friction,
 E - module of elasticity (GPa),
 ν - Poisson ratio,
 $\sigma_{Y(0.2\%)}$ - yield stress (MPa),
 σ_{UTS} - ultimate tensile stress (MPa),
 E_d - energy dissipated at interface per cycle (J),
 V_p / V_s - volume of wear on plane / sphere specimen (m^3).

1 Introduction

Fretting is a phenomenon occurring at interface of two bodies subjected to very small cycling displacement usually in range of a few micrometers. It is mainly caused by vibrations of the system or by cyclic loadings and stresses at interface. In fretting, a relative displacement of the surfaces is very small and in lubricated contact, this will lead to boundary lubrication conditions. Low speed and reciprocating character of motion will prevent formation of

elastohydrodynamic lubricant film [1]. However, due to rubbing action and significant energy dissipation at contact interface an antiwear tribofilm can be formed when zinc dialkyldithiophosphate (ZDDP) or other high pressure additive is present [2, 3 4]. The mechanisms by which the ZDDP tribofilm reduces wear are believed to be [5] (i) protection of surface from corrosion, (ii) its mechanical properties, (iii) support of load, (iv) preferential shearing, (v) removal of hard Fe_2O_3 particles. However, it is important to understand that the formation of tribofilm is a dynamic process, hence the creation rate should be in balance with removal rate of the tribofilm in order to form stable layer and to protect the surfaces.

Depending on the relative displacement of interface, different sliding regimes can be identified. Partial slip sliding regime is associated with a small relative displacement and is characterized by a closed elliptical fretting loop (i.e. evolution of the tangential force (Q) versus the applied displacement (δ)). Fretting scar is characterized by a stick zone in the middle section and sliding zone at the contact extremities. In the partial slip regime the main damage observed at the interface is caused by the cracking phenomenon. For larger relative displacement, full sliding of the contacting surfaces takes place and fretting loop is characterized by a quadrilateral dissipative shape [6]. This gross slip regime is related to the wear damage of contacting surface. Those two fretting regimes and associated fretting loops are presented in Figure 1.

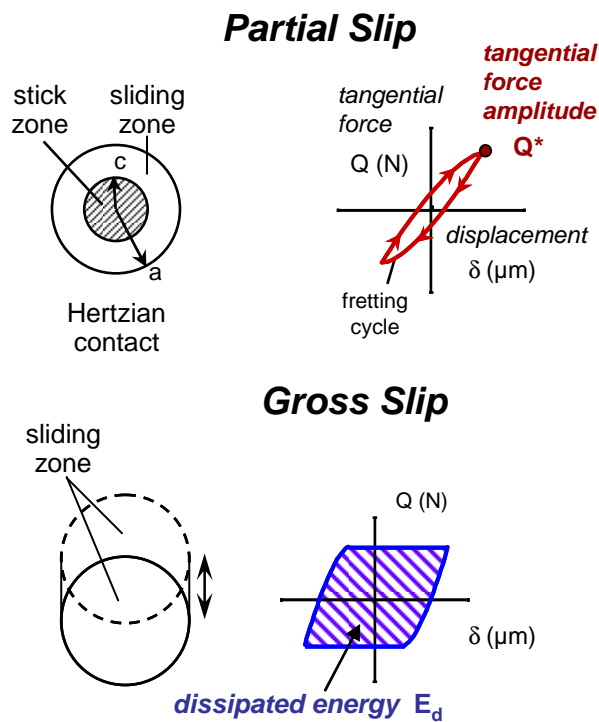


Figure 1: Schematic diagram of sliding regimes in fretting condition, sphere/plane contact configuration is assumed resulting in circular interface of contacting bodies.

Influence of surface roughness on tribofilm formation has been analysed mainly in ball-on-disc configuration under relatively high sliding speed [7, 8]. Also, topography covered was limited to very smooth surfaces in range of few micrometers and coated systems were investigated [9, 10]. Up to best of authors' knowledge, there is no such analysis available in fretting conditions and such high range of

topography which covers surfaces prepared by milling and grinding.

The aim of this research is to analyse influence of surface topography on antiwear tribofilm formation in boundary-lubricated contact such as fretting.

2 Experimental procedures

2.1 Materials and surface preparation

The substrate material used for plane specimens was low carbon alloy AISI 1034. It is very common ferrous material widely used in industry. As a counterbody in fretting tests a ball bearing steel AISI 52100 was used. Chemical composition and mechanical properties of those materials are summarized in Table 1 and Table 2 respectively.

To analyse the influence of surface topography on tribofilm formation a range of rough surfaces were prepared on plane specimens. Topographical features were prepared by milling and wet grinding. Because the surfaces created by milling process are highly anisotropic, also the grinding process was carried out to create a unidirectional surface texture. The direction of sliding in tribological tests were perpendicular to the surface texture. The roughness of prepared surfaces were measured by white light interferometer (Wyko NT3300). Initial roughness was measured using single scan (2.35x1.76 mm), however surfaces with fretting scars were much larger and therefore several scan were taken and stitching function in profilometer software was used to reconstruct entire measured area. Morphologies of initial surfaces prepared on the plane specimens are presented in Figure 2 and roughness parameters in Table 3.

Table 1: Chemical composition of tested materials.

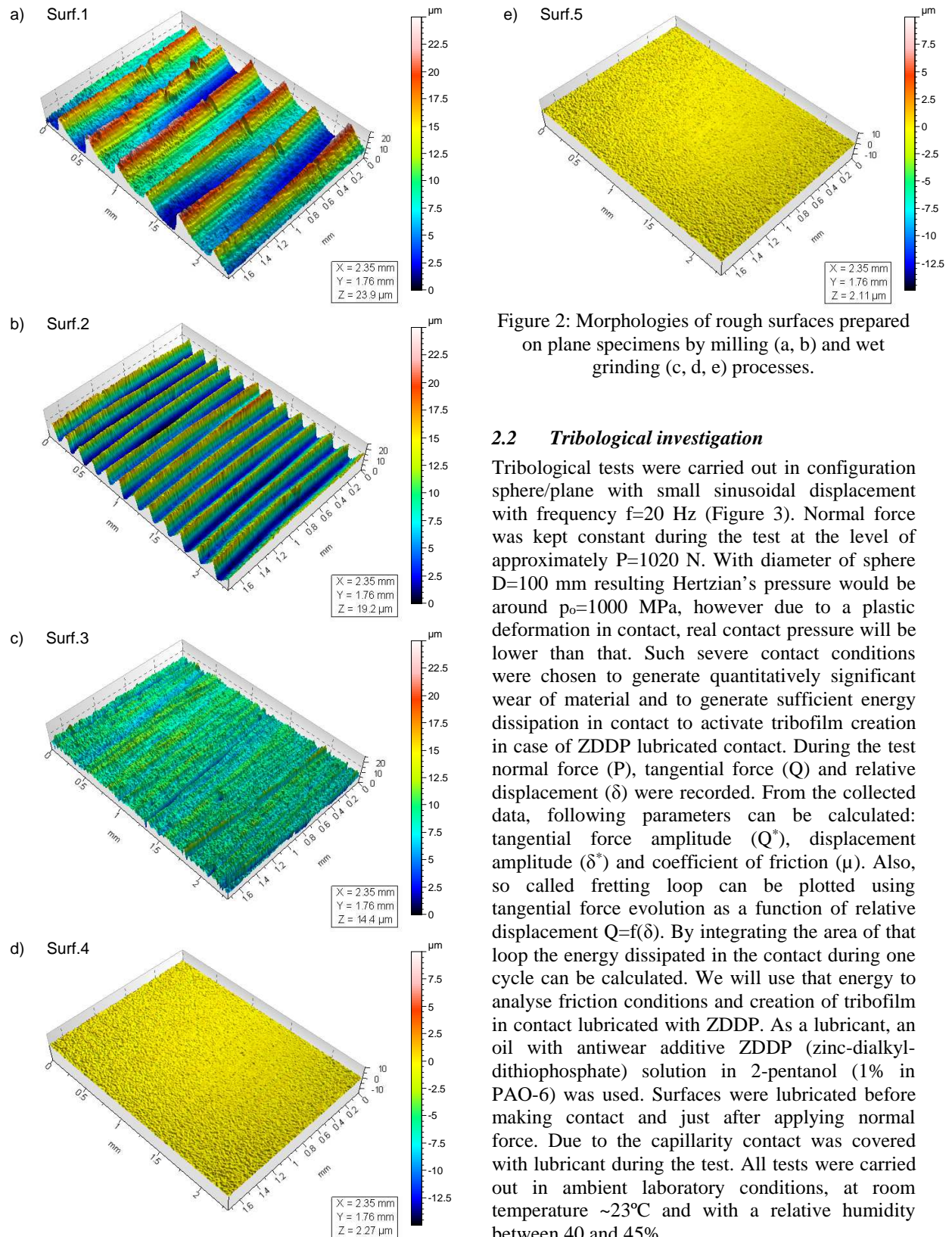
Materials		C	Mn	Cr	Ni	Ti	Cu	Si	P	S	Mo	V
AISI 1034 (plane)	max (%)	0.38	1.2	0.3	0.3	-	0.3	0.5	0.02	0.02	0.08	0.06
5210 (sphere)	max (%)	1.0	0.3	1.5	0.4	1.0	1.0	0.2	0.02	0.02	0.1	0.3

Table 2: Mechanical properties of tested materials.

Materials	E (GPa)	Poisson ratio ν	$\sigma_{Y(0.2\%)} (MPa)$	$\sigma_{UTS} (MPa)$
AISI 1034 (plane)	200	0.3	350	600
52100 (sphere)	210	0.3	1700	2000

Table 3: Initial roughness parameters of tested surfaces.

Surface reference	Surface roughness		Machining process
	$S_a (\mu m)$	$S_q (\mu m)$	
Surf. 1	4.12	5.02	Milling
Surf. 2	3.56	4.14	Milling
Surf. 3	1.17	1.52	Grinding
Surf. 4	0.17	0.21	Grinding
Surf. 5	0.14	0.18	Grinding



2.2 Tribological investigation

Tribological tests were carried out in configuration sphere/plane with small sinusoidal displacement with frequency $f=20$ Hz (Figure 3). Normal force was kept constant during the test at the level of approximately $P=1020$ N. With diameter of sphere $D=100$ mm resulting Hertzian's pressure would be around $p_0=1000$ MPa, however due to a plastic deformation in contact, real contact pressure will be lower than that. Such severe contact conditions were chosen to generate quantitatively significant wear of material and to generate sufficient energy dissipation in contact to activate tribofilm creation in case of ZDDP lubricated contact. During the test normal force (P), tangential force (Q) and relative displacement (δ) were recorded. From the collected data, following parameters can be calculated: tangential force amplitude (Q^*), displacement amplitude (δ^*) and coefficient of friction (μ). Also, so called fretting loop can be plotted using tangential force evolution as a function of relative displacement $Q=f(\delta)$. By integrating the area of that loop the energy dissipated in the contact during one cycle can be calculated. We will use that energy to analyse friction conditions and creation of tribofilm in contact lubricated with ZDDP. As a lubricant, an oil with antiwear additive ZDDP (zinc-dialkyl-dithiophosphate) solution in 2-pentanol (1% in PAO-6) was used. Surfaces were lubricated before making contact and just after applying normal force. Due to the capillarity contact was covered with lubricant during the test. All tests were carried out in ambient laboratory conditions, at room temperature $\sim 23^\circ\text{C}$ and with a relative humidity between 40 and 45%.

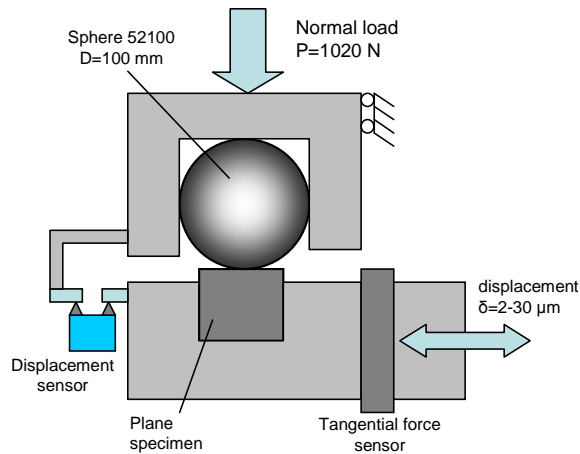


Figure 3: Contact configuration used in tribological analysis.

The test methodology used in this study consist of incremental increase of displacement amplitude ($\Delta\delta^*=0.2 \mu\text{m}$) every 1000 cycles. So that the test starts with small displacement of $\delta^*=3 \mu\text{m}$ and it is gradually increased up to $30 \mu\text{m}$. Schematic diagram of the method is presented in Figure 4. At small displacement, the surfaces are in partial slip and as displacement increases, larger contact area will be in relative sliding. Contact conditions will change from partial slip, through the transition and into gross slip, where entire area of contacting surface is in sliding. In case of contact lubricated with ZDDP, a tribofilm will be formed at the rubbing surfaces due to tribochemical reaction.

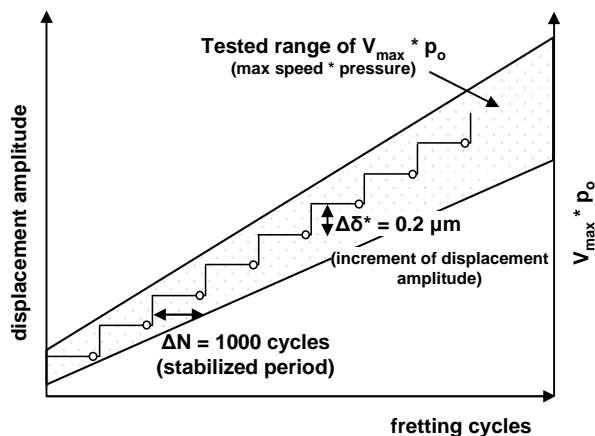


Figure 4: Variable displacement method used in this investigation to analyse friction and initial roughness.

3 Results and discussion

On surfaces prepared by milling and grinding processes a series of tribological test were carried out in dry and ZDDP lubricated contact conditions. Previously published [4] analysis of friction and sliding conditions carried out in very similar contact configuration revealed that maximum value of coefficient of friction was observed at the

transition between partial slip and gross slip. In addition, higher coefficient of friction was noted in ZDDP lubricated contact than in dry friction suggesting that there is some additional mechanism related to formation and removal of tribofilm. In this study, influence of initial surface topography on tribofilm formation will be analysed using energy dissipated at interface. That energy dissipated in contact at every fretting cycle (E_d), corresponds to the area of fretting loop. Therefore, for higher displacement more energy will be provided to system and subsequently dissipated. Presented in Figure 5 is a graph of evolution of dissipated energy in variable displacement test. For dry contact condition it can be noted that after initial activation period energy dissipated in contact increases linearly with fretting cycles. The activation period corresponds to energy required to create tribologically transformed structure [11], activate wear and create wear debris. It has been found [12] that activation energy is higher for smooth surfaces therefore more so called cumulated energy have to be dissipated at the interface to transform the material tribologically and activate wear associated mechanisms. It means more fretting cycles for smooth surface. In case of dry contact configuration after that initial period energy dissipated in contact is increasing as variable displacement test progress (Figure 5). However, this relation during the activation period can not be linear as initially in partial slip, only external part of contact is sliding and central part remains in stick condition. For dry contact, only small variation due to initial surface roughness can be observed and there is no clear trend, but rough surfaces show slightly higher value of dissipated energy.

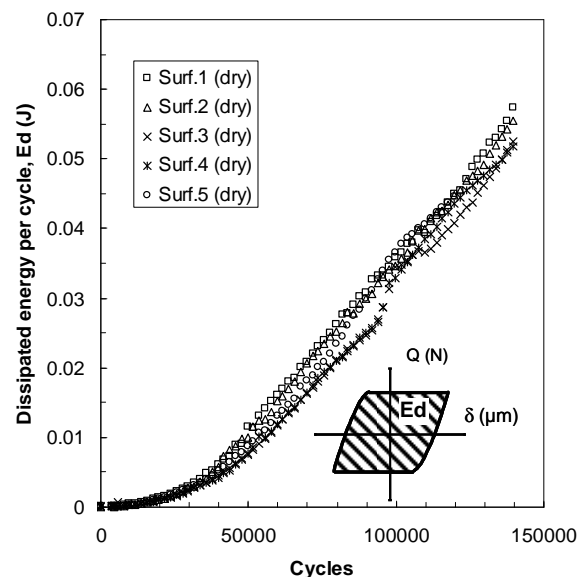


Figure 5: Evolution of energy dissipated (E_d) in a single fretting cycle as a function of fretting cycles, tests conducted using variable displacement method in dry contact configuration.

In case of contact lubricated with oil, containing ZDDP a protecting tribofilm can be formed at the surface of contacting bodies. This tribofilm is commonly known as a 'blue film' due to its intensive blue colour and can be easily identify using simple optical microscopic image (Figure 6).

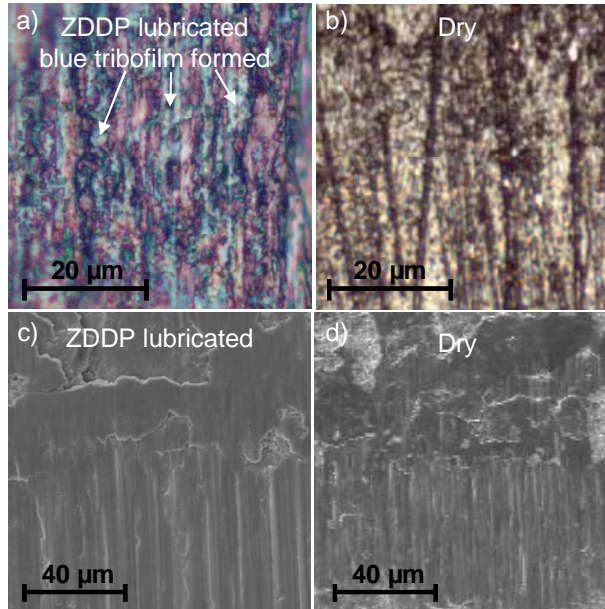


Figure 6: Microscopic images of wear scars: a) optical microscope with blue tribofilm formed at surface, light areas pointed by arrows indicate examples of formed tribofilm; b) optical microscope in dry conditions; c) SEM image of fretting scar with ZDDP lubricated contact; d) SEM image of dry contact.

In case of ZDDP lubricated contact conditions Figure 7 shows that at higher displacement corresponding to 10-20 μm , a tribofilm has been formed leading to lower shear stresses and therefore decreased coefficient of friction is resulting in lower energy dissipation in contact. This time the initial surface roughness has a significant influence on energy dissipation and tribofilm formation. On rough surface tribofilm forms much faster than on smooth surface (Figure 7). Initial conditions before the tribofilm is formed are very similar to condition in dry test, also after the tribofilm formation for all surfaces, dissipated energy converges to very similar value $E_d = 1.48 \times 10^{-2}$ J. However clear influence of initial roughness can be noted. Analysing the local pressure it can be assumed that much higher pressure can be observed at peak asperities in case of rough surface. Also the effective contact area will be smaller due to contact taking place only at roughness asperities. This lead to sliding at lower imposed displacement. Once the contacting surfaces are rubbing a tribofilm can be formed and will successively grow covering the surface in contact and preventing the wear. However, the friction now takes place between the

surfaces covered by tribofilm and therefore mechanical properties of that film will dictate contact behaviour.

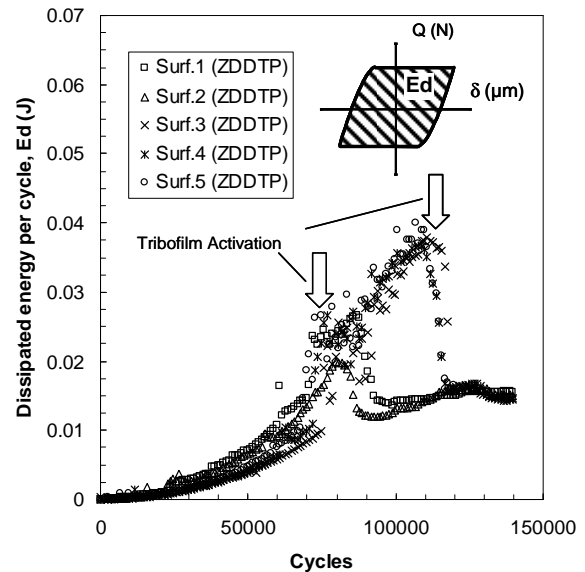


Figure 7: Evolution of energy dissipated in a single fretting cycle E_d as a function of fretting cycles, tests conducted using variable displacement method in ZDDP lubricated contact configuration.

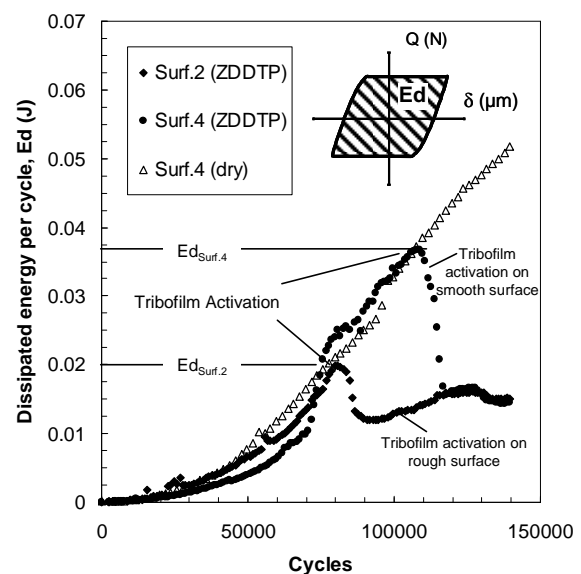


Figure 8: Influence of initial surface roughness on tribofilm formation and comparison with dry contact configuration.

Influence of initial surface roughness on energy associated with tribofilm formation is presented in Figure 8. In first stage of test, the evolution of dissipated energy E_d is similar for dry and lubricated contact up to a point when protecting antiwear tribofilm is formed and E_d is decreasing

significantly.. Analysis of topography of worn surfaces revealed that in dry friction an abrasive wear mechanism is dominant and wear of material can be observed on both plane and sphere specimens (Figure 9). Calculated wear volume of plane surface is $V_p=28.891 \times 10^{-12} \text{ m}^3$ and of sphere $V_s=31.143 \times 10^{-12} \text{ m}^3$ which leads to total wear volume of $60.034 \times 10^{-12} \text{ m}^3$ after dry test. The results where both contacting bodies are worn are usually observed in dry fretting. In this case, a small material transfer from plane to sphere as well as displaced material around the wear scar on plane specimen can be also observed.. However, in lubricated contact very significant transfer of the material can be

observed from plane to sphere. The wear volume of plane specimen is $V_p=60.138 \times 10^{-12} \text{ m}^3$ and the material transferred (negative value of V_s) on sphere specimen is $V_s=-54.674 \times 10^{-12} \text{ m}^3$. Therefore, most of material was transferred to sphere. Mechanism of transfer lays beyond the scope of this paper but it should be noted that the bond created between the tribologically transformed surface and tribofilm is much stronger that resistance to shear stresses of the bulk material of plane specimen. Gradually more and more material is removed and transferred from plane surface to sphere.

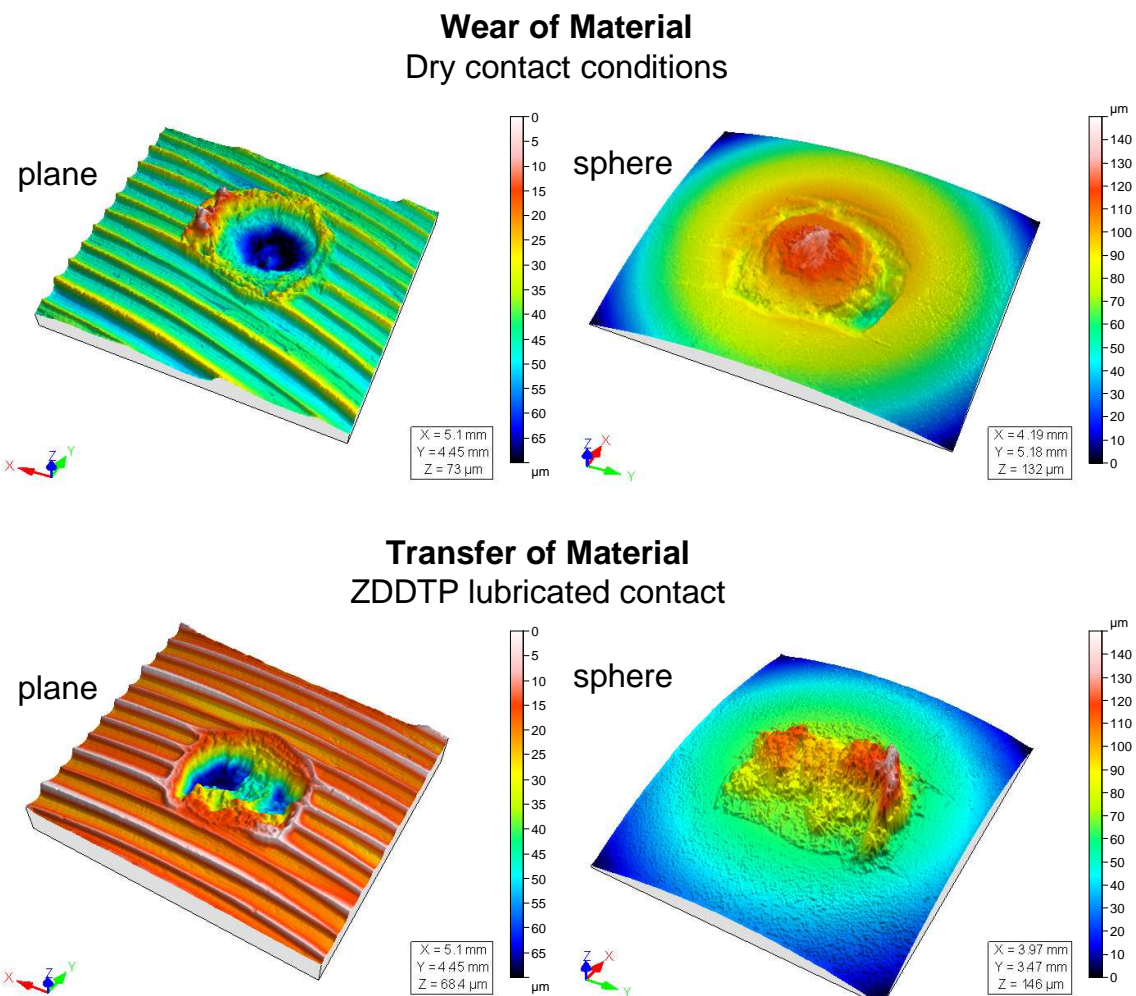


Figure 9: Surface morphologies after fretting test on plane and sphere specimens, wear and transfer of material can be observed for dry and ZDDP lubricated contact respectively.

4 Conclusions

Influence of initial surface topography on tribofilm formation in ZDDP lubricated contact was analysed using small displacement fretting tests. A range of surfaces with different roughness were prepared by milling and grinding processes. Tests were carried out using variable displacement method where imposed displacement was gradually increased ($\Delta\delta^*=0.2\text{ }\mu\text{m}$) every 1000 cycles from 2 to 30 μm . The surfaces after tribological tests were measured by interferometric profiler. Analyzing obtained data the following conclusions can be formulated:

- initial roughness has significant influence on antiwear tribofilm formation in boundary lubricated contact such as fretting conditions,
- tribofilm forms faster and requires less energy to activate in case of rough surface obtained by milling process than for grinded surface,
- transfer of material and therefore higher wear on plane specimen was observed in ZDDP lubricated contact conditions than dry contact conditions.

Presented here results provide initial and interesting inside in the phenomena taking place at interface in lubricated fretting contact conditions, however more detailed analysis of tribofilm formed on rubbed surface using chemical composition equipment e.g. XPS is needed.

5 Acknowledgements

K.J. Kubiak would like to thank Dr Ardian Morina and Dr Siegfried Fouvry for support and fruitful scientific discussions provided for this work.

6 References

- [1] J. Lord, R. Larsson, Film-forming capability in rough surface EHL investigated using contact resistance, *Tribology International*, 2008, **41**, 831-838.
- [2] H Fujita, H A Spikes, The formation of zinc dithiophosphate antiwear films, *Part J: J. Engineering Tribology* 2004 **218**, 265-277.
- [3] J.M. Georges, J.M. Martin, T.G. Mathia, Ph. Kapsa, G. Meille, H. Montes, Mechanism of boundary lubrication with zinc dithiophosphate, *Wear*, 1979, **53**, (1), 9-34.
- [4] K.J. Kubiak, T.G. Mathia, Influence of roughness on contact interface in fretting under dry and boundary lubricated sliding regimes, *Wear*, 2009, **267**, (1-4) 315-321.
- [5] M. Burkinshaw, A. Neville, A. Morina, M. Sutton, Lubrication of aluminium-silicon surfaces with ZDDP and detergents, *Tribology - Materials, Surfaces & Interfaces*, 2012, **6**, 53-58.
- [6] C. Rapiejko, S. Fouvry, B. Grosgeat, B. Wendler, A representative ex-situ fretting wear investigation of orthodontic arch-wire/bracket contacts, *Wear*, 2009, **266**, 850-858.
- [7] F. Svahn, Å. Kassman-Rudolphi, E. Wallén, The influence of surface roughness on friction and wear of machine element coatings *Wear*, 2003, **254**, 1092 - 1098.
- [8] I. Krupka, P. Svoboda, M. Hartl, Effect of surface topography on mixed lubrication film formation during start up under rolling/sliding conditions, *Tribology International*, 2010, **43**, 1035-1042.
- [9] J. Olofsson, J. Gerth, H. Nyberg, U. Wiklund, S. Jacobson, On the influence from micro topography of PVD coatings on friction behaviour, material transfer and tribofilm formation *Wear*, 2011, **271**, 2046-2057.
- [10] P. Harlin, P. Carlsson, U. Bexell, M. Olsson, Influence of surface roughness of PVD coatings on tribological performance in sliding contacts, *Surface and Coatings Technology*, 2006, **201**, 4253-4259.
- [11] E. Sauger, S. Fouvry, L. Ponsonnet, Ph. Kapsa, J. Martin, L. Vincent, Tribologically transformed structure in fretting, *Wear*, 2000, **245**, 39-52.
- [12] K.J. Kubiak, T.W. Liskiewicz, T.G. Mathia, Surface morphology in engineering applications: Influence of roughness on sliding and wear in dry fretting, *Tribology International*, 2011, **44**, 1427-1432.