



Role of Roughness Parameters on the Tribology of Randomly Nano-Textured Silicon Surface

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This experimental work is oriented to give a contribution to the knowledge of the relationship among surface roughness parameters and tribological properties of lubricated surfaces; it is well known that these surface properties are strictly related, but a complete comprehension of such correlations is still far to be reached. For this purpose, a mechanical polishing procedure was optimized in order to induce different, but well controlled, morphologies on Si(100) surfaces. The use of different abrasive papers and slurries enabled the formation of a wide spectrum of topographical irregularities (from the submicro- to the nano-scale) and a broad range of surface profiles. An AFM-based morphological and topographical campaign was carried out to characterize each silicon rough surface through a set of parameters. Samples were subsequently water lubricated and tribologically characterized through ball-on-disk tribometer measurements. Indeed, the wettability of each surface was investigated by measuring the water droplet contact angle, that revealed a hydrophilic character for all the surfaces, even if no clear correlation with roughness emerged. Nevertheless, this observation brings input to the purpose, as it allows to exclude that the differences in surface profile affect lubrication. So it is possible to link the dynamic friction coefficient of rough Si samples exclusively to the opportune set of surface roughness parameters that can exhaustively describe both height amplitude variations (R_a , R_{dq}) and profile periodicity (R_{sk} , R_{ku} , l_c) that influence asperity–asperity interactions and hydrodynamic lift in different ways. For this main reason they cannot be treated separately, but with dependent approach through which it was possible to explain even counter intuitive results: the unexpected decreasing of friction coefficient with increasing R_a is justifiable by a more consistent increasing of kurtosis R_{ku} .

Keywords: Tribology, Roughness, Silicon, AFM, Wettability.

1. INTRODUCTION

Tribology is historically the science of rubbing and is deserved to be considered an ancient craft discipline with a quite modern scientific formulation, as the full interdisciplinary multi-scale description of the interaction between surfaces in relative motion and the involved mechanisms.¹ Motivations surge wherever and whenever friction, wear, lubrication, and related topics assume a huge importance on human life and activities, and the control of such phenomena is required so as to provide strategies for improving item performances and allowing energy and raw material savings at all levels and ranges of applications.

In particular, for proper design of contact surfaces, it is crucial to understand the impact of surfaces roughness and topography on friction. As a matter of fact, engineered surfaces prepared by various machining processes, for both meso-scale objects and micro-nano devices, are not ideally smooth, but with surface irregularities whose amplitudes span from few nanometers to few microns.^{2,3}

Early pioneering works have been shown that the friction between surfaces is substantially affected by the surface texture:⁴⁻⁶ topography and density of peaks/valleys are expected to significantly influence tribological properties, especially when rough surfaces act as lubricated contacts. More recently, regular micro-scale surface texturing^{7,8} and nano-scale surface patterning⁹ have been observed to affect sliding behaviours. Under hydrodynamic lubrication, surface roughness and topography guide the capacity to form

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the lubricant film that fully separates mating surfaces so as to behave as the major load carrying mechanism. Furthermore, under mixed lubrication regime, where the average film thickness is of the same order of magnitude as (or smaller than) surface profile peaks, asperity–asperity contacts alternate with fluid regions between irregularities. Thus, even regarding this scenario, an important role on frictional dissipation is played by peaks amplitude and periodicity, since they determine both the coexisting load supporting mechanisms: contacting performances and lubricant film formation/breakdown.

A still debated question concerns the set of parameters to be exploited for describing random surface textures. Average surface roughness (R_a) draws a very good overall description of profile heights variation and is usually studied together with the average slope of the asperities (R_{dq}). Plenty of published works assert that the load-carrying capacity decreases as R_a and R_{dq} increase, inferring that the increasing of the coefficient of friction primarily depends on the overall shear stresses required to overcome the asperities during sliding.^{4,5,10–12}

However, this latest conclusion is still far to guarantee an exhaustive comprehension of the problem. R_a , R_{dq} (and other strictly interrelated twin parameters) are amplitude parameters that do not sufficiently describe the topography of the surface, because they are purely sensitive to the height deviation from the main profile, but they do not give any information about symmetry, waviness and periodicity. In this sense, recent researches suggest to make use of opportune topography-sensitive parameters: in particular, skewness (R_{sk}) and kurtosis (R_{ku}) were predicted and observed to be strictly related to the load bearing ratio, maximum contact pressure, and effective average lubricant film thickness during sliding contact.^{3,12} Both R_{sk} and R_{ku} are linked to the autocorrelation length (l_c), that is widely recognized as one of the most effective parameter to describe the profile periodicity, since it indicates the statistical distance over which every couple of points can be treated as independent in a random profile.¹³ R_{sk} is a pure number that statistically quantifies the degree of specularity of a surface profile across the main line: $R_{sk} = 0$ describes symmetrical height distribution; positive values indicate the major presence of high peaks above broad valleys; on the contrary, negative values indicate the major presence of deep scratches with the loss of narrow asperities. R_{ku} is a pure number that statistically weights the probability density sharpness of a surface profile across the main line: $R_{ku} = 3$ indicates a Gaussian distribution; $R_{ku} < 3$ indicates the prevailing alternation of broad low peaks and valleys; on the contrary $R_{ku} > 3$ indicates the prevailing alternation of sharp high peaks and scratches.^{14,15}

The aim of the present paper is to give a contribution to the investigation on the not yet clearly defined influence between lubricated friction and surface roughness parameters, through AFM and ball-on-disk characterizations of

rough silicon samples whose surface morphologies were modified with reliable and accurately controlled polishing procedures in order to induce various surface textures with different distributions and shapes of random nano-irregularities. In order to complete the study, contact angle measurements have also been performed for checking the wettability of the same samples, a property which also relates to surface roughness and lubrication. The contact angle measurement is a standard procedure in this context. Depositing a small drop, with size smaller than the liquid capillary length in order to neglect gravity, the angle formed between the tangent of the liquid drop at the contact and the surface itself gives us quantitative information about the hydrophobic/hydrophilic surface behavior.

2. EXPERIMENTAL PROCEDURES

For the purpose of this investigation, well accurate and reproducible mechanical polishing protocols were optimized in order to prepare a set of silicon samples, with different surface roughness and topographies. Commercial flat silicon samples were glued on a piston holder. The holder was loaded through a bound spring, whose elastic force could be calibrated in order to control the normal pressure applied to the sample during machining. In this way, the sample surface is perpendicularly secured to a rotating polishing disk covered by abrasive pads (SiC papers, or velvet rugs imbued by Al_2O_3 slurry). A further device, consisting in a radial arm equipped with a pair of pulleys, was coupled to the piston holder, so that the silicon sample also experienced the autorotation during its rubbing against the abrasive medium. Through this custom rig, reliability, isotropy and uniformity of the produced textures are ensured.

Three different sample were machined (labeled: “180”, “400”, “P3”) keeping constant the operative conditions (normal pressure, lapping speed, polishing time) but varying the abrasive medium. Table I summarizes the manufacturing conditions of each machined sample.

Sample were analyzed through a three-step experimental protocol: microscope imaging, tribological testing, contact

Table I. Main processing parameters exploited during polishing procedure for silicon rough samples.

Sample	Applied pressure [KPa]	Speed rotation [rpm]	Lapping time [min]	Abrasive medium
“180”	7	50	20	SiC paper (grit: 180)
“400”	7	50	20	SiC paper (grit: 400)
“P3”	7	50	20	Al_2O_3 slurry (grain size: 3 μ m)
“FLAT”				Unmachined

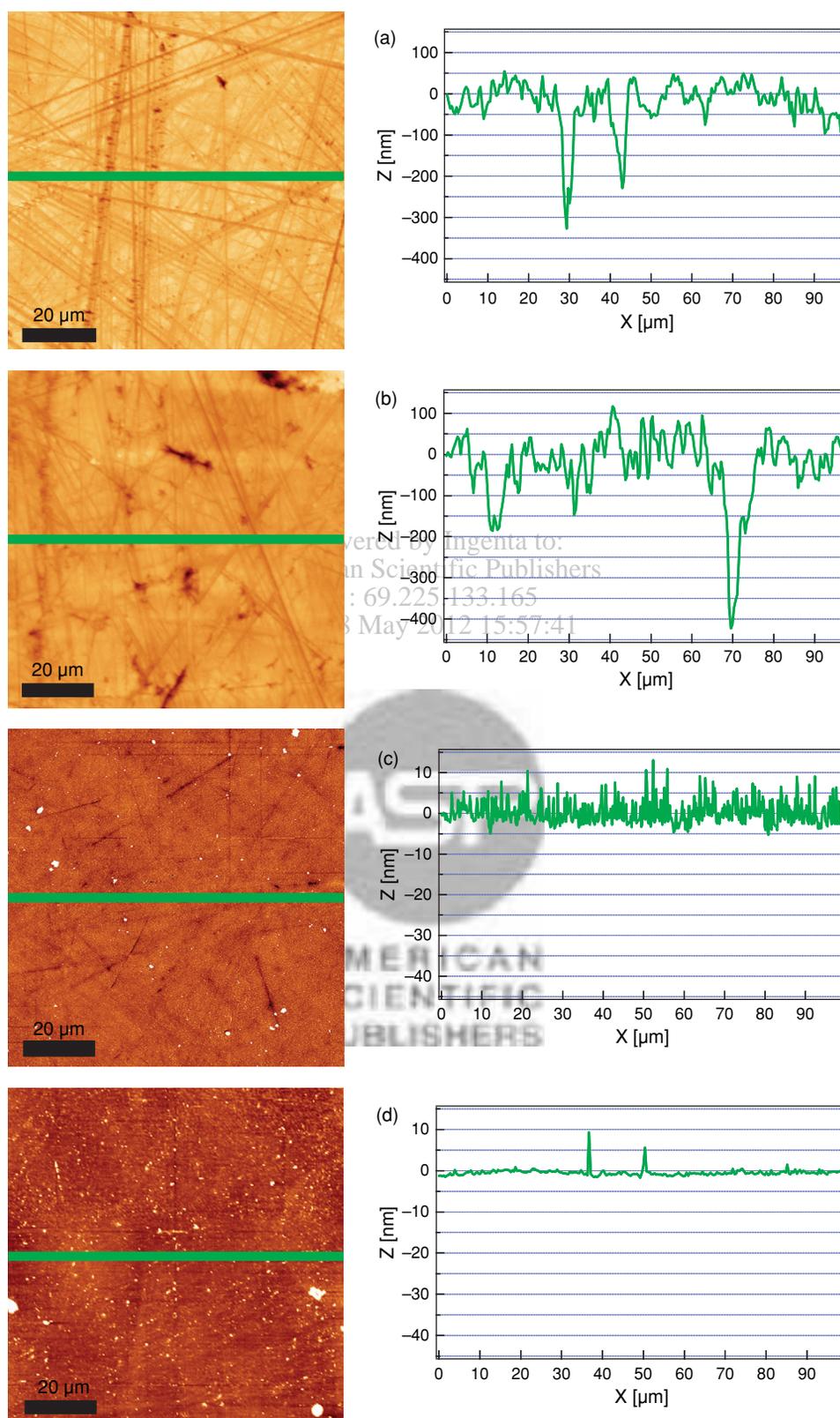


Fig. 1. AFM-based surface characterization. ($100 \times 100 \mu\text{m}^2$ 2D-maps and 1D-profiles averaged on a reduced area (see the stripe over the map). (a) sample "180"; (b) sample "400"; (c) sample "P3"; (d) sample "FLAT". Note that the Z-scale profile relative to "P3" (c) and "FLAT" (d) is reduced by a factor 10 with respect to the Z-scale profile relative to "180" (a) and "400" (b).

angle measurements. A flat unmachined reference silicon sample (labeled: “Flat”) was also characterized.

The tribological campaign was carried out by a ball-on-disk microtribometer (UMT2-CETR). Friction coefficient measurements were performed at constant normal load (250 mN), sliding speed (1.5 cm/s), and elapsed time (1 hour). Steel 100Cr6 balls (diameter: 1.6 mm) were chosen as static counterparts. A bath of distilled water (99% pure) was continuously refilled to act as lubricant between sliding bodies. In order to get a statistically representative collection of friction coefficient data, each sample was tested three times under the same abovementioned conditions. Average friction coefficients with standard deviations as error bars were finally calculated.

Topographical characterization was performed by Atomic Force Microscope (AFM, Veeco Digital Instrument Enviroscope Nanoscope IV). AFM is the most powerful technique for the surface morphological characterization related to its in-plane and out-of-plane spatial resolution. The in-plane resolution is related to the radius of curvature of the tip, stated to be less than 10 nm. To exclude any tip-shape influence on the morphological measurements, the results from two different AFM tips were compared and no differences were found. The vertical spatial resolution is mainly related to the instrumental noise, which has been measured to be ± 0.2 nm. For each sample three separate (100×100) μm^2 areas were scanned through a matrix of (256×256) columns by rows. Such a wide scan size was chosen to include and statistically weight also those structures with low spatial periodicity, and taking into account of the value of the contact area explored in tribological tests (about $500 \mu\text{m}^2$ in agreement to hertzian approximation). Indeed, the values of the measured roughness parameters do not change increasing the spatial density of the scan matrix, acquiring (512×512) pts images and comparing them to (256×256) pts ones. From AFM topographical images, five roughness parameters were monitored and averaged (with standard deviation as error bar) so as to show appreciable differences in dependence of the type of surface preparation. Two of them are height-amplitude parameters: R_a (absolute average height with respect to the midline), and R_{dq} (mean square of average profile slopes with respect to the midline); the other three are R_{sk} (skewness), R_{ku} (kurtosis), l_c (autocorrelation length) and they provide additional topographical information to the previous ones. Other roughness parameters were excluded from the dissertation since they did not show any significant trend, or they can not be considered fully relevant for the average properties of the samples (for example: R_{max} , defined as the maximum height difference between the absolute higher peak and the absolute lower valley, is strongly affected by local defects).

Finally, hydrophobic/hydrophilic behaviours were quantified for each sample through contact angle measurements. A standard single use syringe was exploited to

perform series of five drops of distilled water, deposited on random areas of each ethanol-cleaned sample. Drops volumes spare from 2 to 20 μl . Indeed, drops radii range from 0.8 to 1.6 mm. Hence, average drop sizes are significantly larger than typical dimension of the observed nano-irregularities. For this reason the selection of the sample area to be wetted could be considered almost irrelevant. The contact angle was recorded with a digital photcamera (OLYMPUS MJU 1010) and then measured and statistically analyzed with ImageJ 1.41o software.

3. RESULTS AND DISCUSSION

Figure 1 highlights the main results carried out from AFM-based surface topography characterizations. The comparison among 2D maps and related average 1D profiles shows the peculiar differences of the textures induced by lapping with respect to the commercial flat ones.

Table II summarizes the statistical analysis of the campaign. Samples “180” and “400” exhibit similar values of almost all surface roughness parameters. Also samples “P3” and “Flat” shows similar values of the surface roughness parameters, although very different with respect of those of “180” and “400” samples. Going into details: R_a values attest an increasing of average amplitude variance by a factor 25 from flatter samples (“P3”, “Flat”) to rougher samples (“180”, “400”) which exhibit visible scratches randomly oriented accordingly to the almost isotropic lapping procedure (see Figs. 1(a and b)); on the other hand, topography sensitive parameters outline even more distinct differences between the couple of twin textures: “180” and “400” samples show negative skewness values and quasi-gaussian density sharpness of peak/valleys ($R_{ku} \sim 3$). On the contrary, “P3” and “Flat” samples display an opposite type of waviness with smaller periodicity (since l_c values are lower with respect to “180” and “400” ones) and prevalence of sharp peaks and broad valleys, since R_{sk} values are positive and density sharpness distribution is barely leptokurtic ($R_{ku} \gg 3$).

All water lubricated ball-on-disk tests were nearly under “mixed” or almost “quasi-hydrodynamic lubrication,” since only soft elastic deformations of the counterparts occurred during sliding; in fact, it was impossible to identify wear scars on all the involved sliding counterparts (silicon disks, and steel balls). This observation

Table II. Main results from AFM-based surface characterization. Comparison of surface roughness parameters.

Sample	Height-amplitude parameters		Topography-sensitive parameters		
	R_a [nm]	R_{dq}	R_{ku}	R_{sk}	l_c [μm]
“180”	(38 ± 9)	(0.14 ± 0.05)	(8 ± 6)	$-(1.5 \pm 1.0)$	(1.3 ± 0.5)
“400”	(77 ± 8)	(0.185 ± 0.014)	(5 ± 2)	$-(1.2 \pm 0.6)$	(1.6 ± 0.3)
“P3”	(2.9 ± 0.3)	(0.030 ± 0.002)	(310 ± 90)	$+(11 \pm 3)$	(0.33 ± 0.05)
“FLAT”	(1.4 ± 0.3)	(0.012 ± 0.003)	(130 ± 80)	$+(7 \pm 4)$	(0.41 ± 0.14)

allows to conclude that original textures were not altered, but retained their influence on contact modes during all tests. Thus, the correlation between tribological behaviours and surface topography properties can be considered significant. “180” and “400” twin rougher samples exhibit nearly equal average friction coefficients (respectively: $\mu_{180} = (0.028 \pm 0.001)$, $\mu_{400} = (0.031 \pm 0.003)$) but lower with respect to “P3” and “Flat” twin flatter samples average friction coefficients (respectively: $\mu_{P3} = (0.067 \pm 0.003)$, $\mu_{FLAT} = (0.069 \pm 0.003)$).

In Figure 2, average friction coefficients are plotted against main surface roughness parameters. To rationalize

these data distributions it is necessary, rather than useful, to take into account the competitive contact mechanisms that occur under “mixed” or “quasi-hydrodynamic” lubrication: friction force is the sum of two components since the total normal load is shared by the counterparts asperity interacting force, and the lubricant hydrodynamic lifting force. Thus, the observed decreasing of friction coefficient with decreasing of R_{sk} and R_{ku} (see Figs. 2(c and d)) are in agreement with literature:^{3,12} lower values of skewness and kurtosis optimize maximum contact pressure and effective average lubricant film thickness, so as to favour the hydrodynamic support and minimize asperity–asperity

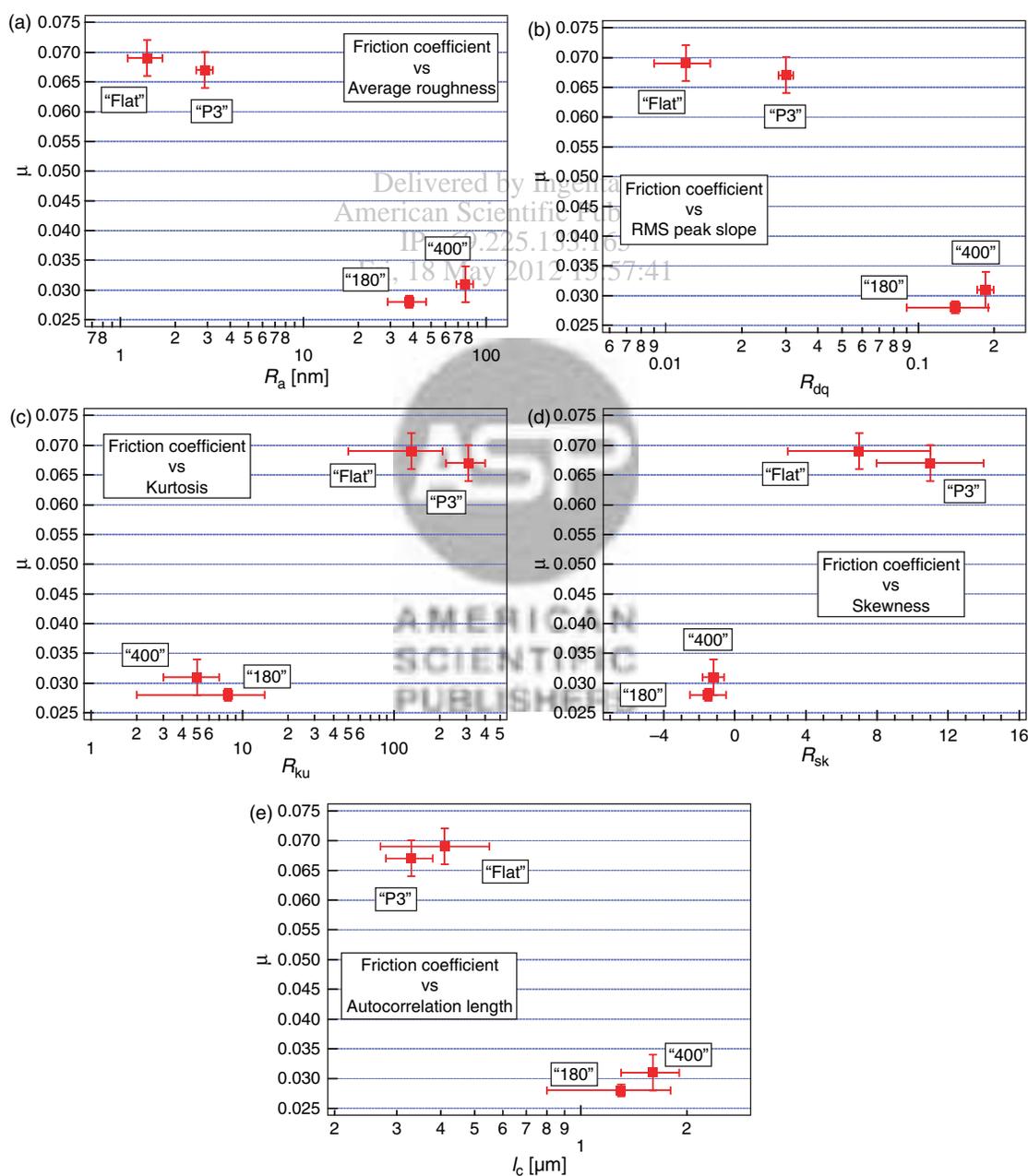


Fig. 2. Tribological characterization. Average friction coefficients of each sample are plotted against main roughness parameters: (a) R_a ; (b) R_{dq} ; (c) R_{ku} ; (d) R_{sk} ; (e) l_c .

interactions. The same considerations could be extended to l_c (Fig. 2(e)) reminding the inverse correlation that links periodicity to R_{sk} and R_{ku} . On the other hand, the decreasing of friction with increasing of R_a and R_{dq} (Figs. 2(a and b)) is in contrast to what expected, since previously reported conclusions^{6,10} infer that higher average profile amplitudes and more pronounced slopes require higher overall shear stresses to be overcome, thus justifying higher frictional dissipations. In order to explain these latest counter intuitive results, height-amplitude parameters and topography-sensitive parameters cannot be discussed separately, but with dependent approach. In fact they are linked to the two competitive components of friction force: R_a and R_{dq} quantify the asperities distribution, while R_{ku} , R_{sk} and l_c describe profile symmetry, waviness, and periodicity, which guide lubrication film formation/breakdown and hydrodynamic lift effectiveness, as explained earlier.

With the support of the available data, a preliminary model was theorized for a numeric interpretation of the discussed correlation between friction and roughness:

For simplicity, the first approximation approach invokes only two of five parameters but no less than representative: R_a (height-amplitude sensitive) and R_{ku} (topography-sensitive).

R_a increases by increasing the peak heights, whereas R_{ku} increases by decreasing the autocorrelation length and thus the wavelength of the profile which lower values are predicted to optimized at least the effective average lubrication during sliding contact.^{3,9} Accordingly, it can be assumed:

$$\mu \propto R_a^\alpha R_{ku}^\beta \quad (1)$$

and thus:

$$\frac{d\mu}{\mu} = \alpha \frac{dR_a}{R_a} + \beta \frac{dR_{ku}}{R_{ku}} \quad (2)$$

Consequently positive or negative variations of the friction coefficient are expected according to:

$$\frac{d\mu}{\mu} > 0 \Rightarrow \alpha \frac{dR_a}{R_a} > -\beta \frac{dR_{ku}}{R_{ku}} \quad (3)$$

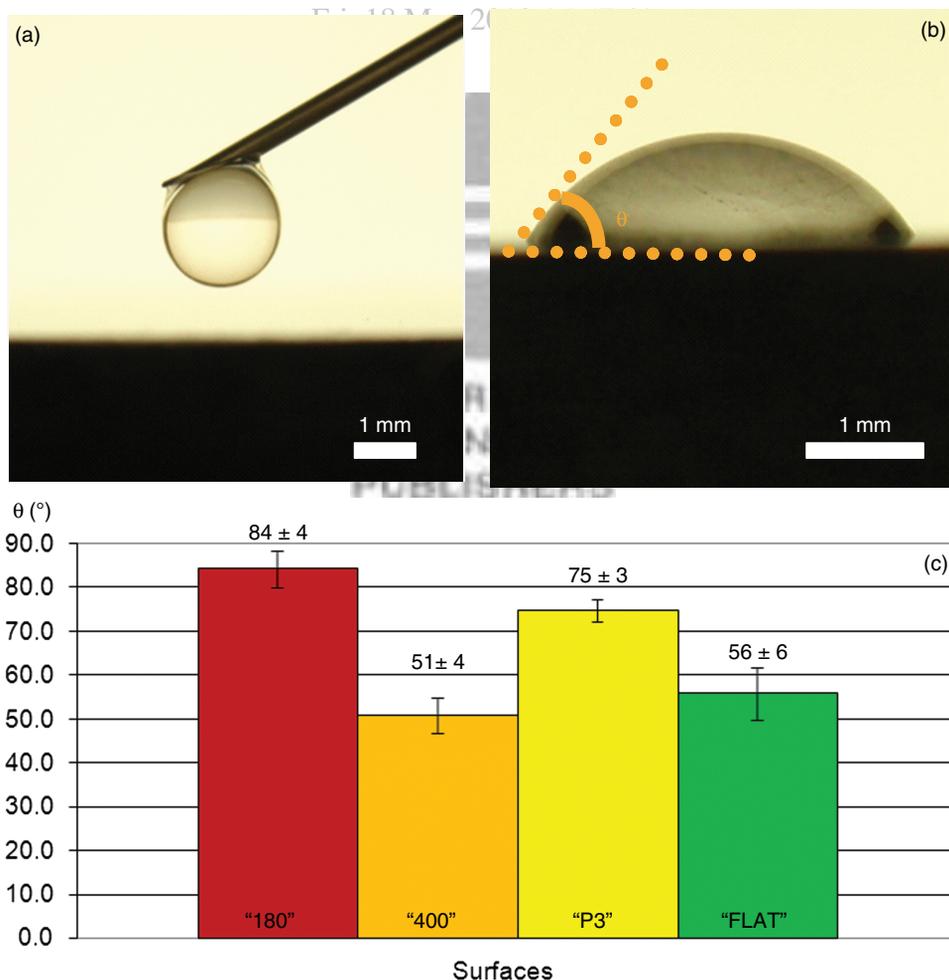


Fig. 3. Contact angle measurements. (a) A random-volume drop of distilled water is deposited with a standard single use syringe on the ethanol-cleaned samples. (b) The contact angle θ is recorded through a digital photocamera, and gives quantitative information about the wettability surface behaviour: hydrophobic surfaces exhibit $\theta > 90^\circ$, hydrophilic surfaces exhibit $\theta < 90^\circ$. Results are shown in diagram (c).

We can rationalize the experimental results with $\alpha \cong \beta \cong 0.2$. Thus Eq. (3) can explain the unexpected decreasing of friction coefficient with increasing R_a by a more consistent increasing of kurtosis R_{ku} .

Contact angles measurement were further investigated in order to understand if friction variation are also linked to wettability behaviours, since it is well known that wettability affects lubrication.^{9,16,17} No significant relationship between roughness parameters and contact angles have been observed (Fig. 3). In particular estimating the Wenzel^{18,19} roughness parameter r as $r \cong (\sqrt{4R_a^2 + (l_c/4)^2}) / (l_c/4)$ as geometrically suggested (and confirmed experimentally by the validity of $R_a \cong (l_c/4)R_{dq}$) the inequality $\cos \theta_{exp} \neq r \cos \theta$ was found, in contrast to what one would expect according to the Wenzel model.¹⁸ Here θ_{exp} is the measured contact angle, whereas θ is the intrinsic one. To rationalize this discrepancy further investigations are running, in particular to check the chemical state of the Si samples, that could also affect wettability.

In any case, this set of characterizations verified no correlation between wettability and tribological properties, as water affinity of the investigated surfaces is not consistently observed to be influenced by the different textures. This further conclusion allows to ascribe the observed friction coefficient behaviours exclusively to the differences in morphology and topography that affect load support mechanisms and thus frictional dissipations under “mixed” or “quasi-hydrodynamic” lubrication regimes.

4. CONCLUSIONS

A set of silicon surfaces with different surface nano-textures was prepared developing a well accurate and reproducible polishing procedure.

A reliable and highly resolved AFM-based surface characterization showed that isotropic method allowed the formation of a wide spectrum of isotropic and uniformly distributed topographical irregularities (from the submicro to the nano-scale) and a broad range of surface profiles. Although gradual changes in both amplitude and topography parameters cannot be obtained through a random micro/nano-structuring process, at least one order of magnitude separated textures were realized, so that some consistent dissertations justified by the two-limits scenario can be argued.

As a matter of fact, ball-on-disk tests highlighted a significant correlation between roughness and “mixed” or “quasi-hydrodynamic” lubricated friction.

Furthermore, roughness and topography were not observed to consistently influence water drop contact-angle. All the samples revealed a hydrophilic character and no clear correlation with height-amplitude and topography-sensitive parameter emerged, allowing to suppose that, in this case, lubrication regimes were not affected by

wettability. Thus the observed differences in friction coefficient can be exclusively ascribed to the differences in surface profile and topography that affect load support mechanisms.

Lower values of topography sensitive parameters (R_{ku} , R_{sk}) optimize maximum contact pressure and effective average lubricant film thickness, so as to favour the hydrodynamic support and minimize asperity–asperity interaction, thus validating previous literature. The same considerations could be extended to l_c .

Apparent counter intuitive results in the correlation between friction coefficient and height-amplitude parameters (R_a , R_{dq}) can be rationalized invoking a dependent approach with topography-sensitive parameters.

In agreement to the obtained data distribution, a first approximation model explains the unexpected decreasing of friction coefficient with increasing R_a by a more consistent increasing of R_{ku} .

In summary, this preliminary model confirms the main idea: for an exhaustive comprehension of the topic, the study of a representative set of surface roughness parameters is necessary. Height distribution (amplitude, slope), and topography (symmetry, waviness, periodicity) cannot be discussed separately, as the former influence asperity–asperity interactions and the latter guide lubrication and hydrodynamic lift effectiveness.

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References and Notes

1. B. Bhushan, *Modern Tribology Handbook*, CRC Press, Boca Raton, Florida (2001), Vol. I.
2. D. Zhu, *Tribol. Trans.* 46, 44 (2003).
3. W.-Z. Wang, H. Chen, Y.-Z. Hu, and H. Wang, *Tribol. Int.* 39, 522 (2006).
4. C. A. Gladman, *Microtechnic* 9, 229 (1962).
5. N. O. Myers, *Wear* 5, 182 (1962).
6. M. M. Koura and M. A. Omar, *Wear* 73, 235 (1981).
7. I. Etsion, *J. Tribol.* 127, 248 (2005).
8. A. Borghi, E. Gualtieri, D. Marchetto, L. Moretti, and S. Valeri, *Wear* 265, 1046 (2008).
9. D. Marchetto, A. Rota, L. Calabri, G. C. Gazzadi, C. Menozzi, and S. Valeri, *Wear* 268, 488 (2010).
10. B. Bhushan and M. Nosonovsky, *Nanotechnology* 15, 749 (2004).
11. P. L. Menezes, Kishore, and S. V. Kailas, *Sadhana* 33, 181 (2008).
12. M. Sedlacek, B. Podgornik, and J. Vizintin, *Wear* 266, 482 (2009).
13. Y. Zhang and S. Sundararajan, *J. Appl. Phys.* 97, 103526 (2005).
14. N. Pugno and E. Lepore, *J. Adhesion* 949 (2008).
15. N. Pugno and E. Lepore, *Biosystems* 218 (2008).
16. Y. Ando, *Tribol. Lett.* 19, 29 (2008).
17. Y. Wang, Y. Mo, M. Zhu, and M. Bai, *Surf. Coat. Technol.* 203, 137 (2008).
18. R. N. Wenzel, *J. Phys. Chem.* 53, 1466 (1949).
19. N. Pugno, *J. Phys. Cond. Mat.* 19, 395001 (2007).

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