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Nano- and microtribological characterization of silanes deposited on cobalt substrate

M. Cichomski[a,*], K. Kośla[a], J. Grobelny[a], W. Kozłowski[b], P.J. Kowalczyk[b,c], A. Busiakiewicz[b], W. Szmaja[b], J. Balcerski[b]

[a] Department of Chemical Technology and Environmental Protection, University of Łódź, Pomorska 163, 90-236 Łódź, Poland
[b] Department of Solid State Physics, University of Łódź, Pomorska 149/153, 90-236 Łódź, Poland
[c] The MacDiarmid Institute for Advanced Materials and Nanotechnology, Department of Physics and Astronomy, University of Canterbury, Private Bag 4800, Christchurch 8140, New Zealand

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ABSTRACT

In this paper, vapor phase deposition was used to grow fluoroalkylsilane films on cobalt surface. The films were characterized by contact angle analyzer for hydrophobicity and X-ray photoelectron spectroscopy (XPS) for identification of fluoroorganic monolayers deposited on the surfaces. Adhesion and friction measurements were performed using atomic force microscope (AFM) and compared with measurements made by microtribometer operated in millinewton (mN) applied load range.

Nano- and microtribological measurements show that cobalt modified by fluoroalkylsilanes has lower adhesion and coefficient of friction. The investigation also indicates a decrease of friction coefficient with increasing fluoric alkyl chain length. Covalently bonded fluoroalkylsilanes with longer alkyl chains are found to be a prime candidate for practical use as a lubricant.

1. Introduction

Cobalt based alloy substrates have been extensively investigated for several years because of their potential application in many technological fields. They are used as new materials in microelectromechanical systems (MEMS), especially in magnetic actuators field. They are also used in biomedical implants because of their excellent mechanical properties, corrosion resistance and biocompatibility [1].

Large adhesion, friction and wear in the case of MEMS can cause some limitations in their production and potential use. For the purpose of better mechanical and chemical properties, their surfaces are modified with lubricants. An ultrathin lubricant film minimizes adhesion, friction and wear between surfaces in contact. One group of the lubricants used in MEMS are silanes. Fluoroalkylsilanes as a self-assembled monolayer (SAM) consist of three building groups: a head group that reacts with a substrate, a backbone molecular chain group, and a terminal group that interacts with the outer surface of the film [2,3]. Fluoroalkylsilanes are formed through hydrolysis reactions, to make bridging siloxane bonds that anchor to the surface and also crosslink to form stable interconnections with adjacent molecules [4–10]. In recent research, the modification of surface properties and functionality through changes in the terminal group has been studied [11–13]. The terminal group determines the surface character. The change of the surface character from hydrophilic to hydrophobic results in decreasing capillary force, better thermal stability, enhanced wear resistance and reduced van der Waals interactions. Such change could be achieved by the coverage of the substrate with monolayer of fluoroalkylsilanes [14–17]. Maboudian et al. [18] reported that perfluorinated silanes show an increase of thermal stability in comparison with nonperfluorinated silanes.

In this paper, we report a comparative study of the nano- and microscale properties of nonperfluorinated and perfluorinated silanes with different carbon chains length. Properties of fluoroalkylsilanes on Co/Si, such as hydrophobicity, surface free energy, adhesion and friction, are evaluated by contact angle measurements, atomic force microscopy (AFM), microtribometry, and X-ray photoelectron spectroscopy (XPS). In this study, for the first time Co substrate was coated using fluoroalkylsilanes.

2. Experimental

2.1. Sample preparation

Silicon pieces were cut from a commercial p-type Si wafer (Cemat Silicon S.A.) and cleaned using ethyl alcohol, de-ionized water and dry argon (Ar) gas. Polycrystalline cobalt films with the thickness of 100 nm were deposited on oxidized Si(1 0 0) substrates, using the process of thermal evaporation at an incidence angle of 0° (with respect to the surface normal direction) in a system maintained at a base

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* Corresponding author. Tel.: +48 42 6355836; fax: +48 42 6355832.
E-mail address: mcichom@uni.lodz.pl (M. Cichomski).

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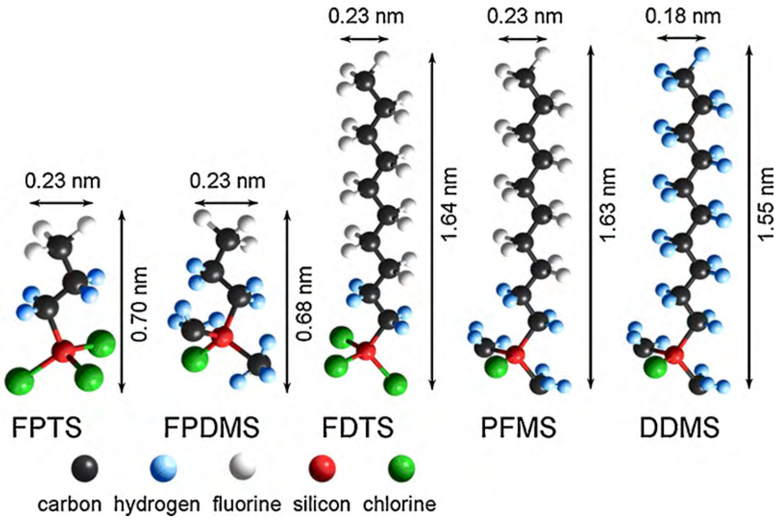

Fig. 1. Chemical structure of various fluoroalkylsilanes deposited on Co substrate.

pressure of about 10^{-5} mbar. The film thickness was determined by a quartz crystal microbalance. The next preparation step was the use of oxygen plasma to activate the surface before modification by fluoroorganic compounds.

2.2. Modification procedure

Deposition process parameters were optimized to produce the most hydrophobic surface possible. Static contact angle was measured to determine the degree of the hydrophobicity. Based on the measured contact angle, optimum deposition conditions were selected [19]. The cobalt surface activated by oxygen plasma was placed into a vapor phase deposition system and kept under low pressure (0.1 Pa). Then the specimen was kept in the modifier vapor for 20 min at room temperature and finally outgassed at low pressure for 1 h at 40 °C to remove any of physisorbed and unreacted molecules [20,21]. The samples were removed from the vacuum chamber and transferred into a vacuum desiccator until characterization.

The modification of the Co substrates was performed with five kinds of fluoroalkylsilanes precursors (Fig. 1): 1H, 1H, 2H, 2H perfluorodecyltrichlorosilane (FDTS), 1H, 1H, 2H, 2H perfluorodecyldimethylchlorosilane (PFMS), n-decyldimethylchlorosilane (DDMS), (3, 3, 3 trifluoropropyl)trichlorosilane (FPTS) and (3, 3, 3 trifluoropropyl)dimethylchlorosilane (FPDMS). These chemicals were chosen to compare the effect of chemical structure on tribological performance at the micro- and nanoscale. The effects of reduced bonding at the surface, carbon chain length, as well as the number of fluoride atoms in the chain were investigated. All precursors were ordered from the ABCR, GmbH & Co. KG, Karlsruhe.

2.3. Measurement techniques

2.3.1. Contact angle and surface free energy measurements

Static contact angles (SCAs) were measured in air by use of a sessile-drop method using a contact angle goniometer. A drop of proper liquid (water, glycerine and diiodomethane) was deposited on the substrate with the use of microsyringe. The image of the droplet was obtained by a digital camera. Images obtained were analyzed using Motic 2.0 software. All measurements were performed at (45 ± 5%) relative humidity and (22 ± 2) °C. The surface free energies of Co and surfaces modified by fluoroalkylsilanes were calculated using the Van Oss–Chaudhury–Good method [22].

2.3.2. Nanotribological characterization

The adhesion and friction measurements were performed with an AFM apparatus (Solver P47, NT-MDT) operating in air under ambient conditions. Nanotribological measurements were performed using a rectangular Si_3N_4 cantilever with a spring constant calibrated by the Sader method (k = 0.62 N/m) [23,24]. The adhesive force was obtained from the force–distance curve after reckoning the pull-off force [25–27]. The friction force was calibrated using the method described by Ruan and Bhushan [28]. The coefficient of friction was obtained from the slope of the friction force versus normal force plots. Applied loads typically ranged from 5 to 100 nN. The mentioned plots were linear in the investigated range of applied load. Friction force measurements were performed at a scan rate of 1 Hz and a scan size of 1 μm × 1 μm. Each measurement was repeated three times on different places of the sample surface. The obtained values of coefficient of friction were comparable. The average data are presented.

2.3.3. Microtribometer measurements

To compare the AFM and microtribological investigations, the samples were frictionally tested on a reciprocating ball-on-flat microtribometer, constructed in the Department of Chemical Technology and Environmental Protection, University of Łódź, Poland [21,29]. The microtribometer was operating in ambient conditions (relative humidity 45 ± 5% and temperature 22 ± 2 °C). Measurements were performed with the use of silicon nitride sphere (Si_3N_4) with diameter of 5 mm over a normal force range from 30 to 80 mN. The ball moved parallel with respect to the sample surface with velocity of 0.42 mm/s and the traveling distance of 5 mm. Each series of measurements were repeated three times in three different locations of the sample surface. The adhesive force was calculated from the negative horizontal intercept of the friction force versus applied load curve (the negative applied load value where the friction force is zero) [30]. The average data are presented.

2.3.4. X-ray photoelectron spectroscopy

X-ray photoelectron spectroscopy was carried out using Omicron UHV system working at the base pressure lower than 5×10^{-8} Pa, equipped with the EA 125 HR hemispherical analyzer with the resolution better than 0.8 eV. The XPS investigations were carried out using Mg K$\alpha_{1,2}$ line with the power set at 75 W in all experiments. The two-point correction of the energy scale based on Au $4f_{7/2}$ (83.95 eV) and Ag $3d_{5/2}$ (368.22 eV) lines was applied to all spectra [31]. The XPSPEAK package was used to perform the quantitative analysis.

3. Results and discussion

3.1. Contact angle and surface free energy studies

Fig. 1 shows the chemical structure of five types of SAMs used in this study. The thicknesses of layers determined from the ellipsometric measurements on the cobalt surface modified by different compounds are comparable with the values obtained by the theoretical model determined using HyperChem 7.5 (Fig. 1). For FDTS, PFMS, FPDMS, FPTS and DDMS, they were found to be 1.95, 1.8, 0.71, 1.27 and 1.68 nm, respectively. These molecules were composed of vertical stacked chains in the trans–conformation of the close packed ~4.5–5 groups/nm^2 [32]. The thickness of FPTS layer on the cobalt surface is much larger than the theoretical value. This is probably due to vertical polymerization and formation of multilayers and agglomerates of the FPTS compound as well as ellipsometry measurement error (it could be up to ±0.5 nm).

Water contact angle and surface energy were measured to quantify the surface hydrophobicity and these results are compared in Fig. 2. All the fluoroalkylsilanes deposited on Co substrate increased the water contact angle, implying a higher surface hydrophobicity and directly proving the presence of the modifier on the surface. The compounds with fluorocarbon terminal group and backbone chain show a higher contact angle and lower surface free energy than methyl groups. Molecules with

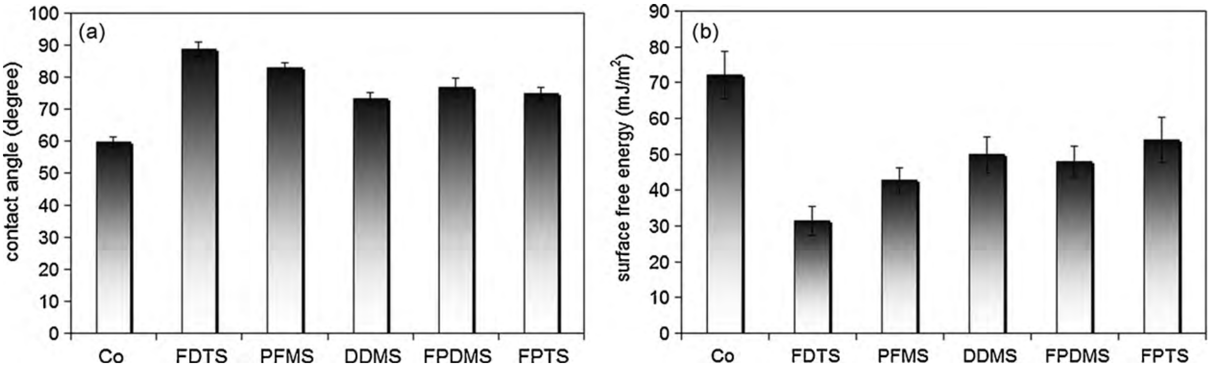

Fig. 2. Data before and after modification by fluoroalkylsilanes: (a) contact angle and (b) surface free energy.

longer hydrofluorocarbon chains exhibit higher contact angles and lower surface free energy than molecules having short chains. The FDTS film on the Co surface shows the largest value of SCA (∼90°) among the perfluoroalkylsilanes. From the measured values of this study, it can be observed that the trifunctional silane (FDTS) exhibits a higher degree of hydrophobicity than the monofunctional fluoroalkylsilanes (PFMS). The difference with monofunctional fluoroalkylsilanes is likely due to the angle of molecular attachment that results from a single covalent bond between the silane and the surface compared with the trivalent formation of a cross-linking between monomers as well as hydrogen bonds between silanols groups, as suggested by Genzer et al. [33].

It is well established that the surface roughness has an effect on the SCA. Table 1 shows the root mean square (rms) surface roughness values measured using AFM for various fluoroalkylsilanes. The rms roughness value of the Co samples was measured as 0.70 nm. After treatment of the Co surface by different fluoroalkylsilanes compounds, the roughness increased. The rms roughness values of the FDTS, PFMS, DDMS, FPDMS and FPTS films were measured as 1.62, 1.55, 1.51, 1.45 and 1.37 nm, respectively. These data suggest that monolayers have an effect on the surface roughness, and consequently have an effect on the value of the SCA. In other words we can say that the first factor to obtain a hydrophobic surface is its roughness. The second factor determining the change in the value of the static contact angle is chemical composition, as expected.

3.2. XPS results

To additionally confirm the presence of modifying agents on the surface and to verify the correctness of the modification procedure, XPS measurements were performed.

The results of XPS investigation are summarized in Figs. 3 and 4. Survey spectra recorded on clean cobalt substrate and fluoroalkylsilanes samples are shown in Fig. 3. In the case of the pure cobalt surface, the spectra inform about the presence of oxidized forms of cobalt (CoO) on the surface. The cobalt surface is mainly in low oxidation state, indicated by shake-up characteristic for Co^{2+}. The presence of oxidized forms on the surface enabled us to perform chemical modification of the surface by different compounds. Peaks for the unique elements such as fluorine, F $1s_{1/2}$ and F KLL, which were observed only after the deposition of fluoroalkylsilanes compounds, confirm the presence of these molecules on the Co surface after modification. For all fluoroalkylsilanes, the intensity of the F $1s_{1/2}$ peaks increases with increasing carbon chain (FDTS, PFMS). The intensities of C 1s, Si $1s_{1/2}$, Si $2s_{1/2}$ and Si $2s_{3/2}$ were significantly higher than those of the Co surface. Also the intensity of Co, specific to the substrate, decreased after fluoroalkylsilanes deposition.

The low intensity peaks of Si $1s_{1/2}$, Si $2s_{1/2}$ and Si $2s_{3/2}$, similar to the literature results [34], are presented for the structure of siloxane (–Si–O–) film on tantalum oxide. Specifically, the bonding between silane and metal oxide mainly occurs through Si–O–metal linkages [34]. In the literature, the peak of Si 2p at 102 eV was assigned to Si–O–Si cross-links [35–39] and the peak at 102.6 eV was assigned to Si–O–metal linkages [36,40,41]. In our study, the peak of Si 2p was observed at 102.8 eV, which also suggests the formation of Si–O–Co linkages. More detailed studies about surfaces modification by all five kinds of modifiers are shown in Fig. 4. Comparison of the CoO peak location for fluoroalkylsilanes with trifunctional group shows the displacement from 780.6 eV (CoO) to 780.9 eV (FDTS). In the case of other fluoroalkylsilanes with longer length, the peak displaces to 781.2 eV (PFMS), 781.3 eV (DDMS). For fluoroalkylsilanes with short carbon chain length and with trifunctional group the peak displaces to 783.5 eV (FPTS), and in the case of monofunctional group the peak displaces to 781.5 eV (FPDMS). This also suggests the formation of Si-O-Metal linkages, and in the case of compounds with trifunctional groups at least two such groups form bonds with the surface. An example of the occurrence of such a horizontal fluoroalkylsilanes polymerization has been shown by Fadeev and McCarthy [32].

Table 1
Surface roughness values of various samples.

Sample	Co	FDTS	PFMS	DDMS	FPTS	FPDMS
rms (nm)	0.70	1.62	1.55	1.51	1.37	1.45

3.3. Nano- and microtribological studies

The measurements of adhesion force and coefficient of friction at the nanoscale performed under ambient conditions are illustrated in Fig. 5. The adhesion and coefficient of friction were always higher for the unmodified surface, compared to those of fluoroalkylsilanes modified surfaces. Among the fluoroalkylsilanes, FDTS showed the lowest adhesive force and coefficient of friction. The decrease of the adhesive force and coefficient of friction is due to the occurrence of longer chains in the deposited films. The SAMs with longer chains produce a less flexible monolayer. When the AFM tip penetrates such a layer, the stronger inter–chain interaction causes a smaller contact area for the same applied load during sliding compared to films with short carbon chains. Thus, for these SAMs the adhesive force is lower.

FDTS, which has the highest water contact angle, showed the lowest adhesion. This tendency of adhesion is very consistent with the change in surface energy. This behavior arises from the fact that monolayers having larger chain lengths are more ordered and more densely packed in comparison to monolayers having smaller chain lengths. As a consequence, longer chain monolayers enable

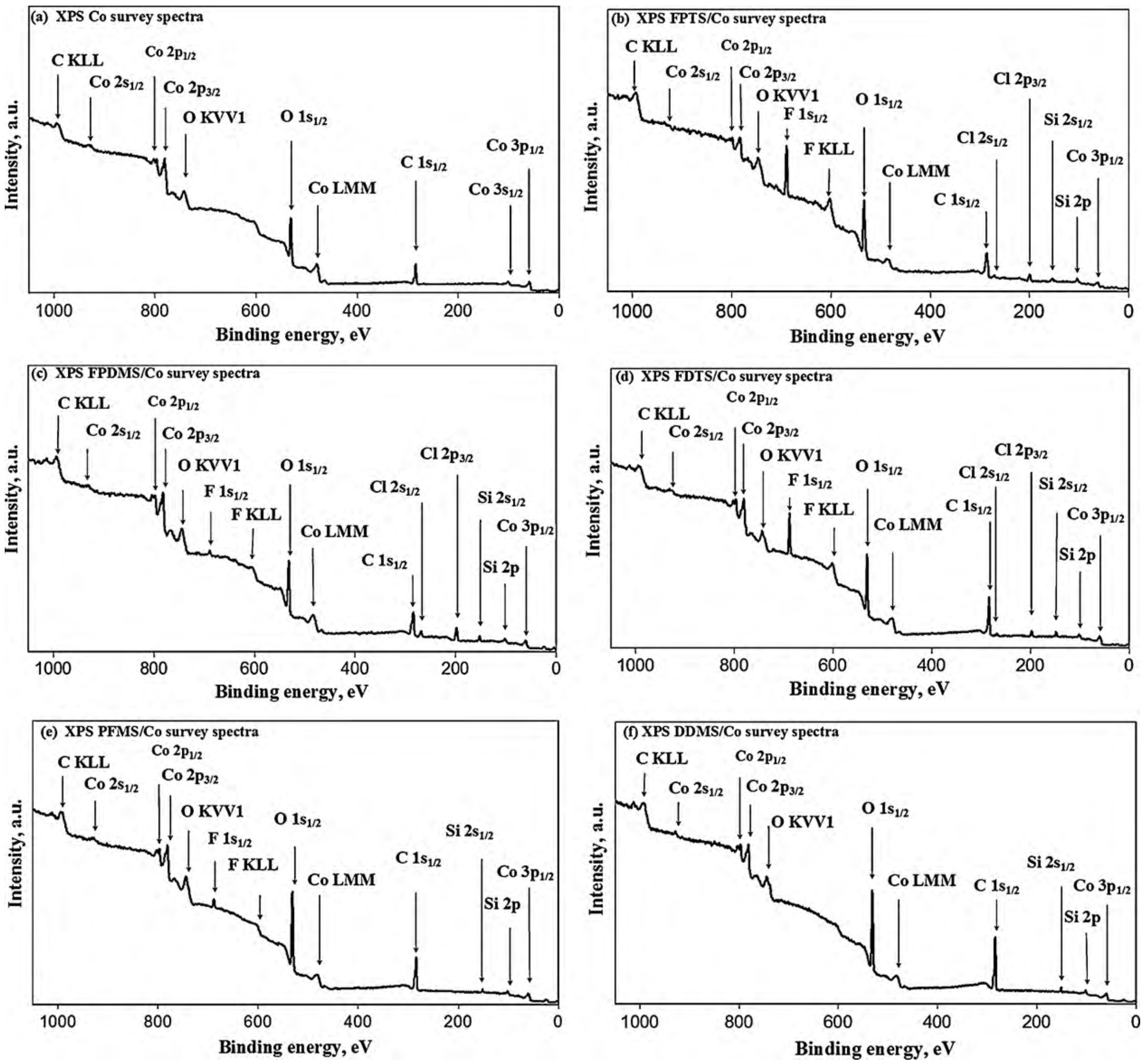

Fig. 3. XPS spectra from Co surface (a) before and after modification by: (b) FPTS, (c) FPDMS, (d) FDTS, (e) PFMS, (f) DDMS.

molecular order to be maintained during sliding, that leads to a lower friction.

The silanes with fluorocarbon terminal group (FDTS, PFMS) exhibit lower coefficient of friction than compounds with methyl group (DDMS). The differences between these compounds were considered to be the effect in the packing density and defects of the alkylsilanes [41]. The larger size of the F atoms compared to the H atoms suggests a more densely packed layer. The number of defects present in SAMs depends on the local ordering and packing density of the film. Defects such as gauche defects in the chains or vacancies in the films increase the coefficient of friction. Mikulski and Harrison [41] studied alkane chains on diamond, comparing fully packed

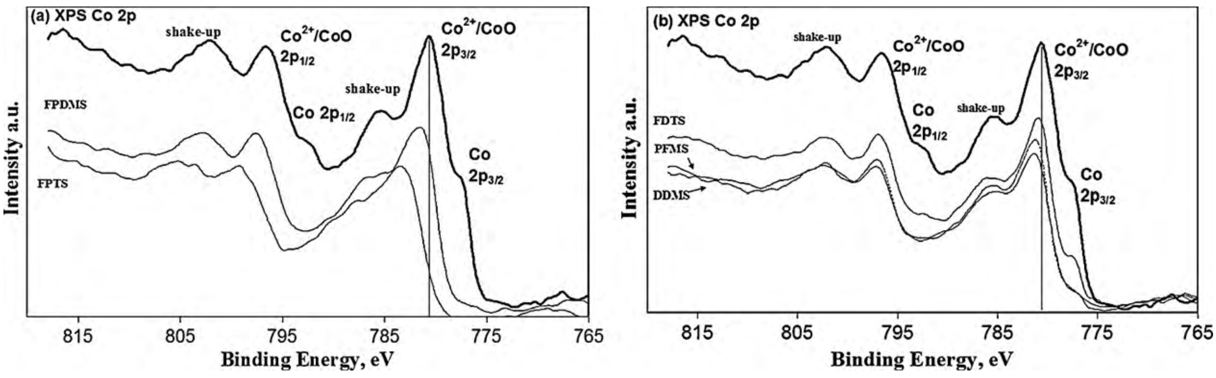

Fig. 4. XPS spectra from Co surface modified by fluoroalkylsilanes with: (a) small chain length and (b) large chain length.

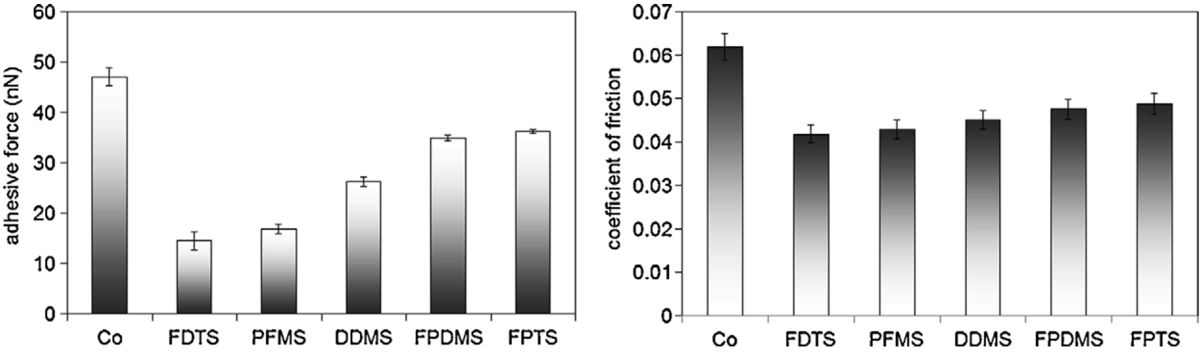

Fig. 5. Nanotribological data: (a) adhesion and (b) coefficient of friction.

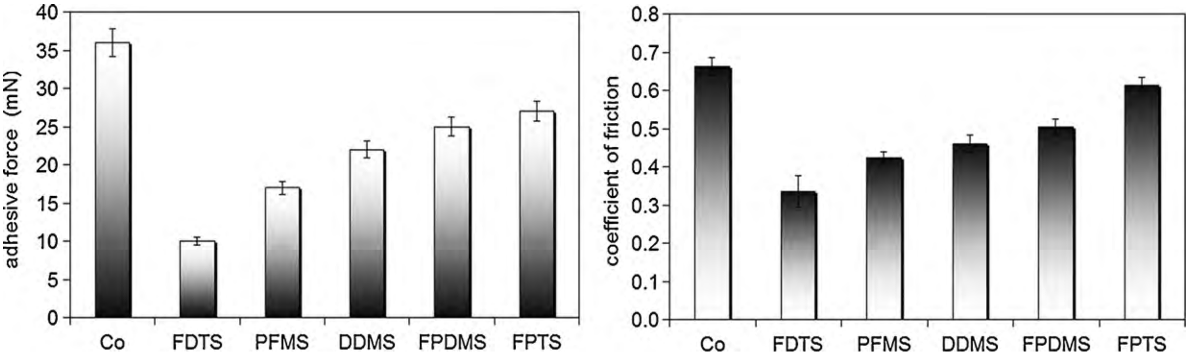

Fig. 6. Microtribological data: (a) adhesion and (b) coefficient of friction.

configurations with a monolayer with 30% of the chains removed, and observed that decreased packing density was correlated with increased friction force.

In the case of the comparison of tribological properties of the trifunctional (FDTS) and monofuncional (PFMS) silanes at the nanoscale, the former silanes exhibit lower adhesion and coefficient of friction. The trend in adhesion and coefficient of friction data agrees well with the contact angle and surface free energy results. The more hydrophobic surface has lower surface energy and adhesion, and in consequence the trifunctional fluoroalkylsilanes (FDTS) have a higher degree of hydrophobicity and a lower coefficient of friction than the monofunctional fluoroalkylsilanes (PFMS). In the case of FDTS lower coefficient of friction is due to the better surface coverage and cross-linking between different molecules. Nevertheless, as the silanes with short chains are compared to each other (FPTS, FPDMS), no significant differences in the tribological properties at the nanoscale are observed.

Fig. 6 shows the adhesion and coefficient of friction measured for various films at the microscale. The values at the microscale are higher than those at the nanoscale. This is due to different contact stresses, plastic deformation, applied load and counterpart radii. To a lesser extent, this can be due to scanning speed of the counterpart. However, in our case we have only technical friction. In this kind of friction no influence of scanning speed on the coefficient of friction value is observed.

At the microscale, all fluoroalkylsilanes exhibit improved friction behavior over the uncoated cobalt surface. The uncoated cobalt shows the highest adhesion and coefficient of friction values. At this scale FDTS presents the best tribological properties. Similarly to the nanoscale, the adhesion and coefficient of friction of the components decrease with the increase in chain length. Trifunctional fluoroalkylsilanes, e.g. FDTS, show a lower friction coefficient than the layers formed of nonfluorinated molecules (DDMS). Comparison of the type of head group indicates that the trifunctional groups (FDTS) exhibit better tribological properties than the monofunctional groups (PFMS). This is attributed to the increase in the packing density with longer chain length.

As the silanes with short chains are compared to each other, significant differences in the tribological properties are observed. In this case, monofunctional fluoroalkylsilanes (e.g. FPDMS) represent better tribological properties. The improvement in the microscale properties can be explained by the fact that monofunctional groups have lower surface free energy as compared to trifunctional groups.

4. Conclusions

A vapor phase deposition system was designed to apply uniform fluoroalkylsilanes films on cobalt surfaces. The results obtained using contact angle and XPS techniques proved that the used method of surface modification was successful.

The surfaces modified by the fluoroalkylsilanes films consistently have larger water contact angles, lower surface free energy and lower coefficient of friction at the nano- and microscale than the uncoated Co surface. The lowering of friction coefficient was observed when the hydrofluorocarbon chain length increased. The silanes with fluorocarbon terminal groups exhibited lower coefficient of friction than compounds with methyl groups. In the case of comparison of tribological properties at the nano- and microscale, the trifunctional silanes exhibit lower adhesion and coefficient of friction than the monofunctional ones. These results led us to conclude that covalently bonded fluoroalkylsilanes with longer alkyl chains are a prime candidate for practical use as a lubricant. The best antifriction properties of modified surface have been shown by FDTS, which is composed of trifunctional reactive groups, ten carbon atoms and a fluorinated carbon backbone.

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