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VALIDATION OF TEMPERATURE SENSITIVITY OF SUPERHYDROPHOBIC SURFACES AS APPLIED ON 316L SS

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ABSTRACT

This paper investigates the viability of commercially available superhydrophobic coatings applied over 316L Stainless Steel in extreme temperature environments. 316L SS or Marine Steel has immense importance in structural applications. Commercial superhydrophobic coatings are gaining importance in industry due to self-cleaning characteristics and effective lubrication. The coatings after application were investigated in a range of temperatures through dynamic-mode imaging of the surface by Lateral Force Microscopy (LFM). LFM revealed changes in the surface roughness and lateral force behaviour under the extremes of temperature. The analysis has been used to evaluate a value of friction coefficient which demonstrates wear behaviour due to isothermal temperature changes in the environment. This is directly associative of the optimal temperature for the coating to remain intact and indirectly predicts the ideal temperature till which the base substrate would remain protected. The analysis aims to validate the temperature sensitivity of these commercial coatings for use in structural applications in demanding environmental conditions.

Keywords: 316L Stainless Steel; Lateral Force Microscopy; Superhydrophobic Surfaces, Oleophobic Surfaces, Temperature Sensitivity of Coatings

1 INTRODUCTION

AFM was introduced as a combination of the principles of STM (Scanning Tunneling Microscope) and Stylus Profilometer [1]. Since its introduction, this technology has been widely adapted for the study of frictional effects, surface properties and changes in material properties for different conditions ranging from micro scale to nano scale. At these scales, the performance and operating lives of the micro/nano tribological devices is reduced due to high surface forces, such as adhesion and friction [2].

The need to modify AFM was to cater for the high surface forces that occur at nano level. Therefore, LFM was developed and is now considered as one of the most effective and informative technologies to measure adhesive and frictional effects. Friction and wear rate are fundamental characteristics of a machine with relative motion. LFM has played an important role in investigating coefficient of friction and other tribological properties for various materials under different conditions at nano scale [2 - 6]. AFM can also be used to study surface roughness of a material [7 - 9] because irrespective of the material, the coefficient of friction can be altered by modifying its surface roughness [7].

Coating is a form of surface modification where one material is deposited on the other to improve properties such as hardness, roughness and wear resistance [10]. Right after the introduction of AFM technique, O. Marti, D. Brake and P.K. Hansma in 1987, carried out experiments using conducting and non conducting surfaces covered with oil and used AFM to analyze them. This research demonstrates the potential of AFM technique. [11]

Metal coatings can be hydrophobic 'water repellent', hydrophilic 'water loving' and similarly oleophobic and oleophilic. Some of the coating methods of a metal are Dip coating, Spraying, Oxidation and electrochemical deposition. There has been extensive research on different types of coatings on a metal. A small thickness chromium nitride coating on a steel substrate when oxidized at a temperature higher than 450 °C, forms a separate Cr₂O₃ layer on top of the nitride coating and as the temperature is increased, the thickness of the formed layer increases [12]. The word oleophobic literally corresponds to the 'fear of oil'. Water is considered as the most oleophobic substance. Applications of oleophobic surfaces include anti-fouling, self-cleaning, low drag and oil-water separation. Oleophobic coatings have been studied

for aerospace applications under harsh environment [13]. There is a wide variety of such coatings available for composites and metals and the research for the same still continues with the development of superoleophobic and superhydrophobic coatings [14].

316L SS has been widely used in industrial applications for its corrosion resistance property and high strength. However, because of its low hardness and wear resistance their applications are limited [15]. Therefore the surface of SS 316L is hardened to improve wear and hardness resistance of the material without changing its other desirable properties. The tribological and corrosion behavior of Amorphous Carbon coatings over widely used 316L SS was investigated recently [9]. In the same year, wear test was conducted on 316L SS deposited with a layer of TiN against Titanium Alloy (Ti-6Al-4V) under different modified conditions [10].

The adhesive forces in a dry sliding contact generate high surface temperature therefore the effect of these forces under elevated temperature condition was observed by A. Gaard using Ti, Stainless Steel and low alloyed steel samples. It was found that the adhesive forces and the surrounding temperature are linearly related [16]. The effect of heat treatment over 316L SS dip coated by thin film of ZrO₂ was investigated using SEM and AFM and it was observed that there was a major decrease in thickness of the film up to 700 °C and almost constant behaviour between 700 °C to 900 °C [17].

Despite its widespread usage little work has been done to confirm the effective properties and consequently the coefficient of friction at nano-level of commercially available oleophobic coating when applied over 316L Stainless Steel. In this work, 316L SS, when coated with oleophobic coating, are observed to determine the coefficient of friction and the changes in its value when the samples are kept at elevated temperature of up to 250 °C for 08 hours. LFM technique is used to record the frictional and lateral forces and consequently the coefficient of friction which will help in enhancement of surface properties and material life prediction. The analysis aims to validate the coefficient of friction value as marketed by the coating manufacturers.

2 EQUIPMENT

The equipment used in this study is the ezAFM by Nanomagnetics Instruments as shown in Figure 1.

The equipment is capable of LFM scanning in contact-static mode and provides meaningful data through its output channels comprising of FN, FL and FT which in turn is used to evaluate the friction coefficient using Amantons' Law. PPP-LFMR probes are used in this investigation. The low force constant of the PPP-LFMR probes enables more rigid tip-sample behaviour which provides accuracy of the scanning results. A low force constant also means that even minute tip deflections are carefully recorded by the detector in the ezAFM.



Figure 1. ezAFM at DHA Suffa University

Isothermal heating of samples was accomplished by ESCO Isotherm OFA-32-8 in Figure 2 that allows temperature rise up to 300 °C and is capable of isothermal heating.



Figure 2. ESCO Isotherm OFA-32-8 at DHA Suffa University

3 EXPERIMENTAL METHODOLOGY

Twenty-Four (24) samples each measuring 25 mm in diameter and 8 mm in height were cut from peeled round bar of 316L SS. The samples' surface was grinded using emery papers of grit size 120, 240, 320, 600 and 800 on Metkon Forcipol 2V Wet Grinding Disc maintained at 400 RPM.

Grinded samples were then labelled 1 to 24 as given in Table 1. Labelled samples were scanned by ezAFM in LFM mode to evaluate initial value of coefficient of friction, results of which are presented in Table 2. For each scanning, sample surface was divided into 10 parts and both front and back scanning was accomplished for accuracy of the results.

Scanning was accomplished on a $5\mu\text{m} \times 5\mu\text{m}$ area at 64 pixels resolution. The scanning frequency was maintained at $5\mu\text{m/s}$ for the first run which was reduced to $1\mu\text{m/s}$ later on for recording results.

The final value of the coefficient friction of each sample surface is therefore a mean of 640 points. Anomalous values were removed from the average to maintain precision. This procedure was kept constant for all samples.

After the scanning was accomplished, labelled samples were then coated with their respective set of coatings as discussed in Table 1. Only one surface was coated on each sample as shown in Figure 3.

Aerosol based coating was positioned 8 inches from the sample surface and coating was performed in a lateral motion from left to right in 02 continuous cycles. For EnduroShield Stainless Steel Coating and Rust-Oleum NeverWet Multi-Surface Liquid Repelling Treatment, base coat was first applied followed by top coat as stated in the manufacturer's manual. For DuPont Teflon Non-Stick Dry-Film Lubricant Coating, a single coat was recommended. Each coat was applied twice in former case while four coats were applied for the latter one to make the number of coats equal.

LFM scanning was repeated on the coated samples and the results are presented in Table 3.

Oven preparation was made at the temperatures as mentioned in Table 4. The oven was preheated for two(02) hours before the final temperature was reached in the heating cycle. Coated samples were then put inside the hot oven and incubated for next 08 hours in the oven. The samples were then cooled and observed with LFM since in-situ scanning was not possible with our equipment. Scanning results are presented in Table 5.

Water repellency test was also performed on the treated samples.

This approach is in continuation to our systematic approach to generate useful data on oleophobic/superhydrophobic surfaces for aerospace and commercial marine applications in Pakistan and would be viably applied for varied applications other than those recommended by the manufacturer once sufficient data has been obtained.

Due to lack of sufficient practical applications data available for these coatings, investigations are being carried out with the said purpose of improving surface finish and corrosion resistance of metallic parts in high-performance applications.



(a)



(b)



(c)



(d)

Figure 3. a) Original sample surface appearance without coating, b) Sample 2 surface appearance with Enduroshield Stainless Steel Coating, c) Sample 6 surface appearance with DuPont Non-Stick Dry Film Lubricant Coating and d) Sample 10 surface appearance with Rust-Oleum NeverWet Multi-Surface Liquid Repelling Treatment

Table 1. Labelling of Grinded Samples based on Coating

Sample Number	Coating Applied
1,2,3,4,5,6,7,8	Enduroshield Stainless Steel Coating
9,10,11,12,13,14,15,16	DuPont Teflon Non-Stick Dry Film Lubricant Coating
17,18,19,20,21,22,23,24	Rust-Oleum NeverWet Multi-Surface Liquid Repelling Treatment

Table 2. Mean values of Coefficient of Friction as obtained from LFM on Uncoated samples

Sample Number	Coefficient of Friction
1	0.154
2	0.182
3	0.167
4	0.140
5	0.158
6	0.162
7	0.155
8	0.173
9	0.180
10	0.151
11	0.164
12	0.167
13	0.176
14	0.149
15	0.157
16	0.179
17	0.144

18	0.175
19	0.160
20	0.171
21	0.158
22	0.179
23	0.161
24	0.147

4 RESULTS AND DISCUSSION

4.1 Physical Appearance

The samples were left to cool after eight 08 hours of temperature incubation. No significant difference in texture was visible in the samples.

4.2 Water Test

Samples were tested for water repellency. EnduroShield Stainless Steel Coating and Rust-Oleum NeverWet Multi-Surface Treatment showed water repellency even after treatment at 250°C. Dupont Non-Stick Dry Film Lubricant Coating showed decreased water repellency after treatment.

4.3 Lateral Force Microscopy

Lateral Force Microscopy revealed that coefficient of friction values for the coated sample is significantly lesser as compared to the coefficient of friction values for uncoated samples. This is shown in Table 3. A comparison of Table 2 and Table 3 results is shown in Figure 4.

Table 5 shows how these values change further when treated in a convection oven.

Table 3. Mean values of Coefficient of Friction as obtained from LFM on Coated samples

Sample Number	Coefficient of Friction
1	0.148
2	0.145
3	0.151
4	0.144
5	0.152
6	0.150
7	0.151
8	0.140
9	0.124
10	0.131
11	0.128
12	0.134
13	0.136
14	0.133
15	0.129
16	0.121
17	0.144

18	0.158
19	0.142
20	0.155
21	0.148
22	0.152
23	0.141
24	0.148

Table 4. Oven temperature for coated samples

Temp. (°C)	Sample Number
75	1, 9, 17
100	2, 10, 18
125	3, 11, 19
150	4, 12, 20
175	5, 13, 21
200	6, 14, 22
225	7, 15, 23
250	8, 16, 24

The coefficient of friction value for samples treated between 75 to 150 remain fairly in range of their previous values. However, samples treated at 200°C and above show a significant change in the coefficient of friction value.

Dupont Non-Stick Dry Film Lubricant Coating shows the maximum change in coefficient of friction value at 200°C. While for EnduroShield Stainless Steel Coating and Rust-Oleum NeverWet Multi-Surface Treatment it is at 250°C.

Our previous investigation results from coating and testing value of COF on Inconel 718 samples in sand-storm conditions are given in Table 6. The conditions of this investigation are fairly different from our previous study. However, for comparison we can relate the difference in values of COF based on different surface interaction between the coating and base surface. Their closeness to our previous results suggest that our values are well in range.

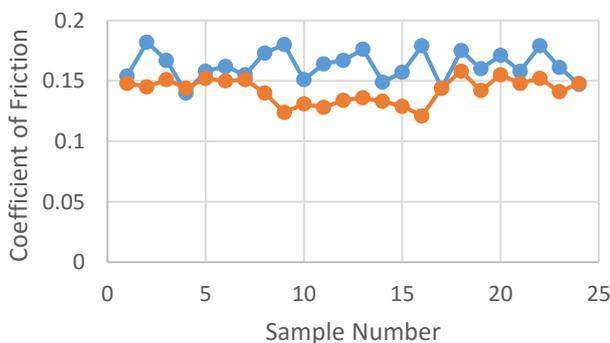


Figure 4. Coefficient of friction values against Sample Number. Blue line shows the coefficient of friction value before coating

while orange line shows the values as a result of coating application

Table 5. Change in coefficient of friction values before and after isothermal heat treatment in convection oven

Coating	Sample Number	Temp. (°C)	Coefficient of Friction	
			Before	After
EnduroShield Stainless Steel Coating	1	75	0.148	0.146
	2	100	0.145	0.144
	3	125	0.151	0.155
	4	150	0.144	0.150
	5	175	0.152	0.157
	6	200	0.150	0.149
	7	225	0.151	0.157
	8	250	0.140	0.201
Dupont Non-Stick Dry Film Lubricant Coating	9	75	0.124	0.133
	10	100	0.131	0.136
	11	125	0.128	0.139
	12	150	0.134	0.141
	13	175	0.136	0.139
	14	200	0.133	0.241
	15	225	0.129	0.244
	16	250	0.121	0.250
Rust-Oleum NeverWet Multi-Surface Treatment	17	75	0.144	0.145
	18	100	0.158	0.152
	19	125	0.142	0.150
	20	150	0.155	0.156
	21	175	0.148	0.151
	22	200	0.152	0.157
	23	225	0.141	0.161
	24	250	0.148	0.247

On a general trend it is evident that the coefficient of friction changes drastically between the temperature ranges of 200°C - 250°C whereas in the lower ranges of temperature 75°C - 150°C, no significant changes are observed.

The lateral and normal forces from each of the 64 points in a scan were obtained using 'Contact mode' analysis capable of generating friction force. The ratio of the forces data obtained is related by the Amonton's Friction Law,

$$\mu = \frac{F_L}{F_N} \quad (1)$$

where μ is the coefficient of friction, F_L is the lateral friction and F_N is the normal loading force.

5 CONCLUSIONS

This research aims to investigate the coefficient of friction of 316L SS samples when coated by three different superhydrophobic coatings, kept at elevated temperature for 08 hours and observed using FFM. The results obtained were:

- The surrounding temperature and adhesive forces are linearly related to each other as confirmed from the literature.
- The coefficient of friction value for samples treated between 75 to 150 remain fairly in range of their previous values.
- Samples treated at 200°C and above show a significant change in the coefficient of friction value.
- Dupont Non-Stick Dry Film Lubricant Coating shows the maximum change in coefficient of friction value at 200°C.
- EnduroShield Stainless Steel Coating and Rust-Oleum NeverWet Multi-Surface Treatment maximum change in coefficient of friction is observed at 250°C.

6 FUTURE WORK

Building upon our earlier work, we have provided a baseline on service temperature of these coatings. This essential step would help us analyse more coatings that we have identified during this research work.

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Table 6. Mean values of coefficient of friction from investigating coated samples of Inconel 718 in sand-storm conditions [13]

Type of Coating	Coefficient of Friction	
	Before Sanding	After Sanding
No Coating	0.156	0.227
Enduroshield Stainless Steel Coating	0.161	0.209
DupontTeflon Non-Stick Dry Film Lubricant Coating	0.145	0.250

Rust-OleumNeverWet Multi-Surface Liquid Repelling Treatment	0.151	0.222
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