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Influence of Surface Treatment on Dry Sliding Wear Behavior of Hydrophobic Silane Coating on AISI 304

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Abstract. Henicosyl-1,1,2,2-tetrahydrododecyltrichlorosilane is a hydrophobic silane compound coated on AISI 304 substrate. The surface of the substrate was treated in two ways (silicon oxide and plasma oxide) for adhesion and interface between the inorganic substrate and organic coating. The wear performance of the coating as a comparison to the AISI 304 was investigated using vacuum tribometer and characterized by scanning electron microscope and atomic force microscope. The result showed that the silane compound did improve the wear property of the AISI 304 stainless steel. It also proved that silane can be used as wear resistance agent.

1. Introduction
Wear can simply be attributed to damaging, gradual removal or deformation of material at solid state due to mechanical process under the condition of sliding, rolling and/or repeated impact. Wear process is one of the factors that cause failure of material application in service [1]. Wear types (abrasive, adhesive, corrosive, fatigue and erosive) are specific to each material and/or the subjected condition [2]. AISI 304 belongs to austenitic stainless steel that suffers lowest wear resistance (both in sliding wear and under lubricated wear) [3]. However, when it is in contact with itself or other materials, the wear volume is always very significant and usually undergoes either abrasive wear or plastic deformation [3]. A number of processes and methods have been developed to reduce the wear rate of AISI 304 which includes but is not limited to; thermal spray [4], anodizing process [5], electroplating [6], radio frequency magnetron sputtering, high-velocity oxy-fuel spraying (HVOF), ceramics, electrophoretic deposition [7], and sol-gel method [8].

The sol-gel has been applied or deposited unto the metal surfaces through many methods which include physical layer deposition (PLD), chemical layer deposition (CVD), dip-coating, spin-coating [9] and recently a hybrid of CVD and atomic layer deposition (ALD) [10]. ALD has shown to be a much precise instrument for delivering precursors for smoother surfaces to the target [10]. This method of coating assists in the mechanical properties of sol-gel as it atomizes the coating layers of thin film and produces good crosslinking and self-limiting surface reaction [11].
However, silane-based sol-gels have been reported to have poor wear and mechanical properties on the substrate except by addition of inorganic nanoparticles but have shown to be effective in the area of corrosion inhibition only [8]. Silicon oxide (SiOx) has shown to be a good wear resistance film [12][13]. Plasma oxide, however, has found its applications as an adhesion promoter and anti-ageing film [14].

In this regards, surface pre-treatment would be used to test the integrity of the coating and it is expected that it would play an important role in reducing the wear rate. The aim, in this present study, is to develop and characterize a new silane compound that will improve the wear resistance of an AISI 304.

2. Experimental

2.1 Material
The tridecafloro-1,1,2,2-tetrahydrooctyltrichlorosilane hydrophobic compound was developed, produced and coated on commercially available AISI 304 by Integrated Surface Technologies Inc., USA.

2.2 Method
The surface treatments and the organic coating were deposited by atomic layer deposition (ALD) technique using RPX-540 manufactured by Integrated Surface Technologies (IST). The surface treatments are of two types, the plasma oxide treatment and the silicon oxide treatment. These pre-treatments also serve as adhesion between the inorganic metal substrate and the hydrophobic organic coating. After curing and drying, the resulting thickness was found to be approximately 20nm using ellipsometry on a reference Si wafer.

2.3 Characterization
A sample size of 1cm² was used for the investigation with 3 different scans made on each sample to determine the range and repeatability of the data. Rtec MFT 5000 (Rtec Instruments, San Jose, CA, USA) multifunctional vacuum tribometer was used for the wear test by adhering strictly to ASTM G99 - 17 standard. The ball on disk test was performed using 440C steel ball, grade 24 of 3.967 mm diameter. The duration of the test was at 1 min, the load was at 9.5 N, wear length of 3 mm, constant velocity at 4mm/s and acceleration at 0.1mm/s². The test was conducted at room temperature. The surfaces of the worn samples were characterized using scanning electron microscope (SEM) (Tescan’s Vega 8) via nanospace software and BX51M Olympus microscope.

3. Results and Discussion
The wear test was carried out on the coated surface to ascertain the integrity of the coating and to determine the effect of silane and/or the surface treatment on the wear resistance performance of the coating process (i.e. coating + treatment + substrate). The system’s simulated worn surfaces are presented in Figure 1 and the data generated are tabulated in Table 1.

The Rtec MFT 5000 software measured the volume and the wear depth of the material while the wear rate and wear resistance are calculated using Equations 1 and 2 [15].

\[
\text{Wear rate}(\text{mm}^3/\text{m}) = \left( \frac{\text{Volume loss}}{\text{Sliding distance}} \right) \times 1000 \tag{1}
\]

\[
\text{Wear resistance}(\text{m/mm}^3) = \left( \frac{\text{Sliding distance}}{\text{Volume loss}} \right) \times 1000 \tag{2}
\]
Figure 1: Wear track of a) uncoated SS 304, b) plasma oxide, c) silicon oxide, and profiler aii) SS 304 bii) plasma oxide and cii) silicon oxide.

Table 1: The summary of the wear results performed at room temperature.

| Material           | Volume (mm³) | Wear Depth (μm) | Wear rate(mm³/m) | Wear resistance (m/mm³) |
|--------------------|--------------|-----------------|-------------------|-------------------------|
| AISI 304 substrate | 1.301E-2     | 1.782E0         | 4.337E-3          | 2.306E+8                |
| Plasma Oxide       | 4.729E-3     | 0.035E0         | 1.576E-3          | 6.344E+8                |
| Silicon Oxide      | 1.675E-3     | 0.039E0         | 5.583E-4          | 1.791E+9                |

The Z position (wear displacement) was monitored and logged throughout the test. This was used to compute the specific wear rate coefficient using sphere-on-plane Hertzian contact pressure formula in Equation 3 [16].

\[
k_i = \frac{h_1}{p s}
\]

Where, \( h_1 \) = wear displacement (using Z-height data), mm3; \( k_i \) = specific wear rate coefficient; \( p \) = contact pressure = \( F/A_{wear\ track} \), N; \( s \) = slide distance = slide rate \( \times \) time = \( v \times t \), m.

From the data captured from the test, there was no change in Z position. This means that as a result of a minimal load applied to the surface and the wear resistance property of the ball’s material, the wear will have less impact on the ball, this is in agreement with Siddaiah et al work [16]. From Table 1, the uncoated surface SS304 has the highest volume wear, lowest wear resistance, highest wear depth and wear rate as compared to the coated surface. This is in agreement with the study on the wear characteristics of SS 304 [17]. However, silicon oxide posed to have the best wear property as compared with AISI 304 and plasma oxide.

3.1 Coefficient of friction
The coefficient of friction responses of the base material and the two surface treatments were studied and are presented in Figure 2.
Figure 2: Comparison of the coefficient of friction of parent material and coated samples against time.

It is very surprising that both the base metal and the coated samples have raised coefficient of friction for the first three (3) seconds and then dropped coefficient at about seven (7) seconds. The first 3 seconds is called a wear track run-in phase and after 7 seconds is referred to as steady-state region [18]. However, AISI 304 and silicon oxide later rose abruptly to the coefficient of about 0.10 with a considerable slope as compared to plasma oxide before they start to normalize. The plasma oxide rose to the coefficient of 0.12 at first 4 seconds, while SS 304 and SiOx pre-treated coating rose to 0.06 and 0.07 respectively at 3 seconds. The plasma oxide started to normalize at around 15 seconds while SS 304 started at around 20 seconds. The rising coefficient of friction on both surface treatments is an indication that the coating adheres strongly to the base material and there was no dilapidation or pill-off of the film.

The investigation was further extended to observe if correlation does exist between the coefficient of friction and the wear rate, but no relationship could be established. It is expected that higher coefficient of friction should generate higher wear rate. The results of the coefficient of friction do not in any way correlate to the volume loss or the wear rate. This may be in agreement with the conclusion of Rus et al., [18]. It is thought that the high coefficient may be attributed to the surface roughness [20], the ball used was carefully chosen to be able to penetrate into the substrate in order to ascertain the integrity and impact of the coating and pre-treatment on the base material. Therefore, the coefficient was not as a result of the roughness of the surface but as a result of the resistance of the coating process and the surface treatment. So, both surface treatments showed an improvement in wears resistance as against the proposition of Figueira et al’s literature survey [21]. This is in agreement with the work of Longhi et al [22] by successfully using tetraethoxy-silane (TEOS) and methyl methacrylate (MMA) to improve the wear resistance of the galvanized steel substrate.

3.2 SEM and Optical Microscope
The wear track was investigated under SEM (Figure 3) and an Optical microscope (Figure 4) to determine the behaviour of the worn surface and wear mechanism.
From the SEM analysis, there was no crack in the film neither did the coating get decapitated. This is an indication that the pre-treatment has good adherence to the surface of the substrate and the coating. The worn surface of the as-received AISI 304 substrate showed abrasive/erosive wear as predicted by [17]. However, the plasma oxide showed normal wear track and less resistance to the penetration of the ball. On the other hand, silicon oxide showed a bit of resistance to the ball as the wear track revealed less penetration of the ball.

Optical images also show the similar characteristics in comparison to the SEM images. Figure 4a shows a wear track that is non-uniform while 4b and 4c show uniform wear track but less penetration in Figure 4c. It also appears that after wearing process, the surface tends to initiate corrosion of the surface after just 3 days of the test (Figure 4b).

4. Conclusion
From the above results, it is certain that the surface treatments have an impact on the wear resistance of the substrate. It is evident that the coating and good surface treatment can reduce the erosive wear of the AISI 304. It is also clear that silane base organic coating can perform beyond corrosion inhibition tendencies as it can also be applied where there is a requirement for wear resistance. From the characterizations discussed above, the SiOx surface treatment resists wear rate more than the plasma oxide. Form the discussion above, the coefficient of friction is not the ultimate determinant of the rate of wear and wear resistivity.

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