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Evaluation of Microstructure, Corrosion and Wear Properties of Inconel 625 Film Deposited on TI6Al4V Surface with Magnetron Sputtering

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ABSTRACT

Several manufacturing industries recognise the value of titanium and its alloys in terms of useful application as petroleum tubes due to the high specific strength, low modulus of elasticity and seawater erosion resistance they offer. However, in a range of aggressive environments with high H2S and Cl− ions, pitting corrosion, can occur. For the purpose of useful surface engineering applications, this study adopted the Radio Frequency (RF) magnetron sputtering approach to a deposit Inconel 625 as the target on titanium grade 5 substrate for an enhanced service operation. The parameters were varied within 100W to 200W power rating, 100^{0}C to 200^{0}C temperature and 60 to 90 minutes of deposition time. The samples were divided into four categories for tribological test to determine the wear behaviour, immersed in a corrosive medium to evaluate the response and the use of a scanning electron microscope (SEM) to analyse morphologically. The results presented that the titanium sample coated at 150W and 100W exhibited enhanced wear and microstructural characteristics compared to the control, but had a reduced corrosion resistance over increased potential. The optimal TI6Al4V sputtered Inconel possessed the corrosion current density (jcorr) of 3.2404E-06 A/cm2 and corrosion rate of 0.03765 mm/year.

1. Introduction

Titanium and its alloys are used for general engineering applications because of its corrosion resistance and high strength [1,2]. The titanium grades that are of most interests in several engineering applications are the commercially pure titanium grades [3,4]. Titanium started gaining proper recognition in the aerospace industry in the early 1940's owing to its lightweight advantage which is a very significant consideration in the aerospace industry [5]. The major rationale for growth in the rate of utilization of titanium is its very strength to weight ratio with a density that falls in between iron and aluminium [6,7] Titanium and its alloys are widely utilized in several industrial

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applications which include fasteners, vessel, hubs, discs, rings, blades, and other high-performance applications various part of race engines like connecting rods and gear boxes. The suitability of titanium in the medical field is well pronounced in the area of metal implants where there is direct contact with bones and flesh tissues [8]. The high corrosion resistance characteristic of titanium makes it highly biocompatible such as cobalt chrome [9,10].

Inconel is a nickel-based superalloy, and superalloys are multiservice materials with good mechanical strength (Table 2) and very stable for surface applications with a preference for high temperatures and cryogenic temperature [11]. Its temperature adaptability qualities make it super applicable as a multipurpose material in areas of varying temperature applications. Although there are three major classifications of superalloys, which are cobalt-based, nickel-based, and iron-based superalloys [12,13], however, nickel-based superalloys have wide coverage of application amongst three classes. One of the prominent nickel-based superalloys is Inconel 625 with very reliable, sustainable applications in the field of engineering like aerospace, seawater ambiance, petrochemistry, and chemical plants [14] It has very impressive mechanical properties like high tensile strength, good thermal properties, high corrosion resistance, and many more [15]. The excellent corrosion resistant trait in Inconel is that it is well attributed to the abundant amount of molybdenum and chromium content (Table 1) in the chemical composition [16]. At present, Inconel 625 coatings are widely used to enhance corrosion resistance, hardness and wear performance of metallic surfaces [17,18], and it worthy of note that Inconel 625 is one of the important superalloys produced by forging, powder metallurgy and additive manufacturing [19].

Table 1
Chemical Composition of Inconel 625 (wt%) [30]

Material	Ni_{max}	Nb_{max}	Fe_{max}	Mo_{max}	Mn_{max}	Cr_{max}
Inconel 625	58.0	3.2-4.1	5.0	8.0-10-0	0.5	20.0-23.0

Looking at a few experimental excerpts from the bank of literature, Verdi *et al.,* [18] evaluated the hardness of Inconel 625 and Inconel 625-Cr_3C_2 coated on steel substrate using Vickers microhardness tests, following ISO 6507-1 standard and using a load of 300 gf and a dwell time of 12 s. Three indentation outlines were performed to get the mean value of the hardness for each distance from the coating surface. While the adhesion or bonding strength of coatings is also easily measured using the nano-scratch tests [20], it is complex to examine the creep, wear, fatigue, and fracture toughness in thin coatings due to their nature. Besides the nano-scratch tests, Singh *et al.,* [21] performed an adhesion-strength test on Inconel 718 coatings using a conventional tensile rig [22]. By applying a tensile load to the linked cylindrical stems at a constant rate, until fracture takes place, the force needed for separation was noted. Bond-strength was obtained by dividing the utmost load by the area of the cross-sectional of the sample. The coated sample was inspected after failure, and the mode of failure was checked to find out if the coating failed within, detached from the substrate (adhesive failure), or remain glued to the substrate. Despite a few exploits discovered from previous studies, this research work has further espoused the possible improvement to enhance the performance of titanium for useful applications. Most manufacturing industries such as oil and gas still derive great interest in the use of titanium and its alloys especially in gas pipes and petroleum tubes application due to their low modulus of elasticity, high specific strength and resistance to erosion in seawater [23,24]. However, in most aggressive environments containing Cl^- ions and H_2S, pitting corrosion (an enormously localized form of corrosion) takes place on the materials surface, leading to damage of the passivation layer [25,26]. Hence, providing initiation points for corrosion fatigue and stress corrosion crack propagation, which minimize the useful life span of the material [27-29]. Therefore, it was envisaged that the application of Inconel 625 film on Ti6Al4V alloy would enhance the microstructure, corrosion resistance behaviour and the wear characteristics of Ti6Al4V alloy. More so, sparse literature exists on the study of the effect of Inconel films addition to titanium and its alloy.

Table 2
Mechanical Properties of Inconel 625 (wt%) [31,32]

Material	Yield Strength [MPa]	Tensile Strength [MPa]	Elongation [%]
Inconel 625	414-621	827-1034	55-80

2. Experimental Procedure

2.1 Materials

Titanium was the parent material for this experiment because of its considerable application for the intended area of application which is the aerospace industry. However, for the sake of specifics, the titanium alloy that was used is titanium grade 5 alloy (Ti6Al4V) which contains six percentage (6%) of Aluminium and four percentage (4%) of Vanadium. The Titanium served as the parent material also referred to as the substrate which was the surface for the deposition of the thin film target. The high-grade superalloy that was used as the target for this study is Inconel 625. It was deposited on the titanium surface through the magnetron sputtering process to create a thin film on the substrate of the experiment.

2.2 Experimental Procedure

RF Magnetron sputtering machine, with the brand name GVAC and model number MSGC-2540 was used to prepare absolute Inconel coatings as targets on the titanium substrate at varying temperature, power and time. The substrates were polished cut into size at the laboratory to gain a fine and smooth surface without any impediments. Acetone was used for the initial cleaning of the titanium grade 5 alloy substrate. Further cleansing was done using isopropanol and deionized water. Before the commencement of the sputtering, a non-rotating work holder was used to help the material absorption process of the substrate which was kept at a distance of 10 cm away from the target. The sputtering was carefully done for four different samples at varying parameters to understudy the impact of time, power and temperature at different levels of 60 to 90mins, 100 to 200W and 100^{o}C to 200^{o}C respectively. Upon the completion of the sputtering process, the coated samples which is a combination of a substrate and a target in thin film medium were cut into dimensional sizes of 10 x 10 mm for SEM, wear and corrosion analysis.

2.2.1 Corrosion test

Potentiodynamic polarization was also carried out using a Metrohm Autolab Potentiostat/Galvanostat (Model PGSTAT128N). The studies were carried out using a three-electrode configuration cell setup, consisting of a platinum rod counter electrode, an AgCl/Ag reference electrode, and a working electrode with an exposed surface of 1 cm^2. The scan rate for every test was set to one mV/s following the ASTM International (American Society for Testing and Materials) specifications. The potential reading of the open circuit was done after an hour before the experimental polarization. The solution for the experiment was a concentration of 3.5%NaCl. The chosen medium was to examine the experimental behaviour of the coated titanium samples in an extremely corrosive environmental to see the stability limit.

2.2.2 Microstructural analysis

The surface morphological behaviour of the titanium thin film samples was studied through the use of a scanning electron microscope (SEM). The surface topography of the pure TiO_2 nanoparticle powder was characterized using Scanning Electron Microscopy (Model JSM-7600F JEOL, manufactured by JEOL Group Companies Japan) at an accelerating voltage of 20 kV and a working distance of 10 m. The SEM images show that the coated titanium nanoparticles are agglomerated. However, it is not clear if the sample coated each nanoparticle or an agglomeration of nanoparticles, as in super-paramagnetic titanium oxide nanoparticles. Agglomerated titanium nanoparticles are often observed in the metal obtained by co-precipitation. However, the dextran layer should increase nanoparticle size, narrow particle size distribution and improve dispersion.

2.2.3 Tribological behaviour of titanium thin film

The wear analysis that defines the tribological behaviour of the titanium substrate uncoated sample and four other thin filmed samples with different parametric factors of deposition was studied through the aid of a TRB^3 tribometer (manufactured in Austria) of version 8.1.8 with serial number 1000092309 using a ball geometry of 6mm. Each sample of TiO_2 was analysed at an atmosphere parameter of 1014, a target temperature of 24^oC, a relative humidity of 48.25%, 48.02%, 47.78% 47.18%, 46.45% and a varying laboratory ambient temperature of 20.71 ^oC, 21.07 ^oC, 21.48 ^oC, 21.75^oC 22.28 ^oC for the sample (a-d) and substrate (control sample) respectively.

3. Results and Discussion

3.1 Wear Analysis

Abrasive wear has a very important significance in surface engineering applications because of the surface response of the materials during operations which determines the possible failure rate and the service life of the materials. The outcome of the wear response investigation is illustrated in Figure 3 and Figure 4 with distinct behavioural patterns. The wear characteristics presented in Figure 1 can be attributed to the rate and temperature of Inconel deposition on sample-A produced at 100W power rating which resulted into lower values of Coefficient of Friction (COF) through the displacement range of the experiment. This suggest that the control sample could have been less lubricous which could have been a product of the experimental conditions and not the inherent properties of the materials [33-35]. Using the response of the control sample as the reference point, sample-B, as indicated in Figure 2 exhibited higher coefficient of friction (COF) between 0.0 and 1.0 m slide of the test material on sample-B. However, between 1.0 and 1.3 m slide, comparable COF was observed, indicating comparable resistance of the materials to indentation and wear [36,37]. The sample-C with 200 ^oC and 200W power rating deposition parameter initially exhibited similar COF compared to that of the control sample. However, there was a reduction in the COF of the sample-C between the sliding distance of 0.4 and 1.3m. Similarly, sample-D exhibited lower COF compared to the control sample between 0.0 and 1.3 m sliding distance by the test sample. The behaviour of Sample-C and Sample-D indicated that their surfaces exhibited higher lubricity and higher wear resistance compared to the other test samples [38,39]. The low COF sample-C and sample-D also revealed that the force required for sliding of the test piece to occur on the samples is less than the force required for sliding to occur on Sample-A, Sample-B and Control sample, due to high COF exhibited by those aforementioned samples [40,41]. The increased value of the coefficient of friction (COF) of Sample-A and Sample-B could also be attributed to the surface roughness of the samples, sputtering time, power and temperature [42-44]. Considering the trend of the behavioural patterns of the material during the analysis, the response change is not linear. Hence, factor responsible of optimized performance is the parametric combinations during the RF magnetron sputtering process. Coefficient of friction is inversely or directly proportional to the wear response value of a sample in the event that the COF parameters such as operational velocity and contact of surfaces have aligning impact on the wear parameters such as temperature, hardness, wear debris, surface roughness etc. [45]. Any material with excellent wear response must have a low COF and lower wear. This was corroborated by authors' ref. [46]. According to Hua *et al.,* [47] "the most excellent wear resistance is synonymous to the lowest coefficient of friction (COF). Therefore, the low COF of sample-D signified that it exhibited the optimum resistance behaviour to abrasive wear.

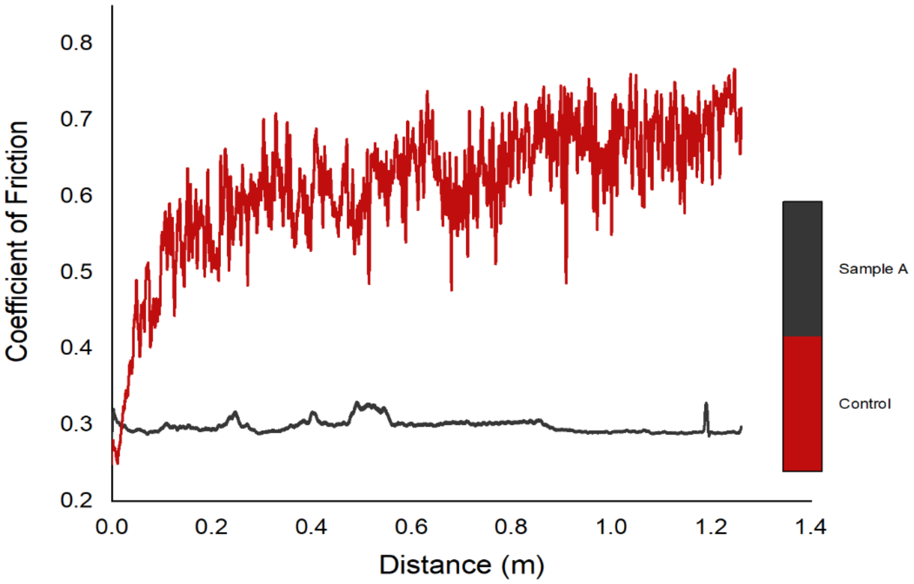


Fig. 1. Wear response profile sample-A titanium coated thin film

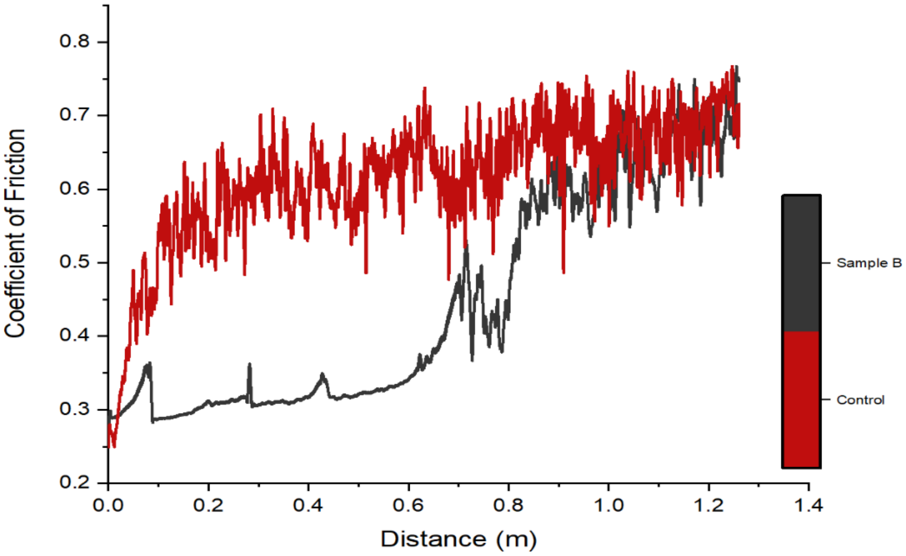


Fig. 2. Wear response profile sample-B titanium coated thin film

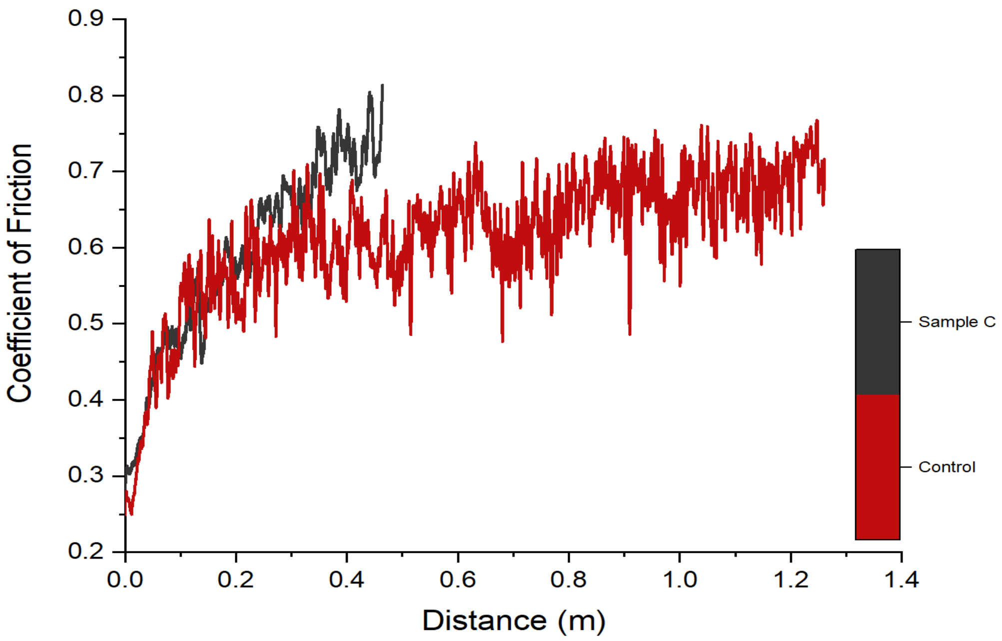


Fig. 3. Wear response profile sample-C titanium coated thin film

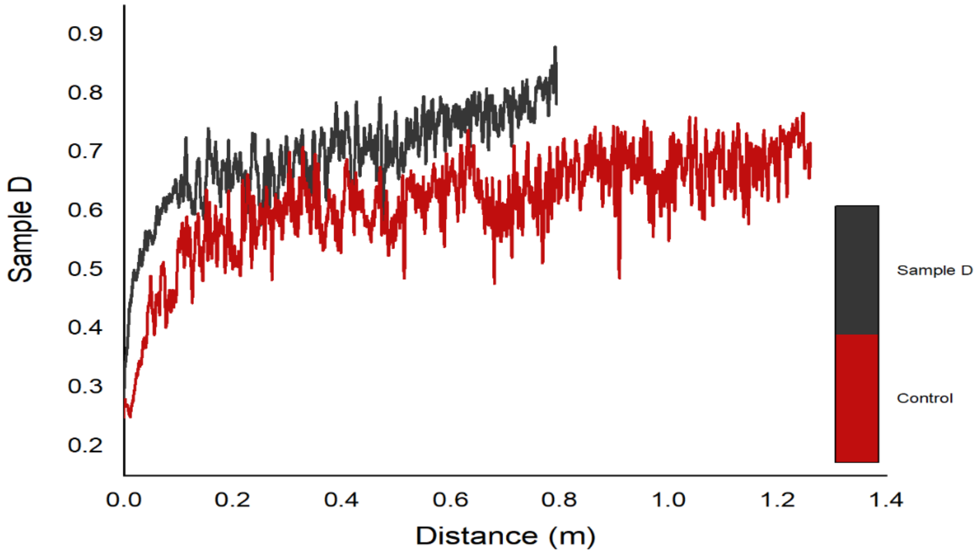


Fig. 4. Wear response profile sample-D titanium coated thin film

3.2 Corrosion Analysis

The polarization data for Ti substrate and Inconel coated Ti (titanium) samples is shown in Table 3. Relative to the other samples, the Ti substrate, which was used as the control samples, exhibited the lowest corrosion rate (Cr) and corrosion current density (jcorr) of 0.03083 mm/year and 2.6532E-06 A/cm^2, respectively. These values of Cr and jcorr exhibited by the control sample (Ti substrate) showed that it offered better resistance to the penetration of the hydrogen and chloride ions from the test solution (HCl) into its active sites as reported by authors' refs. [48,49]. The high polarization resistance (Pr) value of the Ti substrate compared to the Inconel coated Ti (titanium) samples further revealed that it exhibited the best corrosion resistance [50,51]. The Pr values thus indicated that the Ti substrate with the highest Pr value of 149010 Ω provided more passivation in the test medium which culminated in the higher corrosion resistance it exhibited compared to the Inconel coated Ti (titanium) samples [52]. Among the Inconel coated Ti (titanium) samples, the ICT sample 4 sample exhibited the best corrosion resistance performance due to the lowest Cr, lowest jcorr and highest Pr it possessed. By further comparison of the corrosion potential (Ecorr) values of the Ti substrate and Inconel coated Ti (titanium) samples, it was observed that the Inconel coating had more effect on the cathodic reaction, leading to the shift in the potentials to the more negative region during the polarization experiment [53-55].

Table 3
Polarization data for Ti substrate and Inconel coated Ti Samples

Sample	Ecorr (V)	jcorr (A/cm²)	Cr (mm/year)	Pr (Ω)
Ti substrate	-0.44829	2.6532E-06	0.03083	149010
ICT sample A	-0.93872	7.4718E-06	0.08682	7420.3
ICT sample B	-0.97056	9.7326E-06	0.11309	5729.7
ICT sample C	-0.79807	1.4199E-05	0.16499	4770
ICT sample D	-0.80464	3.2404E-06	0.03765	22126

Figure 5 further indicated the linear polarization of the Ti substrate and Inconel coated Ti samples exposed to HCl solution. The slope at the over-potential or Ecorr = 0 gives the linear polarization resistance value. The linear polarization resistance is equal to the ratio of the change in corrosion potential to the corrosion current density. In Figure 5 the slope or linear polarization resistance (ΔE/Δj) for the Ti substrate (control) sample was observed to be the lowest. Since the linear polarization resistance is inversely proportional to the corrosion current density [56], it can therefore be inferred that the reactivity of the Ti substrate was lesser in the corrosive environment, compared to the Inconel coated samples [57], leading to reduced corrosion rate. The result further confirms the assertion of [58], that Inconel 625 has a very good corrosion resistance ability but begins to fail in extremely ash environmental conditions.

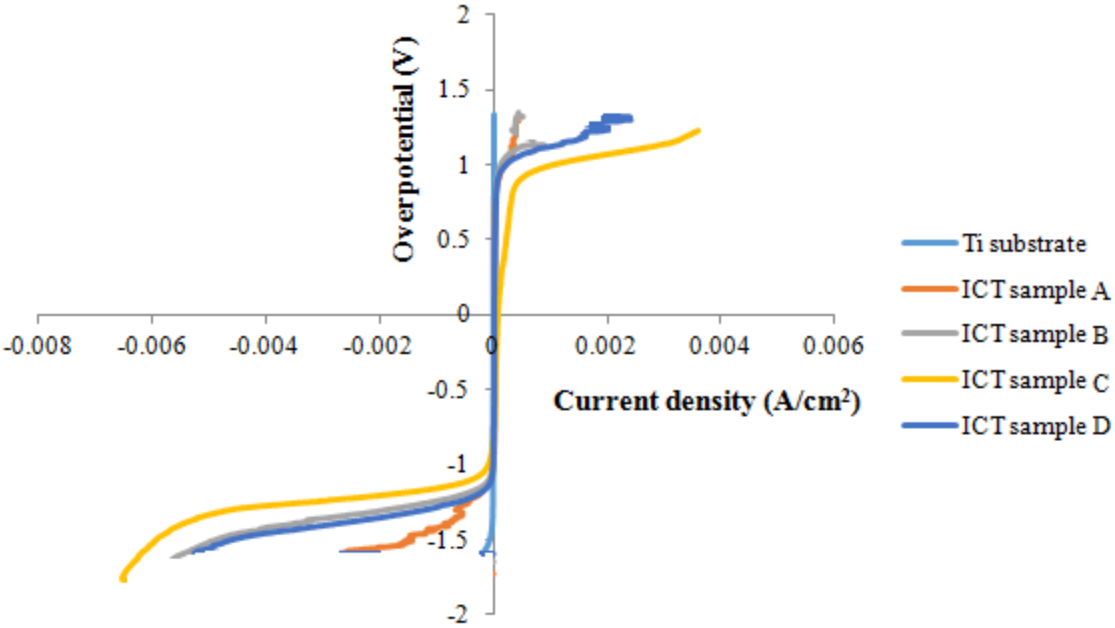
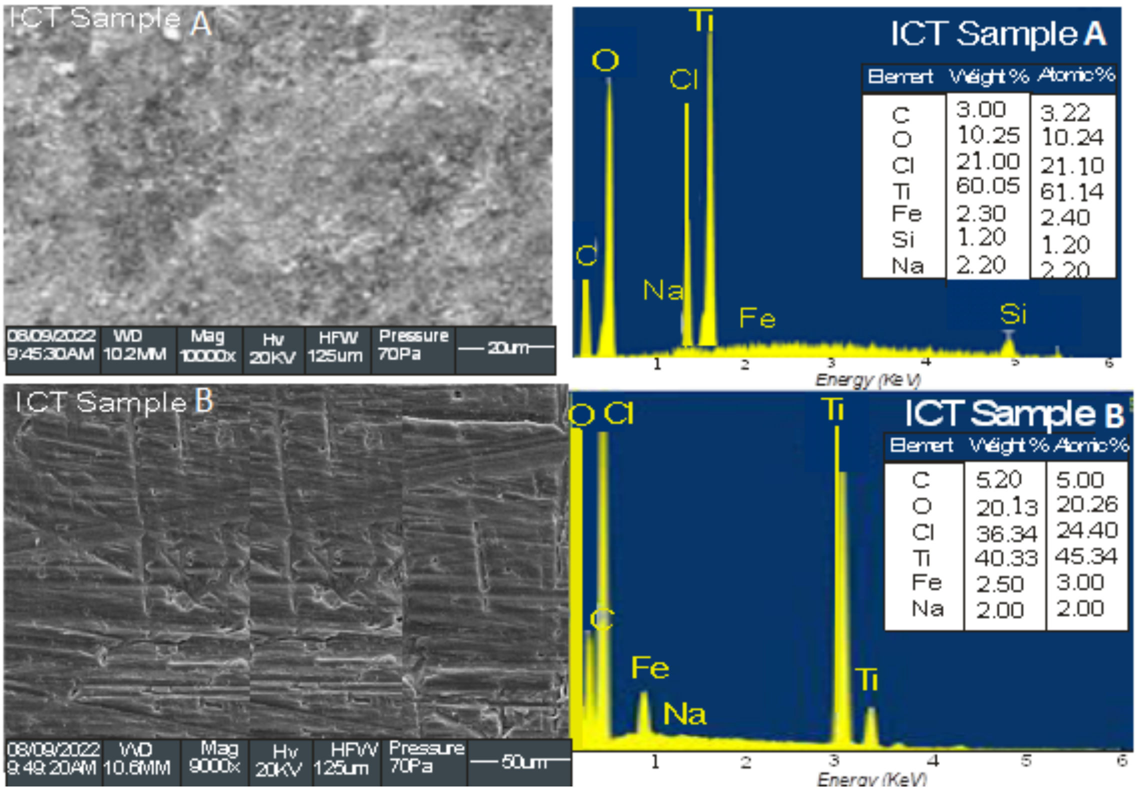


Fig. 5. Linear Polarization plots of Ti substrate and Inconel coated Ti Samples Exposed to HCl

3.3 Microstructural Analysis

Figure 6 shows the morphology of the coated Titanium surface with Inconel 625 target after the corrosion testing. A few brittle cracked layers filled with corrosion products were observed on the all the sample surfaces, indicating the pitting effect of the corrosive medium [59,60]. However, the very obvious cracks formation on Sample-D is attributed to the internal stresses caused by the released gases during the cathodic reactions, or it can be formed as a result of the salt layer [61,62]. Also, as seen in the figures, the coated titanium surfaces are filled with many pits of different sizes. The interpretation from the corrosion morphological image in Figure 6 corroborates the experimental outcomes of stability peak of the coated samples before the decline commenced as shown in Figure 5. Also, the coating on Sample-A was observed to be the most stable after conducting the corrosion test, exhibiting few shallow cracks cause caused by the ingression of hydrogen and chloride ions as affirmed by Tian *et al.,* [63]. Compared to the other samples, Sample-D exhibited more cleavage, indicating the penetration effect of the chloride ions [64]. The SEM image of Sample-D also showed that the coating on the samples' exhibited low passivation [65,66]. However, the EDS images of the samples indicated that the samples still retained their beneficial elements in spite of the corrosive effect of the medium.



ICT Sample A

Element	Weight %	Atomic %
C	3.00	3.22
O	10.25	10.24
Cl	21.00	21.10
Ti	60.05	61.14
Fe	2.30	2.40
Si	1.20	1.20
Na	2.20	2.20

ICT Sample B

Element	Weight %	Atomic %
C	5.20	5.00
O	20.13	20.26
Cl	36.34	24.40
Ti	40.33	45.34
Fe	2.50	3.00
Na	2.00	2.00

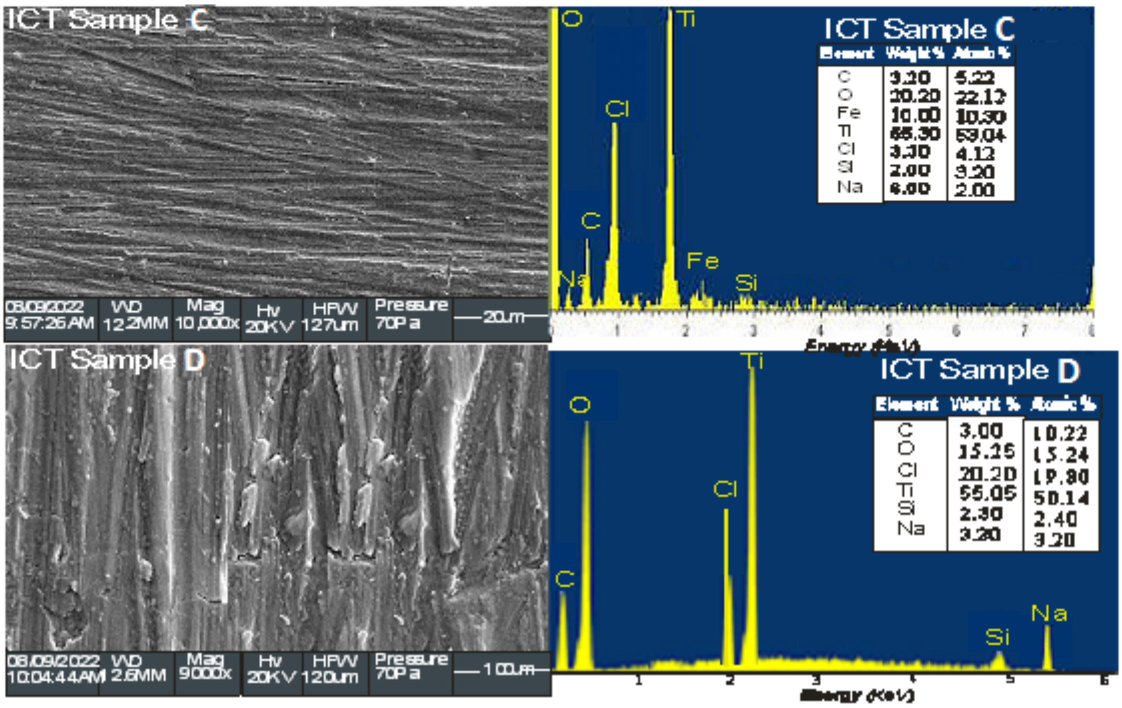


Fig. 6. Imaging of the corroded samples after HCL medium interaction

4. Conclusions

The RF magnetron sputtering procedure was done and completed following the experimental parameter distribution for the surface coating of the various samples. It was observed that the Inconel coating influenced the surface wear, corrosion, and microstructural properties of the titanium. The following conclusions can be drawn:

i. The low COF exhibited by the Inconel coated titanium indicated that the force required for sliding of the test piece to occur on the samples is less than the force required for sliding to occur on the control sample, due to high COF exhibited by the control sample. The behaviour of the Inconel coated titanium also revealed that it is more lubricous than the control sample.
ii. The optimal TI6Al4V sputtered Inconel possessed the corrosion current density (jcorr) of 3.2404E-06 A/cm^2 and corrosion rate of 0.03765 mm/year. The behaviour of Inconel 625 is an indication that it is capable of been used for titanium properties improvement for advanced applications.
iii. Also, the SEM images showed that the coating on the Samples A-C were more stable after conducting the corrosion test, exhibiting few shallow cracks caused by the ingression of hydrogen and chloride ions. The SEM images also showed that the coating on the samples' exhibited low passivation. The EDS images of the samples indicated that the samples still retained their beneficial elements in spite of the corrosive effect of the medium.
iv. The significance of the above finding is to add to the scholarly guide in the bank of literature for scholars intending to further extensive studies in this area and for industrial partners of engineering materials for prudent decision making.

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