# Reduce Effect of Erosion-Corrosion on Separator Heavy Fuel Oilby (Ni-P-SiC) Nano Composite Electroless Coating

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**ABSTRACT**

On a substrate made of (AISI 316 stainless steel), the electroless nickel composite (ENC) with silicon carbide content was deposited.The micro-hardness and erosive-corrosive behavior of composite coating samples with variable coating thickness (9µm to 26µm) were studied. Erosive-corrosive test was carried out at nine different period time (15,30,45,60,75,90,105,120,135 minutes) by local laboratory device to know the resistance of coating layer to erosive-corrosive environment.(FESEM) Field emission scanning electron microscopy with energy dispersive spectrometer(EDS)is exploited to show the morphology of surface coating , analyze the elements of coating layer and to determined coating thickness. Heat treatment for 1-hour at 600°C was carried out to increase the compacting and crystalline of coating layer. X-ray diffraction(XRD) revealed the phases which may satisfied the desired goal.Micro-hardness test was applied in surface of coating samples. The results showed super increasing in the values of hardness exceeded 1090.8HV for coated heat-treated samples. One of the important results, there was a clear decrease in the values of erosion rate from($98.42 \times 10^{-6}$ g/min) for bare sample to ($21.73 \times 10^{-6}$ g/min)for treated coated sample ,when the expose time to erosion fluid was135minutes.

**Keywords:** Electroless Plating,Erosive-Corrosive,(Ni-P-SiC),Hardness, FESEM,EDS.

## I.Introduction

Erosion-corrosion is the accelerated rate of material deterioration brought on by the interaction of mechanical wear and electrochemical corrosion processes. Whereas erosion is a process of mechanical wear, corrosion is a process of material degradation brought on by electrochemical activity. Erosion is a mechanical wear process, whereas corrosion is a material deterioration process that results from electrochemical activity. Erosion-corrosion is the name given to the interaction of these two processes, especially in marine environments. It is one of the main causes of failure in nuclear power plants, chemical and petrochemical industries, as well as marine environment and separators where coupled erosion and corrosion phenomena occur. [1-6].An oil/gas separator is a pressure vessel used for separating a well stream into gaseous and liquid components. They are installed either in an onshore processing station or on an offshore platform. Based on the vessel configurations, the oil/gas separators can be divided into horizontal, vertical, or spherical separators[7,8].The centrifugal pump used in the separator is a dynamic circulation pump with an impeller whose purpose is to push fluid under pressure to increase fluid pressure.Some parts of centrifugal pumps expose to corrosion, erosion-corrosion and wear so the mechanical properties are reduced[9,10]. The most important way for increasing corrosion ,wear and erosion resistance is coating the surface by suitable way according to the application, so electro-deposition or electroless plating is used[11,12].Electroless coating processes are chemical reactions that have been in use for more than 100 years and have been used to enhance carbon steel's and stainless steel's surface characteristics.Electroless coating processes may be using metallic alloys( binary, ternary or quaternary). Amongst Ni electroless coats, Ni-P electroless plating is of significant category[13,14].By coating engineered materials with a micro/nano composite of nonmetallic and metallic particles like $ZrO_2$, SiC, WC, $TiO_2$, $Si_3N_4$, and $Al_2O_3$, they can also be reinforced and protected against corrosion and abrasion phenomena [15].In nature, the composite materials stated

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above are hard, delicate, and only have a limited ability to form strong bonds when used alone as plating materials. In order to keep these particles together and provide a strong bind to the non-metal or metal surface being plated, an electroless Ni-P metal matrix is applied in order to solve this difficult problem. The SiC nanoparticles are also the least expensive of the aforementioned nanoparticles and are rigid, nonreactive, and have both inorganic (Si) and organic (C) characteristics.The use of nanoparticles has also significantly improved the Ni-P alloys' high temperature oxidation resistance[15]. This mechanism further put forward that electroless Ni-P-SiC nanocomposite plating should also offer high hardness, good wear,super resistanc to erosion-corrosin and corrosion resistance [16].By using the electroless plating process, superfine silicon carbide (SiC) particles reinforced nickel-phosphorus matrix composite coatings were created. It was demonstrated that SiC particles co-formed uniformly, and the deposited Ni-P-SiC composite coatings had an amorphous structure. The matrix of composite coatings crystallized into nickel crystal and nickel phosphide ($Ni_3P$) after undergoing specific heat treatment. At higher temperatures, nickel interacted with SiC to form nickel silicides that contained free carbon.There have been reports on the study of nano-sized SiC particulates in Ni-P coatings, but the majority of them focused on how SiC particles affected the coatings' wear resistance or corrosion resistance while very few of them discussed how the structure and corrosion resistance of the Ni-P-SiC composite coating were affe [16].The mechanical and physical properties of composite coatings are improved by the inclusion of particles in electroless deposited metals, which is a practical way of coating preparation.SiC, $Al_2O_3$, and other materials are used in wear-resistant composite coatings. SiC is a type of practical electronic material that has high strength ceramic material and great resistance to corrosion and erosion.Thus, electroless Ni-P-SiC composite coatings are appealing and have been researched in the past. Superfine particles can be disseminated and suspended in an electroless bath for their large specific area and small-scale influence because the sedimentation velocity of particles in this medium is slow with small size.Few research have been published to date that go into great detail on the crystallization and reactivity behavior of electroless composite coatings affected by superfine particles. [17,18]. In the present work, electroless Ni–P–SiC composite coatings with superfine particles were prepared to increase the resistance of surface samples to erosion effect and improve the hardness.

## 2. EXPERIMENTAL DETAILS
*A.         Substrate Preparationand Coating Process*
Composite(Ni-P-SiC) was deposited with thickness of 9-26 μm by incorporating superfine particles of silicon carbide, SiC, in the nickel-phosphorus matrix using autocatalytic process on circular substrate specimens namely as AISI 316 grade stainless steel (15 mm diameter x 10 mm thickness and with side hole 2 mm diameter).Thestainlesssteelhavingchemicalopusas(Cr=17.61%,Ni=11.55%,C=0.258%,Mo=2.35%   , Mn=1.043%,Cu=0.261%,P=0.0409,Al=0.0428,Si=0.041%,S=0.0005%andFe=balance).The silicon carbide (SiC) grinding sheets(100,150,180, 220, 320, 400, 600, 800, 1000, 1200, 1500, 2000, 2500,grit size)  used to grind these samples following the aforementioned grinding and polishing processes, the specimens require extensive treatment as follows:

- To get rid of dust, finishing compound, and other contaminations, clean for 30 minutes with acetone in an ultrasonic machine.

- To remove contaminations, grease, and any residues of oil from surfaces, an alkaline cleaning solution (60g/L NaOH + 38g/L Sodium Phosphate + 38g/L Carbonate of Sodium) is made at 65-70ºC and low voltage (3-5volt) (DC power supply).

- To remove the thin oxide metal film from the specimen's surface and increase the adhesion between the bare surface and the thin plating, special activation is applied. This procedure uses a unique nickel strike in an electrochemical reaction that contains [240 g/L nickel chloride ($NiCl_2$) and 126 ml/L hydraulic acid (HCl), with the specimens linked to the power source as the anode for 2-4

minutes at room temperature.The cathode, however, is nickel pole. Following this, the polarity is reversed for 3 to 5 minutes, after which distilled water is sprayed on the samples.

For coating Ni-P-SiC, the solution is made according to the Table 1 concentration. The specimen is hung inside the solution through a side hole. The specimens are coated at two distinct times, one hour and two hours.With a 150 r.p.m magnetic stirrer, the solution is homogenized.After the grinding, cleaning, polishing, and activation operations are complete, the bath is heated to 85°C and the pH of the solution is maintained between 4and 6. During the coating process, drops of sodium hydroxide or hydrochloric acid are introduced to control the pH of the solution. Hydrochloric acid is added to lower pH, while sodium hydroxide is supplied to raise pH.

**Table 1: Conditions and Components of (Ni-P-SiC) Nano Composite Coating**

| Conditions and Components | Concentrations |
|---|---|
| Nickel Sulphate | 30 |
| Sodium Hypophosphite | 40 |
| Superfine SiC particles | 5-10 |
| Sodium Citrate | 25 |
| Temperature | 85±2 °C |
|  | 4-6 |

All samples are rinsed with distilled water after the electroless composite coating is finished, and they are dried by a hot dryer.

*B. Heat Treatment*

For heat treatment, the specimens are kept at the specified temperature for 1 hour before being allowed to cool to room temperature. Specimens are placed in a tiny cup and heated to 600ºC for one hour in a vacuum furnace (Electrical Tube Furnace Type MTI-(GSL1600X)).Figure 1 shows electrical tube furnace.



**Figure 1: Electrical Tube Furnace**

*B.        Hardness Test*

Hardness is measured by the Vickers device, which is based on the traces of the diamond pyramid. The measurement of the hardness of the base metal is completely different from the measurement of the hardness for  thickness of the coating layer, where a load is applied in the case of the base metal

ranging between 200-300g/cm$^2$ at 15s, while when measuring the hardness of the coating layer, the load is between 30-50g/cm$^2$ at 10s.

*C.        Measurement of Coating Thickness*

In this study, it is necessary to know the thickness of the coating in order to know the appropriate thickness for the specific application or examination.By SEM examining or microscope, the thickness of the coating layer can be measured. In this study, the scanning electron microscope was used to find out the thickness of the coating.

*D.        Erosion Test*

This test was carried out with a laboratory device consisting of a high-speed pump whose speed ranges approximately between 80-92 m/s and includes a nozzle with a diameter of 2 mm and a basin that includes the specialized fluid, which is crude oil. In this important test, nine different time periods were used to know the erosion rate. These periods are as follows (15,30,45,60,75,90,105,120,135minutes).The impact angle between chemical solution and surface specimen is 90º.Figure 2 a view the device of erosion-corrosion.The erosion rate can be determined in the equation below:The losses in weight=original weight-weight after test:

Erosion rate=the losses in weight/exposure time (g/min)…………………3.1
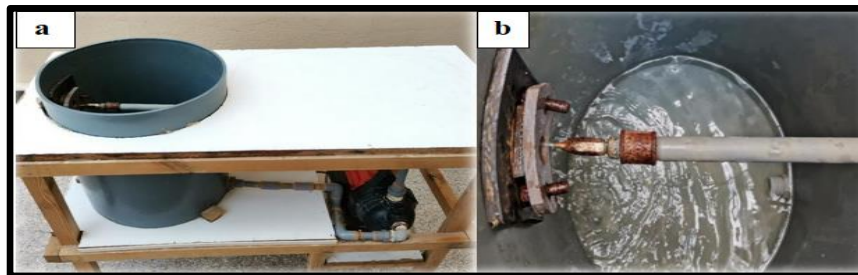


**Figure 2: (a)Erosion-Corrosion Device(b) Jet Nozzle**

## 3.RESULTSANDDISCUSSION

*A. Hardness and Heat Treatment Results*

The effect of heat treatment was clear in this study, as the hardness was much higher than it was without heat treatment.The presence of SiC particles which has a super hardness  up to 2000 Vickers, this certainly leads to an increase in the hardness of the coating layer.On the other hand, when the coated samples are heated to 600 °C for one hour inside the furnace, it leads to the formation of new phases and intermetallic compounds ($Ni_xP_y,Ni_xSi_y$) that are very important in hardness resistance, as well as in the increasing of the erosion resistance. Figure 3 shows the hardness before and after heat treatment at two different time.The hardness of coating specimens in 2-hrs.time of coating registered high magnitudes of hardness than the coating specimens in1-hr.time of coating in two stages before and after heat treatment respectively. Figure 3 shows the variation of hardness before and after heat treatment.
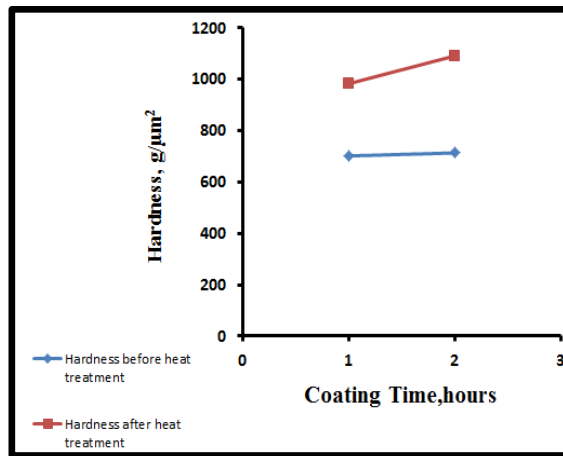
**Figure 3 : Hardness Variation with Coating Time Before and After Heat Treatment at (600ºC) for 1hr.**

*B.Morphology of Surface and Qualitative Analysis*

The morphology of Ni-P-SiC nano compsite coating was observed by using a field emission scanning electron microscope (FESEM device model-MIRA-TESCAN) with an EDS instrument model (X-Max), and an EDS attachment was used for qualitative elemental chemical analysis.By electroless plating, ultrafine SiC particles were effectively co-deposited in a matrix of Ni-P alloy. The amount of nanoparticles in the composite coating increase the plating rate  is dropped .About 8-12 µm/h was the average deposition rate for composite coatings containing 90-100nm SiC particles.The SEM picture (Figure4) of Ni-P-SiC platingstates the presence of small white globules with assimilation of apparently second phase nanoparticles on the basic substrate surface.The nano composites coating are smooth and moderately brilliant when first applied, and may be seen with the naked eye. These were identified as SiC nanoparticles because they were too obvious in the EDAX data (Table 2, Figure 5). They become embedded in the Ni-P coatings when 5-10 g/L silicon carbide nanoparticles are added to the electroless bath. Although silicon carbide nanoparticles have been included decreases the typical size of spherical nodules.
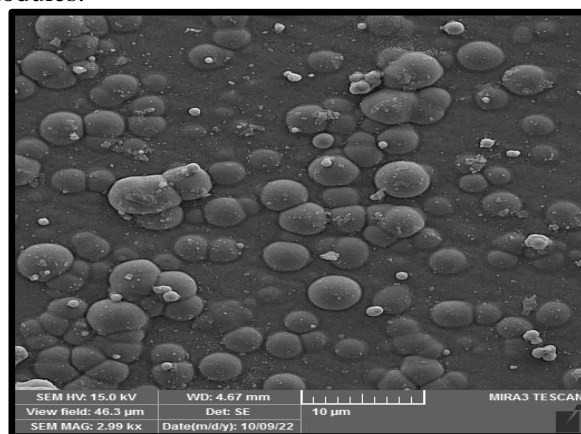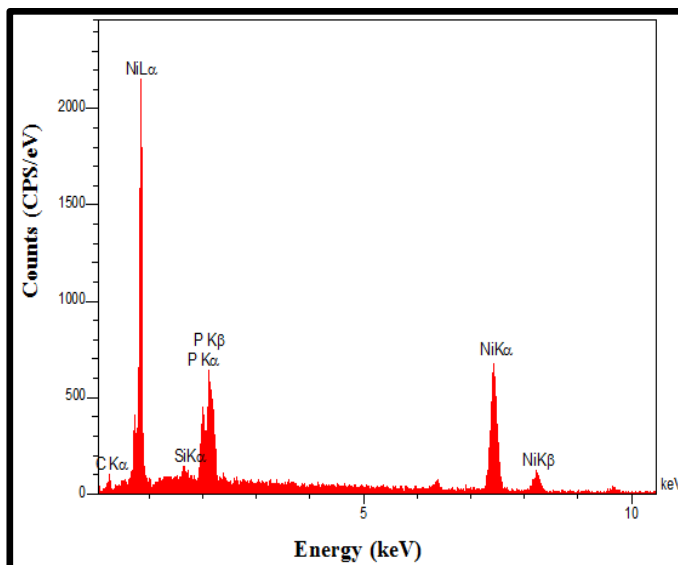


**Figure4: SEM of Ni-P-SiC Platings**

**Figure5:EDAX Chart of Ni-P-SiC platings on Stainless Steel 316**

**Table 2: EDAX analysis of Ni-P-SiC plating**

| Elements | Weight % |
|----------|----------|
| C | 11.24 |
| Si | 0.57 |
| P | 7.57 |
| Ni | 80.63 |
| Total | 100 |

*C. Analysis of Coating Phases*

Figures 6 and 7 show the XRD diagrams of the samples before and after heat treatment, respectively. The amorphous Ni-P structure's distinctive peak, which was, can be seen in the XRD spectra of the untreated specimens. An as-plated Ni-P specimen has a broad peak in 43.4º that also includes peaks from the Fe substrate.
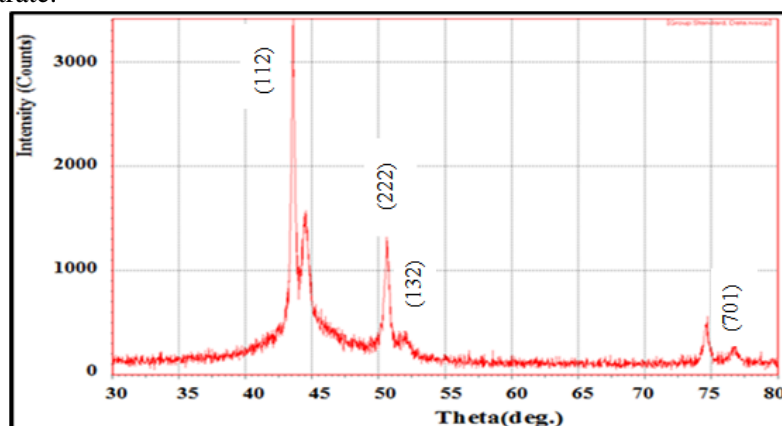


**Figure 6:XRD Pattern of Un-Treated Coated Sample**

The as-plated X-ray diffraction does not show any notable alterations. The heated plated specimen at 600°C displays additional new peaks as a result of the crystallization and precipitation of intermetallic

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Ni-P alloys (bct) and Ni-P/Ni-Si intermetallics.The amorphous or ultrafine crystalline microstructures of the Ni-P matrix are indicated by the wide character of as-plated coatings.It was demonstrated that the Ni-P-SiC nano composite coating's as-deposited structure was amorphous, and that heat treatment transformed it into nickel crystals, nickel phosphide ($Ni_3P$, $NiP$), and silicide compounds ($Ni_xSi_y$).
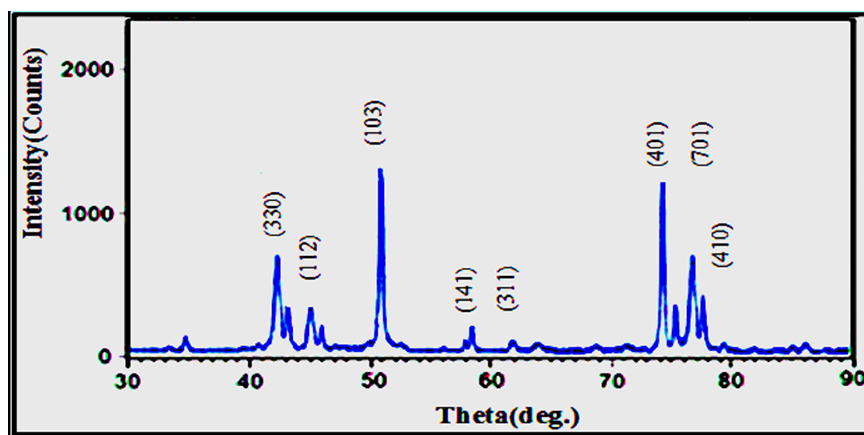


**Figure 7:XRD Pattern of Heat-Treated Coated Sample at 600°C**

*D.        Coating Thickness*

From SEM examination the thickness of coating by Ni-SiC-P nano composite plating has been measured.It revealed that the thickness of the coated samples with a coating time of 1 hour ranges between 8-12 μm, while the samples with a coating time of two hours reach twice the thickness in the first time. Figure 8 a and b shows the thickness of the coating with a time of one and two hoursrespectively.
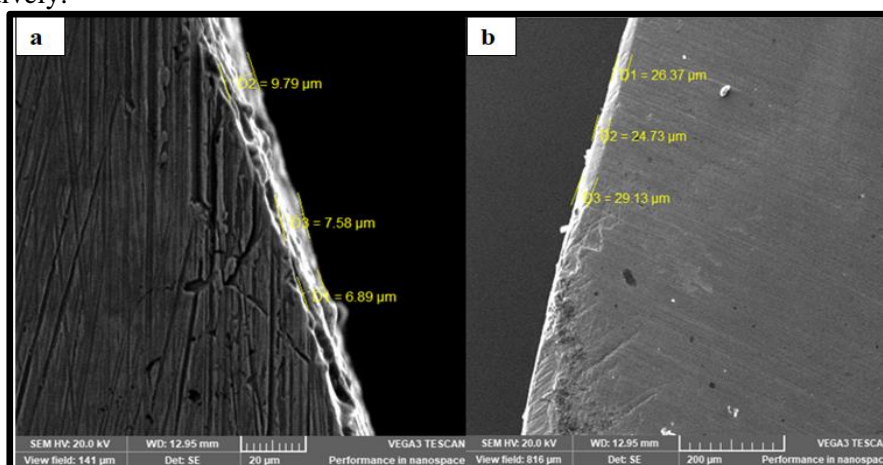


**Figure 8:SEM Images of Coating Specimens for (a): Coated Specimen with 1-hr. Time of Coating and (b): Coated Specimen at 2-hrs time of Coating**

*E.        Erosion Results*

Figure 9 display the results of erosion-corrosion rates with respect to time for bare specimens, coating specimens by (Ni-SiC-P) at two different times. The results show that behavior of bare metal or coated metal passes in four stage; incubation stage, acceleration stage, deceleration stage, and steady state. In incubation stage the change in weight is very low because high resistance of metal due to have high hardness. In the second stage the erosion rate increases due to the obtain reaction between the solution and metal surface to form metal oxides such as $Fe_2O_3$ and chlorine. These oxides play important role in erosion corrosion rate because it sticks with metal surface. In the third stage clearly

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decline appears in the erosion rate because most formation oxides and contamination on the surface metal may be removed due to high velocity of stream solution from jet of erosion advice. Also in this stage some of grooves may be appear in the surface metal .The last stage which called the steady state stage, in this stage there is no significantly increasing or decreasing in weight of metal therefore the rate of erosion stays constant. In this last stage the little change in rate of erosion resulted from the ionic drawing as well as the ability of the metal to form protective strong oxides. Figure 9 below shows the rate of erosion-corrosion in heavy fuel oil medium.
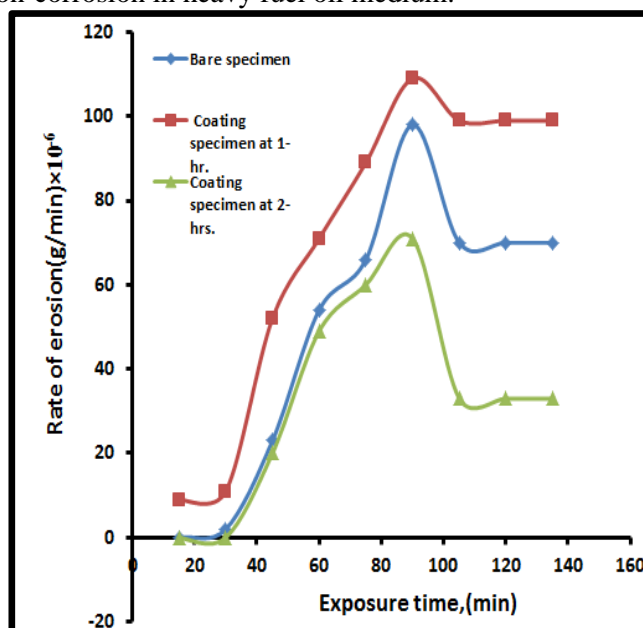


**Figure 9:Rate of Erosion-Corrosion in Heavy Fuel Oil Medium**

Figure 9 show high different in the rate of erosion especially at the bare metal while the coating metal show less erosion rate. It is clear that the heavy crude fuel contains various forms of impurities, plankton and solid particles, in addition to that it contains minerals and chlorides, some of which may be harmful to the parts of the separators. These harsh environments increase the rate of erosion.

By comparing the results of the previous Figure, the nickel-phosphor-silicon carbide coating shows a significant improvement in erosion resistance, because the presence of SiC particles especially when coating specimen at 2hrs. .The specimen has 2hrs. time of coating and thickness ranging between 16-28μm shows less erosion rate when compare with bare specimen and coating specimen at 1-hr. which has low thickness of coating. The low thickness of coating result from short period time that not enough to resistance erosion corrosion.

After the completion of erosion-corrosion test, SEM is carried out in some of specimens to show the effect of the erosion in the coating layer and bare specimens. Figure 10 shows the effect of erosion in heavy crude oil in bare a specimen.
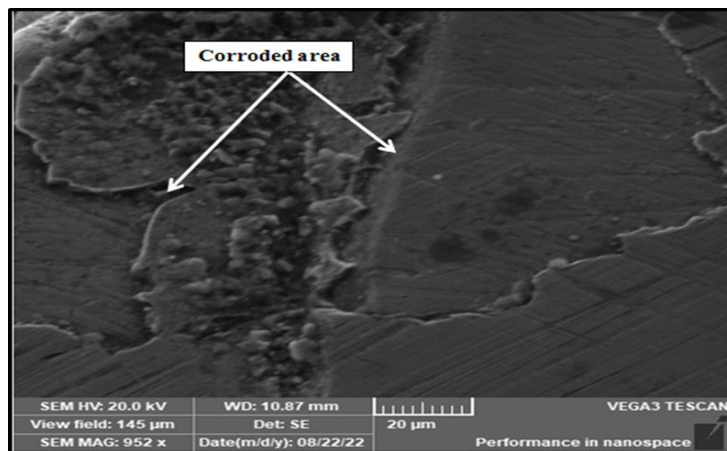
**Figure10:SEM image of Erosion-Corrosion for Bare Specimen**

On the other hand Figure11shows the erosion-corrosion SEM images for coated specimen at 1-hr. and 2-hrs. time of coating.
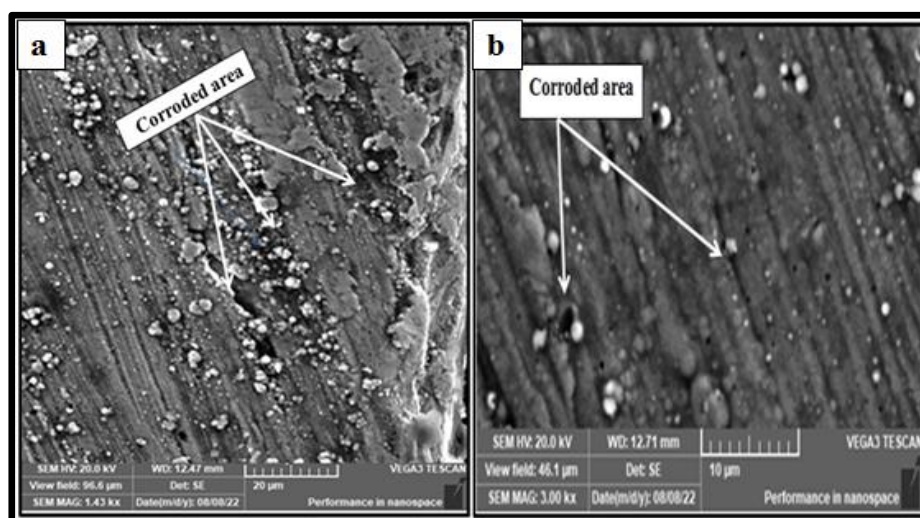


**Figure 11:SEM Images of Erosion-Corrosion for (a): Coated Specimen with 1-hr. Time of Coating and (b):Coated Specimen at 2-hrs Time of Coating**

SEM shows that the surface layer is more corroded in bare than coated specimens also the erosion-corrosion for coated specimen is characterized in appearance with grooves, waves, rounded holes, and/or shaped grooves. However, the resistance of samples coated with a time of 2-hour was higher than the resistance of samples coated with a time of 1-hour by a small margin.

**4. Conclusions**
• The inclusion of SiC nanoparticles in Ni–P binary alloy matrix was done successfully from an electroless plating bath.
• Surface morphology of both pure and composite coated specimens
changed significantly after heat treatment.
•Both of the deposits heated to 600º C demonstrated the existence of metastable and stable phases.
• The composite coatings exhibited higher micro-hardness than pure alloy.
• Erosion-corrosion test showed that the annealed Ni–Sic-P coatings have high erosion resistance in heavy crude oil fuel compared to bare alloy.

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