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Ceramic Coating for High Temperature Corrosion Protection of Steel

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Abstract: In high temperature environments where oxidation occurs which effects the metal in multiple ways. High temperature corrosion is typically seen in ductile steel. This can cause abrupt failure and reduce a metal's mechanical properties. A variety of ceramic coatings are available that can be applied to enhance functionality and prevent corrosion, particularly at high temperatures. The majority of ceramic coatings are typically electrically nonconducting, making them excellent insulators. It also has a high melting temperature of up to 4000°F and remarkable abrasion resistance. Due to these characteristics, they offer exceptional corrosion resistance and strong abrasion resistance in high temperature conditions. In this research, the team synthesize ceramic coating which may provide exceptional abrasion resistant as well high temperature oxidation resistance. Before selecting the ingredients, it needs to be examined the compatibility between mild steel (sample) and constituents of ceramic coating, majorly the thermal expansion characteristics. It must be identical otherwise fractures can be produced on the coated surface of a metal. Samples were made as per demand, following collecting a sample and cleaned accurately to attach the coating appropriately. After application of coating on sample it should place in furnace which promote diffusion on a metal surface and construct protective barrier. Further, dipping the sample in 3.5% NaCl solution for 21 days and execute the OCP and EIS test of naked sample and coated sample correspondingly for 5 days. Potentiostat provides several graphs viz OCP, Bode plot, Nyquist impedance plot and Tafel Extrapolation. High temperature oxidation resistance of the ceramic covering was investigated using TGA analysis. The weight gain of coated sample is roughly 1mg/cm^2 compared to about 4 mg/cm^2 for plain sample in TGA curve. This illustrates that the ceramic coating is averting oxidation of steel surface at high temperature of 500°C.

Keywords: ceramic coating, high temperature corrosion, impedance, oxidation resistance.

I. INTRODUCTION

One sort of corrosion that doesn't require a liquid electrolyte is high temperature corrosion. Instead of reacting with ions in a solution at high temperatures (more than 400^0C), metal ions in this kind of corrosion directly interact with gaseous molecules in the environment. The term High temperature corrosion has an impact on a number of industries, including (a) the oil and petrochemical sector (b) the power generating business (c) the heat treatment of steel (d) the aerospace and gas turbine industry (e) the mineral processing industry (f) and the metallurgical industry (g). Chemical sector (i), Paper and Pulp industry (i), and the Automotive sector (h) The negative impacts of high temperature corrosion include (a) a decrease in a metal's ability to support loads due to material loss. (b) A metal may suddenly fail brittle under load conditions if its brittleness becomes excessively because of carburization and nitridation. (c) Precipitation-induced stress on a metal surface can be seen in a metal and can seriously harm a material. (d) A gap is formed in an alloy matrix as a result of microscopic corrosion. Together, these vacancies create a void inside the matrix of an alloy.

This decreases the mechanical strength of a metal and raises the stress concentration factor in the metal. Metal oxide forms on an alloy or metal surface at higher temperatures(Birks et al., 2006). It functions as a stable protective layer that fends against future corrosion, such as high temperature corrosion attacks including carburization, sulfidation, and nitridation. In most cases, the name itself denotes the method of corrosion. For example, the phrase "oxidation" generally refers to oxides, "sulfidation" generally refers to sulphides, "oxidation/sulfidation" often refers to a combination of sulphides and oxides, and "carburization" generally refers to carbides.

The ionic-defect structure, thermodynamic stability, and shape of high temperature oxide scale are important elements in figuring out how resistant a metal or alloy is to oxidation in a specific environment. Two fundamental elements can affect the ceramic coating of a metal or alloy at higher temperatures. The first is thermodynamics, which affects whether or not the corrosion reaction will occur. Kinetics is the second, and it allows us to estimate the rate of corrosion.(Attarzadeh et al., 2021)

II. EXPERIMENT

A. *Preparation of Sample/Specimen*

Before applying a ceramic coating, a sample must be prepared. To ensure that the ceramic coating adheres well to the sample, it is crucial to take this procedure. It first requires sand paper with grit sizes ranging from P300 to P1500 according to ISO/FEPA specifications. It helps to improve surface finish and get rid of the extremely thin layer of oxide layer creation on the metal surface as the grain size grows from P300, which is very coarse, to P1500, that is very fine in nature. Dimensions of samples are (15X0X0.5) and (5X3X0.5) in cm. Specification of mild Steel.

Chemical composition of Mild Steel:

- CARBON 0.16 to 0.18 % (maximum up to 0.25% is allowable)
- MANGANESE 0.70 to 0.90 %
- SILICON maximum 0.40 %
- SULPHUR maximum 0.04 %
- PHOSPHOROUS maximum 0.04 %

B. *Cleaning of a Sample*

Cleaning the steel surface thoroughly before applying a ceramic coating will increase the adherence of the metal surface to the coating. Metal surfaces must be cleaned using a 10% HCL solution to get rid of any last-minute residues of metal oxide and other microbes that can interfere with the adhesion of the coating to the metal surface. The metal is submerged in 10%HCL solution for approximately 10 minutes. Acid content in the solution should be low to prevent corrosion, even though it is intensely corrosive to metals. The pitting agent ferric chloride, which is generated by HCL, is particularly potent. The most popular acids that can be used for acid pickling include nitric acid (HNO3), hydrochloric acid (HCL), sulfuric acid (H2SO4), and hydrofluoric acid (HF). This technique is known as acid marinating.

C. *Preparation of a Ceramic Coating*

The preparation of Ceramic coating is prepared by mixing of the chemical ingredients in chronological order as shown below in a table.

SNo.	Chemical Ingredients	Quantity
1	H_2O	25ml
2	H_3PO_4	10ml
3	MgO	2gm
4	$\mathrm{M}_gC_rO_4$	9gm
5	$k_2C_{r_2}O_7$	4gm
6	Triton X-100	A few droplets

Table 1 Composition of ceramic coating

After the ceramic coating has been prepared, a standard brush is used to uniformly apply the coating to both sides of a metal surface. Make sure the coating is even and that no blank spots are left, particularly on metal corners. The sample is now inserted inside a muffle furnace and kept there until the furnace reaches a temperature of 350°C. When a furnace reaches a temperature of 350°C, power should be interrupted and allow the sample to cool inside the furnace. A protective layer is created when a coating is adhered to a metal surface. Further,25ml water is taken into beaker and placed the beaker on a solution agitator under room temperature condition at a medium to high rpm (revolution per minute) as per requirement. 10ml Phosphoric acid is adding in a beaker progressively and after couple of minutes until solution became homogeneous throughout, 2gm MgO is added in a solution. Exothermic reaction occurs and MgO neutralizes the phosphoric acid ($H3PO4$) further adding magnesium chromate (Mg $CrO4$) in a solution after some time until the solution become clear and acquire a room temperature. Then we add potassium dichromate ($k2Cr2O7$) in a solution and agitate continuously until the solution becomes homogeneous. It will take approximately 1-2 hours. Before application of this coating on a steel sample adding a few droplets of Triton X-100 will enhance the binding characteristics of a coating on metal surface(Jha, 2021).

D. *Application of Coating on Metal Surface (sample) and Baking Process*

After the preparation of ceramic coating, the coating is applied evenly on both sides of a metalsurface by normal brush. Make sure uniform coating is there and no blank spot is left behind especially on the corners of a metal. Now, sample is put into a muffle furnace and kept inside until temperature of a furnace reach up to 350ºC. When temperature of 350ºC is achieved we cut the power of a furnace so that sample get cooled.. Coating is fused on a metal surface and forms a protective layer.

Figure 1:(a) bare sample (b) 10% HCL solution for cleaning (c) Ceramic coating preparation (d) Ceramic coating applied on a sample before baking process (e) Ceramic coating on a sample after baking process

E. Corrosion Monitoring and Testing Procedure

Corrosion testing and monitoring of a Coated metal sample is required for analysing the anti- corrosion property or corrosion resistance of a coating. An electrochemical technique provides a quick, reliable and comparatively inexpensive method to obtain the electrochemical properties of a material. These techniques are based on the ability to detect corrosion of a metal by observing the response of a charge transfer process to a controlled electrochemical disturbance.

F. Potentiostat

It is device which is used to conduct various types of corrosion test such as OCP, EIS by controlling three main electrodes. Potentiostat generates polarization curves which is used for predicting the corrosion characteristics of a material or coating. Polarization scan allows for the extrapolation of kinetic and corrosion parameter of a sample. While scanning, the reduction or oxidation of an electro active species can be limited by charge transfer and the movement of reactants. All these factors are incorporated by the polarization scan; hence it is a great importance of having a system that can produce reliable and repeatable polarization curve across multiple no of cycles(*Basics of EIS_ Electrochemical Research-Impedance Gamry Instruments*, n.d.).

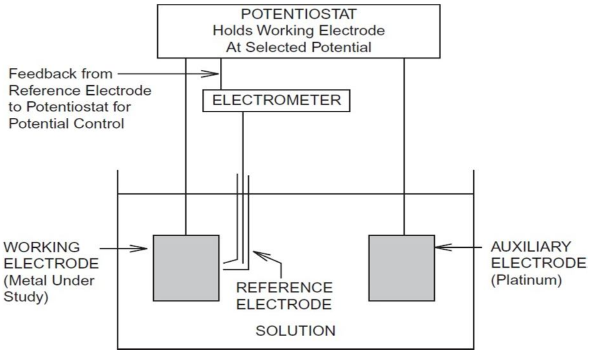

Figure 02 Potentiostate circuit diagram/polarization-cell arrangement.(*Basics of EIS_ Electrochemical Research-Impedance Gamry Instruments*, n.d.)

Setup for conducting open circuit potential test

It is based on three electrodes configuration system in which reference, working and counter electrodes. Firstly, working electrode is prepared in our case mild steel is used as a working electrode. Working electrode is place centrally beneath the cylindrical vessel and tighten the setup to prevent any leakage of an electrolyte (NaCl solution,3.5% by weight or 0.6M) which is filled in a cylindrical vessel. Reference electrode is made up of graphite rod and loosely bound with connecting wire. Here, Ag/AgCl is used as a reference electrode. Run the setup which is connected through computer to generates various graphs. Now, establish a steady state open circuit potential for 1-hour duration to generates the OCP curve for sample, after that proceeding for electroscopic impedance spectroscopy (EIS) test, EIS gives bode plot, Nyquist impedance plot and Tafel extrapolation curve which can be used in calculation the corrosion current density of a sample corresponding to corrosion potential or impedance

G. Tafel Extrapolation

The most essential method for experimentally estimating I(corrosion current) is by Tafel extrapolation. This process needs the occurrence of a linear or Tafel portion in the E(Emf) vs log current density graph. A potential scan of nearly 6300 mV about E(corrosion potential) is usually crucial to determine if a linear portion of at least one period of current should be present, so that a practically accurate extrapolation can be made to the E (corrosion potential). Such linear portions are demonstrated for experimental cathodic polarization graph. It governs the irreversible behavior of an electrode.

H. Dipping the sample in NaCl solution

After application of ceramic coating on mild steel. The ceramic coated sample dip in 3.5% (by weight) NaCl solution at room temperature for 21 days which result in no sign of corrosion on the surface of sample after 21 days.OCP test is conducted on bare sample for reference.

Result and analysis

The following graph shows the OCP of bare metal surface**.**

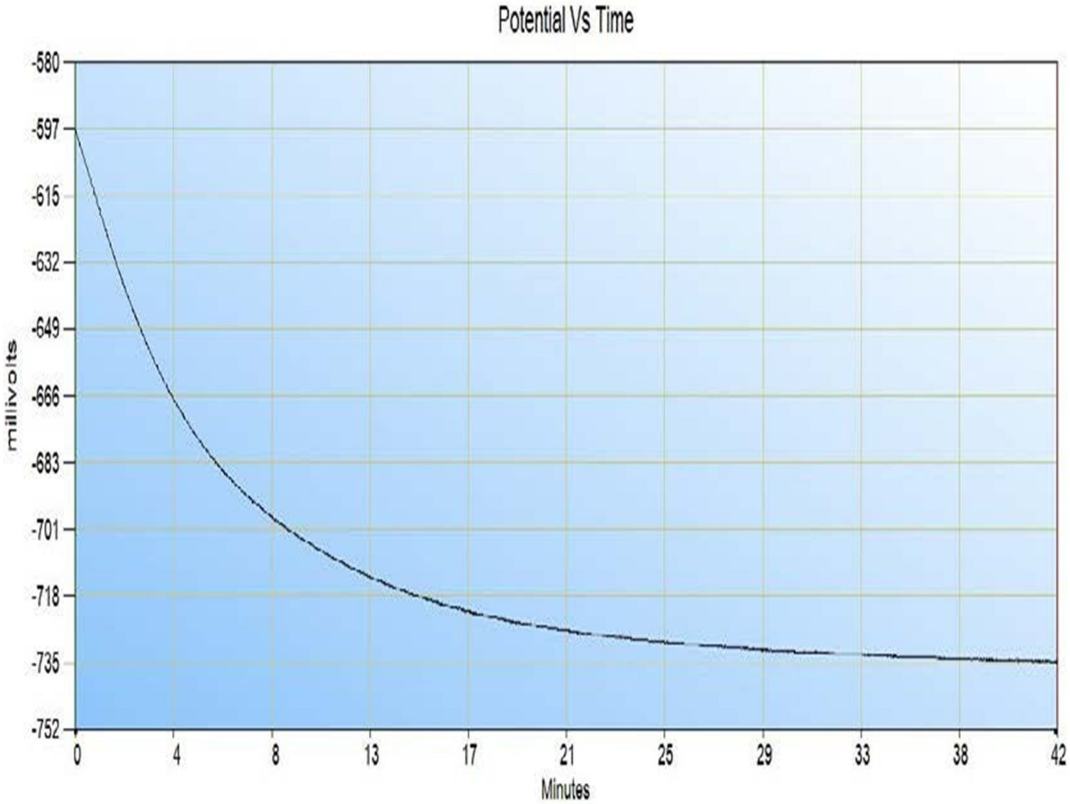

Figure 03 Variation of impedance with respect to immersion time of bare sample at 0.1Hzand 3.5%

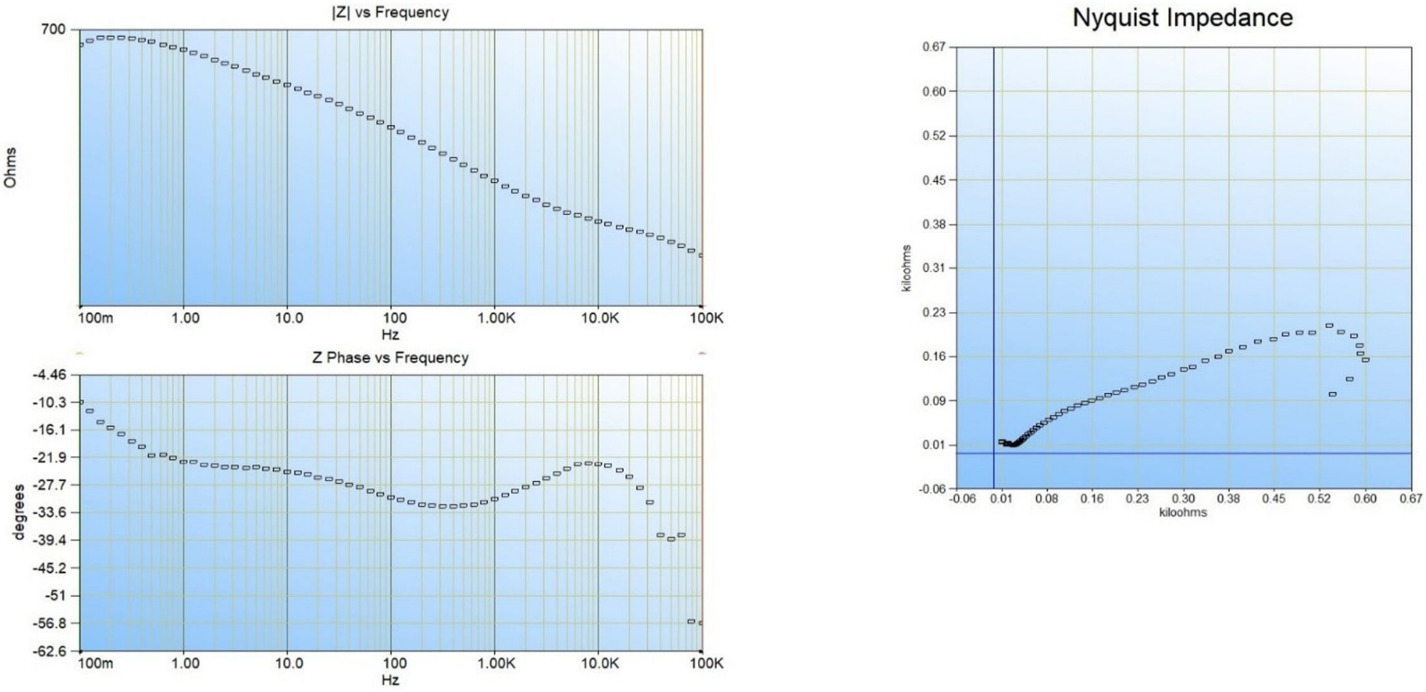

Figure 04 Bode plot Z(Ohms) vs frequency (Hz) curve, Nyquist impedance curve and phage angle (in degree) vs frequency (Hz) curve of bare sample. Here we can observe that initially the OCP curve of bare sample is not stable it become stable after 40 minutes of immersion time in 3.5% wt. NaCl.

I. *EIS curves of Bare Sample*

EIS test is conducted on bare sample for reference. The following graph shows the bode plot and Nyquist impedance plot and Tafel extrapolation curve of bare sample.

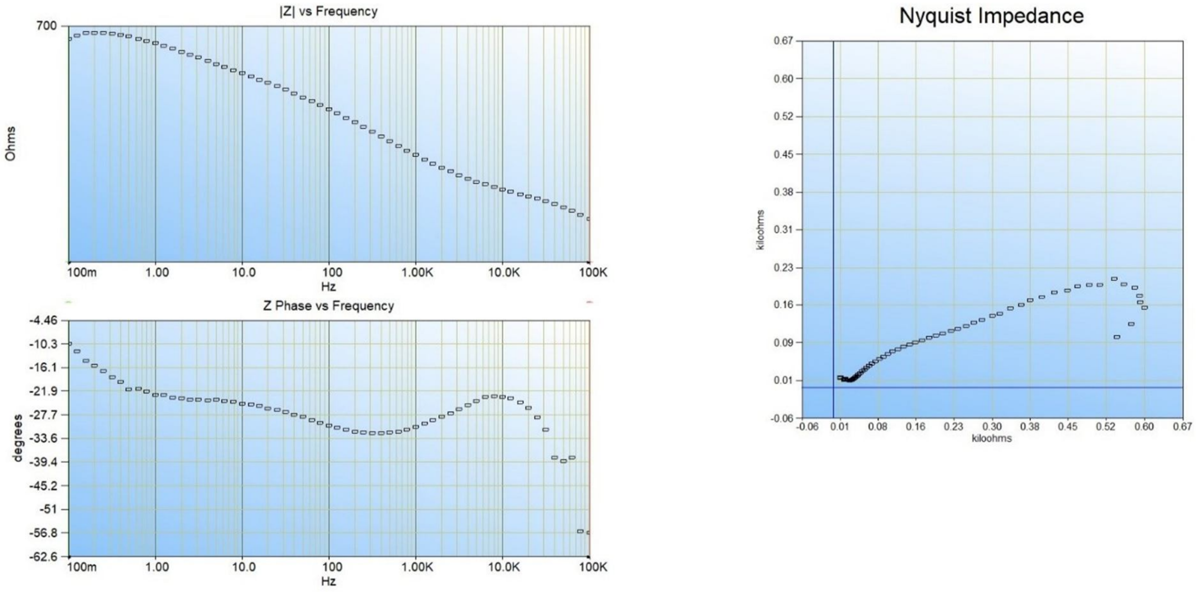

Figure 05 Bode plot Z(Ohms) vs frequency (Hz) curve, Nyquist impedance curve and phase angle (in degree) vs frequency (Hz) curve of bare sample.

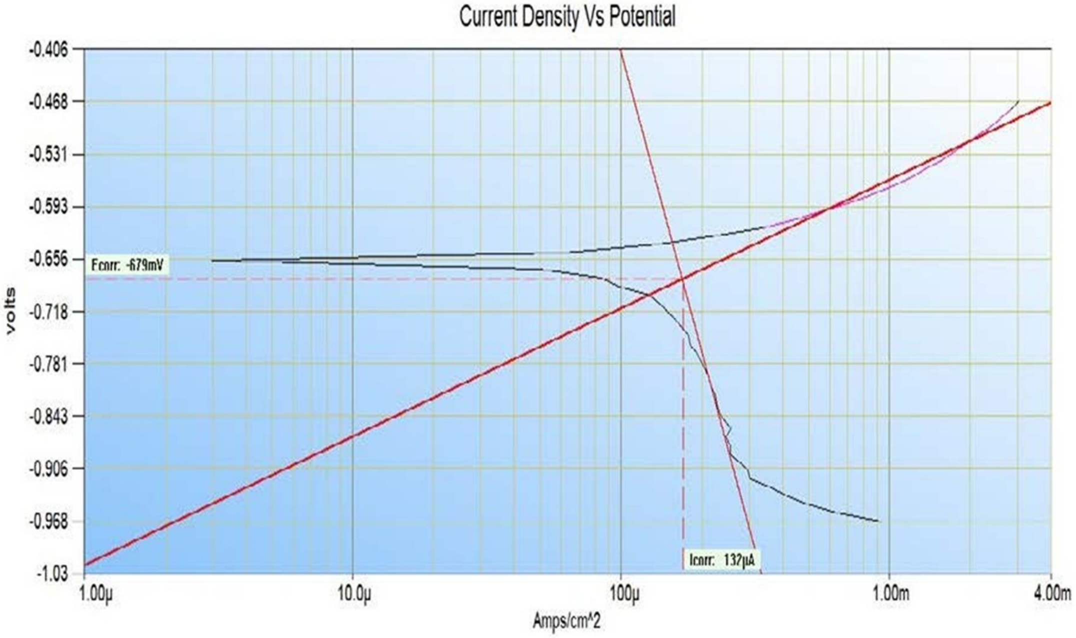

Figure 06 Impedance vs current density (Amps/cm^2) plot of bare sample at 0.1Hz and 3.5%wt. NaCl electrolyte. From figure 05 it can be concluded that the rate of corrosion or current density is of bare sample is 132µA(micro ampere) at open circuit potential of -679mV.

J. Open Circuit Potential graph of Ceramic Coated Sample

Open circuit potential test performed on ceramic coated sample. The following curve represent the potential vs time graph of ceramic coated sample.

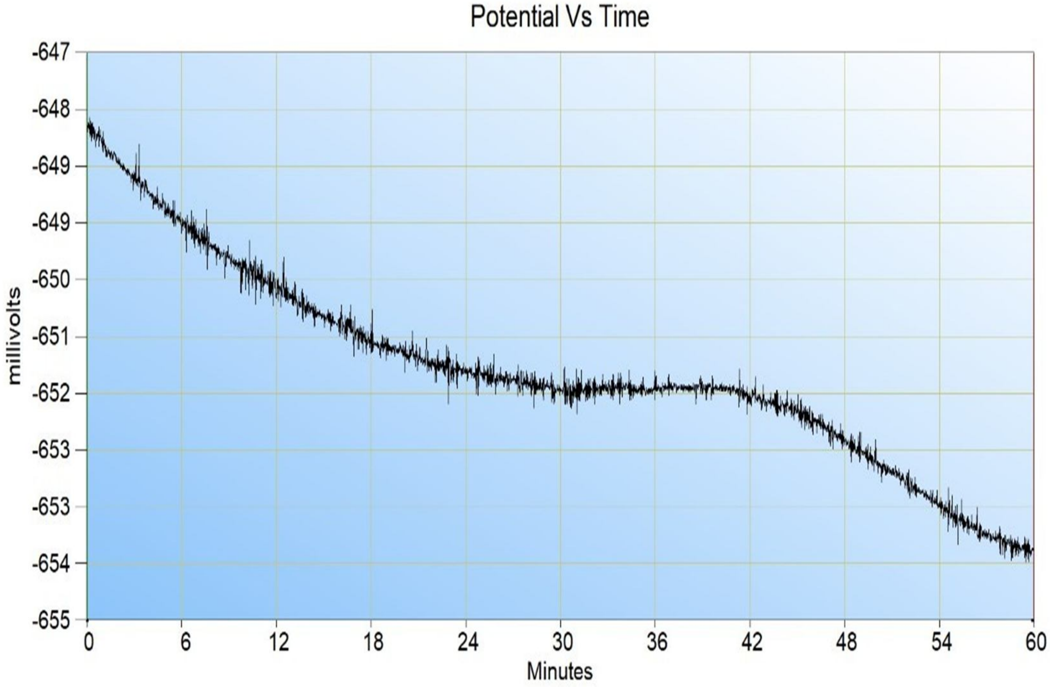

Figure 07 Variation of OCP with respect to immersion time of ceramic coated sample at 0.1Hz and 3.5% wt. NaCl electrolyte.

K. EIS Curves of Ceramic Coated Sample

Electrochemical impedance spectroscopy test performed on ceramic coated sample.Following curve represent bode plot, Nyquist plot and Tafel extrapolation.

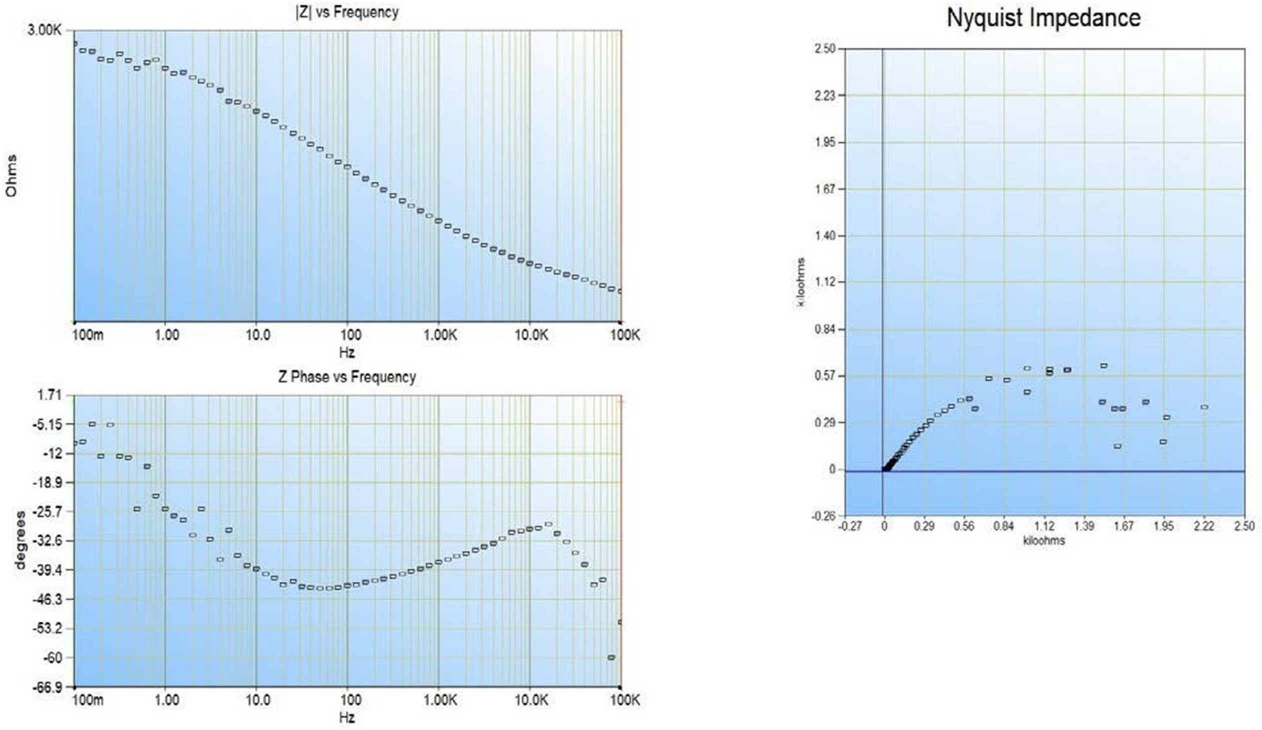

Figure 08 Bode plot Z(Ohms) vs frequency (Hz) curve, Nyquist impedance curve and phage angle (in degree) vs frequency (Hz) curve of ceramic coated sample at 0.1Hz and 3.5% wt.NaCl electrolyte.

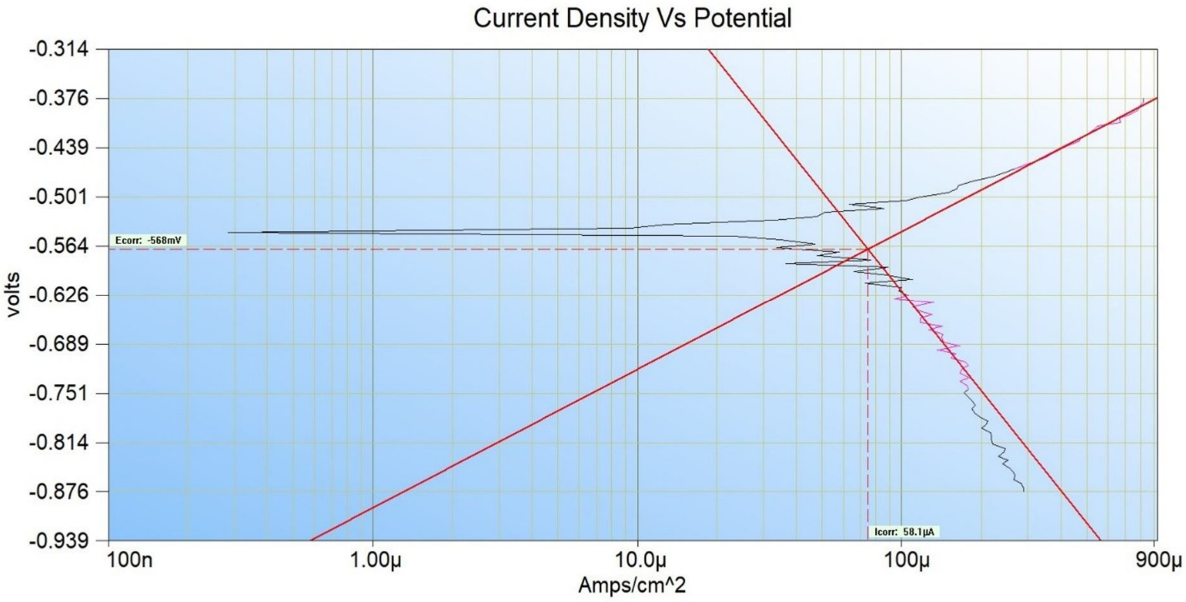

Figure 09 Impedance vs current density (Amps/cm^2) plot of ceramic coated sample at 0.1Hz and 3.5% wt. NaCl electrolyte

Here; it can be concluded that the rate of corrosion or current density is of ceramic coated sample is 58.1µA at open circuit potential of -568mV.

L. Combined OCP graph of ceramic coated sample.

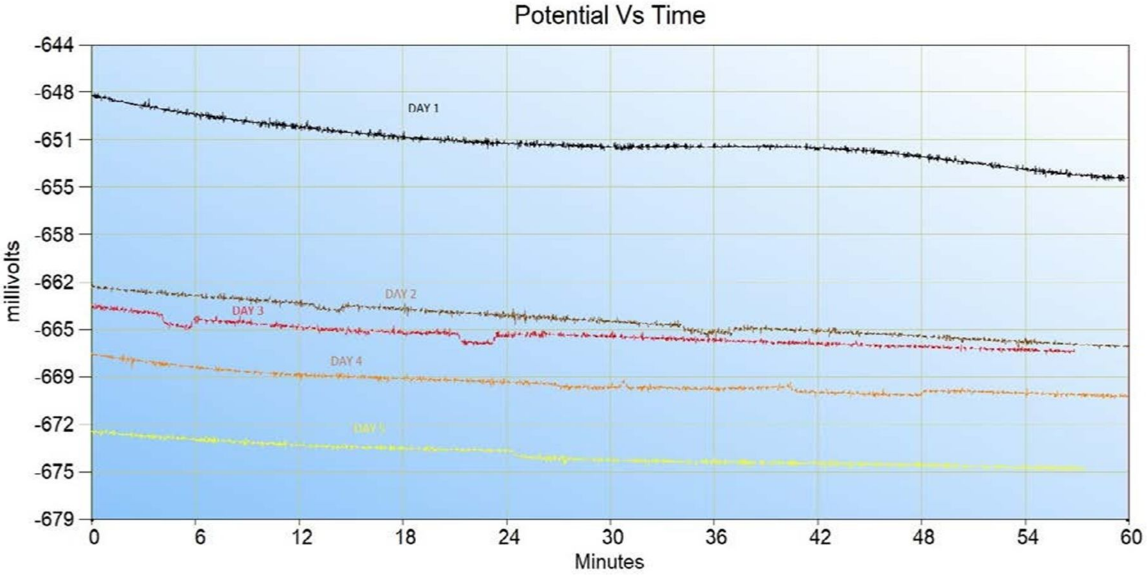

Figure 10 Variation of impedance with respect to immersion time of ceramic coated sampleat 0.1Hz and 3.5% wt. NaCl electrolyte for 5 days at 24 hours of interval.

It is clearly seen that the OCP curves of ceramic coated sample are stable corresponding to distinct OCP test conducted in 5 days at 24 hours of interval with little variation in impedance between values obtained in 24 hours of interval.

M. Combined EIS graph of ceramic coated sample

Electrochemical impedance spectroscopy test performed on ceramic coated sample. The following curve represent bode plot and Nyquist plot corresponding to EIS test conducting for 5 days at 24 hours of interval.

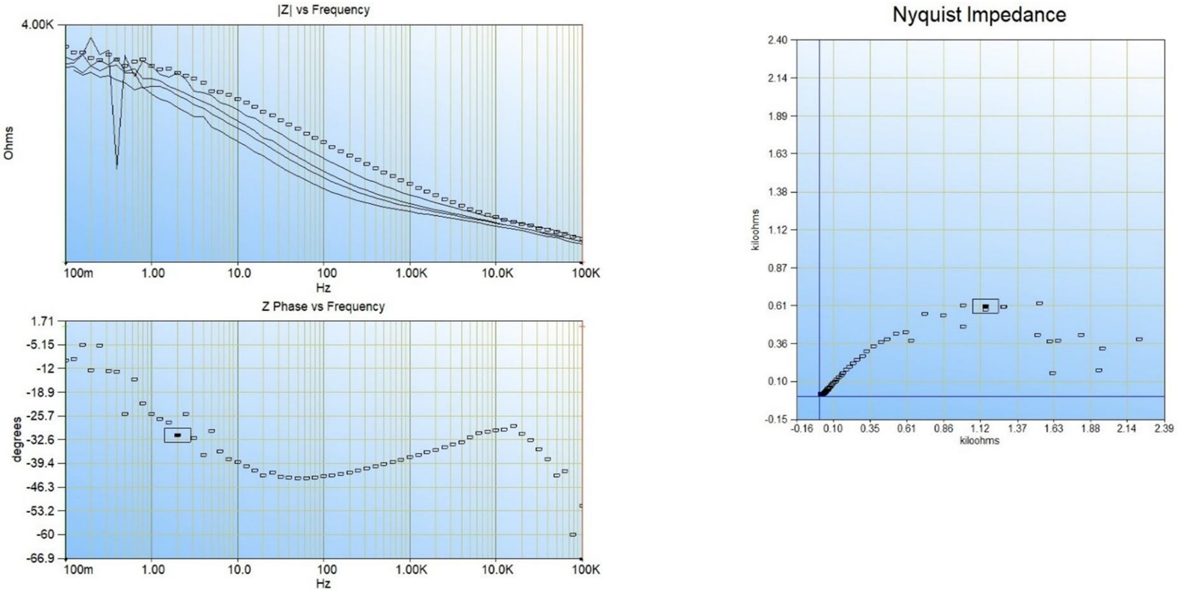

Figure 11 Bode plot Z(Ohms) vs frequency (Hz) curve, Nyquist impedance curve and phage angle (in degree) vs frequency (Hz) curve of ceramic coated sample corresponding to 5 days (24 hours of interval) at 0.1Hz and 3.5% wt. NaCl electrolyte.

The same pattern goes in case of EIS curves, the stable curves are obtained except a variation in between day 1 and day 2. Apart from rest of the curves are stable. Nyquist curve of ceramic coated sample is semicircle which shows less tendency to corrosion.

N. High Temperature Oxidation Resistance

High temperature oxidation resistance of the ceramic coating was evaluated using TGA (Thermogravimetric Analyzer) furnace. Mild steel samples were coated with the synthesized ceramic coating and its oxidation resistance at 500 ^{0}C was evaluated using TGA oxidation furnace. The sample was kept at 500^{0}C for 50 hours to measure gain in weight due to formation of oxide layer on the metal surface.High temperature oxidation study of a blank sample of mild steel was also carried out for comparison purpose. The sample for oxidation study was of the size (20 mm X 10 mm X 2 mm).The oxidation TGA (Thermogravimetric analysis) graph of both blank and coated samples is shown in Figure 11

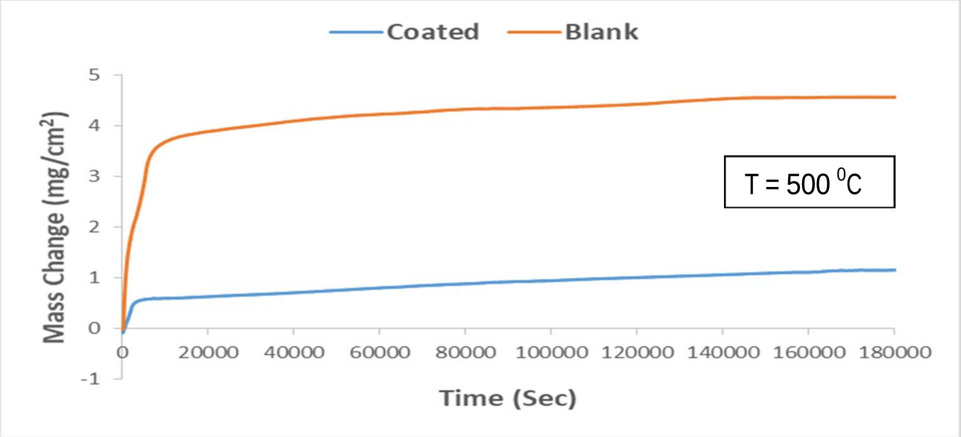

Figure 12. High Temperature Oxidation TGA graph of blank sample and ceramic coated mild steel sample at 500 ^{0}C.

The weight gain of coated sample is about 1 mg/cm^2 compared to about 4 mg/cm^2 for blank sample in TGA curve. This indicates that the ceramic coating is preventing oxidation of steel surface at high temperature of 500 ^{0}C(Kane et al., 2021).

III. CONCLUSION

OCP curves of ceramic coated sample are stable as compared to the bare sample with addition to bode plot and Nyquist. Corrosion current density is less in ceramic coated sample (58.1μA corresponding to -568 mV) as compared to bare sample (138μA corresponding to -652 mV). The weight gain of coated sample is about 1 mg/cm2 compared to about 4 mg/cm2 for blank sample in TGA curve. This indicates that the ceramic coating is preventing oxidation of steel surface at high temperature of 500°C(Boissonnet et al., 2023). On the basis of obtained data, it can be concluded that the ceramic coating improves the corrosion r resistant of mild steel sample.

IV. ACKNOWLEDGEMENT

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