International Journal of Research in Engineering, Science and Management
Volume-3, Issue-8, August-2020
journals.resaim.com/ijresm | ISSN (Online): 2581-5792 | RESAIM Publishing

Corrosion and Hardness Study of AISI1010 Sputtered with ZrO_2

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Abstract*: Production and textile industries have some of the hazards in nature. There are various hazards associated with various process in textile industry like blow room, carding, drawing, roving, spinning, cone winding, chemical storage room which cause increased rate of frequency and severity of accident. Corrosion is one of them. This study brings forward the concept of reducing the corrosion on mild steel pipeline affected by caustic soda lye in textile industry. In this paper, the preparation steps of ZrO_2 and pellet for the coating on low carbon steel (AISI 1010) by sputtering coating technique is explained and corrosion test was done. SEM images confirm the uniform coating of ZrO_2 on 1010 steel. By referring various acts and standards and recommended action has been suggested to reduce the risk associated with the operation of process in textile industries. *Hardness is also measured.

***Keywords*: Zirconium Dioxide, Sputtering, Corrosion, Mild Steel, EIS.**

1. Introduction

Corrosion is a natural process that converts a refined metal to a more chemically stable form, such as its oxide, hydroxide, or sulphide and it gradually destructs the metals by chemical and electrochemical reaction with their environment due to the reaction of the surface with substances in its external environment. It occurs whenever a gas or liquid chemically attacks an exposed surface of metal and is accelerated by warm temperatures. In textile industry, corrosion is the major problem because of using corrosive chemicals. Aiqin Gao et al. [1] discussed about silicone nano micelle dyeing using the nano emulsion containing highly dispersed dyes for polyester fabrics. Bai Xiaomin et al. [2] discussed about the corrosion resistance by Al-Al_2O_3 composite coatings used on carbon steel pipe surfaces under thermal insulation. Al-Al_2O_3 coatings were produced on the carbon steel pipe surface by cold spray (CS) technology. The results showed that when α-Al_2O_3 was added in spraying powder, the coating could obtain higher hardness and a denser microstructure. Edison Gil Pavasa et al. [3] discussed about the borosilicate glass coatings obtained on mild steel substrates by the dip-coating sol-gel technique. Different coating solutions were used in fresh and aged conditions. Anwar et al. [4] discussed about Morphology evaluation of ZrO_2 dip coating on mild steel and its corrosion performance in NaOH solution. Sathish et al. [5] discussed about the corrosion behavior of plasma sprayed Al_2O_3, ZrO_2, Al_2O_3/ZrO_2 and ZrO_2/Al_2O_3 coatings fabricated on low carbon steel. Coating was confirmed with the microstructure and the phase composition of the as- sprayed coatings was examined by scanning electron microscopy (SEM) and X-ray diffraction (XRD), respectively. The polarization test, salt spray test and immersion test were used to investigate the corrosion behavior of the coatings in 3.5% NaCl solution. The results suggested that the bi-layer of Al_2O_3/ZrO_2 coating exhibits superior corrosion resistance when compared with the other coatings. Sebahattin Kirtay et al. [6] discussed about the preparation of hybrid silica sol–gel coatings on mild steel surfaces and evaluation of their corrosion resistance. In textile industry, due to hazardous chemicals used in dying process corrosion is a major problem. In that industry, caustic soda lye is stored in the tanks to distribute for mercerizing, bleaching and dyeing processes. A report of an medium textile industry states that 45,000 liter caustic Soda lye per month was used in the range of 1452 liter/day. The pipe line used for transporting chemical is made of mild steel. The aim of this study is to reduce corrosion in pipe and detect the corroded metal due to corrosive chemicals. In an industry the corrosive chemicals are used the most for textile purpose. The total consumption of chemicals is 207018 in kg out of these amount 70991 kg of corrosive chemicals are used in the industry and also 155 chemicals are in different nature of hazard out of these amount eight corrosive chemicals are used in the industry. In our project caustic soda lye corrosive chemical has to be taken. Because it was producing more scale formation in mild steel pipeline. Caustic soda lye (Sodium Hydroxide), which is corrosive, is used for Mercerizing, Bleaching and Dyeing process. Mercerizing is the process of improving properties such as fiber strength, shrink-age resistance. Bleaching is the process to improve whiteness of textiles. Dyeing is to adding permanent color. A Material Safety. Data Sheet (MSDS) is a document that contains information on the potential hazards and how to work safely with the chemical product and safety precautions for employees placed in workplace area.

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2. Materials and Methods

A. Materials

AISI 1010 carbon steel is a plain carbon steel with 0.10% of carbon content. This steel has relatively low strength. The chemical composition of this steel is Iron (Fe) 99.18 %-99.62%, Manganese (Mn) 0.30%-0.60%, Sulfur (S) 0.050%, Phosphorous (P) 0.040% and Carbon (C) 0.080%-0.13%.

Zirconium dioxide (ZnO2), also known as Zirconia is highly resistant against acids, alkaline lyes, and exogenous (chemical, thermal, and mechanical) influences and corrosion. It has high thermal stability. It does not melt below 2680 °C, which is why it is used in high-temperature ceramics such as crucibles or furnaces. Coating means covering that is applied to the surface of an object, usually called substrate. The purpose of applying the coating may be decorative, functional and both. Functional is a type of coatings which may be applied to change the surface properties of substrate such as adhesion, wet ability, corrosion resistant and wear resistant. Coating processes may be classified into various types. There are under vapor deposition, chemical and electrochemical techniques, spraying, roll to roll coating and physical coating processes. The vapor deposition must be followed by Chemical and physical vapor deposition. Physical vapor deposition has types that are magnetron sputtering and vacuum deposition. A Scanning Electron Microscope (SEM) is the type of electron microscope that produces images of a sample by scanning the surface with the help of focused beam of electrons. The electrons facing the atoms in the sample and to be produced various signals that contain information about the surface morphology and composition of sample. From the SEM images of ZnO_2 powder in Figure 1 a & b, the particle size is around 1µm.

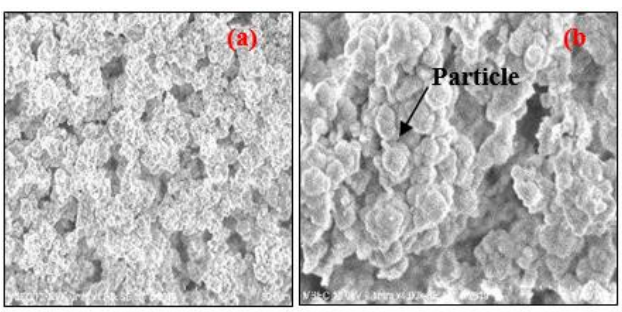

Fig. 1. SEM images of ZrO_2 Powder (a).50µm (b)10µm

B. Sputtering Process

Sputtering is the process of microscopic particles of a solid material ejected from its surface; after the material is itself bombarded by energetic particles of a plasma gas shows in Figure 2a. The target material and the substrate are placed in a vacuum chamber. A voltage is applied between them so that the target is the cathode and the substrate is attached to the anode as per Figure 2a. Plasma is created by ionizing a sputtering gas (generally a chemically inert, heavy gas like Argon). The sputtering gas bombards the target and sputters off the material to deposit.

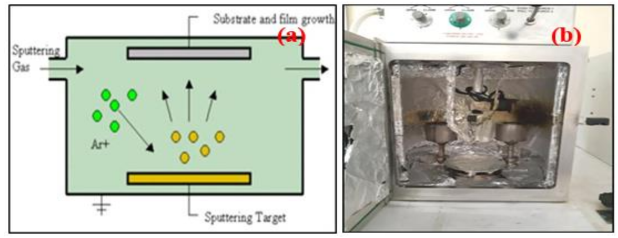

Fig. 2. (a) Sputtering process, (b)Equipment

In this method, Zno_2 was coated on steel using sputtering equipment shown in figure 2b. DC/RF Magnetron Sputtering is the single unit. It is used to making sputtering process of various target material of powder and solid particles. It is a physical vapor deposition coating technique for depositing either metal or insulating layer on a substrate in nm thickness. Sputtering utilizes low pressure plasma to generate positive ions which bombard and dislocate atoms from the solid source or target. A substrate is place in a vacuum chamber opposite to a target. Rotary and diffusing pump are used for vacuuming the chamber. RF frequencies are used in the value of 13.56 MHz and DC rating at 200 watts. The vacuum chamber is placed above the unit. It has contained place that are target place and substrate place. In this work Zirconium dioxide (cathode) in the pellet form is at target place and the substrate (anode) is mild steel plate. Voltage is placed between them. Plasma is created by ionizing a sputtering gas/Argon gas. The sputtering gas bombards the Zirconium dioxide particles and sputters over mild steel metal. After the sputtering process, coating substrate is analyzed.

C. EIS analysis

Electrochemical Impedance Spectroscopy (EIS) is a quantitative technique for the accelerated evaluation of the anti-corrosion performance of protective coatings. Within short testing times, EIS measurements provide reliable data, allowing for the prediction of the long-term performance of the coatings. The result of EIS is the impedance of the electrochemical system as a function of frequency. Impedance is opposition to the flow of AC current because of any three components that is resistive, inductive or capacitive. It is a combination of both resistance and reactance in a circuit. In this study, an EIS instrument having 3 probes CH electrochemical workstation is used and attached with computer.

3. Results and Discussion

A. Pellet Preparation

Here, the ZnO_2 power is prepared as pellet for having the sputtering coating on mild steel in sputtering instrument. The steel to be coated (figure 3a) was mechanically cut into rectangular shape using hand grinding wheel with dimensions of 75 x 25 x 2 mm. Then the sample was rubbed using abrasive sheet and cleaned by acetone to remove rust, dust. Mild steel is one of the materials which is easily corroded by low concentration chemicals. Mild steel is easily corroded caused by long storage, transferring the high concentration chemicals and thickness of the metal to be depended. A pellet die is used to form thin cylindrical pellets from ZnO_2 powder using a pellet press. The dimension of split-up pellet die (figure 3b) is 52 mm diameter and is used to making pellets for Zirconium Dioxide. The 30g ZrO_2 powder has to be taken and filled it in the pellet die initially. The whole setup is placed in the CTM (Compression Testing Machine) shown in figure 4a, and 200 kN compression load is applied for 70-80 sec continuously to get Zirconium dioxide pellet (figure 4b) in dimensions of 52 mm diameter and 4 mm thickness. Muffle furnace (figure 5a) is the instrument that is used to heat prepared pellet at 950 ^{0}C for 30 minutes continuously. Pellet gets more strength and particles are having joined together to have more hardness. The final pellet after taking off from furnace is shown in figure 5b.

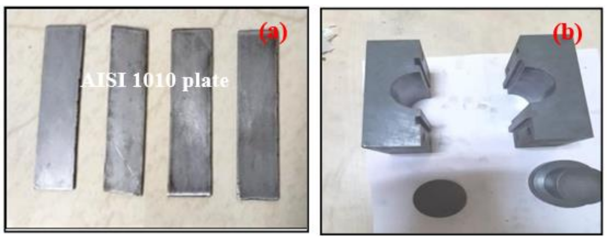

Fig. 3. (a) Mild Steel Sample, (b) Split-up pellet die

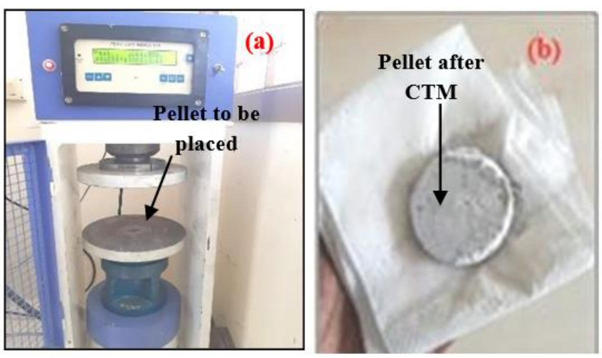

Fig. 4. (a) CTM Machine, (b) ZrO_2 powder pellet

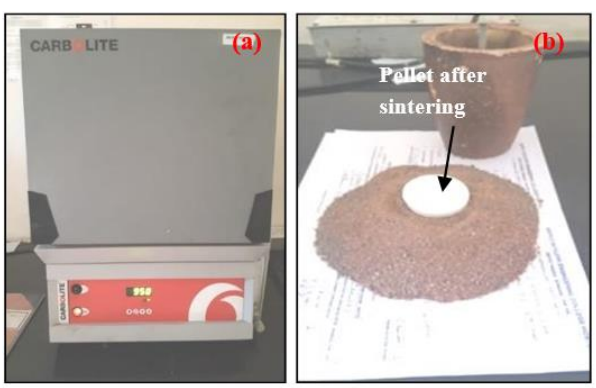

Fig. 5. (a) ZrO_2 powder pellet after Muffle furnace, (b) Muffle Furnace

B. Sputtering and EIS Results

After the pellet is prepared, the sputtering process was done and the sputtered steel is shown in figure 6b and compared to the plate before coating shown in figure 6a.

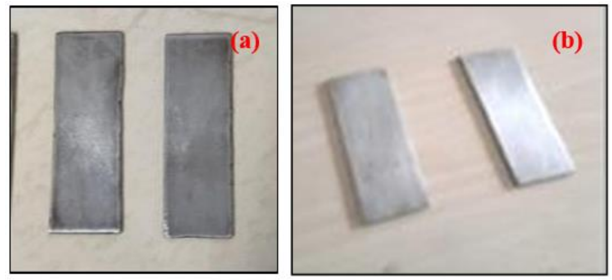

Fig. 6. (a) Steel before coating, (b) After coating

In Tafel Plot (TAFEL), potential is scanned from Initial E towards Final E. The potential may be scanned back. The logarithm of current is recorded as the function of potential. TAFEL measurement in bare metal is -0.6v as shown in figure 7. TAFEL measurement in Zirconium Dioxide coating metal is -0.61v as shown in figure 8. In AC Impedance (IMP), the base potential (figure 9) is held as constant at Initial E. A sine waveform is superimposed to the base potential. The frequency is scanned from high frequency to low frequency with 12 components per decade frequency. The current and the potential are sampled and analyzed to obtain the real and imaginary impedance.

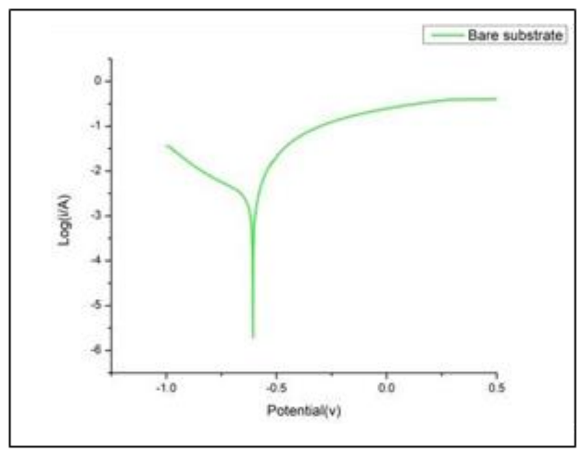

Fig. 7. TAFEL measurement for Bare substrate

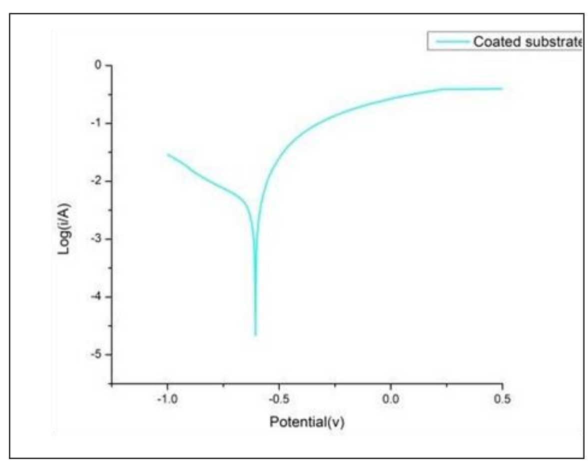

Fig. 8. TAFEL measurement for Bare substrate

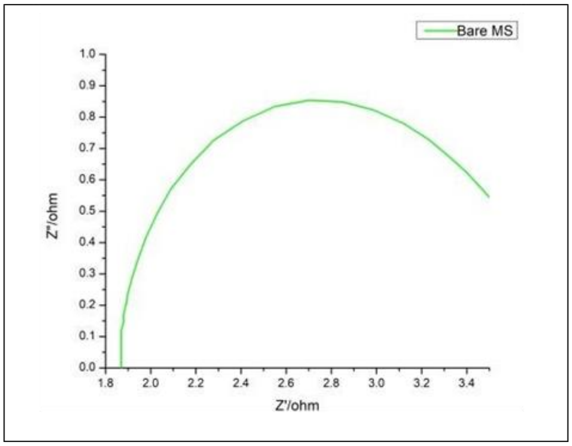

Fig. 9. AC impedance of Bare substrate

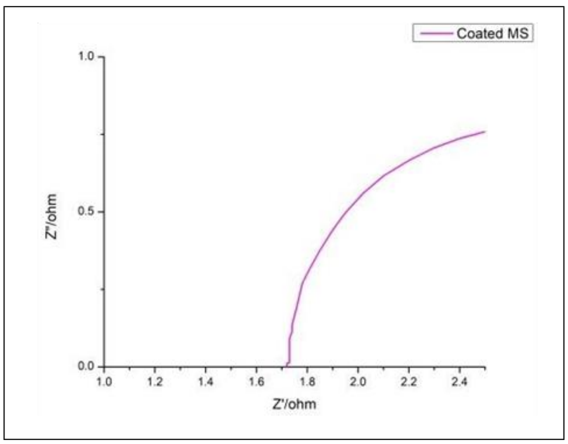

Fig. 10. Zirconia coated substrate (B)

In figure 10, the AC impedance of Zirconia coated specimen is shown and compared with the base substrate value shown in figure 9.

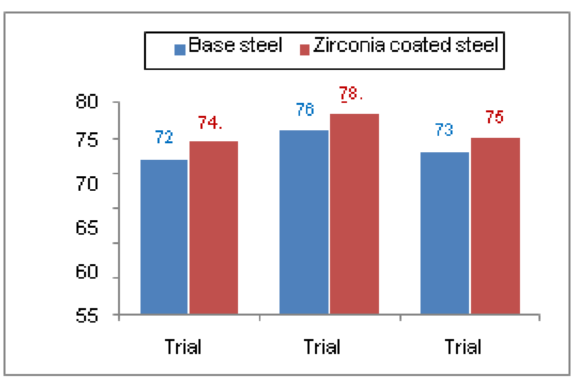

Fig. 11. Hardness value comparison

The hardness values are examined at different locations for both the base substrate and the coated specimen and also the average value of the coated specimen is calculated and listed in figure 11. It is measured by Rockwell hardness machine which is a surface sensitive measurement of hardness, rather than measuring the bulk hardness of the metal. This process is valuable for testing thin sample of materials, sample with hardness gradients at the surface and small surfaces. The results showed ZnO_2 coated samples showed maximum hardness of 78.5 HRC compared to base substrate.

4. Conclusion

In the present work, ZrO_2 pellet was successfully prepared and the same was used in sputtering process to coat ZrO2 over mild steel base metal and analyze the various chemicals in industry which causes corrosion. The outcome of this study is to identify and control the corrosion and risks faced by the employee/workers due to hazardous chemicals during the production at industry. According to that various hazards were analyzed and control measures have been made. According to hardness results, ZrO_2 coated samples showed a maximum of 78.5 HRC which is better than uncoated samples. The corrosion test results also discussed in this paper.

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