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ANTI-CORROSION COATING OF MILD STEEL USING TERNARY Zn-ZnO-Y_2O_3 ELECTRO-DEPOSITON

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ABSTRACT

Mild steel has found many engineering applications due to its great formability, availability, low cost and good mechanical properties among others. However its functionality and durability is subject of concern due to corrosion deterioration. Based on these, Yttria is selected as reinforcing particles using electroplating process to enhance the corrosion and wear behaviors. Bath formulation of Zinc- Yttria was prepared at moderated temperature and pH, to coat the sample. Corrosion and wear behaviour were analyzed using electrochemical potentiostat and abrasive test rig. The composition and microstructure of coated samples were investigated using standard method. The microstructure of the deposited sample obtained at 10 % Yttria, revealed fine-grains deposit of the Yttria on the mild steel surface. The results showed that adding of Yttria particles, improved wear behaviour and corrosion resistance in sodium chloride solution. Microhardness of the coated samples showed increases hardness values before and after heat treatment. This work established that elecrodeposition of mild steel with Yttria is promising in increasing the wear and corrosion resistance.

Keywords: Microhardness, Zinc-Yttria, Coating, Mild steel, Microstructure, Wear and corrosion.

1.0 INTRODUCTION

The modifying of the surface, which involves altering only the surface layers of a material, is becoming increasingly important with the aim to enhance the corrosion resistance of many kinds of materials[1-2]. The advantage of this approach lies in the fact that the natural physical and mechanical properties of the material are retained, while at the same time the corrosion resistance is improved[2]. Mild steel is widely used alloy in the world for different application because of its ease of fabrication, easy availability, low cost and better tensile strength among others[3]. However its functionality and durability are subject of concern due to corrosion and deterioration; as a resulted many researchers have start R & D in this area, in order to developed a coating that will protect carbon steel. Among the works are: Blejan & Muresan[4] introduced Al_2O_3 nanoparticles in the plating. Their work showed increase in corrosion resistance of Zn–Ni layers. An effort made by Tiwari et al [5] to increase the corrosion resistance of mild steel by depositing alumina coating with formulating a suitable conversion coating to provide adherent and protective layer on mild steel substrate for subsequent sol–gel coating. An adaptation coating was formulated and applied on mild steel prior to sol–gel Al_2O_3 by using silica sol and aluminium oxyhydroxide followed by heat treatment at 500°C to created mild steel surface suitable for sol gel Al_2O_3 coating.

Fayomi et al[6] developed a multilayer-modified coating and enhance the surface characteristics of mild steel against chemical deterioration with a ternary Zn-Ni-Al_2O_3 composition induced Mono and Tri ethylamine as surfactant using electrolytic chemical deposition.

Punith Kumar and Venkatesha [7] reported on composite coating of Zinc-TiO_2 on mild steel. The composite Zinc-TiO_2 coating was prepared from bath solution with different amount

of TiO_2 and they were tested for corrosion behavior by electrochemical studies using polarization and Impedance method. Their results showed that TiO_2 incorporated Zinc coatings shown better corrosion resistance towards aggressive media when compared to pure zinc coating. The embedded TiO_2 nanoparticles changed the compactness, microstructure and preferred orientation of the deposit.

Popoola and Co-Worker[8] reported on the surface modification of Zn-ZnO-SiO_2 nano composite coating on mild steel. They studied the effect of SiO_2 on Zn-ZnO using sulphate electrolyte. The microstructure characteristics of the composites coating were investigated using scanning electron microscopy couple with energy dispersive spectroscopy (SEM/EDS), X-ray diffraction and atomic force microscopy (AFM). The corrosion degradation properties in 3.65% NaCl medium were studied using potentiodynamic polarization technique and characterized by high resolution optical microscope (HR-OPM). The results showed that average hardness value of 142.5 and 251.2HV and corrosion rate of 0.13088 and 0.00122 mm/yr were obtained for the 0 and 16wt% SiO_2in Zn-ZnO.

Ghaziof and Gao [9] reported on Zn-Ni-Al_2O_3 nanocomposite coatings on mild steel using a novel sol enhanced electroplating method. The effect of alumina sol on the electrodeposition process, and coating properties were studied using cyclic voltammetry, XRD, ESEM and Tafel test. They found out, that Zn-Ni-Al_2O_3 nanocomposite coatings produced more uniform and compact deposits, with fine grained microstructure when compared to Zn-Ni coatings. The corrosion resistance of Zn-Ni coatings improved significantly by incorporation of alumina nanoparticles into the coatings

Punith Kumar et al[10] work on Zn-SiC composite coatings using sulphate plating bath dispersed with 1, 2 and 3 g $L^{-1}$of 64.28 nm SiC nanoparticles. The morphology and microstructure were observed using scanning electron microscopy, X-ray diffraction spectroscopy and texture co-efficient calculations for SiC incorporated zinc coatings. The electrochemical corrosion behavior of zinc and Zn-SiC composite coatings were studied by potentiodynamic polarization and electrochemical impedance analysis. They found out that SiC incorporated zinc coatings shown improved corrosion resistance and micro-hardness to pure zinc coating.

Fayomi et al [11] reported on the microstructural, mechanical and anti-corrosion properties of nanocomposite Zn–Al coating containing SnO_2 nanoparticles prepared from sulphates electrolyte by electrodeposition. The anticorrosion behaviour of the coating prepared with different concentrations of SnO_2 (7 and 13 g/L) and potential of (0.3 and 0.5 V) were examined in 3.65% NaCl solution by using linear polarization techniques. They performed wear and hardness test of the coatings under accelerated reciprocating dry sliding wear test and diamond micro-hardness tester. Their findings showed that incorporation of SnO_2 in the plating increases corrosion resistance and mechanical properties of Zn–Al–SnO_2composite coatings. The SEM images showed a homogeneous grain structure and finer morphology of the coatings.

Senthil Kumar et al[12] studied EN8 based metal exposed to Nitriding in Cyanide salt bath with temperature of 560^{o}C. The Ni-Zn coating was done on both Nitrided and Non-Nitrided EN8 specimens by pulse electrodeposition. Fatigue behavior of each category was studied through Cantilever type Rotary Bending Fatigue machine. The results showed that coating on nitrided specimens produces high fatigue resistance than coating on non-nitrided specimens.

Fayomi[13] reported on Co-deposition of zinc metal matrix with ZnO composite using electrolytic chloride based coating consisting of 20-40g/L ZnO particle. The composite coatings were characterized using high optic microscope (OPM). The corrosion resistance properties of Zn-ZnO composite coatings were measure using linear polarization in 3.5% NaCl solution. The results showed that addition of ZnO particles in the deposition bath obviously increase hardness values and corrosion resistance significantly

Also according to Amuda[14] stated that deposition of yttrium is beneficial to wear resistance of steel layers or surfaces in corrosive environments. Based on the forgoing it was observed that little work was documented on the use of Zn-Y_2O_3 of mild steel, since[14] reported that Yttria can formed a better surface coating than oxide of Ce and Ti. Hence the need to carryout research in this noble area became very imperative. Hence, the aim of this work is to study the mechanical properties, corrosion behavior of mild steel using Zinc-Yttria electro-deposition method.

2.0 Materials and Method

2.1 Materials

A flat plate mild steel (20 mm x 20 mm) substrate was used in this research. Other materials employed for the purpose of this work include pure zinc plate anode, Zinc chloride was purchased from Triveni intrechem Gujarat India, Boric acid was purchased from PVT ltd Gidc Vapi, Gujarat India, Glycine, Thiourea, Zinc oxide(55nm, 95 %purity) was purchased from Suvidhi industries, Vapi – 396195, Gujarat India and Yttrium oxide (45nm, 98 %purity) was purchased from by GH Chemicals Ltd, Saint Hyacinthe, Québec Canada.

2.2 **Method**

2.2.1 Sample Preparation

The Mild steel plate was sectioned using automatic struers high precision cut-off machine which is connected to lubricant supply to cool the blade and sample during the cutting process. The mild steel plate was cut into equal plates of about five samples with dimensions of 20mm by 20mm.

2.2.2 Electro-deposition

The mild steel sample was dipped into 1M of HCl solution for 10 seconds followed by rinsing into the de-ionised water. Analytical grade chemicals and de-ionised water were used to prepare the plating solutions at 35˚C, to easily dissociate any agglomerate in the bath. During the plating, the solution were stirred using the magnetic stirrer for the solution to plate well on the mild steel. The bath compositions admixed for the coatings are shown in Table 1 and they were prepared two weeks before the plating. Boric acid was added to obtain a pH of 4.5. An innovative Static current electro-deposition was used to produce Zinc- Yttria coating. A current density of 0.5A/cm^2 was used for the deposition. The rinsing was done in distilled water for 5 seconds then later air dried.

2.2.3 Characterization of the Electrodeposited Samples.

The X-ray diffraction (XRD) patterns of the samples were obtained with an X‟PertPro PANalytical, LR 39487C. XRD diffractometer using Cu Kα radiation (40 kV, 40 mA). Stepwise increase for small angle was 0.01° over the range of 1 to 8° and wide angle rate of 1° 2θ min^{-1} over the range of 8 to 90° (2 θ).

The microstructure of the samples before and after heat treatment was analyzed using optical microscope. The samples were placed on the microscope stand and the microscopic

lenses, were focused on the surface. A magnification of x 1000 was achieved using the optical microscope. Also the morphology of the deposited sample was further examined by scanning electron microscopy using a JEOL JSM 6390 electron microscope.

Emco-test micro-hardness tester machine was used in determining the hardness values of the samples. Indentation of five points and 15 seconds dwell with 100 g load were used in this work.

2.2.4 Corrosion Test

The samples were embedded in epoxy resin leaving a working area of 0.785 cm^2. The working surface was ground with grinding papers from 600 to 1000 grit, cleaned with distilled water and ethanol. A conventional three electrode cell, consisting of Ag/AgCl, Platinum and coated mild steel was used as: reference, counter and working electrodes respectively. The electrochemical measurement was done with Autolab PGSTAT 101 Metrohm potentiostat/galvanostat. An electrolytic cell containing 50 ml of 3.65 wt.% NaCl solution, with plated sample, a Platinum which works as counter electrode and Ag/AgCl as reference electrode were used. The potentiodynamic potential scan was fixed from -1.5 V to +1.5 mV with scan rate of 0.012 V/s. The electrochemical corrosion test was performed at room temperature in a static solution.

2.2.5 Abrasive wear Test

Abrasive wear occurs when a hard rough surface slides across a softer surface. The samples were cut to 10mm x 10mm and they were weighed before the test. The sample was inserted in sample holder and tightened to avoid falling of sample. The test was run for 200 revolutions with automatic stop. The sample was pressed into the wheel by a 5N weight loaded lever and 10N after the first test is done. The samples were cleaned with ethanol solution before and after each test. Average mass loss was estimated by measuring the mass loss (Δm) in the specimen after each test divide by the time (see Equation 1).

$$W_r = \frac{\Delta m}{T} \text{ ---------- (1)}$$

Where Δm is mass loss, T=time in minute

3.0 RESULTS AND DISCUSSION

From SEM/EDS analysis in Figure 1 showed that, there is no present of yttrium in the coating but in Fig. 2, yttrium peaks was well evidence in the EDS at 10 wt% Y_2O_3–ZnO-Zn samples. This implies that yttrium, after released from yttria, possibly appeared in formed of passive films of Y_2O_3 on the mild steel.

Fig. 3, Fig. 4 presented the AFM topographical view of the composition matrix coating in two (2D) and three-dimension (3D). The uniformity of the coating was achieved in Fig. 4 than Fig. 3, hence the topography is more uniform due to the formation of fine structure and sub-grains.

The X-Ray Diffraction analysis of the Zn-ZnO is showed in Fig.5a, while Fig. 5b showed the XRD pattern of the Zn-ZnO-10Y_2O_3. One can observed great differences between the two spectrums. The Fig. 5a mainly showed the presence of Fe, Zn and ZnO phases while Fig. 5b revealed phase composition of: Zn, Fe, ZnY_2, $ZnOY_2$, Zn_3Y_2 and ZnOY. From the XRD spectrum it was cleared that there is hard phases of ZnY_2, $ZnOY_2$, Zn_3Y_2 and ZnOY. This is in par with the previous work of [15-17].

The particle size of the coating was calculated using the Scherres equation

$$D = \frac{K\lambda}{\beta Cos\theta}$$

Where θ is the angle between the incident and diffracted beams (degree), β is FWHM (full width at half maximum), D is the particle size of the sample (nm) and λ the wavelength of the X-ray.

The XRD pattern of Zn-ZnO-Y has similar features to that of Zn-ZnO but with more diffraction peaks and phases, which could be attributed the present of Y_2O_3 in the coating formulation. The average grain sizes of the particles were found to be: 102.5nm and 95.5nm for Zn-ZnO and Zn-ZnO-Y coating respectively.

The optical microscope was used to study the microstructures of the samples before and after heat treatment. The optical image was taking at x1000 magnification (see Fig.6, Fig. 7). From Fig. 6, it can be seen clearly that as wt% of Y_2O_3 addition increases in the formulation, the microstructures becomes more finely and uniformly, for example compare Fig.6a with Fig. 6e. This implies that addition of Y_2O_3 help to refine the grain; this can be use to support the earlier claimed that Fig.5b is more uniform and fine than Fig. 5a. Also the optical microstructures of samples after heat treatment were illustrated in Fig.7. From Fig. 7, it was observed that the microstructure appearance still finer and uniformly. The heat treatment stabilizes the material matrix and the firmness of the deposited sample with Zn-10ZnO-10Y_2O_3. This was an improvement from the work of [13] which observed that thermal treatment lead to coarse and cracks surface deposited surface.

Micro-hardness test was performed to determine the hardness values of differeent samples(see Fig.8). It was observed that the hardness of the samples increases with an increase in the weight percentage of yttria content. The hardness value obtained for mild steel was 115 Hv, the average hardness values of 155.76 and 156.4 Hv were obtained for zinc-yttrium deposited samples before and after heat treatment. The finer microstructure formed due to the addition of yttria and the various phases formed after coating: ZnY_2, $ZnOY_2$, Zn_3Y_2 and ZnOY is the main factor responsible to the improvement in the hardness values of the deposited samples. Decrease

in hardness after heat treatment at higher percentage of yttria content could be mainly attributed to the dissolution of the ZnY intermetallic compound as a second phase after heat treatmemt.

Wear abrasion test rig was used to perform the experiment using load of 5N and 10N respectively on the coated and uncoated samples. The results of wear test are showed in Fig. 9. The coated samples shown great resistance to wear abrasion with the average mass loss of 0.005g/min compared to the control sample with 0.035g/min average mass loss(see Fig. 9). The sample of Zn-10gZnO-10Y_2O_3 composition shown unusual expectation with the lowest mass loss of 0.002g/min at 5N load and have a best wear resistance compare to all the samples. This results obtained can be attributed to the hard phases of ZnY_2, $ZnOY_2$, Zn_3Y_2 and ZnOY formed after Yttria addition, this increased the hardness values and wear resistance of the materials. The wear resistance of the samples was observed to be the best at lower load of 5N. This observation of increases in wear rate with increases in load is in par with earlier work of [18-19].

Fig. 10 and Table 2 show corrosion potential versus current. The values of the corrosion potential in the presence of Yttria is more positive than the uncoated sample, for example the potential and current density changes from -1.547 to -1.341 V and 12.404 to 1.802 mA/cm^2 for the control and Zn-10gZnO-15Y_2O_3 samples respectively. Therefore one can conclude that the presence of Yttria in the coating shifted the corrosion potential of the steel in positive direction. The improvement of the corrosion stability of the steel is as a result of the action of the effective Yttria cathodic coating in the stabilization of the passive state of the steel. This shows that the coated sample have more corrosion resistance than the uncoated mild steel in this order: Control> Zn-10ZnO> Zn-10ZnO-5Y_2O_3> Zn-10ZnO-10Y_2O_3> Zn-10ZnO-15Y_2O_3

The optical micrographs of the corroded samples are illustrated in Fig. 11. It can be observed in the microstructures that as the percentage of Yttria increases in the bath there is less

corrosion attack on the material. This support the earlier claimed that the uncoated sample has a higher corrosion rate that the coated sample.

4.0 CONCLUSIONS

From the results and discussion above the following conclusions can be made:

1. Hardness results increases after the mild steel was coated with Zn-ZnO-Yttria(115 to 156.4Hv).
2. The Zn-ZnO-Yttria coating has a higher corrosion and wear resistance
3. The finer microstructure formed due to the addition of yttria is the main factor responsible to the improvement in the hardness values, corrosion and wear resistance.
4. It have been established that Yttria can be use to improve the surface hardness, wear and corrosion resistance of mild steel coating.

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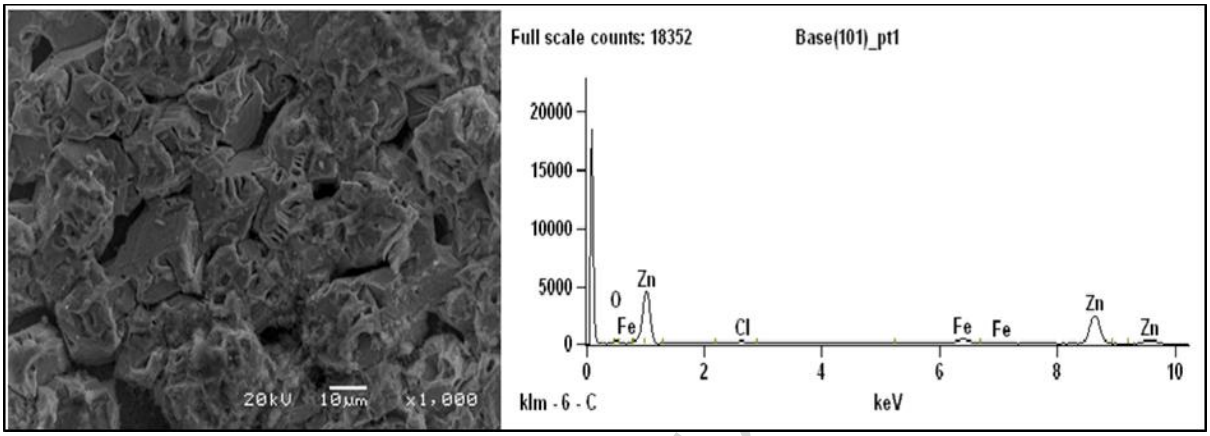

Fig. 1: SEM Surface morphology of deposited Zn-10ZnO

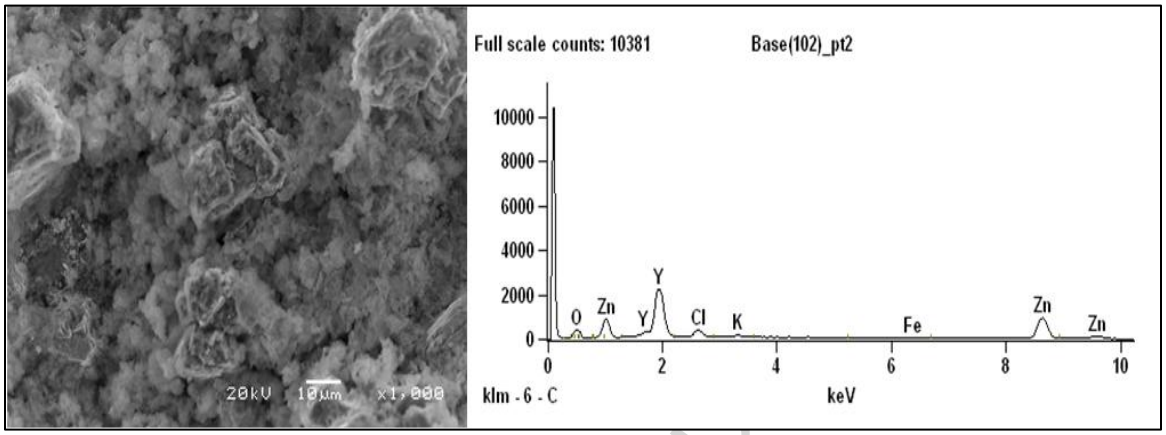

Fig. 2: SEM Surface morphology of deposited Zn-ZnO-10Y_2O_3

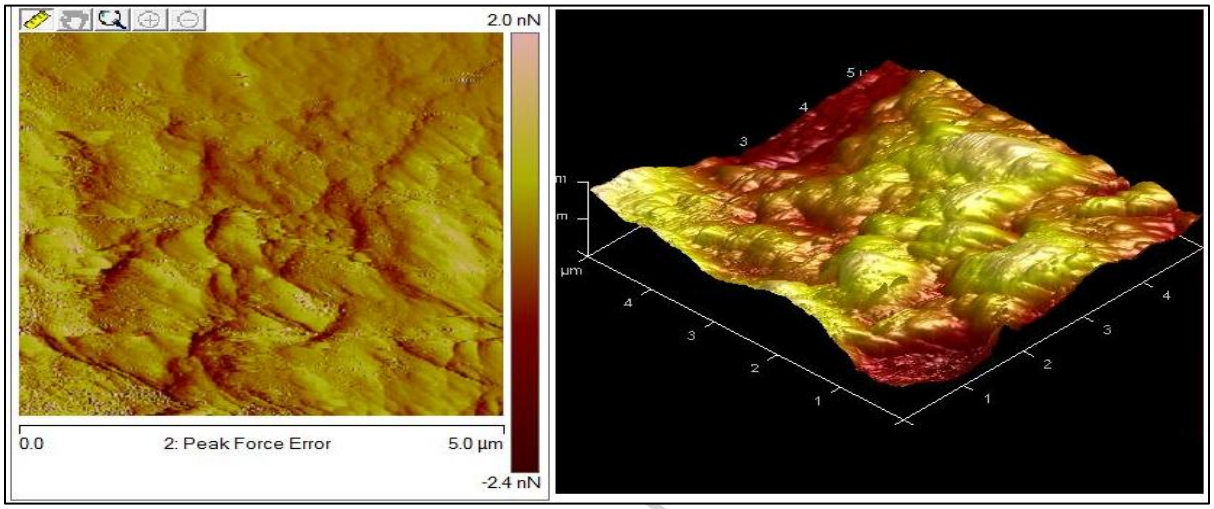

Fig.3: AFM image of the surface film of Zn-10ZnO

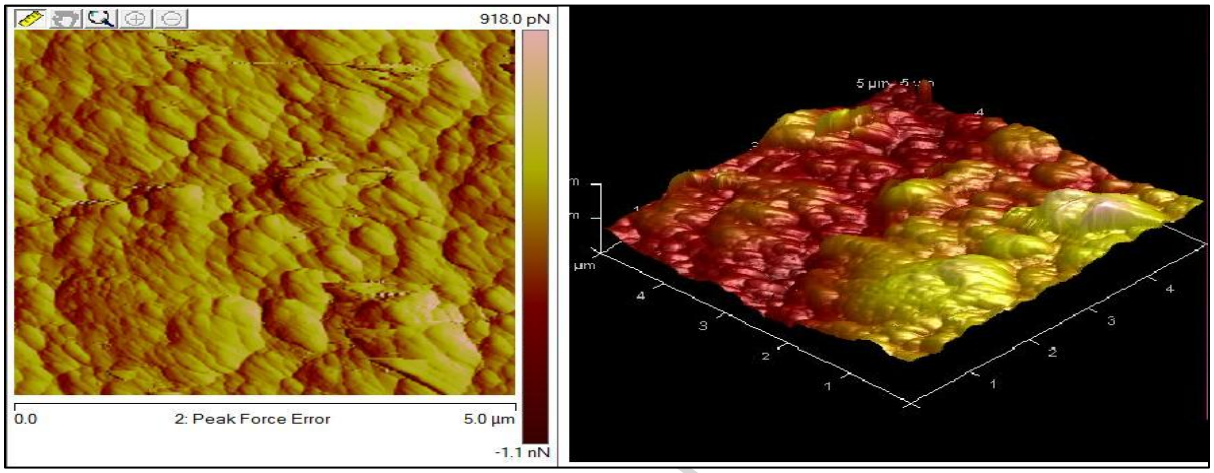

Fig. 4: AFM image of the surface film of Zn-10ZnO-10Y_2O_3

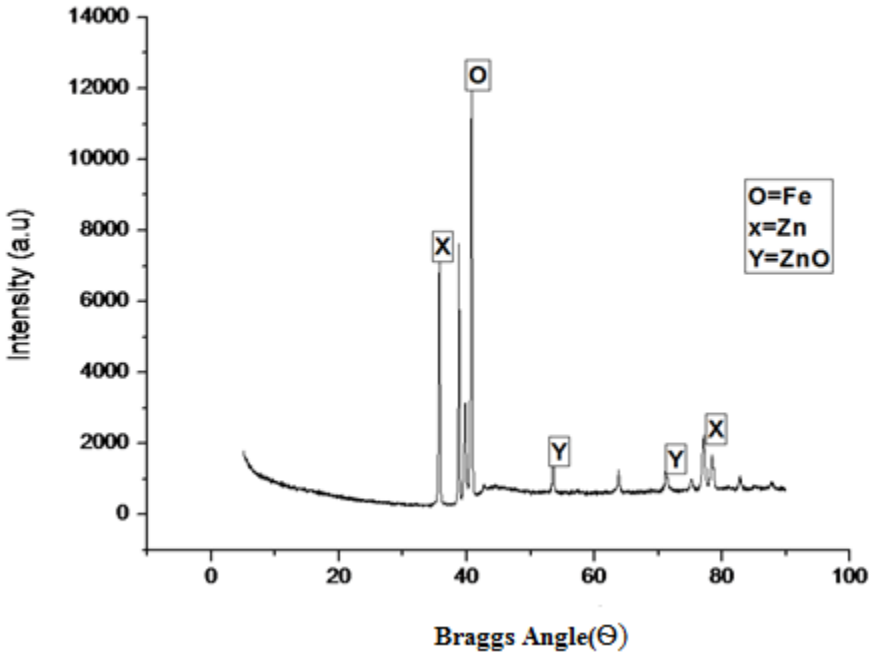

Fig. 5a: X-ray Diffraction of Zn-10ZnO composition

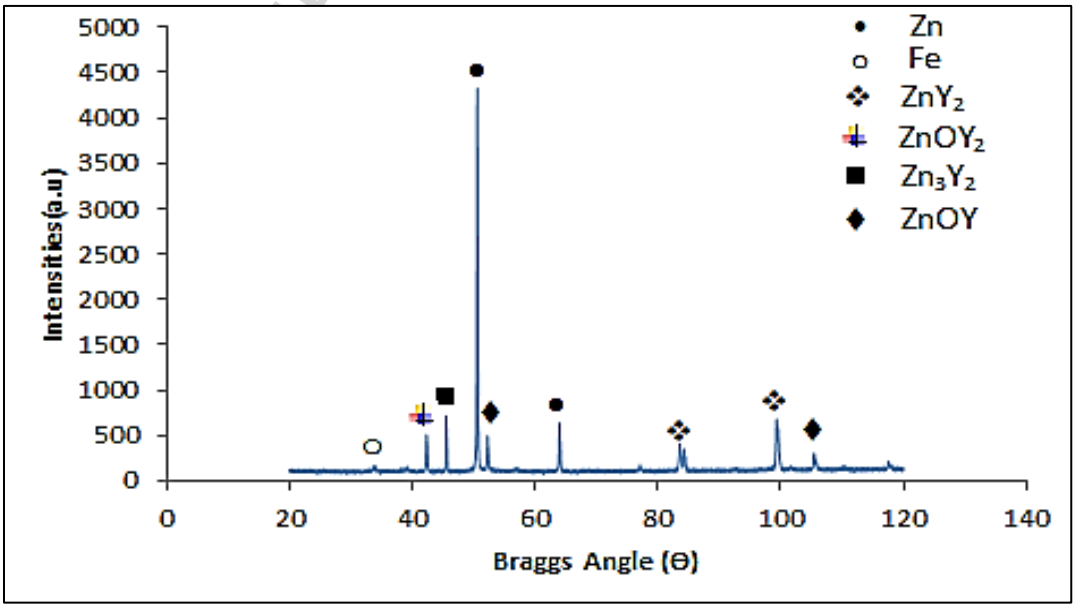

Fig. 5b: X-ray Diffraction of Zn-ZnO-10Y_2O_3 composition

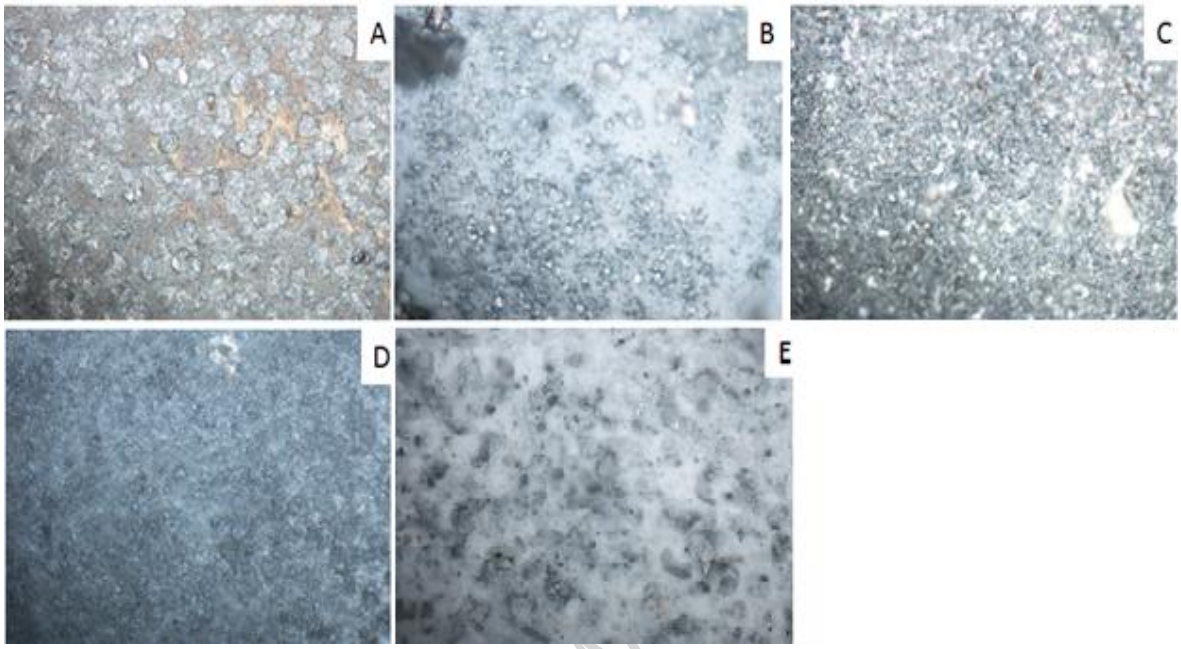

Fig. 6: Optical images of coating of (a) Zn-10ZnO, (b) Zn-10Y_2O_3, (c) Zn-10ZnO-5Y_2O_3, (d) Zn-10ZnO-10Y_2O_3 and (e) Zn-10ZnO-15Y_2O_3 with mag x1000

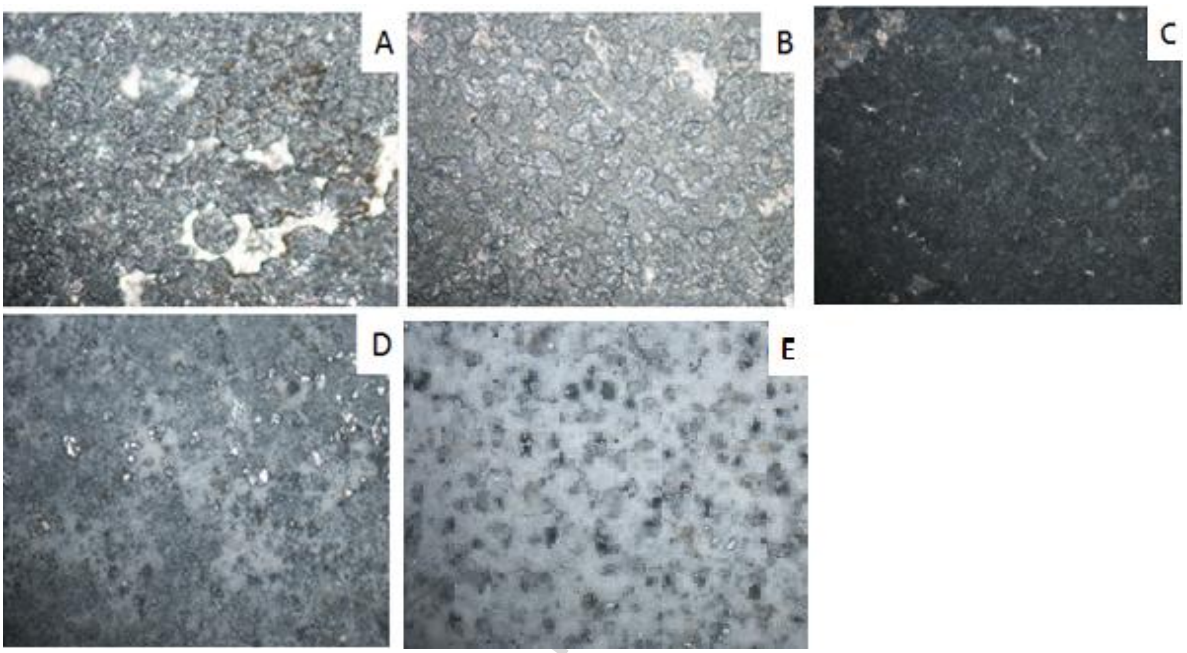

Fig.7: Optical images of coated and heat treated of (a) Zn-10ZnO, (b) Zn-10Y_2O_3, (c) Zn-10ZnO-5Y_2O_3, (d) Zn-10ZnO-10$Y_2O_3$and (e) Zn-10Zn-15Y_2O_3 with mag x1000

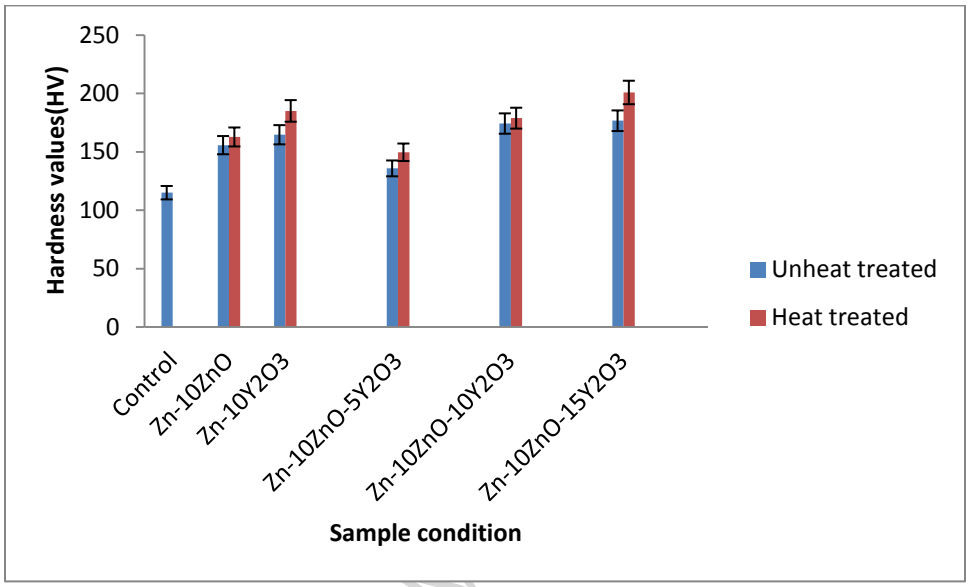

Fig. 8: variation of the hardness of plain mild steel and ZnY coated matrix.

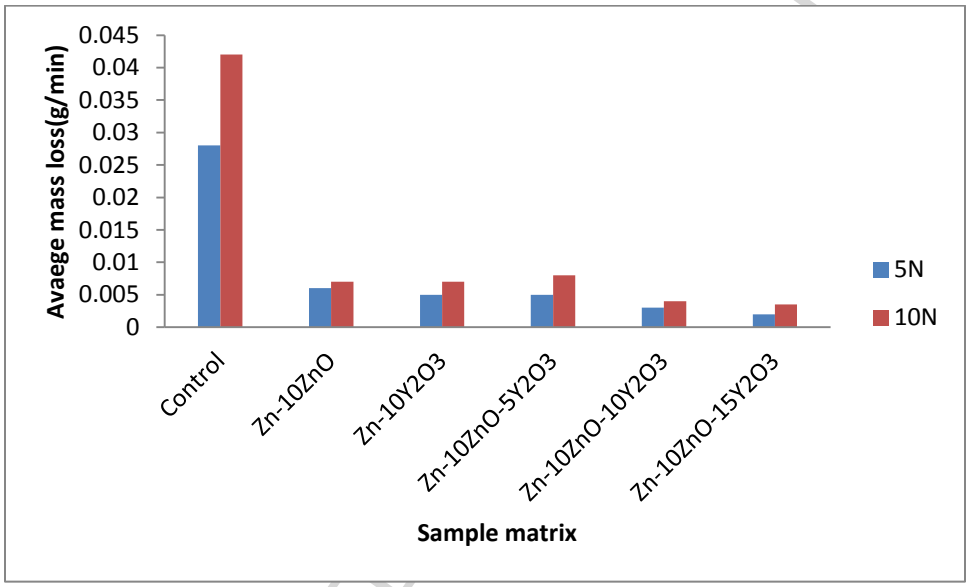

Fig. 9: Variation of average mass loss with sample condition

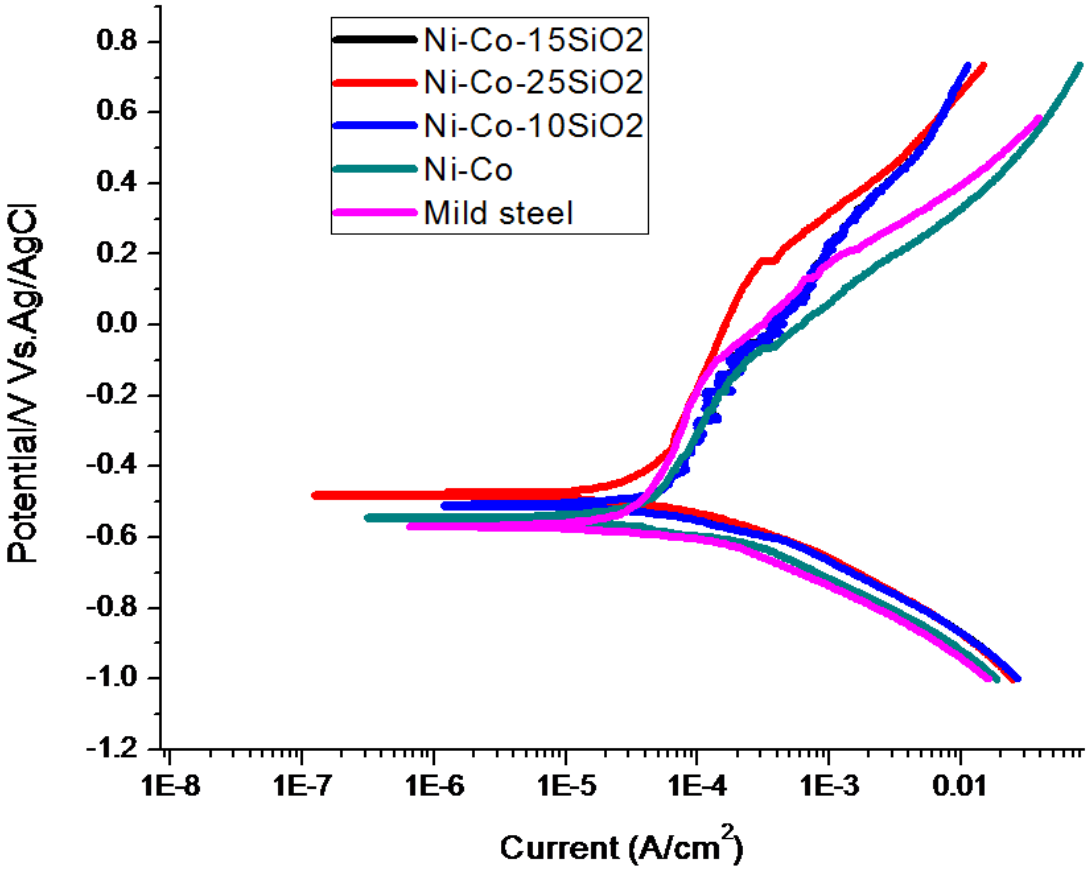

Fig.10: Potentiodynamic linear polarisation curves of the samples

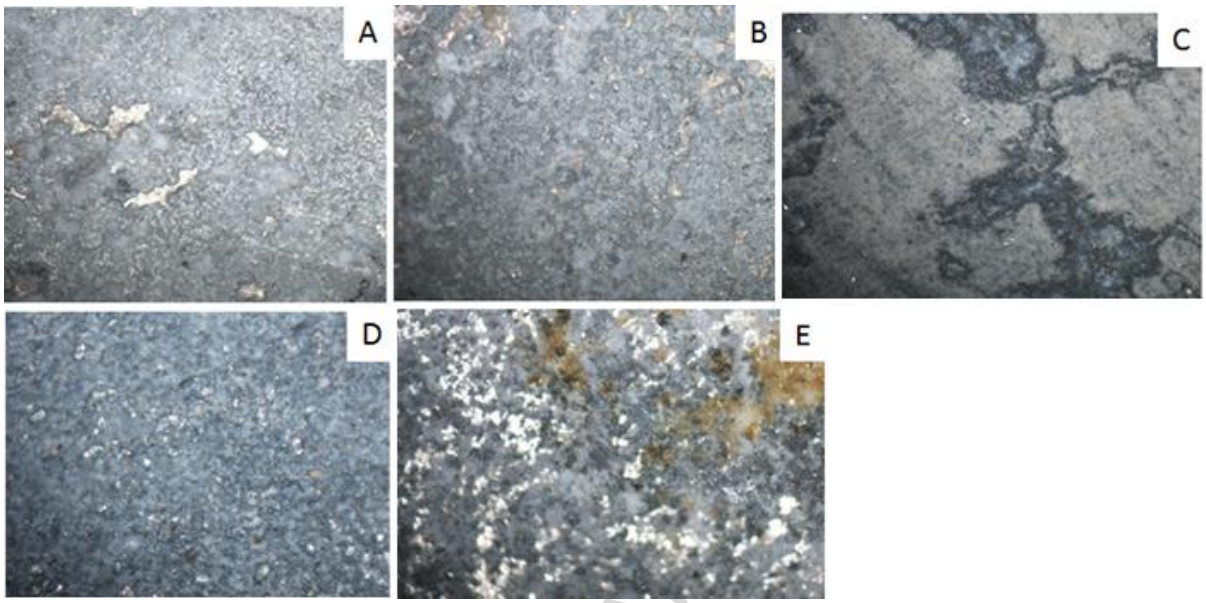

Fig. 11: SEM images of corroded surface of (a) Zn-10gZnO, (b) Zn-10Y_2O_3, (c) Zn-10gZnO-5gY_2O_3, (d) Zn-10gZnO-10g$Y_2O_3$and (e) Zn-10gZnO-15gY_2O_3 with mag x1000

Table 1: Chemical composition and concentration for bath solution

Composition	concentration (g/L)
Zinc Chloride (ZnCl)	70
Zinc Oxide (ZnO)	10
Boric Acid	5
Glycerin	5
Yttrium Oxide (Y_2O_3)	5-15
Thiourea	8
pH	4.5 pH
Temperature	40 ºC
Stirring rate	250 rpm
Time	10 minutes
Current density	0.5 A/cm^2

Table 2: Results of potentiodynamic polarization

	Ecorr, Obs/V Vs.Ag/AgCl	**jcorr (mA/cm²)**	**Corrosion Rate (mm/year)**	**Polarization resistance (Ω)**
Control	-1.547	12.406	1.134	6.784
Zn-10ZnO	-1.376	10.602	0.085	11.951
Zn-10ZnO-5Y_2O_3	-1.359	6.505	0.066	13.670
Zn-10ZnO-10Y_2O_3	-1.355	1.901	0.058	16.101
Zn-10ZnO-15Y_2O_3	-1.341	1.802	0.019	17.904

highlight

- Hardness results increased after plain mild steel was coated with zinc-yttrium.
- Corrosion resistance of the Zinc-Yttria deposition was achieved
- The enhancement of Zinc-Yttria coating revealed higher wear resistance
- The entrenched of enormously appropriate microstructure has modified surface attack