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Experimental Study and Effect of Particulate Interference on the Microhardness, Wear and Microstructural Properties of Ternary Doped Coating

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Abstract. This paper studies effects of the composite particle infringement of ZnO/Cr₂O₃ on zinc rich ternary based coating. The corrosion-degradation property in 3.5% NaCl was investigated using polarization technique. The structural characteristics of the multilayer produce coatings were evaluated by scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS). The mechanical response of the coated samples was studied using a diamond base Dura –Scan) micro-hardness tester and a MTR-300 dry abrasive wear tester. The combined effect of the coatings gave highly-improved performance on microhardness, corrosion and wear damage. This also implies that protection of wind-energy structures in marine environments can be achieved by composite strengthening capacity.

INTRODUCTION

The challenge of component during service as a result of mechanical degradation and chemical deterioration is no news a concern to materials expert, in such a way that it affects both the domestic, industrial and national development [1]. Corrosion scientists and engineers are continually seeking for techniques and methods to effectively combat corrosion by learning the phenomenon and its control with the aim of finding better ways of using engineering materials. The failure of these materials are majorly in application like construction, aerospace, automobile, solar cells, ship building, and energy conservative. With the increasing way to oppose this catastrophic, promising methods of mitigation had been employed ranging from vapour deposition, laser coating, cold spray coating and so on. Surface enhancement of engineering materials is necessary for preventing service failures and corrosion attack in industry [1, 2]. Electrodeposition technique had been proved to give superior advantages due to cost effectiveness, processing and nature of characteristics. Zinc coatings add corrosion resistance to steel in several ways [4]. As a barrier layer, a continuous zinc coating separates the steel from the corrosive environment. The sacrificial properties of zinc can be seen in a galvanic series where the potential of zinc is less noble than steel in most environments at ambient temperatures. Zinc, like all metals, corrodes when exposed to the atmosphere. Infringement of particulate into bath rich such as zinc-composite had been seen as a promising way of getting better performance in term of corrosion protection, wear resistance and other metallurgical properties [5-7]. Hence, this paper focuses on the effects of Cr₂O₃ particulates on the morphology of the composite coatings. The hardness and corrosion resistance of the Zn-ZnO-Cr₂O₃ coatings are also evaluated.

EXPERIMENTAL SECTION

Preparation of substrate

Flat steel specimens of dimension (30 mm x 20 mm x 1 mm) sheet were used as the substrate and zinc sheets of (40 mm x 30 mm x 2 mm) were prepared as the anodes. The initial surface preparation was performed using progressively finer grades of emery paper, as described in our earlier publications [3, 4]. The samples were properly cleaned with sodium carbonate, pickled and activated with 5% HCl at ambient temperature for 10 seconds, followed by instant rinsing in deionized water. The specimens were obtained from Metal Sample site, Nigeria. The chemical composition of the sectioned samples analyzed on a spectrometer were C 0.15%, Mn 0.45%, Si 0.18%, P 0.01%, S 0.031%, Al 0.005%, Ni 0.008%, Fe Balance.

Formation of deposited coating

The prepared Zn-ZnO-Cr₂O₃ composite bath was heated for 2hrs and stirred intermittently to obtain a clear solution before it was prepared for electrolytic deposition on the steel. The prepared cathode and anodes were connected to the D.C. power supply through a rectifier as described by [3, 4]. Deposition was carried out at varying applied current density around 1.0A/cm² for 15 minutes. The distance between the anode and the cathode and the immersion depth were kept constant. Thereafter, the samples were rinsed in water and then dried. The formulated design plan for the coating is described in Table 1.

TABLE 1: Formulated designed bath composition of Zn-ZnO-Cr₂O₃

Sample order	Matrix Sample	Time of deposition (min)	Current density(A/cm ²)
1	Zn-8ZnO	15	1.0
2	Zn-8ZnO-16Cr ₂ O ₃	15	1.0

Structural characterization of the coatings

The structural studies and elemental analysis of the fabricated alloy samples were verified using a high-tech TESCAN scanning electron microscope with an attached energy dispersive spectrometer (SEM/EDS).

RESULTS AND DISCUSSION

Surfacemorphology

Figure 1a and b shows the structural properties of Zn-8ZnO and Zn-8ZnO-16gCr₂O₃ composite coating respectively. The EDS pattern of the composite coating confirms the crystalline phase of the film as identified in Figure 2. The nano particulates were found to be agglomerated when analyzed by scanning electron microscopy (Figure 1a) studies. This is due to the high surface energy of the particles. In general it can be seen that the coating on the mild steel plate resulted into a good appearance, better plating and good adhesion. The nature of the surface morphology and orientation in Figure 1b revealed the non- homogeneous appearance but good disatches as expected. There are no pores and cracks at the interface, which shows that the interface bonding is firm. But the observed improvement may not be far from the possibility that the deposition behaviour and the adhesion strength of any particular plating often based on the current density, potential, plating time and strengthening effect of the grain present in the matrix [5] Figure 2 revealed the morphology of the coating, indicate fine grain size, uniform arrangement and crystal growth for the Zn-8ZnO-16gCr₂O₃ coating. Unlike the Zn-8ZnO deposition where good surfacedeposition was achieved but irregular or inhomogeneous dispersion of deposit was observed.

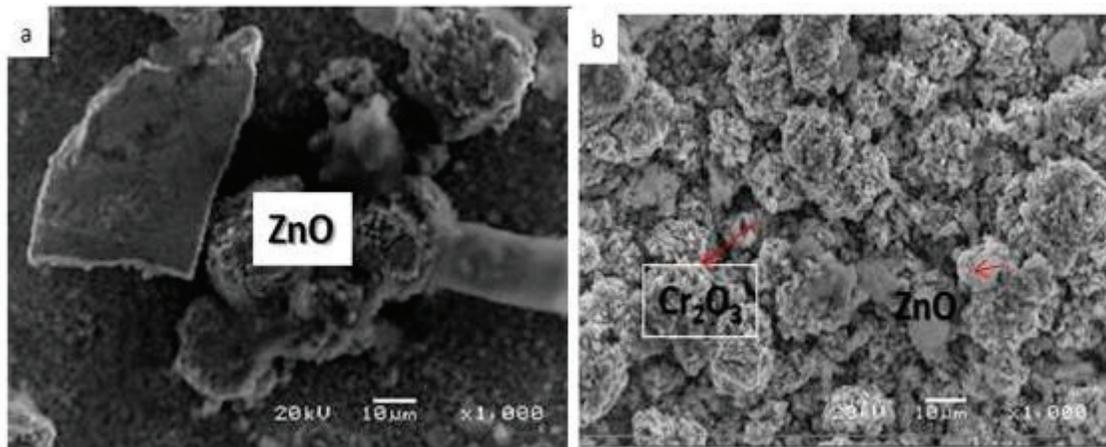


FIGURE 1. SEM images of the a) Zn-8ZnO and b) Zn-8ZnO-16gCr₂O₃ composite coating

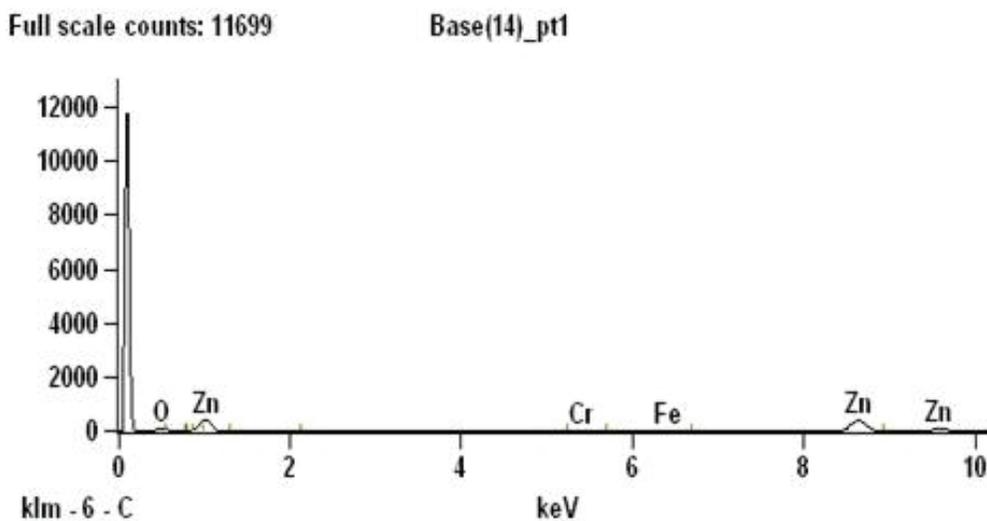


FIGURE 2. Energy Dispersive Spectral Zn-8ZnO-16gCr₂O₃ hybrid

Wear and Microhardness property

Figure 4 shows the wear loss of Zn-ZnO coating and Zn-ZnO-Cr₂O₃ composite coatings with different Cr₂O₃ contents. It is obvious that the wear resistance of the composite coatings is more than that of Zn-ZnO coating. Though the wear resistance of the composite coatings did not increase with increase in the Cr₂O₃ particles content, as it can be seen that Zn-ZnO-8Cr₂O₃ displayed the optimum wear resistance, but the addition of Cr₂O₃ in the matrix has immensely contributed to the wear resistance. The thermal deformations were observed in 250°C for 4hrs and mechanical responses of the coated samples were investigated. A good increase in the micro hardness value was observed in the Zn-ZnO-Cr₂O₃ composite coating even before heat-treatment as shown in Figure 3. The microhardness value for the Zn-ZnO material was 134.0 HVN and almost doubled for the value of Zn-8ZnO-8Cr₂O₃ composite coating (which was the optimal performance) with 344 HVN. Figure 4 illustrates the wear analysis of the coatings. The improved wear resistance can be attributed to the strengthening effect. The hardness process parameter and microstructural behaviour are the parameters which affect the wear resistance as indicated by [6]. The wear resistance increases with

increasing hardness of the matrix. The tendency of Zn-ZnO-Cr₂O₃ composite coatings for plastic deformation is less than Zn - ZnO coating. The results suggest that the wear resistance was improved by the addition of Cr₂O₃ particles. According to [7], the embedded Cr₂O₃ particles can significantly improve the tribological performance of Zn-ZnO-Cr₂O₃ composite coatings.

Figure 3 shows the Microhardness variation of the nano-composite coating before heat treatment. The increase in hardness of the Zn-ZnO-Cr₂O₃ composite coated specimens was due to the presence of the Cr₂O₃ as reinforcement additive, despite the fact that the increase in the quantity has limit.

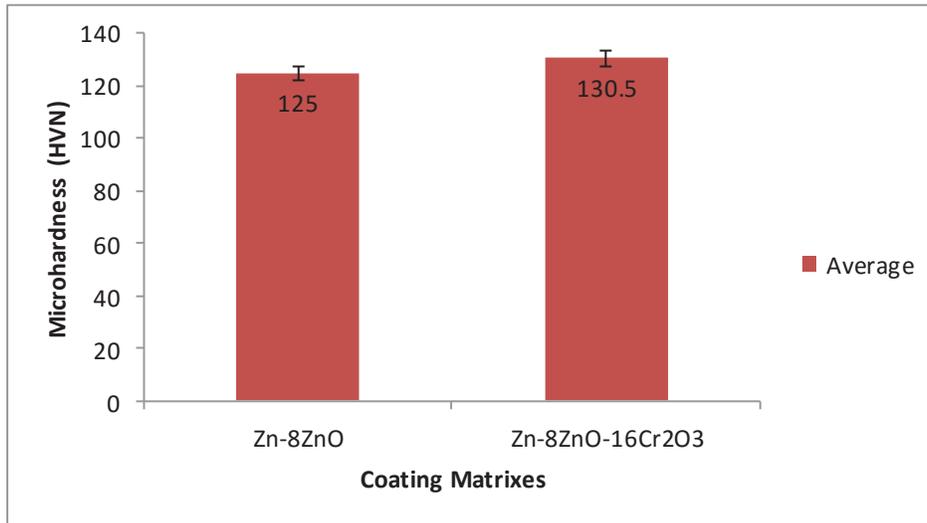


FIGURE 3. Variation of hardness property with coating matrix.

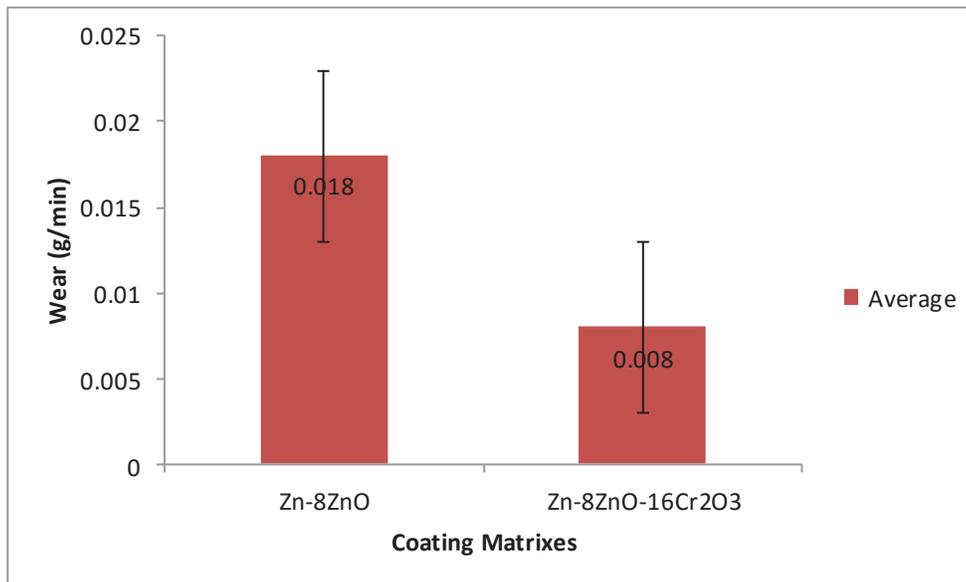
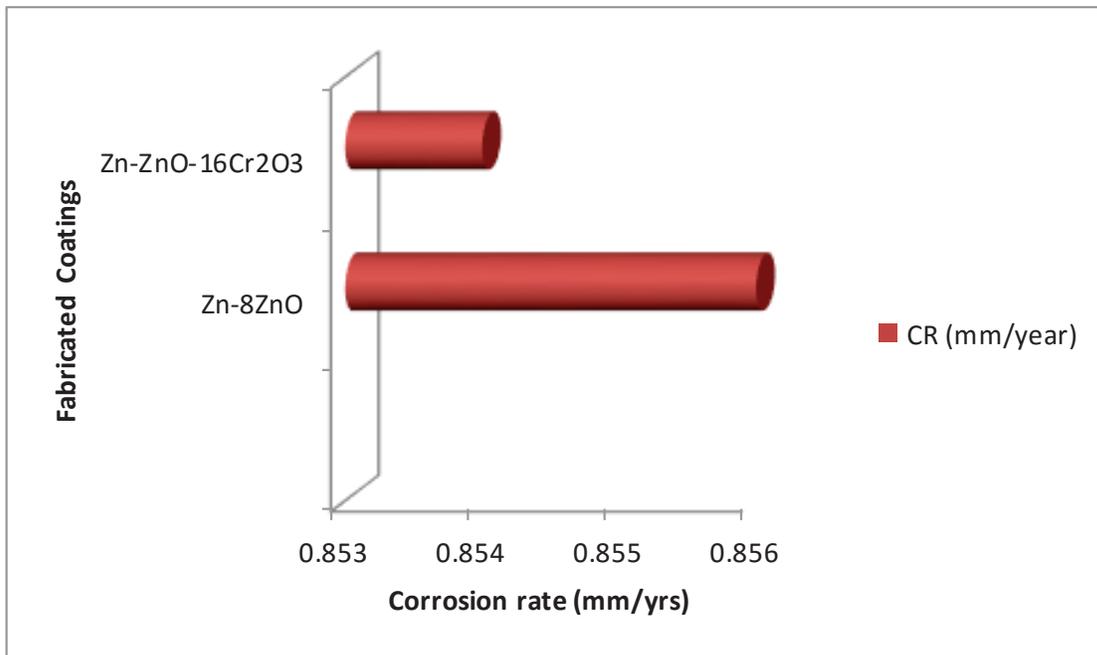


FIGURE 4. Variation of wear rate with time.

Polarization measurements

The values of E_{corr} , j_{corr} , corrosion rate (CR) and polarization resistance (R_p) of the samples before heat-treatment are extrapolated from Tafel slope as presented in Table 2. The results revealed the corrosion resistance behaviour of the coatings in 3.65% NaCl static solution. From the polarization curves of Zn-ZnO- Cr_2O_3 composite coating in Figure 5, it was found that the addition of Cr_2O_3 altered the shape of the polarization curve but causes a considerable increase in the value of the E_{corr} . The features from the linear polarization test (Figure 5) indicate that an increase in additive concentration intensifies this process. However, the optimal value for the coating with least corrosion rate was Zn-8ZnO-8 Cr_2O_3 , which implies that addition of Cr_2O_3 gives better effect of corrosion resistant property. According to [9], this could be attributed to the nature and tenacity of the passive film produced by Zn-8ZnO-8 Cr_2O_3 on the surface of the coated steel and the drop in the potential thereafter could be traced to slight passivity breakdown experienced when the concentration of Cr_2O_3 was further increased, which is in line with [8].



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FIGURE 5. Potentiodynamic polarization curves of Zn-ZnO- Cr_2O_3 composite coating on mild steel in 3.65% NaCl solution.

TABLE 2: Polarization data extrapolated from Tafel slope for matrix Zn-ZnO- Cr_2O_3 composite coating.

Samples No	$E_{\text{corr, Obs}}$ (V)	j_{corr} (A/cm ²)	CR (mm/year)	R_p (Ω)
1. Zn-8ZnO	-1.3067	0.003029	0.854270	23.583
2. Zn-ZnO-16 Cr_2O_3	-1.3706	0.003102	0.856890	20.390

CONCLUSIONS

The following conclusions were drawn from the study:

1. Composite coatings consisting of Zn-ZnO and Zn-ZnO-Cr₂O₃ particles were successfully prepared by means of an electro-deposition technique onto carbon steel substrates.
2. The plating of Zn with incorporation ZnO and Cr₂O₃ in the coating was confirmed by the EDS and XRD.
3. The co-deposition of nano-sized Cr₂O₃ particles in a metal deposit modified the surface morphology of the substrate.
4. The developed composite coating was mechanically and thermally stable.
5. The presence of Cr₂O₃ particles in the matrix of the coating system increased adhesion, corrosion resistance and micro-hardness of the surface, compared to that of plain carbon steel.

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