Potential of Composite Incorporation on the Mechanical Behavior of Multilayer Coatings

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Received 13/12/2016; accepted 30/10/2020
https://doi.org/10.4152/pea.2021390301

Abstract

In this paper, the performance of a composite particle for the deposition of ZnO/Cr₂O₃ on a zinc electrolyte was examined. Its susceptibility to corrosion in 3.5% NaCl, using linear polarization, was investigated. The developed crystal was characterized by using scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS). The strengthening properties of the coated samples, i.e. the mechanical response, were studied using a high sensitive diamond microhardness indenter and a MTR-300 rigid wear tester. From the results, the effect of the composite coatings interestingly influenced the performance regarding microhardness, durability, corrosion mitigation and wear damage. In general, the micro-hardness value for the Zn-ZnO material was 125.0 HVN, while it was 130.5 HVN for Zn-8ZnO-16Cr₂O₃ composite coating. For Zn-8ZnO-20Cr₂O₃, a better hardness performance was noted with 138.0 HVN. From the wear study, Zn-8ZnO shows 0.018 g/min dissociation against the counter body with the best wear performance obtained at 0.005 g/min. The corrosion properties of the developed composite coating also tend towards a more positive region, with a corrosion rate of 0.850 mm/yr. This shows that the role of composite particulates maximally contributes to improve the strengthening characteristics of the developed coating.

Keywords: materials processing, microstructure, manufacturing, co-deposition and wear.

Introduction

Components failure due to mechanical and chemical degradation has been a major consideration over the years. These enormous challenges range from industrial to domestic applications [1,2]. No doubt, mild steel has been a significant metal, and its industrial usage has been widespread, owing to its...
physical and mechanical resilience. Efforts by researchers on environmental control and selection of appropriate materials have been made [3,4]. The failure of these materials mainly occurs in areas such as construction, aerospace, automobile, solar cells, ship building, and energy conservation [5]. With increasing ways to oppose this catastrophe, promising methods of mitigation have been employed, ranging from vapor deposition to laser coating, cold spray coating, and so on [6,7]. Surface enhancement of engineering materials is necessary for preventing service failures and corrosion attack in industry [8].

Electrodeposition technique has proven to give superior advantages, due to its cost effectiveness, processing and special characteristics. Zinc coatings add corrosion resistance to steel in several ways. As a barrier layer, a continuous zinc coating separates steel from the corrosive environment. Zinc properties can be seen in a galvanic series, where its potential is less noble than steel, in most environments, at ambient temperatures. Zinc, like all metals, corrodes when exposed to the atmosphere [9,10]. Infringement of particulates into a bath rich in such a zinc-composite has been seen as a promising way of getting better performance, in terms of corrosion protection, wear resistance and other metallurgical properties.

Protection of metallic materials and alloys against corrosion is one major way of reducing the maintenance costs and hazardous environmental impacts. Previous studies on chemical bath compositions and fluxes to improve processes, corrosion resistance and mechanical behavior, revealed that Zn and Zn-based coating composites are good diffusion driving substances [10]. 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 were also evaluated.

Experimental procedure
Preparation of substrate
Flat steel specimens of dimensions 30 mm x 20 mm x 1 mm were used, and the substrate and zinc sheets of 40 mm x 30 mm x 2 mm were prepared as anodes. The initial surface preparation was progressively performed using finer grades of emery paper, as described in our earlier publications [2]. The samples were properly cleaned with sodium carbonate, pickled and activated with 5% HCl at ambient temperature, for 10 sec, 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 was: C 0.15%, Mn 0.45%, Si 0.18%, P 0.01%, S 0.031%, Al 0.005%, Ni 0.008%, and Fe balance.

Formation of a deposited coating
The steel substrates were prepared by dipping them into a 10% HCl solution for 10 sec, followed by rinsing in distilled water. Analytical grade chemicals and distilled water were used to prepare the plating solution at room temperature. The
formulations were stirred for one day, while heated at 40 °C, to allow for the dissolution of any agglomerates in the bath solution. The bath produced from the formulation, shown in Table 1, was concurrently stirred and heated for several hours before plating, at a temperature of 40 °C.

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

| Sample order | Matrix sample | Time of deposition (min) | Current density (A/cm²) |
|--------------|---------------|--------------------------|------------------------|
| 1            | Zn-8ZnO       | 15                       | 1.0                    |
| 2            | Zn-8ZnO-16Cr2O3 | 15                      | 1.0                    |
| 3            | Zn-8ZnO-20Cr2O3 | 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). 1000 magnification was considered when the samples were subjected to microscopic view.

Results and discussion
Surface morphology
Fig. 1a, 1b and 1c show the SEM images of the EDS patterns of Zn-8ZnO, Zn-8ZnO-16Cr2O3 and Zn-8ZnO-20Cr2O3 surfaces’ composite coatings. The EDS pattern of the composite coating confirms the crystalline phase of the film. The nano particulates were found to be agglomerated when analyzed by scanning electron microscopy (Fig. 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 solid crystal.

The nature of the surface morphology and orientation in Fig. 1b and 1c revealed a sequential homogeneous appearance. There are no pores and cracks at the interface, which shows that the interface bonding is firm. In addition, Fig. 1c shows a more compact and better grains distribution. Moreover, the deposition strength may result from possible process parameters. The elemental quantification was done with massive revelation of essential representative elements at the interface (Fig. 2).
Figure 1. SEM images of a) Zn-8ZnO, b) Zn-8ZnO-16Cr$_2$O$_3$, and c) Zn-8ZnO-20Cr$_2$O$_3$ coatings.

Figure 2. Energy dispersive spectral: a) Zn-8ZnO and b) Zn-8ZnO-20Cr$_2$O$_3$ hybrids.

**Wear and microhardness property**

Fig. 3 shows the wear loss of a Zn-ZnO coating and Zn-ZnO-Cr$_2$O$_3$ composite coatings. It is obvious that the wear resistance of the composite coatings with Cr$_2$O$_3$ is higher than that of the Zn-ZnO coating. As it can be seen, Zn-8ZnO-16Cr$_2$O$_3$ and Zn-8ZnO-20Cr$_2$O$_3$ displayed significant optimum wear resistance because of Cr$_2$O$_3$ presence. The improved wear resistance can be attributed to the strengthening effect. The hardness process parameter and microstructural behavior are the parameters which affected the wear resistance, as indicated by
The wear resistance increased with the increasing hardness of the matrix.

![Figure 3](image.png)

**Figure 3.** Variation of wear rate with time.

![Figure 4](image.png)

**Figure 4.** Variation of hardness property with the coating matrix.

Fig. 4 shows the microhardness trend for the Zn-ZnO coating and Zn-ZnO-Cr$_2$O$_3$ composite samples. Interestingly, a good increase in the micro hardness value was observed in Zn-ZnO-Cr$_2$O$_3$. As expected, this is not far fetch from the result obtained from wear characteristics. The micro hardness value for the Zn-ZnO material was 125 HVN; the value for Zn-8ZnO-16Cr$_2$O$_3$ composite coating was 130.5 HVN, while Zn-8ZnO-16Cr$_2$O$_3$ had a value of 138 HVN. In summary, one can vividly see the massive role of Cr$_2$O$_3$ as a reinforcement additive.

**Polarization measurements**

Fig. 5 represents the deterioration and mitigation characteristics of the deposited coatings in 3.65% NaCl, using linear polarization techniques. Corrosion rate (CR) values show that the additive concentration separates the mitigation characteristics of the coatings. In other words, Cr$_2$O$_3$ addition enhances corrosion resistant properties. According to [3], the presence of a nano particulate infringement most often influences passive films characteristics, and reduces corrosion penetration into the coating interfaces [10,12].
Fig. 5. Potentiodynamic polarization curves of Zn-ZnO-Cr$_2$O$_3$ composite coating on mild steel in a 3.65% NaCl solution.

Fig. 6 showed SEM analysis with EDS images for both Zn-ZnO and Zn-ZnO-20Cr$_2$O$_3$ composite coatings, after corrosion. The presence of the pits (Fig. 6a) and cracks (Fig. 6b) reveals the corrosion effects. That is, the fairly compacted morphology, noticed in Figs. 1a and 1c (before corrosion), has been negatively altered after corrosion. However, the presence of key elements could still be confirmed.

Conclusion

From the research work, it can be concluded that: Zn-ZnO and Zn-ZnO-Cr$_2$O$_3$ coatings were successfully prepared by co-deposition route onto mild steel; good structural modification was obtained with the infringement of nano-sized Cr$_2$O$_3$ particles in to the zinc rich lattices, and the coatings produced good stability, improved microhardness tendency and good resistance to mechanical deformation.
Acknowledgments
This material is based upon work financially supported by the National Research Foundation. The authors acknowledge the support from Surface Engineering Research Centre, (SERC) Tshwane University of Technology, Pretoria, South Africa.

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