EGYPTIAN JOURNAL OF BASIC AND APPLIED SCIENCES 1 (2014) 120–125

Available online at www.sciencedirect.com
ScienceDirect
journal homepage: http://ees.elsevier.com/ejbas/default.asp

Short Communication

Study of Al_2O_3/SiC particle loading on the microstructural strengthening characteristics of Zn–Al_2O_3–SiC matrix composite coating

O.S.I. Fayomi [a,b,*], A.P.I. Popoola [a]

[a] Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology, P.M.B. X680, Pretoria, South Africa
[b] Department of Mechanical Engineering, Covenant University, P.M.B 1023, Ota, Ogun State, Nigeria

ARTICLE INFO

Article history:
Received 27 March 2014
Received in revised form
20 May 2014
Accepted 25 May 2014
Available online 19 June 2014

Keywords:
Microstructure
Composite
Micro-hardness
Interfacial effect
AFM

ABSTRACT

In this paper, the microstructure, and mechanical performance of Zn–Al_2O_3–SiC film co-deposited on mild steel substrate were produced by electrodeposition method. The structural characteristic of the composite coating was analyzed by scanning electron microscope (SEM) equipped with energy dispersive spectrometer (EDS), X-ray diffraction (XRD) and atomic force microscope (AFM). Mechanical examination was done using durascan hardness tester. The result showed that the influence of individual particle loading greatly alter the structural properties and hardness behavior. The increase in hardness is attributed to the perfect homogeneity and characteristics of the particulate which led to the formation of uniform distribution, coherent and interfacial precipitation within the zinc lattice.

1. Introduction

The existence of Zn coating over year has tremendously been attested to exhibits resilient mechanical and corrosion resistance to a considerable level [1,2]. Meanwhile their service performance decreases due to scar piled up in mechanical application and aggressive medium resulting from corrosion product [3–7]. Metal and ceramics composite deposition are appreciated surface modification technology

* *Corresponding author*. Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology, P.M.B. X680, Pretoria, South Africa. Tel.: +27 719811277.
E-mail addresses: ojosundayfayomi3@gmail.com, fayomio@tut.ac.za (O.S.I. Fayomi), popoolaapi@tut.ac.za (A.P.I. Popoola).
Peer review under responsibility of Mansoura University

Production and hosting by Elsevier

http://dx.doi.org/10.1016/j.ejbas.2014.05.003

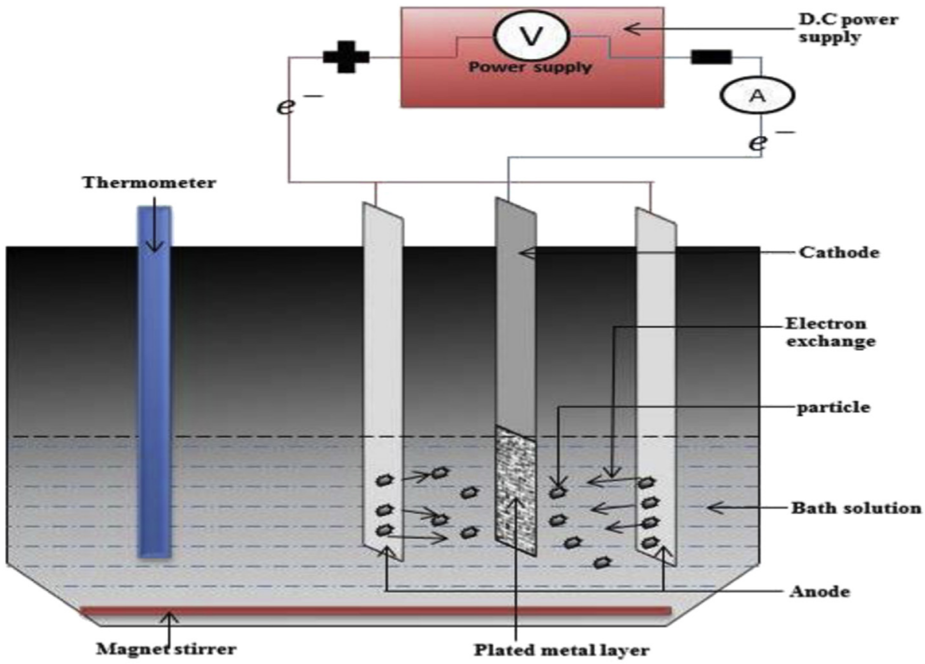

Fig. 1 – Schematic diagram of electrodeposited system.

to obtained significant reinforced properties. Their engineering relevant varied based on the individual characteristics such as wear resistance, increase in stiffness, high temperature properties, good strength behavior and enhanced corrosion properties. The majority of this characteristic plays a priority in machinery, chemical industry, light space application, automobile, defense, spaceflight aviation and photonics [8,9].

Effort to enhance the promising option for zinc based alloy instead of passivation technique is the introduction of dispersed composite and nano-composite materials like TiO_2, SiO_2, ZrO_2, CNTs, WC, Al_2O_3, and SiC which are extensively used to modified coating and electrode. Some report by authors attested that composite coating with silica and mica particle in Zn interface increased hardness and considerable improvement in corrosion resistance properties [10–14].

This composite induced metal and ceramic particle possess outstanding performance with composition in both metal and the second phase particulate. Considerable study has been carried out in view to develop suitable materials for better compatibility and good hardness behavior could be accessed. In view of this, silicon carbide is considered as a major particulate. SiC is an excellent abrasive composed of tetrahedral of carbon and silicon atoms with strong bonds in the crystal lattice [12–14].

The nucleation growth, mechanical and adhesion properties of Zn–Al_2O_3–SiC have not been studied in details in sulphates environments to the best of our knowledge. The objective of the work is to develop a particulate-strengthened bright and adherent Zn–Al_2O_3/SiC deposit; observed the structural and mechanical properties, since attention in ceramic composite is significant because of the roles in engineering and various applications, in recent time.

2. Experimental procedure

2.1. Preparation of substrate

Mild steel specimens of dimension (30 mm × 20 mm × 1 mm) sheet were used as a substrate and zinc sheets of (40 mm × 30 mm × 2 mm) were prepared as anodes. The initial surface preparation was performed with finer grade of emery paper as described in our previous studies [8,12]. The sample were properly cleaned with sodium carbonate, pickled and activated with 5% HCl at ambient temperature for 10 s then followed by instant rinsing in deionized water. The mild steel specimens were obtained from metal sample site Nigeria. The chemical composition of the sectioned samples is as follows C 0.15, Mn 0.45, Si 0.18, P 0.01, S 0.031, Al 0.005, Ni 0.008, and Fe-balance.

2.2. Formation of deposited coating

The mild steel substrate earlier prepared was actuated by dipping into 10% HCl solution for 10 s followed by rinsing

Table 1 – Formulated designed bath composition of Zn–Al_2O_3–SIC.

Sample order	Matrix Sample	Time of deposition (min)	Current (A)
1	Zn–5Al_2O_3–10SiC	15	0.5
2	Zn–5Al_2O_3–10SiC	15	1.0
3	Zn–10SiC	15	0.5
4	Zn–10SiC	15	1.0
5	Zn–15SiC	15	0.5
6	Zn–15SiC	15	1.0
7	Zn–10Al_2O_3–15SiC	15	0.5
8	Zn–10Al_2O_3–15SiC	15	1.0

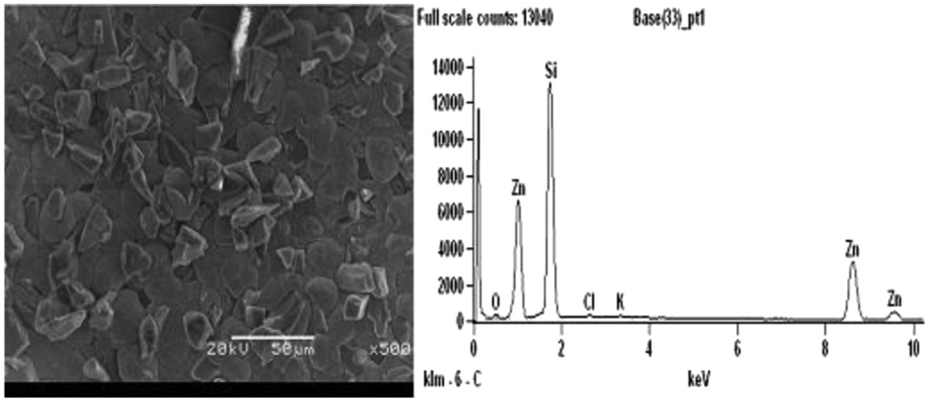

Fig. 2 – SEM/EDS micrographs of Zn–15SiC sample.

in distilled water. Analytical grade chemicals and distilled water were used to prepare the plating solution at room temperature, prior to plating. The formulations were then placed in a stir for a day while heated to 40 °C to easily admix and to allow for dissolution of any agglomerate in the bath solution. The bath produced is concurrently stirred as heating trend lasted for hours before plating [8]. $ZnSO_4.7H_2O$ 70 g/L, Al_2O_3 5–10 g/L, SiC 10–15 g/L, Boric acid 5 g/L, Glycine 5 g/L, Thiourea 5 g/L, Temp 40 °C, pH 4.5, Current density 0.5–1.0 A/cm^2.

2.3. *Preparation of the coatings*

The prepared Zn–Al_2O_3–SiC composite was heated for 2 h and intermittently stirred to obtain clear solution before it was prepared by electrolytic deposition process over mild steel. The prepared cathode and anodes were connected to the D.C. power supply through a rectifier as presented in Fig. 1. Deposition was carried out at varying applied current between 0.5 and 1.0 A/cm^2 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 dried. The formulated design plan for the coating is described in Table 1.

2.4. *Structural characterization of the coatings*

The structural studies and elemental analysis of the fabricated alloy samples were verified using a TESCAN scanning electron microscope with an attached energy dispersive spectrometer (SEM/EDS). The phase property was observed with the help of X-ray diffractogram. The adhesion profile, topography and morphology of the coating were observed with the help of atomic force microscope (AFM). High optic diamond based durascan microhardness tester was used to estimate the average microhardness of the deposit in an equal interval range.

3. Results and discussion

3.1. *SEM/EDS of deposited alloy*

Figs. 2 and 3 show the SEM/EDS structure of represented Zn–Al_2O_3–SiC fabricated ceramic coating with reference to (Zn–15SiC and Zn–10Al–15SiC) matrix respectively. From the two figures it is obvious that the crystal flakes of the deposits were homogeneously dispersed on the interface. It is quite evident that at 1.0 A/cm^2 for Zn–15SiC a noticeable crystal nucleus patched along the interface was observed. Apparently there are two distinctive phases, the initial having a homogeneously uniform stable precipitate and the latter with hexagonal patches. In fact, the incorporation of the SiC along the zinc interfacial could be seen to provide a refine microstructure with dominating nodular particle.

The deposition appearance with coating interferes of Al_2O_3/SiC was quite appreciated in that both particulate

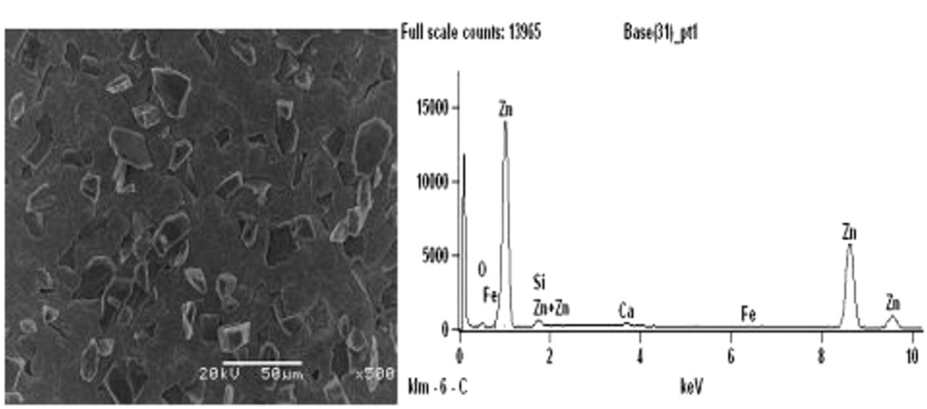

Fig. 3 – SEM/EDS micrographs of Zn–10Al_2O_3–15SiC sample.

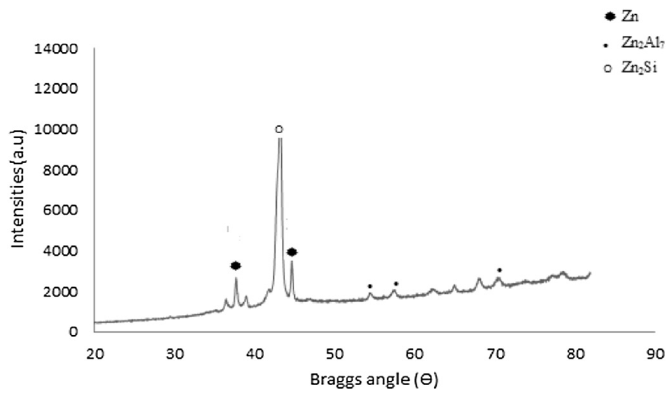

Fig. 4 – XRD pattern of Zn–10Al_2O_3–15SiC sample.

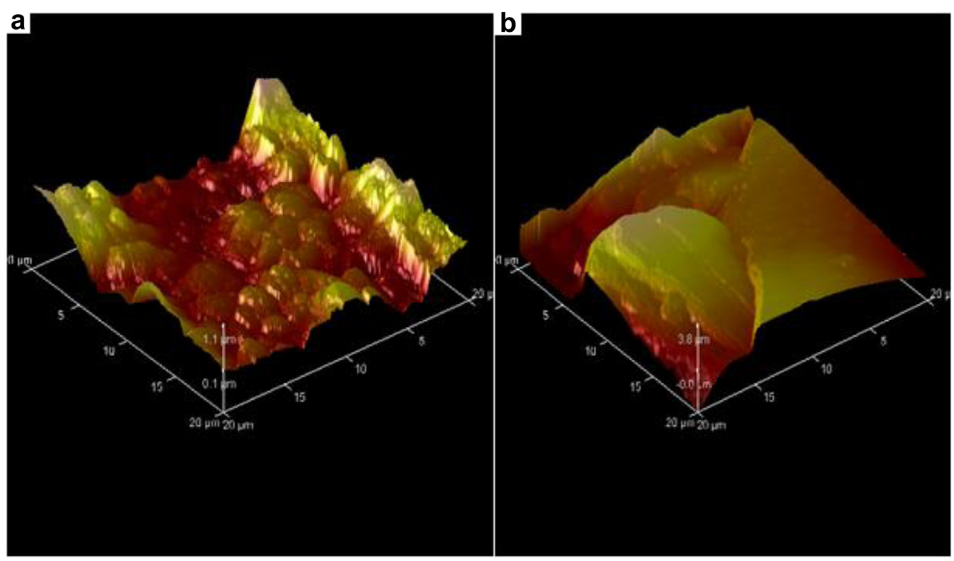

Fig. 5 – 3D AFM images of a] Zn–15SiC b] Zn–Al_2O_3–15SiC particle reinforced.

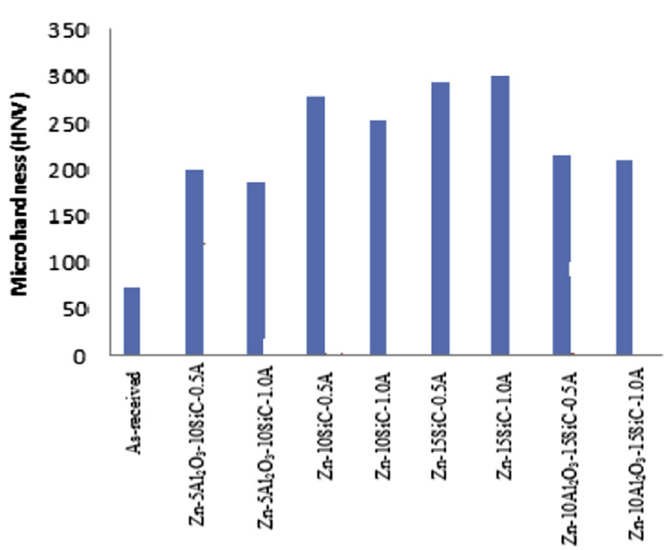

Fig. 6 – Hardness properties of varied loading particle of Zn/Al_2O_3/SiC matrix deposited on mild steel.

strengthen composite find their way expressly doped into the zinc metal matrix. The structural behavior was quite expected because the nucleation process proceed from the zinc metal as load bearer, the dispersion of the particulate cover the nucleation site and strengthening the produced alloy [11,13,14] Fig. 3. In other words, it is necessary to mention that morphological change may be associated to the strong blocking influence of the induced composite leading to good precipitation and better orientation. Sometime the conditioning parameter in relation to the degree of additive impede also play a vital role in re-modification of the crystal orientation and surface texture of a deposited as attested [3].

Fig. 4 shows the XRD pattern of the Zn–Al_2O_3–SiC deposit prepared with 10Al_2O_3/15SiC nanoparticle concentration from the plating bath. The diffractogram give the major diffraction peak as 38°, 42°, 45° 55° and 72°.

The intermetallic growth phases observed are Zn, Zn_2Al_7, Zn_2Si etc. The presence of the individual dispatched metal-particulate phases was noticeable on the coating as it seems to influence the phase change through the help of inter-diffusion mechanism and ion of each particulate [1,14]. The phase orientations of the metal matrix are indication of the harness performance and remarkable effect of the coating produced.

3.2. *Atomic force microscope analysis*

Fig. 5a–b shows the 3D atomic force micrographs of the deposited Zn–SiC and Zn–Al_2O_3–SiC samples. Zn–20SiC matrix had fairly grain size and uniform crystal growth with crystallites. For Zn–Al_2O_3–SiC matrix as observed in Fig. 5b, the inclusion of the composite ceramic Al_2O_3/SiC provide a robust inter-link at the interfacial pool of the Zn based matrix. Although the growing of crystal and topography of a deposit is a combined contribution of diffusion of ions into the nucleus, meaning that the adsorbed atom wanders to a growth point on the cathode and is incorporated in the growing lattice under the influence of the applied voltage and applied electrodeposition time. Ref. [5] affirmed further that surface roughness and adhesion increases most times as a result of the applied voltage on the deposited metal with coalesced crystallite, hence film thicknesses are influenced by metallurgical parameters. Compatibility was seen with Zn–Al_2O_3–SiC adhesion compared to the Zn–SiC matrix with undulation.

3.3. *Microhardness studies*

The microhardness study shown in Fig. 6 was carried out in order to observe the impact of the composite particulate and its concentration loading on the hardness properties of Zn–SiC and Zn–Al_2O_3–SiC coatings. From general observation, all composite fabricated alloys produced an excellent and significant improvement as compared to the as-received mild steel substrate.

The unexpected result seen among the coating matrix is the less hardness observed from the ternary matrix as against the Zn–SiC alloys. Reason for this behavior is not well as certain, however according to Popoola et al. [8] and Rahman et al. [11], the micro-hardness of the electrodeposited layers can depend on many factors such as electrolyte composition, operating conditions and diffusion mechanism. On the hand, the hardness improvement for Zn–SiC with about 290HVN can be linked to the agglomeration of SiC influence rather than the network of additives that constitute the electrolyte composition for Zn–Al_2O_3–SiC coatings that possess 205HVN value. Invariably particle loading might not necessitate dramatic improvement but rather refinement and proper nucleation of the bath constitute.

4. Conclusion

1) In incorporation of the Al_2O_3/SiC ceramics composite particles in the zinc matrix as reinforcement increases the hardness properties of the substrate.
2) The hardness properties increase as the concentration of additive increases.
3) There are good uniform particle distribution and better crystal orientation with compactable structure within the ternary alloy coating.

Acknowledgment

This material is based upon work supported financially by the National Research Foundation. The support from Surface Engineering Research Centre, Tshwane University of Technology Pretoria South Africa is appreciated.

REFERENCES

[1] Fayomi OSI, Popoola API. Chemical interaction, interfacial effect and the microstructural characterization of the induced zinc-aluminum–*Solanum tuberosum* in chloride solution on mild steel. Res Chem Inter 2013;1354:2.
[2] Fayomi OSI, Popoola API, Loto CA, Popoola OM. Morphology and properties of Zn-Al-TiO_2 composite on mild steel. Proc ICCEM 2012;2012:207–12.
[3] Basavanna S, Arthoba NY. Electrochemical studies of Zn–Ni alloy coatings from acid chloride bath. J Appl Electrochem; 39(10):1975–82.
[4] Chitharanjan HA, Venkatakrishna K, Eliaz N. Electrodeposition of Zn–Ni, Zn–Fe and Zn–Ni–Fe alloys. Surf Coat Technol 2010;205:2031–41.
[5] Dikici T, Culha O, Toparli M. Study of the mechanical and structural properties of Zn–Ni–CO ternary alloy electroplating. J Coat Technol Res 2010;7(6):787–92.
[6] Fayomi OSI, Popoola API. An investigation of corrosion and mechanical behaviour of Zn coated mild steel in 3.65% NaCl. Int J Electrochem Sci 2012;7:6555–70.
[7] Mohankumar C, Praveen K, Venkatesha V, Vathsala K, Nayana O. Electro-deposition and corrosion behavior of Zn–Ni and Zn–Ni–Fe_2O_3 coatings. J Coat Technol Res 2012;9(1):71–7.
[8] Popoola API, Fayomi OSI, Popoola OM. Comparative studies of microstructural, tribological and corrosion properties of plated Zn and Zn-alloy coatings. Int J Electrochem Sci 2012;7:4860–70.

[9] Popoola API, Fayomi OSI, Popoola OM. Electrochemical and mechanical properties of mild steel electroplated with Zn-Al. Int J Electrochem Sci 2012;7:4898–917.
[10] Praveen BM, Venkatesha T. Electro-deposition and corrosion resistance properties of Zn-Ni/TiO_2 nano composite coating. Int J Electrochem 2011;261:407–41.
[11] Rahman MJ, Sen SR, Moniruzzaman M, Shorowordi KM. J Mech Eng Trans 2009:40–9.
[12] Shivakumara S, Manohar U, Arthoba Naik Y, Venkatesha TU. Influence of additives on electro-deposition of bright Zn–Ni alloy on mild steel from acid sulphates bath. Bull Mater Sci 2007;30:455–62.
[13] Zheng HY, An MZ, Lu JF. Surface characterization of the Zn-Ni-Al_2O_3 nanocomposite coating fabricated under ultrasound condition. Appl Surf Sci 2008;254:1644–50.
[14] Wang P, Cheng Y, Zhang Z. A study on the electrocodeposition processes and properties of Ni-SiC nanocomposite coating. J Coat Technol Res 2011;8:1–9.