



# Comparative Assessment of the Microhardness and Plastic Degradation Mechanism of Deposited Modulated Coatings on Mild Steel

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**Abstract.** Zinc based coatings modified with aluminium and tin inclusions were electrodeposited in chloride zinc sulfate electrolytes containing a metallic powder of titanium. It was found that presence of these particulates is suitable to obtain ZnAlSn-Ti composites coating that could help increase the microhardness characteristics and wear properties. The hardness and wear properties of the deposited coatings were examined with diamond base micro-hardness tester and CETR reciprocating sliding tester respectively. The structural properties were examined with the help of scanning electron microscope. It was observed that structural coating surface impact on the hardness propagation with increases from 33.4 to 299 kgf mm<sup>-2</sup> (HVN<sub>40</sub>), and shows a considerably higher wear resistance from 2.351g/min to 0.002g/min. It is obvious that plastic deformation of the working steel structure is dependent on protective coating and the concentration of the individual particulate.

Keyword: Microstructure; modulated; composite coating; protection

## INTRODUCTION

Zinc is widely used in engineering applications to mitigate against structural fallout and wear failure in service conditions [1]. However, these properties deteriorate upon subjected to slightly high temperatures application above (300 °C) [2]. The recent studies on literatures show that the properties of electrodeposited single phase metal can be impacted upon with inoculation of some organic additives, by alloying with suitable elements or by adding inert ceramic particles. The overview of particles incorporated with metallic and ceramics particles has been seen to improve the properties and performance characteristics of electrodeposited Zn metal. Several metal and ceramics based composite particles like titanium dioxide (TiO<sub>2</sub>), titanium boride (TiB<sub>2</sub>), silicon carbide (SiC), ceria (CeO<sub>2</sub>), zirconia (ZrO<sub>2</sub>), silicon nitride (Si<sub>3</sub>N<sub>4</sub>), alumina (Al<sub>2</sub>O<sub>3</sub>), Dysprosium oxide (Dy<sub>2</sub>O<sub>3</sub>), to mention but a few have been incorporated to enhance the properties of zinc in its electrolyte form [3-5]. Silicon nitride (Si<sub>3</sub>N<sub>4</sub>), silicon carbide (SiC), Chromium oxide, (Cr<sub>2</sub>O<sub>3</sub>) particles which exhibit high hardness and a low coefficient of friction

are commonly used for wear resistant applications. Major application that required structural modification to impact on mechanical behaviour uses titanium, alumina, tin incorporated on zinc electrolyte on binary single system which is also adopted in our study. Titanium and tin are used in various applications such as aesthetic components, heterogeneous catalysts, and coating materials for thermal protection and corrosion resistance [6-10]. More so, the capacity to produce powders with specific reinforcing particulate with smaller grain has become a new advent in recent time owing to fabrication of new material composite alloy with unique characteristics.

The major constrain is the multistep hybrid bath formation of modulated composite coating and the conditioning parameter. In addition, the codeposition of nanoparticles needed in term weight fraction for uniform incorporation without agglomeration is also a challenge [11-13]. Reasonably less work is reported on the improvement in properties of electrodeposited Zn on quaternary additive by the incorporation of nano alumina, titanium, and tin particles in single bath [14-15]. In this study the effective use of these particulate to obtain a highly improved microhardness and high temperature wear resistance is considered [16]. Investigations will be carried out on the incorporation of nano titanium, tin and alumina in a Co matrix for hardness and wear resistant applications.

## EXPERIMENTAL SECTION

### Experimental procedure:

#### Preparation of substrates

Mild steel was commercially sourced and sectioned into (40 mm x 20 mm x 1 mm) sheet as cathode and 99.5% zinc plate of (30 mm x 20 mm x 1 mm) were prepared as anodes. The initial surface preparation was performed with finer grade of emery paper as described in our previous studies [1, 9]. The sample were properly cleaned with sodium carbonate, pickled and activated with 10% HCl at ambient temperature for 10 second then followed by instant rinsing in deionized water. The mild steel specimens were obtained from metal sample site in Nigeria. The chemical composition of the sectioned samples is shown in Table 1 as obtained from spectrometer analyzer.

**Table 1: Chemical Analysis of Substract (wt. %).**

Element	C	Mn	Si	P	S	Al	Ni	Fe
Composition	0.15	0.45	0.18	0.01	0.031	0.005	0.008	Balance

#### Processed composition:

The electrolytic chemical bath of Zn-Al-Sn-Ti fabricated alloy was performed in a single cell containing two zinc anode and single cathode electrodes. The distance between the anode and the cathode is 15mm. Before the plating, All chemical used are analar grade and de-ionized water were used in all solution admixed. The bath was preheated at 40°C. The processed parameter and bath composition admixed used for the different coating matrix is as follows Zn 75g/L, Al 30g/L, KCl 50g/L, ZnCl 75g/L, Boric acid 10g/L, SnO<sub>2</sub> 7g/L, TiO<sub>2</sub> 7g/L, pH 4.8, time, 20min and tempt 40°C. The choice of the deposition parameter is in line with the preliminary study from our previous work [1]. The prepared zinc electrodes were connected to the rectifier at varying applied potential and current density between 0.3V at 2 A/cm<sup>2</sup> for 20 minutes. The distance between the anode and the cathode with the immersion depth were kept constant as described by [18]. The fabricated were rinsed in distilled water and samples air-dried.

Portion of the coating were sectioned for characterization.

**Table 2: Deposition frame work**

Sample Order	Material sample	Time of deposition (min)	Potential (V)	Current density (A/cm <sup>2</sup> )	Con. of additive (g)
Blank	-	-	-	-	-
Sample 2	Zn-Al-7Sn-Ti-0.3V-Cl	20	0.3	2	7

### *Characterization of coating*

The structural evolution of the deposited composite coating alloy was characterized with VEGA TESCAN Scanning electron microscope equipped with EDS. The phase change was verified with XRD. Micro-hardness studies were carried out using a Diamond pyramid indenter EMCO Test Dura-scan micro-hardness testers at a load of 10 g for a period of 20 s. The average microhardness trend was measured across the coating interface in an interval of 2 cm using screw gauge attached to the Dura hardness tester.

### *Plastic degradation test*

The friction and wear properties of the deposited quaternary fabricated alloy were measured using CERT UMT-2 tribological tester at ambient temperature of 25°C. The reciprocating sliding tests was carried out with a load of 5N, constant speed of 5mm/s, displacement amplitude of 2mm in 20minutes. A Si<sub>3</sub>N<sub>4</sub> ball (4mm in diameter, HV50g1600) was chosen as counter body for the evaluation of tribological behavior of the coated sample. The dimension of the wear specimen is 2cm by 1.5cm as prescribed by the specimen holder. After the wear test, the structure of the wear scar and film worn tracks are further examined with the help of scanning electron microscope couple with energy dispersive spectroscopy (VEGAS-TESCAN SEM/EDS).

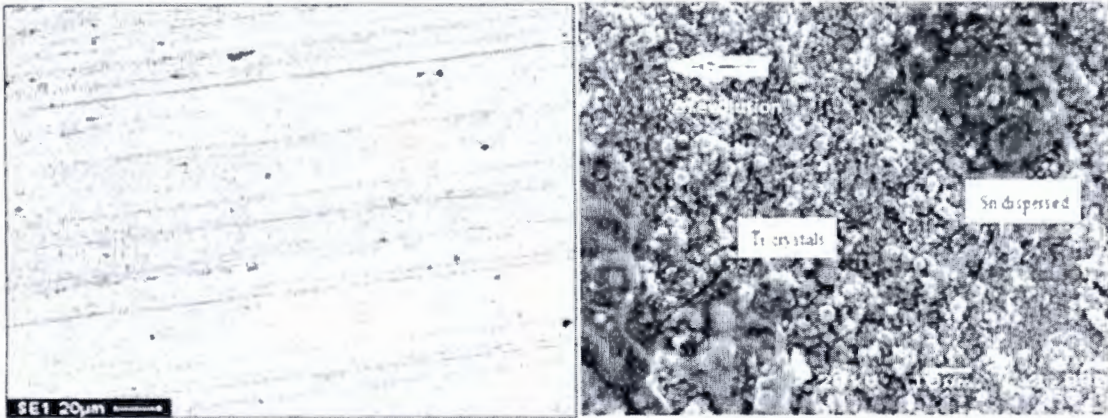


## RESULTS AND DISCUSSION

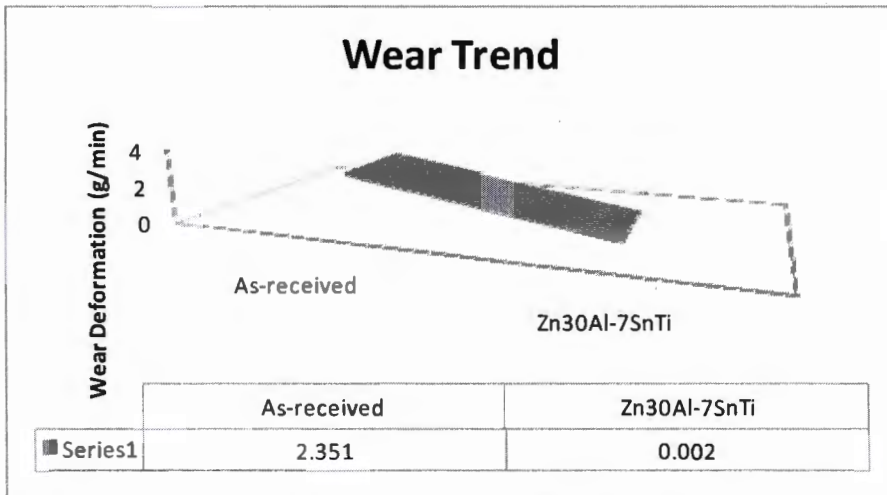
Figure 1 shows structural properties of as-received and fabricated morphology of Zn-Al-7Sn-Ti-0.3V-CI composite coating respectively. We saw a distribution of embedded particulate at the entire interface of the coated mild steel (see Figure 1b). The as-received sample shows a clean clear free to crack surfaces (see Figure 1 a). The distinctive structure indicates a participatory lattice grains crystals, although this is expected since modulation of particles often results into intermetallic adhesion phase resulting from individual additive behavior [13]. The grains were observed to be small and homogeneously distributed within lattice perfect substitution vacancies. The migration of deposited ions that results into the successful build up of composite coating are characterized by process parameter [16] and this is in par with the observation noticed in this study.

Figure 2 shows variation of the wear rate as a function of time between the as-received and fabricated Zn-Al-7Sn-Ti-0.3V-CI. When compared, a remarkable improvement was obtained for Zn-Al-7Sn-Ti-0.3V-CI composite coating as a result of multifaceted influence of the composite particulate as against the working sample. A significant and severe plastic loss was noticed on the as-received sample with about 2.351g/min loss. It is obvious that the non structure of the counter body over the rigid plan surface could have eroded the mild steel leading to severe grooves and asperity seen. With the coated sample, a strengthening structure was seen which obviously lead to better resistance to wear loss.

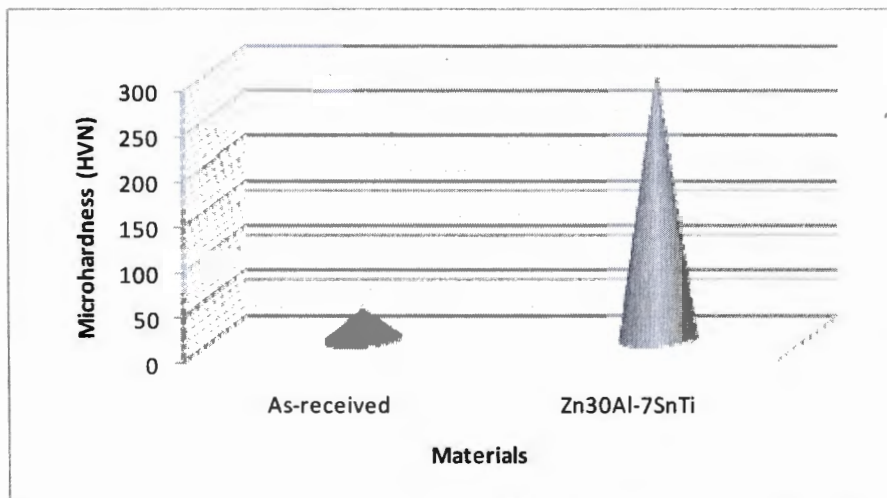
*In* Figure 3, a comparative study between the as-received and Zn-Al-7Sn-Ti-0.3V-CI composite coating was presented in a plot. From the result a geometric increase was noticed from 33.4 HVN to 299 HVN for coated alloy. This significant improvement was observed to be as a result of influence of the relative composite activities and the deposition potential. Although, operating condition is a major factor for enhance structural properties which could also facilitate improved hardness performance [12]. Invariably, structural evolution depends on the processing parameters which are critical to the formation of new phase capable of causing solid solution for improved hardness characteristics.



**FIGURE 1.** SEM image of a) mild steel b) Zn-Al-7Sn-Ti-0.3V-CI at 1000x



**FIGURE 2.** Comparative wear analysis for Zn-Al-Sn-Ti Coatings



**FIGURE 3.** Microhardness progression for Zn-Al-Sn-Ti and as-received sample

## CONCLUSIONS

In the present work, we have tried to produce new materials of metal-ceramics composites with unique properties that used to improve microhardness and wear deformation in manufacturing system. These composites coating can be made to function in a multifaceted environment where instability are a major constrain.

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## REFERENCES

1. J.L. Mo, M.H. Zhu, B. Lei, Y.X. Leng, N. Huang, *Deposition Phys. Vap. Deposition Wear* 263 (2007) 1423
2. O. S. I. Fayomi, A. P. I. Popoola. LR., Kanyane, T. Monyai *Results in Physics*, 7, 2017 644–650
3. A.A., Daniyan, Lr., Umoru, A. P. I. Popoola, , O. S. I. Fayomi. *Results in Physics*, 7, 2017 3222–3229
4. O. S. I. Fayomi, A. P. I. Popoola, A.A. Daniyan, 2016. *Particulate Science and Technology*,35,4,418-425
5. J.L. Mo, M.H. Zhu, B. Lei, Y.X. Leng, N. Huang, *Deposition Phys. Vap. Deposition Wear* 263 (2007) 1423
6. O.S.I. Fayomi, A.P.I. Popool, *Int. J. Electrochem. Sci.* 8 (2013) 11502
7. S. Hongmark, P. Hedenqvist, S. Jacobson, *J. Surf. Coat. & Tech.* 90, (1997) 247
8. K.H. Zum Gahr, *Wear.* 200 (1996) 215
9. A.A. Volinsky, J. Vella, I.S. Adhietty, V.L. Sarihan, L. Mercado, B.H. Yeung, W.W
10. Gerberich, *Mat. Res. Soc.* 649 (2001) 6
11. R. Xu, J. Wang, Z. Guo, H. Wang, *J. Rare Earth* 26 (2008) 579
12. P.A.L. Anawe, O.S.I. Fayomi, *Results in Physics*, 7, 2017, 777-788
13. P.A.L. Anawe and O.S.I. Fayomi Impact of Applied Potential on the Structural and Non-lubricated Wear Composite Coating in Petrochemical Industry Port. *Electrochim. Acta* 35, 5, 297-303
14. O. S. I. Fayomi, A. P. I. Popoola, O.O. Ige, AA. Ayoola, 2016. *Asian Journal of Chemistry* ,29,25,2575-2581
15. Anawe, P.A.L., Raji, O., Fayomi, O. S. I., *Elsevier Procedia Manufacturing* 7 (2017) 556 – 561.
16. Anawe, P.A.L., Raji, O., Fayomi, O. S. I., 2017. *Procedia Manufacturing* 7 (2017) 562 – 566.