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NANOSCALE AND NANOSTRUCTURED MATERIALS AND COATINGS

Nanoparticle Dispersion, Microstructure and Thermal Effect of Multi-doped ZrO_2/SiC from Sulphate Induced Electrolyte[1]

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Abstract—Effort to improve the hardness and thermal resilient properties of coating for advanced engineering applications has necessitated this study. Zn sulphate electrolyte was induced with ZrO_2-SiC composite particulate at varied current density of 1.5 and 2.0 A/cm^2 for 10 minutes. The incorporated composite particles of ZrO_2/SiC were varied in other to examine their mechanical responses on zinc electrolyte. The coated films were characterised with scanning electron microscope with attached electron dispersion spectroscopy (SEM/EDS) and atomic force microscopy (AFM). The micro-hardness properties of the coated and thermal aged alloy were determined with high diamond micro-hardness tester. The anti-corrosion progression was examined using linear polarization technique in 3.65% NaCl. From the results, the incorporation of the composite matrix was found to impact significantly on the surface and microhardness properties. The co-deposition of composite submicron on the zinc electrolyte revealed that homogenous grain structure was obtained. To this end, a boost in the performance characteristics was attained due to effective co-deposition parameters in the electrolyte.

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INTRODUCTION

The durability of any materials has been known to depend on the properties of the coatings and the operating environment [1–4]. The subject of co-deposition of metallic coating containing submicron composite particles are recently invoke for new emerging technological materials [5–8]. Challenge with contemporary coated alloy has been due to selection of particle incorporation, proper bath formulation and process parameter to overcome atmospheric propagation.

However, the need for composite alloy development for the purpose of combating high temperature infringement becomes necessary for researcher [9–14]. According to [15, 16], morphological and structural build-up as results of the co-deposition of a satisfactory amount of non-agglomerated particles often causes formation of harder and more resistant coatings. Although the principle of metal matrix composite for fabrication of sustainable durable alloy consist of the following process of formation which are, behaviour of particles in suspension, particles mass transfer from the bulk of the suspension to the electrode surface and most importantly the particle incorporation and irreversible entrapment simultaneously with the growing metal layer.

Different particle likes Cr_2O_3, SiO_2, ZrO_2, Al_2O_3, SnO_2, CeO_2, ZnO to mention but a few have been successfully used by direct electrolytic-system to offer significant functional individual characteristics [15–17]. However, restrain in developing a multi-link composite induced system and control of formulated variable for advance processing are needed to give good structural build-up against harsh environment. In this present work, a successful functional bath containing ZrO_2/SiC particulate on zinc electrolyte has been used to produce hard alloy that could resist high temperature impunity.

EXPERIMENTAL PROCEDURE

Preparation of the Substrates

Mild steel with (30 × 20 × 1 mm) sheet dimension was used as cathode substrate and zinc sheets of (50 × 30 × 2 mm) were prepared as anodes. The initial surface preparation was performed with fine grade emery paper, properly cleaned with sodium carbonate, descaled/pickled and activated with 10% HCl at ambient

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Table 1. Nominal chemical composition (wt %) of mild steel substrate

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

Table 2. Bath formulation

Composition	Mass concentration
Zn	50 g/L
Thiourea	10 g/L
Boric Acid	10 g/L
ZrO_2	5–10 g/L
SiC	7.5–12.5 g/L
$ZnSO_4$	75 g/L
pH	4.8
Current dens	1.5–2.0 A/cm^2
Time	10 minutes
Na_2SO_4	30 g/L

temperature for 10 seconds, then followed by instant rinsing in deionized water. The mild steel specimens' chemical composition is shown in Table 1. The cathode was made of mild steel coupons and the anode used was commercially 99.99% pure zinc obtained from SERC research centre, Pretoria, South Africa.

Bath Formulation

Single cell containing three electrodes (two anodes and one cathode) were used for the co-deposition series. Zinc plates were used as anodes and the mild steel samples as a cathode. Analytical grade reagent and de-ionized water was used for all prepare plating admixture electrolyte. The bath formulations are shown in Table 2. Table 3 shows the expected design fabricated patterns.

The samples were polished using a emery paper of 1200 μm. After the surface preparation was completed, the samples were dipped in HCl for 10 secs for good adhesion properties and cleaned with distilled water. Cathode and anodes were connected to the D.C. power supply through the rectifier. The deposition was carried out with varying current densities of 1.5 and 2.0 A/cm^2 for 10 minutes. The plating bath was continuously stirred by magnetic method for a stable dispersion for 2 hrs.

Structural Properties of the Deposits

For structural properties VEGA 3 TESCAN scanning electron microscope with an attached energy dispersive spectrometer (SEM/EDS) was used. The adhesion profiles, topography coatings, were observed

Table 3. Formulated design bath composition of Zn–ZrO_2–SiC alloy deposited at 10 min

Sample order	Matrix Sample	Current density, A/cm^2
1	Zn–5gZrO_2	2.0
2	Zn–10gZrO_2	2.0
3	Zn–5gZrO_2–7.5gSiC	2.0
4	Zn–10gZrO_2–12.5gSiC	2.0

with the help of Atomic Force microscopy (AFM). High optic diamond based durascan microhardness tester was used to estimate the mean value of microhardness with a load of 100 g for a period of 20 seconds. The average microhardness trend was measured across the coating interface in an interval.

Thermal/Electrochemical Studies

Heat treatment was done with direct furnace atmospheric system on the sulphate produce composite at 200°C for 2hours to check the mechanical stability. The electrochemical studies were performed with Autolab PGSTAT 101 Metrohm Pontentiostat using a three-electrode cell assembly in 3.65% NaCl static solution at 40°C. The developed composite was the working electrode, platinum electrode was used as a counter electrode and Ag/AgCl was used as a reference electrode. The anodic and cathodic polarization curves were recorded by a constant scan rate of 0.012 V/s that were fixed from ±1.5 mV. From the Tafel corrosion study, the corrosion rate, potential and linear polarization resistance was obtained.

RESULT AND DISCUSSIONS

Microstructural Properties

Figures 1, 2 showed the SEM/EDS micrographs of some developed alloy composite coatings. It is obvious to see a distinctive nodules crystal with Zn–ZrO_2–SiC showing a more closed structure than the Zn–ZrO_2 coatings. Although particle incorporation of ZrO_2 in to the Zn interface is seen to be visible from EDX (see Fig. 1). In other word, the solubility of zirconium in zinc electrolyte from the microstructure was appreciated owing to the homogeneous composites dispersion of the metal-particle on coating contact surface area.

With more pronounced fine crystals ridges with equi-granular structure in Fig. 2, it was observed that the structure may not likely to be aided by particle agglomeration but steady particulate adsorption movement toward the cathode region. More so, it is assumed that with zinc participation, the film growths were assisted by zinc ion interaction with individual grains of other composite oxide/metal. In view of this,