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Electrodeposition of Metal Alloys (Ni, Zn and Fe) and their Characterization: A Review

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Abstract: ***Metal alloy thin film deposition and their properties are briefly reviewed in this article. This includes of structural studies (crystallinity, grain size and surface texture), electrical, corrosive and optical properties (absorbance, transmittance, refractive index and optical band gap) corrosion behavior. It has been experimented that the deposition of thin films dependent on different parameters like deposition technique, deposition temperature, bath composition, deposition time, thickness of the film etc. composition analysis were performed using SEM. Energy dispersive x-ray analysis of the deposit confirmed the presence of iron nickel and zinc in the deposit. The results showed that metal alloys can be produced with good quality adhesive and anticorrosive properties and all films have the same crystallographic structure and different crystallographic orientations. Alloys systems are very eye-catching due to their good corrosion and wear resistance activities, which make them appropriate for significant technological applications.***

Keywords: Thin film, Electro deposition Technique, Alloy Characterization, Corrosion

1. Introduction

Thin film science has grown universal into a main research area. The importance of coatings and the synthesis of new resources for industry have resulted in a great increase of innovative thin film processing technologies [1]. Thin films are generally used as place on a substrate for integrated optical circuit, capacitors, transistors etc. There have been some studies on thin films [2-6]. Metal alloys thin films are main because of their various applications. An alloy is a material that has metallic properties and is comprise of two or more chemical elements, at least one of which is a metal. Examples of alloy coating comprise of gold–copper–cadmium, zinc–cobalt, zinc–iron, zinc–nickel, brass (an alloy of copper and zinc), bronze (copper–tin), tin–zinc, tin–nickel and tin–cobalt. Alloy coatings are produced by plate two metals from the related solution. Metal thin layer can be fabricated by a variety of methods for example chemical vapor deposition (CVD), electro deposition, chemical bath deposition (CBD), thermal evaporation, successive ion layer adsorption and reaction method (SILAR) etc. Electro deposition thin film preparation method is mostly used to deposit metal films [7]. It deals with the combination of solid films from dissolved species by alteration of their oxidation states using electricity [8]. Electroplating is an electro deposition method for producing a dense, uniform, and adherent coating, generally of metal or alloys, upon a surface by the act of electric current [9]. The coating formed is generally for decorative and for defensive purposes, or enhancing specific properties of the surface. The surface can be conductors, for example metal, or nonconductors, such as plastics. Electroplating products are commonly used for various industries, such as automobile, ship, air space, machinery, electronics, jewelry, defense, and toy industries. The studies of these thin films supply directly or indirectly a way for latest areas of research in thin film chemistry and optoelectronic devices.

2. Literature Review

Giz et al.(2003) electrodeposited Ni-Cu-Fe on mild steel substrates having an exposed area of 0.5 cm^2. Ni, Cu and Fe were co-deposited from an acetate bath contain 130 gL^{-1} $(CH_3COO)_2$; 10 gL^{-1} of $FeSo_4$; 0.22 gL^{-1} of $Cu(OOCCH_3)_2$; 25gL^{-1} of H_3BO_3 and 0.75 ml L^{-1} of $HCONH_2$. Vishalakshi et al.(2006) prepared Zn-Ni alloys on mild steel while Electrolytic zinc and Electrolytic nickel were used as anodes. Kim et.al.(2014) have developed a bath consisting of manganese acetate and nickel chloride for the electro deposition of manganese-nickel (Mn-Ni) oxide films like electrochemical capacitor electrodes [22]. Stankovic et al. (2004) investigated XRD data of Zn-Fe alloy and found the structure of alloy same as zinc but with different phase orientation. Bhattarai et.al.(2007) investigated X- ray diffraction of binary W-Ni alloys the apparent grain ranges from 1.5 to 6.0 nm. Ebadi et al.(2010) investigated XRD data of Ni–Co layers with and without PPMF (4.4 T) at different amount of cobalt. Pathak et.al.(2014) studies Zn-Se-Hg layer of Corrosion characteristics of compositionally modulated ZnSeHg deposits were evaluated using Tafel polarization techniques.

3. Methods and Approach

Different methods have been used for electro deposition of metal alloys. Some methods are electrochemical deposition, corrosion measurement techniques, crystallography surface morphology etc.

3.1 Weight Loss Method

In this method weighed clean piece of alloy immersed test solution for a particular time period after the removing alloy piece to determine the loss of weight. The corrosion rate is calculated

$$CR = \Delta W*K/D*A*t$$

CR =corrosion rate (mmpy), ΔW= weight loss of alloy after emersion (g), A= area of sample alloy (Cm^2) t= time in hours.

3.2 Surface Morphology

Scanning electron microscopy (SEM) is a method for high resolution imaging of surfaces. The micro structural and surface morphological thin films will be performed by (SEM). Energy Dispersive X- ray Analysis (EDAX) of elemental composition will be done by Enegry dispersive x-ray spectrometer with SEM.

3.3 Crystallography

X –ray diffraction (XRD) analysis is a method used for determining the atomic and molecular Structure of a crystal, in which the crystalline atoms cause a beam of X –ray to diffract into many specific directions. X-ray crystallography will be used to determine its structure.

4. Experimental Details

Ni, Cu and Fe were co-deposited from an acetate bath contain 130 gL^{-1} $(CH3COO)_2$; 10 gL^{-1} of FeSo4;0.22gL^{-1} of $Cu(OOCCH3)_2$;25gL-1 of H3BO3 and 0.75ml L^{-1} of $HCONH_2$.The deposition were carried out at a current density of 25 mA cm^{-2} for 60 min using nickel foil as counter electrode [10]. Electro deposition of Zn-Fe alloys galvanic statically at 0.5 – 15.0A dm^{-2} on a steel panel or on a rotating disc electrode at 25 and 40 ºc from alkaline bath: 0.09 mol dm^{-3} $ZnSO_4$, 0.01 mol dm^{-3} $FeSO_4$, 0.01 mol dm^{-3} ascorbic acid, ≈ 0.2 mol dm^{-3} triethanolamine, 30 g dm^{-3} Na_2SO_4 and 80 g dm^{-3} NaOH (pH ≈14) [11]. Preparation of Zn-Ni alloys on mild steel while Electrolytic zinc and Electrolytic nickel were used as anodes. For deposition solution, Zinc sulphamate was prepared by dissolving Zno in sulphamic acid (99% purity). Annular grade nickel sulphamate was used. Sulphamate complex brought the deposition potentials of zinc and nickel closer and the alloy deposition was of 98–99% efficiency [12]. The binary W-Ni alloys were prepared through direct current (D.C.) magnetron sputtering on glass substrate [13]. The Zn-Co alloys, with Co content higher than 1%, were electrodeposited on a mild steel surface using an additive-free plating solution (bath 1) containing 1 mol dm-3 $ZnCl_2$, 0.1 mol dm^{-3} $CoCl_2$, 0.5 mol dm^{-3} H_3BO_3 and 2.6 mol dm^{-3} KCl, preheated to 60 °c. Electrodeposited Zn-1 Co coating was obtained at room temperature from a bath that be prepared by just addition 10 ml of star zinc to 1 l of the bath 1 (bath 2) and was carried out at room temperature. The pH of both plating solutions was 5.0.Steel plate was used as cathode and Zn plate as an anode [14]. Ni–P thin films were prepared by galvanostatic electro deposition. Copper substrates were use as working electrodes. The copper electrode and reference electrode were a graphite plate and a saturated calomel electrode (SCE), respectively. The deposition Bath contains 0.2 M $NiSO_4 \cdot 7H_2O$, 0.10 -0.3M NaH_2PO_2, 0.4 M H_3BO_3, 0.7M NaCl and 0.005 M Saccharin at 70°C using current density of 7.5-10 mA/cm^2 [15].The electrodeposition of Ni–Co alloys in the absence and presence of a PPMF was carried out on copper substrates (0.01 × 1 × 2 cm) using an electrochemical cell with a conventional Nickel Watt's solution .The pH was used to 4.0 by adding sulphuric acid. Current density at 75 mA cm^{-2} with temperature 50 -55 °C [16]. Electrodeposition of zinc–iron alloy was carried out from the sulphate bath. The optimized composition of the bath used: 0.28M; $[ZnSO_4.7H_2O]$, 0.41M $[FeSO_4\ (NH_4)_2SO_46H_2O]$ and 1.07 M [KCl], EDTA 0.08M. The deposition were carried out on mild steel panels (5.0 cm × 2.5 cm × 0.05 cm) is cathode and the anode used was electrolytic grade zinc.[17]. Zinc-nickel coating were electrochemically deposited onto carbon steel substrates from a weak acid bath. The bath contained 0.1 mol L^{-1} zinc chloride ($ZnCl_2$), 0.1 mol L^{-1} nickel chloride ($NiCl_2$), 3.0 mol L^{-1} ammonium chloride (NH_4Cl), 0~0.01 mol L^{-1} TEOS, 0.05 mol L^{-1} hydrochloric acid (HCl) and 0.02 mol L^{-1} boric acid (H_3BO_3). Using Current density 10 MA cm^{-2} [18]. Iron & Copper was deposited inside alumina pores from an aqueous electrolyte containing Iron sulphate (0.1 M) and Copper sulphate (0.1 & 0.01 M) through applying high frequency (200 Hz) alternating current. After electrodeposition, the oxide film was removed partially in 0.5 M phosphoric acid at 87°C for 15 second [19]. Studies carried on Zn-Co-Ni alloy coating on mild steel using pure Zn plat as anode from sulphate bath. All depositions were carried out galvano statically in common Conditions of temperature and pH for duration of 10 minutes [20]. The Zn–Ni–Co alloys were deposited galvano statically, at various current densities, from chloride baths of the following composition : 0.38 mol dm^{-3} $ZnCl_2$; 0.24 mol dm^{-3} H_3BO_3; 3.85 mol dm^{-3} KCl and either 0.34 mol dm^{-3} $NiCl_2.6H_2O$ and 0.04 mol dm^{-3} $CoCl_2.6H_2O$, or 0.20 mol dm^{-3} $NiCl_2.6H_2O$ and 0.18 mol dm^{-3} $CoCl_2.6H_2O$, at 25 °C. The working electrodes were Zn–Ni–Co alloys deposited on steel panel (20 mm×20mm×0.25 mm) [21].

4.1 Structural Analysis

Now a day's development of structural analysis technique, it is possible to investigate structure of smallest particles accurately**.** Some scientist investigated XRD data of Zn-Fe alloy and found the structure of alloy same as zinc but with different phase orientation. The reflections of zinc rich phase are present in all investigated deposits [11]. X-ray diffraction studies of the Zn-Ni alloy and X-ray patterns of the electro deposit revealed that reflections were obtained from (330,411) planes of d-Ni_3Zn_{22} plane along with (204) of zinc and (311) of nickel planes. On a few deposits, signals corresponding to (002,311) of $Zn(OH)_2$ were also seen [12].

X- ray diffraction of binary W-Ni alloys the apparent grain ranges from 1.5 to 6.0 nm. The W-Ni alloys containing 23-69 at % nickel having the apparent grain size less than 2 nm. and 78 at % nickel were nano crystalline arrangement having the grain size of 6 nm or less in size. The W-15Ni alloy of amorphous and nano crystalline structures as of XRD pattern having the apparent grain size of 2.5 nm [13]. X-ray diffraction patterns of Ni–P alloy deposit at various NaH_2PO_2 concentrations (0.10 M to 0.20 M) and the FCC structure with three nickel peaks (111), (200) and (220). It is also observed that the 2θ values are faintly shifted to top angles indicating the presence of phosphorous in the films [15]. XRD data of Ni–Co layers with and without PPMF at different amount of cobalt. The grain dimension changing of deposited layers could be calculated from XRD data through

the Debye-Scherrer equation [16]. XRD patterns of Zn-Fe alloys at different current densities. Pyramidal textural intensity was significant than the prismatic and basal textures. Peaks representing specific crystallographic plane gradually shifted either to higher or lower 2θ angles with the change in iron content in the deposit [17]. Researcher reported XRD patterns of the composite Zn-Ni films fabricated by adding different concentrations of TEOS: (a) 0 M, (b) 0.005 M, and (c) 0.01 M. The result indicates that TEOS can act as a structure modifier to change the structure of the deposit [18]. X-ray diffraction of Zn-Co- Ni alloys at different deposition. XRD study found that the Zn (110) is the prominent phase, which responsible for peak corrosion resistances of ternary alloy coating. Large decrease in corrosion rate of ternary alloy is due to significant change in the crystal lattice of the coatings [20].XRD analysis of Mn - Ni revealed that the electrodeposited Mn-Ni oxide film contained separate manganese oxide (MnO_2) and nickel oxide (NiO) [22].

4.2 Morphological and Compositional Analysis

Discovery of electron microscope was revolutionary changes in research of chemistry. Many scientists studied scanning electron micrograph by a magnitude of 10000X for the Ni-Cu-Fe surface just after the electro deposition. It is observed a very uneven surface with a cauliflower structure signifying that the electro deposit has a bigger area when compared with just the Ni-Fe material [10].The chemical composition of the Zn–Fe alloys determined by EDX analysis of the deposits using SEM and EDX analysis, so Fe amount is the greatest in the case of alloy deposited at 10A dm^{-2} (2.2 wt.%, whereas it was 1.3 wt.% for alloy deposited at 4A dm^{-2}).So on the basis of the chemical composition and results. The differences in electrochemical properties among different Zn–Fe alloys rise from different chemical composition and surface morphology of alloys obtained at different current densities. It is well known that Zn coatings deposited by different deposition parameters have differences in porosity, structure and other characteristics, which, in turn, affect the corrosion resistance of the coating [11]. The zinc nickel alloy electrodeposited at a current density of 1.5 A/dm^2 was creating to have a fixed and non uniform surface. The presence of aldehydes in the bath offered deposits with smooth uniform and non porous surfaces [12]. Surface morphology of W-Ni alloys and the surface of the passive films created on the W-23Ni and W-69Ni alloys are very even and highly reflective similar to the surface oxide-films formed on the as prepared sputter-deposited W-Ti alloys [13]. Scanning electron micrographs of Ni–P films deposited at different phosphorous concentrations were recorded. SEM obtained for a film deposited at a NaH_2PO_2 concentration of 0.15 M. The surface morphology is found to be even with spherical shaped grains. The average grain size is found to be in the range between 0.2 and 0.30 µm. The surface morphology appears to be composed of larger nodules of "cauliflower" type structures typical for amorphous materials. As the NaH_2PO_2 concentration was increased to 0.25 M, the grain size increased. EDAX studies of Ni–P alloys reveal an enhance of phosphorus content in the film with an increase in the NaH_2PO_2 concentration in the deposition bath [15]. SEM analysis of the Zn, Co and Zn–Co thin films deposited in different solutions. They found fine-grained deposits in the presence of high zinc/cobalt electrolyte ratios but dendrite deposits when the comparative cobalt concentration was increased. It is seen that Co plays the role of a grain-size refiner in zinc–cobalt alloys. In the pure cobalt deposit, the grains were not visible. The structure was compact [23]. SEM micrographs of Ni and Ni–Co deposits at 75 mA cm^{-2} for 7 min Nickel Watt's bath based on copper plate; Ni in the absence of MF; Ni in the presence of a MF at 4.4T; Ni–Co deposited in same bath solution which; Ni–Co in the absence of MF; in the presence of PPMF 4.4T. Holes on the surface of the cobalt deposit from the effect of hydrogen evolution effect had disappeared with the application of a magnetic field. An increased mass transport of cobalt ions have diminished the effect of hydrogen evolution reaction under magnetic field which gave larger cobalt crystallites, Prevented the appearance of holes attributed to hydrogen evolution reaction and gave more dense Cobalt deposits [16]. The deposit morphology Zn –Fe alloys was influenced by the alloy composition. Three types of morphologies were found. For zinc-rich alloy (<10% iron content), distorted hexagonal (1 A dm^{-2}) morphology noted. For an alloy with finest (10–25%) iron content, triangular pyramidal morphology (2–4 A dm^{-2}) observed. Even and dense deposit with fine-grained morphology (with an iron weight percentage of ≈ 17%) noted at 3A dm^{-2}. Coarser deposits with stacked platelet coloum. Morphology were noted above 5 A dm^{-2} (iron content >25%). Reduction of crystallite size at 3 A dm^{-2} indicates that the nucleation rate was higher than the grain growth rate. This indicates the overall deposition rate was mainly controlled by charge-transfer processes rather than mass-transfer processes [17]. Electro deposition of Fe-Ni-Cr alloys by SEM and EDAX technique gives the result that the upper Cr% in alloy composition without Cr-complexing agent at -1.5V; structural morphology is fine grained dense nodular deposit. The present studies, the morphology of alloy deposited at -1.3V and the corresponding EDAX pattern reveals the presence of Fe-53.7%, Ni- 41.7% and Cr-4.5%. Deposits were darker than one obtained at -1.4 and -1.5V, nodular grain sizes range from 0.3-0.7µm with agglomeration of particles formed are seen [24]. Electro deposition Fe-Ni-Cr alloys by SEM and EDAX technique gives the result that the upper Cr% in alloy composition without Cr-complexing agent at -1.5V; structural morphology is fine grained dense nodular deposit. The present studies show the morphology of alloy deposited at -1.3V and the corresponding EDAX pattern reveals the presence of Fe-53.7% Ni- 41.7% and Cr-4.5%. Deposits were darker than one obtained at -1.4 and -1.5V, nodular grain sizes range from 0.3-0.7µm with agglomeration of particles formed are seen [25]. Observation of the surface morphology of Zn-Ni alloys coatings deposited with 10 mA cm^{-2} at different concentrations of TEOS: 0, 0.005 and 0.01 mol L^{-1}, are compared. This examination of the deposits using SEM indicated that both the morphology and grain size are affected by the presence and concentration of TEOS [18]. Researcher investigated morphological and structural analysis of the Ni-Se alloys were revealed by the SEM. It result that the thin films are composed of largely regular-shaped grains. The grains tend to cover the substrate surface completely. This is due to some crystallites grouped together to form larger grains. Grain size lies below 100nm, while the micro-strain in the samples was determined and which

indicates the presence of compressive stress in some samples and tensile stress in rest of the electrodeposits. The EDAX analysis confirms the inclusion of Nickel, Aluminium and Selenium in the deposited thin films. From EDAX study atomic percentage of deposited elements is obtained [26]. Studied of electro deposition of ZE41 Mg-Zn-Rare Earth Alloy by SEM. SEM micro-image showed that the film formed due to 10 g L^{-1} permanganate treatment is the best among the other concentrations in terms of the coating distribution and surface morphology [27]. Electrodeposition of Fe-Zn alloy (SEM) image of top surface of porous aluminum oxide produced after anodizing the aluminum foil in 0.24M oxalic acid Pores having diameter in the order of 25 to 35 nm were seen uniformly throughout the surface. Energy dispersive x-ray (EDX) analysis was carried out on both the locations for identifying the constituents deposit EDX spectrum indicates the presence of aluminum and oxygen which are from the base substrate and its conversion to aluminum oxide [28]. Study on the electrodeposited films by scanning electron micrograph images. (a) CdHgSe and (b) 0.0002M Tl containing CdSeHg films the films are continuous and homogenous, the films show to be polycrystalline in nature and heavily packed. The morphology of the films is affected by Tl incorporation. The grains of the deposit at improved Tl content appeared clearer and larger than that of CdSeHg [29]. SEM studies showed that the Mn-Ni oxide film was well covered with a variety of nano fibers in a three-dimensional network. Porous spaces were also observed between the nano fibers, which are necessary for electrochemical capacitor applications. The size of the pores in the film was generally distributed in the range of 2–20 nm [23]. The SEM of Co-Ni-Fe alloy electrodeposited at -1.30V shows that the electro deposition occurred evenly. The existence of Co, Ni and Fe peaks for the electro potential of -1.30V (SCE) confirmed the information that the synthesized product is Co-Ni-Fe alloys for of peaks credited to Co, Ni and Fe. EDX spectra examination of the thin films composition show that the Co and Fe content of the alloy decreased with the increase in electro deposition potentials whereas the Ni content increased [30].

4.3 Corrosion Behavior

Corrosion is major problem with metals alloys. Many studies carried on corrosion behavior of metals alloys. Zn–Fe alloys electro deposition on the corrosion resistance of these alloys was obtained that Zn–Fe alloy deposited at 4Acm^{-2} exhibited the lowest corrosion rate, i.e., the longest time of red rust appearance and the lowest [12]. Positive shift in E_{corr} and decrease in I_{corr} values for the Zn–Ni electrodeposits indicates its better corrosion resistance nature. Adsorption of these aldehydes and their inclusion caused a reduction in corrosion rates [13]. Nickel metal acts synergistically with tungsten in enhancing the corrosion resistance of the W-Ni alloys so as to show about two orders of magnitude lower corrosion rate than the corrosion rate of tungsten and even slightly lower than that of nickel in 1M NaOH solution. Zn–Fe layer reveals 10% nobler corrosion potential, −0.987 vs. −0.897 VSCE, and two times minor corrosion current density, 42.0 vs. 19.7 μA cm^{-2}. The proposed baths are much easier for bath control during deposition. It is concluded that the deposited Zn–Fe alloy coating possesses superior anti-corrosion behaviors than that of Zn Coating [31] ZnCo electrodeposited alloys the smallest I_{corr} of the studied substrate/coatings systems, 2.4 μA cm^{-2}, was obtained at I = 10 A m^{-2}. In these conditions, the coatings contained ~ 30 % m/m Co and 70 % m/m Zn and only the γ- ZnCo phase was obtained. The I_{corr} create in the present work is smaller than those generally initiate in the literature for Zn-Co coatings with small Co content [32]. Zn-Se-Hg layer of corrosion characteristics of compositionally modulated ZnSeHg deposits were evaluated using Tafel polarization techniques. The corrosion parameters determined from the polarization curves recorded in 0.5M NaCl solution at room temperature. The corrosion resistance increases on addition of Hg content. The corrosion current decreases with Hg increments [33].

5. Conclusion

Electro deposition thin film study on the different electro deposition method has been review in different metal alloys (Zn,Fe,Ni and other related material). Effectiveness of electrodeposition depends on their choice of suitable electrolytes, concentration of electrolytes, temp, pH, applied potential, additives etc. Electrodeposited thin film alloys investigate the dependence of the structural, compositional, electronic and morphological properties at various electrolyte concentrations. Almost similar data was observed for the films synthesized by different synthesis techniques and conclude that thin film alloy higher resistance to corrosion.

6. Future Scope

In future, it is possible to develop more efficient thin film by the use of combination of different suitable metals alloys, which have high resistance power against corrosion. It is also possible to use cheap metals for preparation of alloys for reduces the manufacturing cost.

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