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# Influence of vanillin on the corrosion behavior of Ni-W alloy electrodeposits and its properties



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#### ABSTRACT

A simple and viable electrodeposition technique has been implemented to fabricate nanocrystalline Ni-W electrodeposits from an alkaline citrate bath with variations of vanillin (additive) concentration. The optimization of additive in the bath was substantiated in terms of its corrosion resistance property (evaluated using Tafel polarization and electrochemical impedance spectroscopy). For alloys deposited in presence of additive (100 ppm) in the bath, resulted in low corrosion rate (C.R) and high charge transfer resistance (Rct). The enhanced corrosion resistance property (i.e. low C.R and high  $R_{\rm ct}$ ) was deduced in terms of its homogenous surface morphology (using scanning electron microscope), reduction in crystalline size (using X-ray diffraction study), diminish in surface roughness value (using atomic force microscopy) as compared with that of electrodeposits obtained in the absence of additive. The inclusion of additive in the electrodeposits during electrodeposition, showed a major impact for its significant corrosion resistance property (C.R. — 0.12 mm/year and  $R_{\rm ct}$ -2177.83  $\Omega$ ·cm²). With an increase in the additive concentration in the bath, the corrosion resistance of the Ni-W coating becomes inferior, due to its porous nature and relative decrease in its capacitive behavior at electrode/electrolyte (corrosive) interface.

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#### 1. Introduction

Mild steel, one of the foremost iron alloy with superior mechanical, physical and electrical properties, makes its usage in diverse industrial and structural applications [1]. Industrial applications include, in the fabrication of reaction vessels, storage tanks and in particular petroleum refineries due to its low cost and good ductility [2]. Nevertheless, its resistance towards corrosion attack is very poor under the appliance of acidic solutions. Protection of mild steel has become one of the key topic in industrial and academic areas, predominantly in acid media [3]. Increased usage of acidic solutions in industry includes oil-well acidizing in oil recovery, acid pickling and descaling, industrial cleaning and in the petrochemical process. Nanocrystalline alloy coatings with improved properties are widely applied as protective coatings in the surface coating community. The usage of metallic alloy coatings with improved corrosion stability are in demand in the present surface technology applications. Nanostructured Ni-W coatings are considered as a versatile choice of protective coatings on various substrates in diverse structural and engineering applications, due to its excellent combinations of tribological, magnetic, electrical, and electro-erosion properties [4]. The corrosion resistance towards strong oxidizing agents and also

\* Corresponding author. E-mail address: pramod7147@gmail.com (P.K. U.). towards aggressive high temperature corrosive environments makes them to use in major industries like automotive, aircraft and in machinery parts. Moreover, it is considered as a strong candidate for the replacement of environmentally hazardous hexavalent chromium plating. Some of the notable applications that contributes for its high temperature resistance are turbine blades, [5] manufacture of injection nozzles in combustion engines [6] and as a substrate for high temperature superconductor [7]. Despite the development of new technologies. traditional electroplating still plays an important role in surface enhancement [8] and it is considered as an intrinsically fast technique which is compatible with patterning and large scale production [9]. The advancement of this technique has been seen from laboratory scale to industrial relevance, due to its feasibility in controlling the process parameters [10,11]. Further this fabrication processes, has provided a smooth platform to fabricate nanocrystalline alloy coatings with tailored properties for the advanced engineering applications. An addition of small amount of additive to the plating bath leads to significant changes in the properties and aspects of electrodeposit [12]. Most of the additives are organic species, that are called as brighteners or grain refiners, which helps in attaining uniform, smooth and homogenous deposits. In consideration, with the Ni-W alloy deposition, researchers claimed that the impact of usage of additives in the plating baths is found to be same and considerable [13-15]. Vanillin is an aromatic aldehyde with a pleasant fragrance and the main compound in vanilla flavoring and can be found in perfumes and other products.

Vanillin, as alone and its condensation product as an additive in plating baths [16–19] and as inhibitor [20,21] in corrosive solutions for the protection of metals is well exploited. Nonetheless, very rare attention has been paid in studying the effect of vanillin in Ni-W alloy plating baths as additive. Thus the present study, aimed to study the effect of vanillin on Ni-W electrodeposits and to optimize the additive concentration in the bath, based on the obtained corrosion results. Further, the cause of high corrosion resistance property is substantiated in terms of structural, morphological, and other surface characterization studies, for the deposits obtained in the absence and in the presence of additive from optimized bath.

#### 2. Materials and methods

The electrodeposition of Ni-W alloy coatings onto mild steel was conducted at constant temperature (60 °C) and current density of 5 A·dm<sup>-2</sup> for 15 min. The bath solution was prepared using analytical grade chemicals with twice distilled water. The electrolyte composition and the operational parameters used to electrodeposit Ni-W coatings are pointed out in Table 1. The electrolyte composition reported in the literature [22], was used as the base bath in this study. The bath solution was prepared at ambient temperatures with incessant stirring. Prior to the electrodeposition, pH of the electrolyte was adjusted to 8, by the respective additions of sulphuric acid or sodium hydroxide. A freshly prepared bath was used for the electrodeposition of Ni-W coatings at various concentration of 0, 50, 100, 250, 500 ppm of vanillin (additive). All the deposition experiments were done in a thermostat glass cell, containing 40 cm<sup>3</sup> electrolytes. Galvanostatic deposition, using direct current was implemented to deposit Ni-W alloy coatings. A two electrode system, containing platinized titanium (25 mm  $\times$  25 mm  $\times$ 1.5 mm) as an anode and mild steel plates of dimension (25 mm  $\times$ 25 mm  $\times$  0.3 mm) were used as cathodic substrates. The selected mild steels were well pretreated right before the deposition. The desired surface of the mild steel, were wiped with cotton gently, to remove the loosely adhered particles, ultrasonically degreased with acetone for about 15 min and then mechanically polished to attain mirror finish with diamond paste using twin disc polisher, followed by electro cleaning in the conventional alkaline cleaner solution (contain 3.5 g/l NaOH and 2.5 g/l Na<sub>2</sub>CO<sub>3</sub>) for about 2 min and then the mild steel is rinsed with twice distilled water and etched in 5% H<sub>2</sub>SO<sub>4</sub> solution, and again rinsed well using twice distilled water and immediately transferred to the beaker containing 40 cm<sup>3</sup> of electrolytes. After deposition the plates were well rinsed, to remove the loosely adsorbed oxide particles and left for air drying. Experiments were done in triplicate for each concentration in order to optimize the additive concentration in the bath solution.

#### 2.1. Electrochemical corrosion testing

The corrosion behavior of the Ni-W coatings was evaluated by employing potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) techniques. A standard three electrode cell system was used to do the corrosion experiments. Ni-W alloy coated steel, with an exposed area of 0.2 cm<sup>2</sup> was used as working electrode. A

**Table 1**Basic bath composition and operating conditions.

Constituents present in the bath	Concentration (M)	Operating conditions
NiSO <sub>4</sub> ·6H <sub>2</sub> O	0.1	Temperature $60 \pm 1$ °C
$Na_2WO_4 \cdot 2H_2O$	0.2	Current density 5 Adm <sup>-2</sup>
$C_6H_5Na_3O_7 \cdot 2H_2O$	0.5	pH 8
NaCl	0.1	Deposition time 15 min
NH <sub>4</sub> Cl	0.5	Anode- platinized titanium
vanillin	0-500 ppm	Cathode- mild steel

saturated calomel electrode (SCE) and platinum foil with large area were used as reference electrode and counter electrode respectively. Electrochemical corrosion studies were conducted at room temperature in  $0.2~\mathrm{M}~\mathrm{H}_2\mathrm{SO}_4$  solution, which serves a corrosive electrolyte. A computer controlled poteniostat/galvanostat CHI 660C (USA) was employed to perform the corrosion studies. Impendence measurements were done at their respective open circuit potential (OCP) in the frequency ranging from 1 Hz to 100 KHz, where the sinuous potential amplitude was 5 mV. Simulation and acquisition of electrochemical data were attained using the software Z-view of version 3.0. Impedance spectra were represented in the form of Nyquist plots. After attaining the steady state open circuit potential (OCP), polarization curves were recorded, with a potential window of  $\pm$  200 mV from OCP with 10 mV/s scanning rate.

The PE was calculated from the corrosion current values using the following equation:

$$P.E(\%) = \frac{i_{corr}^{0} - i_{corr}^{i}}{i_{corr}^{i}} x \, 100\%$$
 (1)

where i<sup>0</sup>corr and i<sup>i</sup>corr denote the corrosion current densities of Ni–W coatings obtained from the baths without and with additive, respectively.

PE was calculated from the charge transfer resistance by the following equation:

$$P.E(\%) = \frac{Rct^{i} - Rct^{0}}{Rct^{i}} x 100\%$$
 (2)

where Rct<sup>i</sup> and Rct<sup>0</sup> denote the charge-transfer resistance of Ni–W coatings deposited in the presence and absence of vanillin in the bath, respectively.

#### 2.2. Physical characterization

The Ni–W deposit which is having high corrosion rate is subjected to the further studies, in comparison with the as such deposits. X-ray diffraction (XRD) patterns of the Ni–W alloy electrodeposits were recorded in the  $2\theta$  range from  $10^\circ$  to  $90^\circ$  using Cu/K $\alpha$  radiation by an X-ray diffractometer Model, Shimadzu XRD 6000 (Japan) system.

The crystallite size of the deposits from the obtained XRD results was calculated using the scherrer equation,

$$d = (0.9\lambda)/(\beta \cdot cos\theta) \tag{3}$$

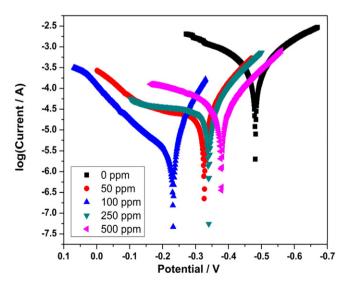


Fig. 1. Tafel plots of Ni-W alloy coatings in  $0.2~M~H_2SO_4$  medium. The electrodeposits obtained in absence and in presence of different concentrations of vanillin in the bath solution.

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