RESEARCH PAPER

Synthesis and Characterizations of Ni-W Alloy

Dipali Potdar[1,2], Sushant Patil[2], Yugen Kulkarni[1,3], Niketa Pawar[1,3], Shivaji Sadale[2], Prashant Chikode[1,*]

* *prashantchikode@gmail.com*

[1] Department of Physics, Jaysingpur College, Jaysingpur-416101, India
[2] Department of Technology, Shivaji University, Kolhapur-416004, India
[3] Department of Physics, Willingdon College, Sangli, 416415, India

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Abstract: *The Nickel tungsten (Ni-W) alloy was electrodeposited on stainless steel (SS) substrate using potentiostatic mode at room temperature. Potentiostatic electrodeposition was carried out by varying the deposition time. The physicochemical properties of Ni-W alloys were studied using X-Ray diffraction (XRD), Electron Microscopy and micro-Raman spectroscopy. Recorded XRD spectra was compared with standard JCPDS card and the presence of Ni was confirmed, no such peaks for W were observed. Further study was extended for micro-Raman analysis. From Raman spectroscopy study the appearance of Ni-O and W^{6+}=O bonds confirms that the Ni-W present in amorphous phase. Several cracks were observed in SEM images along with nanoparticles distributed over the electrode surface. The appearance of cracks may be correlated with the in-plane tensile stresses, lattice strains and stacking faults and maybe related to the substrate confinements.*

Keywords: *Ni-W, Alloy, Electrodeposition, Amorphous, Raman Spectroscopy.*

1. INTRODUCTION

Since last decades a variety of nanostructured materials were explored by many researchers owing to their unique physicochemical properties as compared to bulky structures [(1)-4]. Tungsten alloys along with iron group materials are having peculiar magnetic, electrical, mechanical, thermal and corrosion resistive properties [5–9]. Nickel Alloys composed with tungsten, iron, cobalt and/or phosphorous are explored in the diverse field of micro electromechanical systems. Electrodeposition of compositional Ni based alloys may improve the mechanical features which may facilitates their utilization in various applications.

Nickel and tungsten (Ni-W) alloys have noticeable features such as corrosion resistance, anisotropic magnetic properties, ductility and hardness [5, 10-20]. The composition of second transition group of elements may improve the strength of Ni based alloys, however, in certain cases they may found to be brittle or along with internal stress [21]. Several efforts were escalated towards the electrodeposition of Ni-W based alloys. The precursors of Ni & W both are having different physicochemical characteristics and may require different preparative conditions for the same. Electrodeposition of Ni-W based alloys are intriguing and optimizing their preparative parameters is vital; furthermore, utilization of complexing agents, adjustments of pH values, precursor concentrations may lead to the formation of pure Ni-W alloys.

Numerous synthesis routes are explored for the deposition of Ni-W alloys such vapor deposition, molecular beam epitaxy, magnetron sputtering [22], electrodeposition [23] etc. however, electrodeposition stands to be one of the most prominent methods as compared to others as they may require highly complex apparatus, vacuum etc. In electrodeposition, one can tune the preparative parameters for further modifications in the structural and physical properties of the nanostructured materials. By tunning the surface chemistry and topography of the materials one can improvise the corrosion resistance and wetting properties, mechanical properties and control the bulk compositions. Hence, electrodeposition stands to be most prominent method for the production of alloy coatings with their distinctive properties. Some of the limitations such as intricated chemistry, temperature, and electrode configuration can be hindered by utilizing the proper complexing agents and other preparative parameters which may improvise the quality of deposits and solvent stability [24-26]. Atanassov *et. al.* [27] have

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electrodeposited Ni-W alloy in the sulfamate electrolyte. Schuh *et. al.* [28] observed that nanostructured Ni has poor abrasion resistance than Ni-W alloys. Siraman *et. al.* [29] studied the friction and wear behavior of Ni-W alloy at various current densities. Yamasaki *et. al.* [30] electrodeposited the amorphous Ni-W and reveals that, it exhibits various properties viz. good thermal stability, high hardness, good corrosion resistance etc.

In this present research, we have fabricated Ni-W alloys by electrodeposition method using the tri-sodium citrate as a complexing agent for improvising the stability of the electrolytic bath. The electrodeposition time was varied accordingly and the deposited films were characterized by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and micro-Raman Spectroscopy. Further research is engaged in the analysis of these alloys for their magnetic properties.

2. EXPERIMENTAL DETAILS

2.1. Electrodeposition of Ni-W Films

All the chemicals were purchased by Thomas baker Pvt. Ltd. and are of analytical grade and are used without further purification. Electrodeposition was carried out at room temperature in a two-electrode configuration wherein Stainless-steel (SS) substrate (1 cm × 5 cm) having sheet resistance of 0.6 Ω/sq. was used as working and counter electrode. The precursor solution was prepared by using $NiSO_4.6H_2O$ as a source of Ni, $Na_3C_6H_5O_7.2H_2O$ as a complexing agent and $Na_2O_4W.2H_2O$ as a source of W in 40 ml double distilled water (DDW) having the pH of solution at 8.0. Potentiostatic electrodeposition was performed using DC power supply at room temperature. Prior to this, the stainless-steel substrates were polished with emery paper of 2000 grade. Further, they were cleaned ultrasonically for 10 minutes each using DDW, acetone, ethanol respectively. Electrodeposition was performed on SS electrode having 2 cm2of area of the electrode. After deposition, substrates were rinsed using DDW and dried in air. Deposition time (T_d) was varied for 60, 120 and 180 minutes each. The schematic of electrodeposition is depicted in Figure 1.

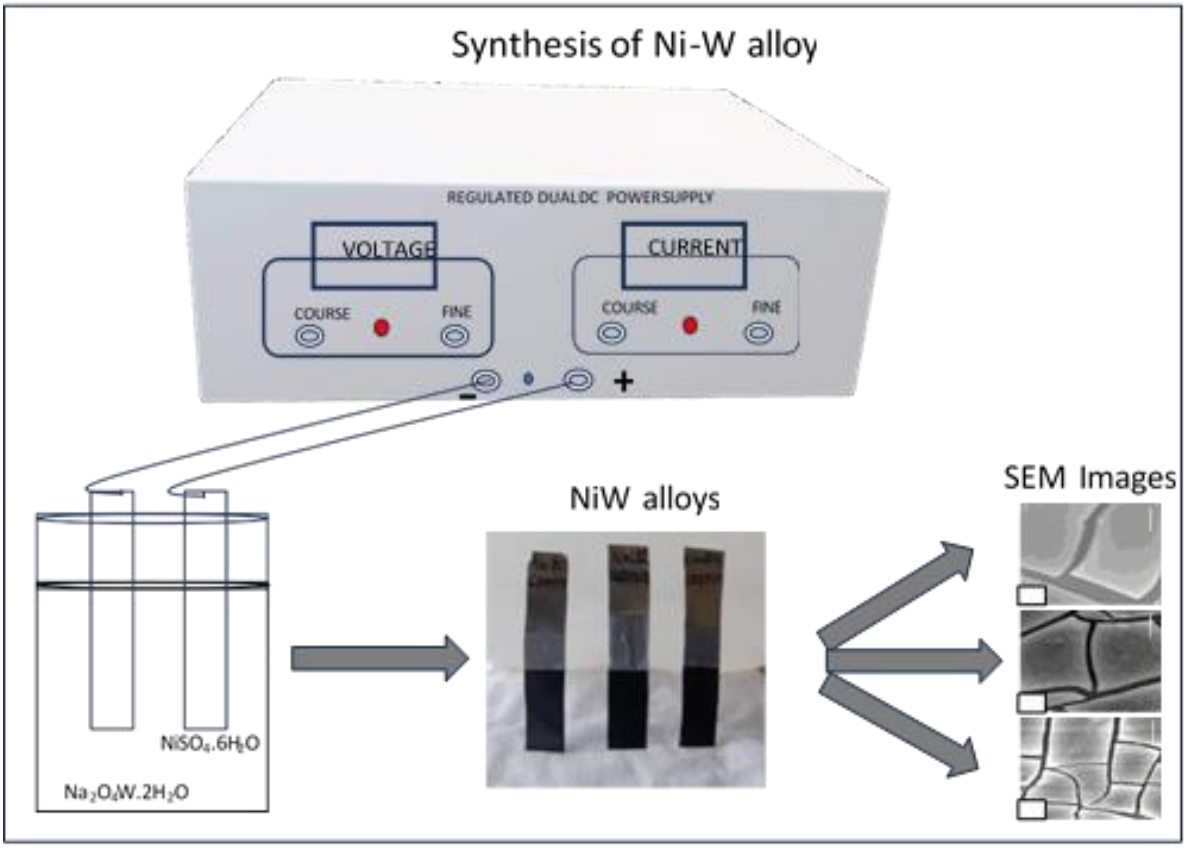

Fig. 1. Schematic Representation of Ni-W Electrodeposition.

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2.2. Characterization

The NiW alloys were analyzed for their structural studies using the X-Ray Diffractometer (Bruker AXS D8 Advance) with Cu-Kα radiation (λ= 1.54 A0). Surface morphology was analyzed using Scanning Electron Microscopy (JEOL JSM-6360) and the chemical states were inspected using the micro-Raman Spectroscopy (Renishaw INVIA0120-02, Renishaw).

3. RESULT AND DISCUSSION

3.1. Structural Characterization

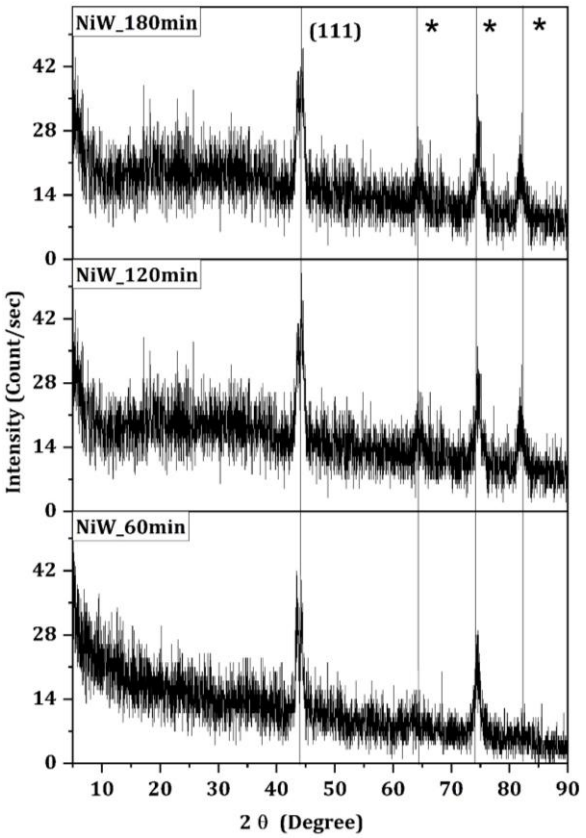

Fig. 2. XRD pattern of Ni-W electrodes.

Figure 2 shows the XRD pattern of Ni-W films deposited on the SS substrate. The diffraction peaks were compared with the JCPDS data and the diffraction peak having 2Θ value at 44.50° is well aligned with the dhkl (111) crystal plane of Ni with cubic structure (JSPDS no. 01-087-0712) which confirmed the presence of nickel (Ni) on SS substrate. No such characteristic diffraction peaks for tungsten (W) were observed in the XRD spectra which maybe correlated with the amorphous phase of the W. Further, the diffraction peaks marked with '*' are corresponds to the SS substrate. No further peaks were observed in the XRD pattern which confirms that the deposited Ni-W films formed and W may be present in amorphous phase. With the increasing deposition time, the intensity of d_{hkl} (111) plane has been improved which maybe corelated with the improved crystallite size. Further structural analysis of the XRD pattern were made by calculating the parameters such as interplanar spacing (d), lattice constant (a), crystallite size (D), dislocation density (δ), lattice strain (ε) and texture coefficient ($TC_{(hkl)}$), and stacking faults (SF).

Interplanar spacing: $d_{(hkl)} = \frac{\lambda}{2\sin\theta}$ (1)

Lattice constant: $a = d_{(hkl)} \times \sqrt{(h^2 + k^2 + l^2)}$ (2)

Debye–Scherrer formula: $D = \frac{K\lambda}{\beta\cos\theta}$ (3)

Dislocation density: $\delta = \frac{1}{n^2}$ (4)

Lattice strain: $\varepsilon = \frac{\beta}{4\tan\theta}$ (5)

Texture Coefficient: $TC_{(hkl)} \frac{\frac{I_{(hkl)}}{I_0}}{\frac{1}{N}\sum\frac{I_{(hkl)}}{I_0}}$ (6)

Stacking Faults: $SF = \left\{\frac{2\pi^2}{45\times(3\tan\theta)^{1/2}}\right\} \times \beta$ (7)

The structural parameters of the XRD pattern were calculated with respect to d_{hkl} (111) plane and the values of the same are summarized in figure 3. and table 1. It is observed that the values of *d*, *a* and *D* are decreased with respect to the increased deposition time, moreover, the values of *δ*, *ε*, $TC_{(hkl)}$, *SF* are increased. The decreased crystallite size along with other parameters such as interplanar spacing, lattice constant which may be due to the increased lattice strain during the deposition of the film which confines the grain growth. The increasing texture coefficient with respect to increasing T_d is related to the kinetic processes involved during the development of texture. As the electrodeposition process continues, the electrolytic atoms having ample kinetic energy are directed towards the charged electrode surfaces which favors the growth of Ni-W alloys and resulting into the improved texture coefficient. Furthermore, the increase of SF along with the increasing T_d indicated the availability of the planar defects and the presence of dislocation density maybe correlated with the structural differences between the electrode surfaces and the deposition layer.

3.2. Morphological Characterization

Figure 4. Illustrates the SEM Images of as

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deposited Ni-W alloy for different time period. The deposited Ni-W coated alloys show cracks on all deposits along with growth of the nanostructured particles over the surface. The observed cracks are due to the trapping of hydrogen over the surface during electrodeposition; as the tensile stress increases hydrogen ions are forced to release from the surface which resulted in to formation of the crack over the electrode surface.
Most of the amorphous Ni-W alloys have reported the similar cracks during the deposition [31-32]; furthermore, the cracks in amorphous Ni-W alloys are correlated with the in-plane tensile stresses, lattice strains and stacking faults and maybe related to the substrate confinements.

3.3. Micro-Raman Analysis

Figure 5. represents the micro-Raman spectra of electrodeposited Ni-W thin films for 60, 120 and 180 min. The micro-Raman spectra was recorded in the spectral range of 200 cm^{-1} to 1200 cm^{-1} using LASER excitation of 532 nm with 5% (2.5 W) LASER power intensity at room temperature. All the three samples have depicted a broad peak in the range of 480 ~ 560 cm^{-1} which can be assigned to the first order phonon mode vibrations of 1P LO modes and Ni- O stretching modes of Ni. Furthermore, the well-defined intense peak at ~955 cm^{-1} can be attributed towards the stretching of W^{6+}=O bond which confirms its amorphous phase. The appearance of 1P LO, Ni-O and W^{6+}=O bonds confirms the presence of Ni-W.

4. CONCLUSION:

The Ni-W alloys were electrodeposited on stainless steel substrate. The Potentiostatic electrodeposition was performed using complexing agent i.e., Tri-sodium citrate. The effect of deposition time was studied. X-ray diffraction confirms the presence of Ni along with presence of W in amorphous phase. The SEM images shows the microcracks along with growth of nanoparticles over the electrode surface.

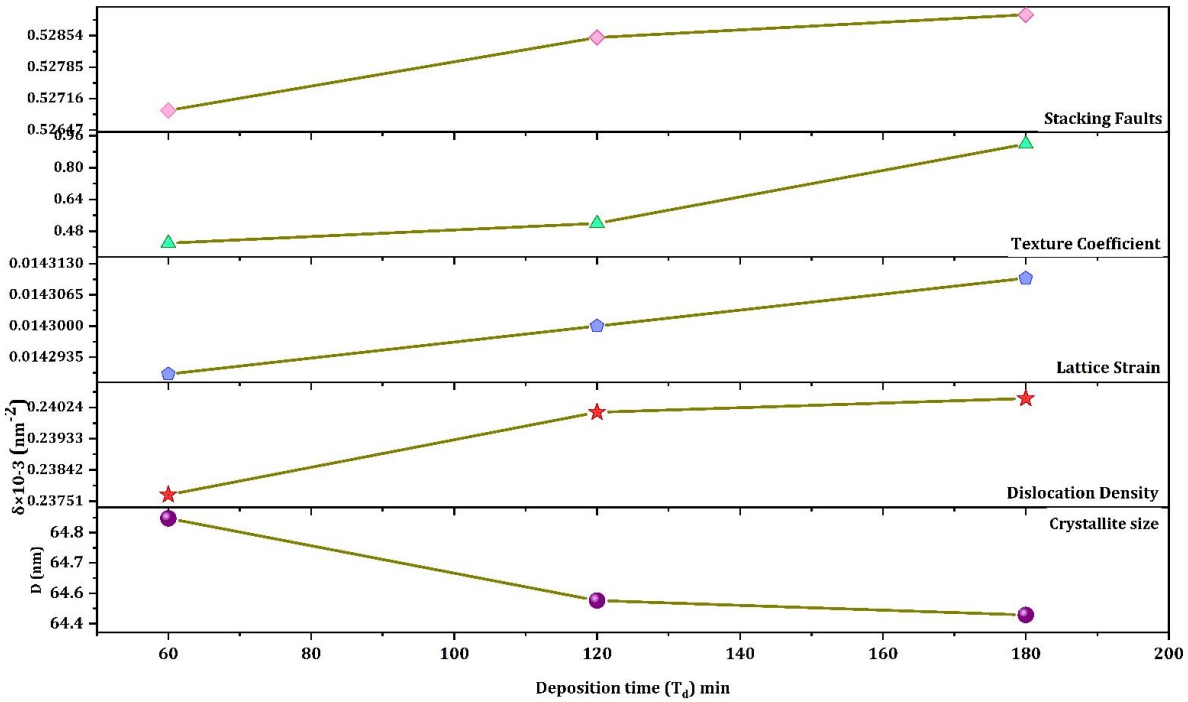

Fig. 3. Various structural parameters of XRD with respect to (111) plane.

Table 1. The Structural parameter of Ni-W alloys with respect to (111) plane

Sr. No.	T_d (min)	Interplanar Spacing (d)	Lattice Constant (a) (A°)	D (nm)	Dislocation Density δ	Lattice strain (ε)	Texture Coefficient {TC(hkl)}	Stacking Fault (SF)
1	60	2.0572	3.5632	64.8566	0.2377	0.01429	0.4198	0.5269
2	120	2.0528	3.5557	64.5316	0.2401	0.01430	0.5194	0.5285
3	180	2.0531	3.5561	64.4748	0.2405	0.01431	0.9194	0.5290

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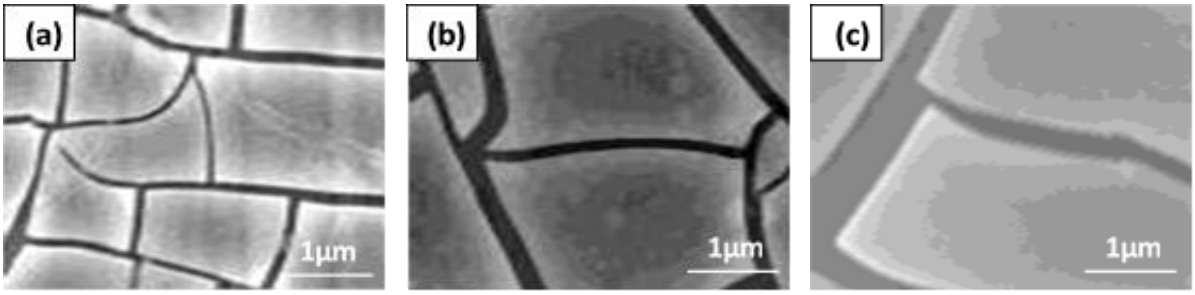

Fig. 4. SEM Images of Ni-W for (a) 60 min, (b) 120 min, (c) 180 min.

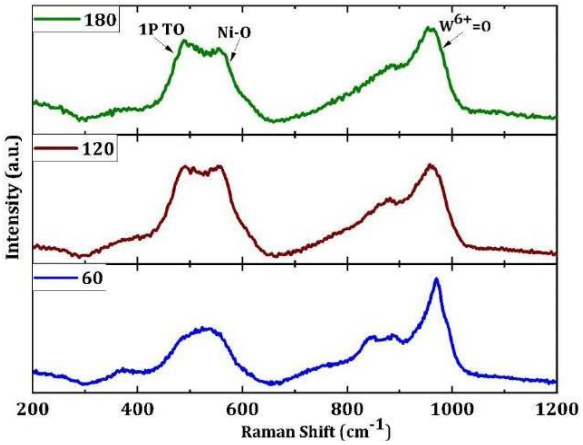

Fig. 5. Micro-Raman Spectra of Ni-W thin films deposited at 60, 120 and 180 minutes.

The observed cracks in the alloys are correlated with formation of the tensile stress during the deposition. From micro-Raman spectroscopy study, the appearance of 1P LO, Ni-O and W^{6+}=O bonds confirms the presence of Ni-W. Further study is engaged in the investigation of the magnetic properties of Ni-W alloys.

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