

Improved mechanical and corrosion properties of nickel composite coatings by incorporation of layered silicates



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HIGHLIGHTS

- 0.05–2% of layered silicates are incorporated into crystalline nickel films.
- Resulting composite films had improved stability and adhesion.
- Corrosion resistance improved for the composite films.
- Hardness improved 20% and young's modulus improved 25% for the composite films.

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ABSTRACT

Layered silicates as exfoliated montmorillonite are incorporated into nickel films by electrodeposition, enhancing both corrosion resistance and hardness. Films were deposited onto stainless steel from a plating solution adjusted to pH 9 containing nickel sulfate, sodium citrate, and various concentrations of exfoliated montmorillonite. The presence of the incorporated layered silicate was confirmed by scanning electron microscopy and energy-dispersive X-ray spectroscopy. The composite films were also compact and smooth like the pure nickel films deposited under the same conditions as shown by scanning electron microscopy. X-ray diffraction results showed that incorporation of layered silicates into the film do not affect the nickel crystalline fcc structure. The nanocomposite films exhibited improved stability and adhesion. Pure nickel films cracked and peeled from the substrate when immersed in 3.5% NaCl solution within 5 days, while the nanocomposite films remained attached even after 25 days. The corrosion resistance of the nickel nanocomposites was also improved compared to nickel films. Nickel-layered silicate composites showed a 25% increase in Young's modulus and a 20% increase in hardness over pure nickel films.

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1. Introduction

In the field of corrosion, there is a constant demand for increase performance at a reduced cost. Harsh environmental conditions eventually degrade protective coatings and lead to many types of corrosion. Nickel is used for corrosion protection due to its abundance, environmentally friendly nature, and ability to resist general corrosion [1–4]. However, poor mechanical properties for the metal films lead to corrosion resistance failure. One way to increase the corrosion resistance of nickel coatings is to form alloys with other metals, such as zinc [5]. Alloy films help improve corrosion

resistance of the coating but typically still have similar mechanical properties as the individual metals. Enhancing the mechanical properties of films can be done by ceramic reinforcement into the metal coating. Nanoparticles such as of SiC, Al₂O₃, TiO₂, and CeO₂ have been incorporated into a nickel metal matrix composite coating to enhance the lifetime and hardness of the coatings [6–15].

Layered silicate nanoclays are a type of ceramic material which have several beneficial properties including high resistance to chemicals, pH, and temperature extremes as well as a high surface area. These layered silicates have been used to reinforce several kinds of materials such as polymers [16–20] and ceramics [21,22] in order to improve mechanical properties of the composite coatings. Researchers have shown that layered silicates enhance corrosion resistance in conductive polymer composite coatings [23,24]. Using

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layered silicates in metals should prove beneficial to increase corrosion resistance as well as hardness of the resulting nano-composite films.

Electrodeposition is one of the more popular techniques to synthesize film coatings because of its convenience, low-cost and use of low-temperatures [25]. Composition, morphology, and structure can also be influenced by the applied potential or current, temperature and pH of the depositing solution [5,25,26]. In this work, nickel-layered silicate nanocomposite films were electrochemically deposited using pulsed potentiostatic conditions from an alkaline plating bath containing exfoliated clay nanoparticles. Alkaline electrodeposition gives a more uniform deposit for some nickel alloys, which results in better corrosion protection of the underlying metal [5]. Scanning electron microscopy (SEM) was used to determine the morphology of the electrodeposited films. X-ray diffraction (XRD) was used to confirm the crystal structure of the films. The corrosion resistance and hardness of the nano-composite film were compared with that of the pure nickel deposits using Tafel polarization, immersion tests, electrochemical impedance spectroscopy and nanoindentation.

2. Experimental

2.1. Materials

All of the solutions were prepared from analytical-grade chemicals dissolved in deionized water. Nickel and nickel-layered silicate films were electrodeposited from a citrate bath at ambient temperature. The plating bath contained 0.1 M $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ (Alfa Aesar) and 0.1 M $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ (Fisher Scientific Company) adjusted to pH 9 with 1 M ammonium hydroxide. Montmorillonite concentrations ranging from 0.05 to 5.0% were vigorously stirred for 24 h by a mechanical stirrer to exfoliate the clay into the individual clay platelets (~ 1 nm thick) prior to introduction into the plating solution. The layered silicate solution was used as the makeup solution instead of deionized water for the plating bath. The working electrode was polished mechanically with 600, 800, and 1000 grit silicon carbide paper, then with 3 and 1 μm diamond solution on felt cloth. After the electrode was polished to a mirror-like finish, it was sonicated in deionized water for 10 min. The electrochemical cell was comprised of three electrodes; chromel wire (counter electrode), saturated calomel (reference electrode) and stainless steel (working electrode). A schematic of the deposition cell is shown in Fig. 1.

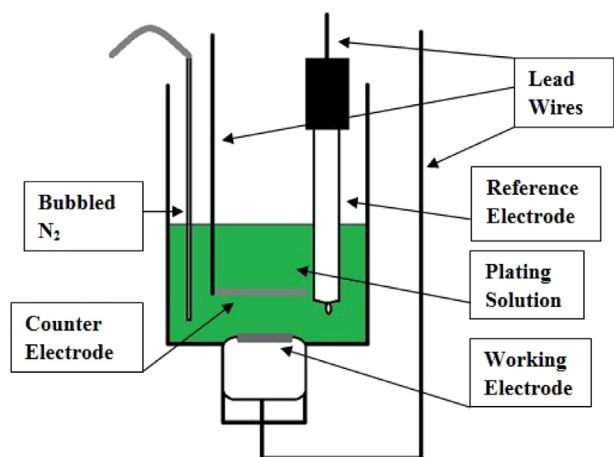


Fig. 1. A schematic of the electrochemical deposition cell used for the deposition of the coatings.

2.2. Electrodeposition and solution studies

An EG&G PAR Potentiostat/Galvanostat Model 273A was used for all depositions, cyclic voltammetry, and corrosion studies. The stainless steel surface was pre-coated with a copper thin film at -0.6 V for 1–2 coulombs to improve the adhesion of the nickel deposit. The Cu pre-coated bath contained 0.01 M $\text{CuSO}_4 \cdot 2\text{H}_2\text{O}$ (Fisher Scientific Company) and 0.01 M $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$. Pulsed electrodeposition for the nickel and nickel-layered silicate films was carried out using a potential of -1.1 V for 10 s then a step to -0.7 V for 3 s until a total charge of 30 C, which has been shown to refine the crystalline size and uniformity [5].

The zeta potential of various solutions containing exfoliated montmorillonite was measured using a Delsa Nano-HC (Beckman Instruments). Montmorillonite is known to have a negative charge on its surface. The size of the particles in the solution was determined by photon correlation spectroscopy using the Delsa Nano-HC.

2.3. Characterization techniques

The surface morphology of the films was characterized with an Environmental FEI Quanta 200 scanning electron microscope (SEM) with an ETD detector. Energy-dispersive X-ray spectroscopy (EDX) was used to determine the presence of the layered silicate platelets in the film by monitoring the Al, Si, and Mg signals. Three random points on the film surface were chosen for analysis then an average value was calculated using the approximate layered silicate formula of $(\text{Na}, \text{Ca}) (\text{Al}_{1.66}\text{Mg}_{0.33})_3 (\text{Si}_4\text{O}_{10})_3 (\text{OH})_6 \cdot n(\text{H}_2\text{O})$ [27]. X-ray diffraction data was obtained on a Siemens D-500 Diffractometer using $\text{Cu K}\alpha$ radiation ($\lambda = 0.1541$ nm) at 35 kV and 24 mA. The scans were run from 2 to $100^\circ 2\theta$ at a step size of 0.05° and dwell time of 1 s.

Nanoindentation experiments were performed with a MTS nanoindenter XP following the constant stiffness method standard hardness/modulus with a Berkovich tip. A combination of a minus k vibration isolation table and a thermal sound vibration isolation cabinet were utilized in order to provide environmental isolation. Prior to testing, the indenter system was calibrated on a sample of fused silica with 25 indents. The specimen was mounted on an aluminum stub, this was fixed to the sample holder stage and with the help of a microscope the indentation spots were identified. 25 indentations were done in square array with a gap of 20 microns between each indent. The percent unload in the stiffness calculation was kept to 50% and allowable drift correction was kept at 0.05 nm s^{-1} with a drift correction of 1. The depth limit was varied from 200, 300, 400 and 1200 nm. Unloading percent was 90 and the strain target was 0.05 s^{-1} . Hardness and indentation modulus were calculated using the Oliver–Pharr method which assumes isotropic material behavior [28]. The elastic properties of the diamond indenter were: $\nu_i = 0.07$ and $E_i = 1140$ GPa.

2.4. Corrosion measurements

Corrosion behavior was studied by electrochemical techniques. A four-electrode electrochemical cell used as a corrosion testing system was composed of the working electrode of the composite film, two graphite rods, and a saturated calomel electrode (SCE). Potentiodynamic polarization measurements were run in a 3.5% sodium chloride solution. A ± 100 mV potential range was applied to the cell without stirring the solution, the resulting current density was then measured. In each scan, the run started at open circuit potential E_{ocp} with a scan rate of 1 mV s^{-1} . The E_{corr} and i_{corr} were determined from the intercepts by Tafel's extrapolation

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