

INVESTIGATION OF MECHANICAL PROPERTIES OF ELECTRODEPOSITED NICKEL-TITANIUM OXIDE-FUNCTIONAL GRAPHENE OXIDE COMPOSITE COATING

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Abstract: In this study, the nanocomposites of Ni-TiO₂-Functional Graphene Oxide were coated on mild steel by the method of electrodeposition. Both the TiO₂ and Functional Graphene Oxide particles exhibit enhanced mechanical properties with superior bonding properties. Nickel is appended to the synergetic combination of TiO₂-Functional Graphene Oxide particles to achieve better corrosion and mechanical properties. The influence of particles on coating surface properties and mechanical properties is examined. Characterization of electrodeposited coatings is accomplished with X-ray diffraction (XRD), scanning electron microscopy (SEM), atomic force microscopy (AFM) and Vickers's hardness tester. Addition of TiO₂-Functional Graphene Oxide particles to the coating improved corrosion and mechanical properties of Nickel. Surface morphological analysis at room temperature reveals the presence of granular structure with finely filled microholes and crevices. The Ni-TiO₂-Functional Graphene Oxide (1g/l) exhibited an excellent barrier against corrosion with a 49 % reduction in corrosion rate. Minimal exfoliation of the coating material was observed without any adhesion cracks for an applied load up to 20 N.

Keywords: Nanocomposites, Protective coating, Electrodeposition, Surface morphology, Titanium dioxide, Corrosion, FGO

INTRODUCTION

In recent years, nanomaterials have attracted the interest of scientists from a variety of various fields of science, including hybrid nanostructures and 2D nanomaterials. Hybrid nanostructures consisting of metal nanoparticles (NPs) assembled on semiconductors are being investigated for use in a wide range of technologies, including optics, catalysis, sensors, and biotechnology [1]. These characteristics are a result of their complex architecture and the presence of metal nanoparticles with surface plasmon resonance properties (SPR). The presence of metal nanoparticles decreases the TiO₂ bandgap energy and increase the separation of charge between the agitated electron (e⁻) and hole (h⁺), resulting in a stronger interaction between metal nanoparticles and titania, and an increase in TiO₂ photocatalytic activity [2]. As a pioneering 2D nanomaterial, graphene has been widely applied in biological applications due to its ultrathin structure, good conductivity, and large surface area. One of the most researched 2D nanomaterials is Titanium Carbide (TiC), which has a huge surface area, strong metallic conductivity, good biocompatibility and is simple to functionalize. MXenes have an assortment of applications, including biosensors, wearable electronics, electromagnetic interference shielding, water purification and energy storage devices [3, 4].

Metallic coatings such as Cu, Ni, and Zn, along with alloy coatings such as Ni-Co, Zn-Ni and others, have gained prominence. Protective layers containing nanoparticles can significantly improve corrosion resistance [5, 6]. The protective coatings have increased hardness, lubrication, and photocatalytic characteristics, as well as better corrosion resistance [7]. Ni-based composite coating has proven to be an important coating because of its superior hardness, lubrication, wear, and corrosion resistance qualities [8]. Thermal spray, electrochemical deposition, molecular mixing, sputtering, vapour deposition, plasma spray coating, plasma induction polymeric coating, laser cladding, and electrodeposition coating are some of the coating techniques available [9]. Electroplating is one of the most efficient ways of producing metal coatings, and it has been widely employed in industries owing to its easy and steady procedure [10]. The permutations and combinations of nanoparticles in the metal matrix can be used to tailor the coating properties [11]. Various nanoparticles like (SiO₂) [12], (TiO₂) [13, 14], (CeO₂) [15], (Al₂O₃) [16, 17-18], ZrO₂ [19], MoS₂ [20], SrSO [21], Gr [22] carbides (SiC,

B4C) [23-24] , nitrides (Si_3N_4) [25, 26], polymers (PTFE) [27,28], and even metals (Cr, Co) [29-31] have been used in combination with Ni to enhance the corrosion and mechanical characteristics.

Titanium dioxide (TiO_2), the second most commonly used oxide after Al_2O_3 and ZrO_2 , has captivating properties resembling the ideal semiconductor. Considering its high stability and low cost, it is used in a wide variety of applications, such as anticorrosive coatings, self-cleaning coatings, optical devices, solar cells, sensors, environmental detoxification, electrolysis of water, photocatalysis, pesticide devaluation, hydrogen generation as an energy source, etc. In the protective layer, nanoparticles improve corrosion resistance significantly [32-36]. There are numerous applications of Ni- TiO_2 coatings, including automotive components, printed circuit boards, electrical connectors, awards, soft metal gaskets, decorative door, light, and bathroom accessories, aerospace components, artificial joints, marine structures, agricultural equipment, nuclear equipment [34-38]. The functional groups in the FGO can bind to the substrate and the coating material, increasing surface areas of contact by strengthening the bond between the coating and substrate, the coating becomes more durable and resistant to environmental factors [39]. GO nanosheets can be functionalized with organic and inorganic materials. GO-based fillers have received a lot of attention in this regard because they achieve a higher degree of nanofiller dispersion because of the presence of oxygenated groups [39, 40].

EXPERIMENTAL

An electrodeposition of nanocomposite on a mild steel substrate with dimensions of $2 \times 3 \text{ cm}^2$ was conducted using a nickel bath containing calcined TiO_2 -FGO particles. The ultra-pure chemicals of analytical grade were utilized, and the electrolyte was produced prepared with distilled water. The optimized bath parameters and deposition conditions for the process are shown in Table 1.

Table .1. Bath composition and Deposition conditions

Bath Composition	Concentration, gl^{-1}
NiSO_4	300
NiCl_2	60
H_3BO_3	45
$\text{NaC}_{12}\text{H}_{25}\text{SO}_4$	4
Deposition conditions	
Current density	5 Adm^{-2}
Temperature	50°C
pH	3-4

The electrolyte was stirred by a magnetic stirrer at 300 rpm prior to electrodeposition to ensure an even distribution of the particles. During the process, mild steel is the cathode and nickel is the anode. Atouch-up was done on the test samples with emery paper grades 80, 800, 1000, and 2000. To prepare mild steel substrates for polishing, a 10 % HCl solution was infused for a few minutes, followed by acetone. Temperature was set to 50°C and current density was set to 5 A dm^{-2} . SDS ($\text{NaC}_{12}\text{H}_{25}\text{SO}_4$) was used as the additive in order to prevent pin pore formation and facilitate the growth of metallic ions [41, 42]. The duration of each trial experiment was determined to be 20 mins. The electrodeposition experiments were conducted using a range of TiO_2 -FGO concentrations, specifically 0.25, 0.50, 0.75 and 1 gl^{-1} .

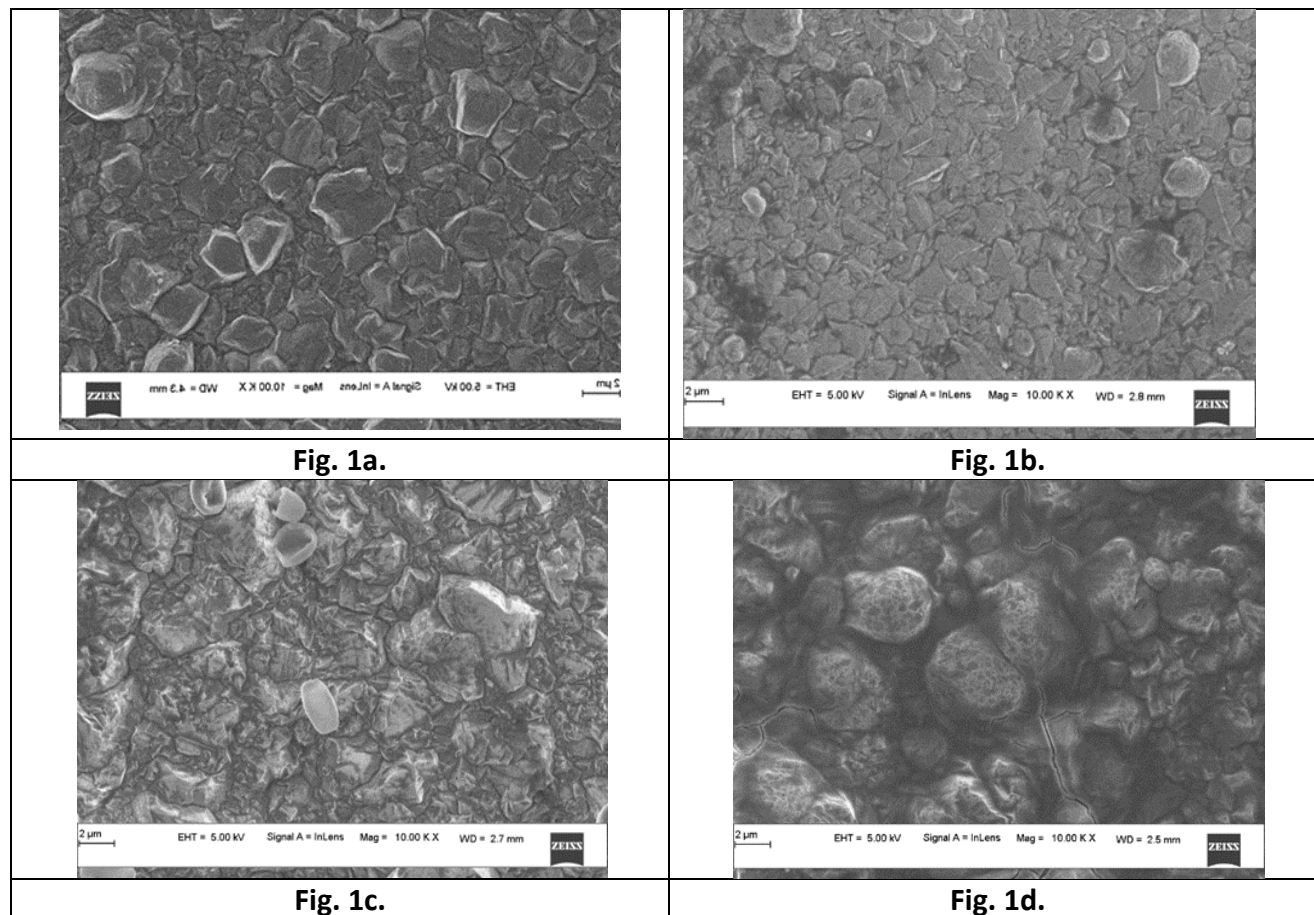
A. Characterization techniques

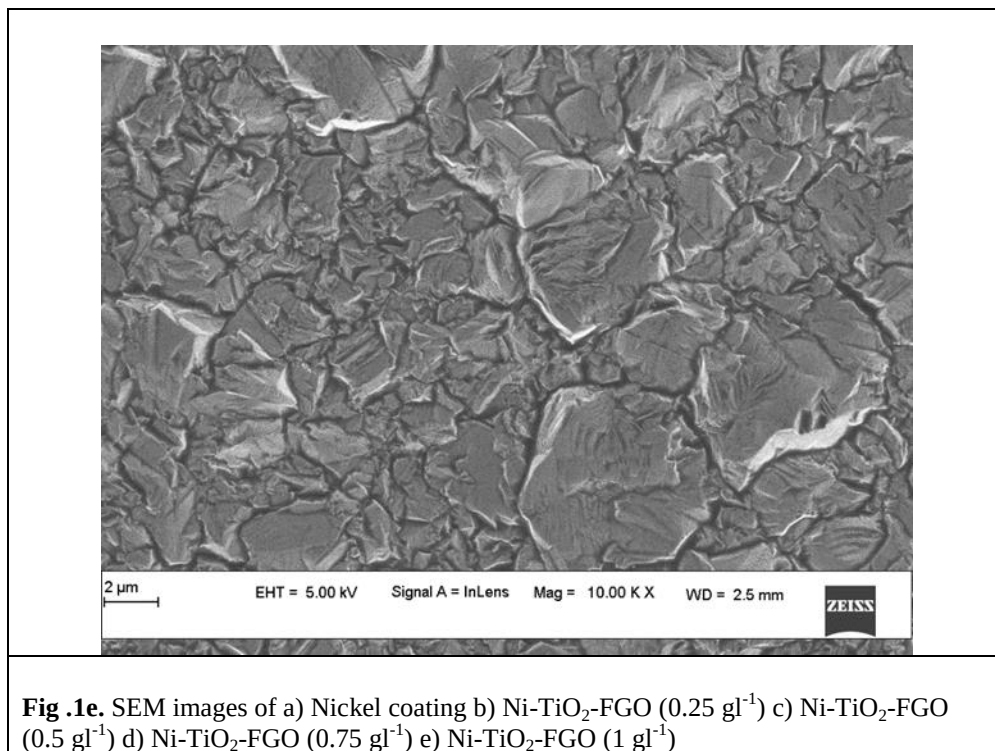
To obtain X-ray diffraction patterns, the Malvern PanalyticalEmpyren 3rd Gen is used with Cu $K\alpha$ radiation ($\text{Cu} = 0.1540598 \text{ nm}$). Surface structure was examined using a Carl Zeiss Sigma FESEM. The MMT-X7A MatsuzowaVicker's hardness tester was deployed to measure the indentation and microhardness. Images were obtained using the Innova SPM atomic force microscope. Corrosion tests were performed with a CHI 608D electrochemical workstation at 27°C . Nanonew ST 400 profilometer was used to scan coating profiles. The DUCOM unitest 750 scratch tester was used for the scratch testing. The contact angle of the coated sample was found using Drop shape Analyzer-100.

RESULTS AND DISCUSSIONS

A. Scanning Electron Microscopy

Figure 1 shows the surface topography of electrodeposited samples using SEM images. Fig. 1 displays the surface morphology of Nickel coating and Ni -TiO₂-FGO solutions of contents of 0.25 gl⁻¹, 0.50 gl⁻¹, 0.75 gl⁻¹, and 1 gl⁻¹ concentrations. A fine granular structure was observed on the surface. The gaps, crevices and microholes were all found to be reinforced with the composite coating.





As shown in Figure 2, Nickel DC deposited coatings and Ni-TiO₂-FGO DC deposited coatings exhibit similar XRD patterns. Nickel peaks with varying intensities can be seen at the (111), (200), and (220) planes when examining the deposits. The International Centre for Diffraction Data (ICDD) card numbers 01-070-1849, 01-088-2321 and 01-089-3440 were used to index the XRD peaks of nickel, TiO₂ and graphene elements, respectively. The plots clearly show a lack of Titanium and Graphene peaks since TiO₂-FGO is present in a much lower amount than nickel in the coating. A TiO₂-FGO peak is less intense than that of nickel and cannot be distinguished by X-ray diffraction [32]. An EDAX analysis of the composite coating in Fig.3 indicates the presence of Nickel, Titanium, and Graphene.

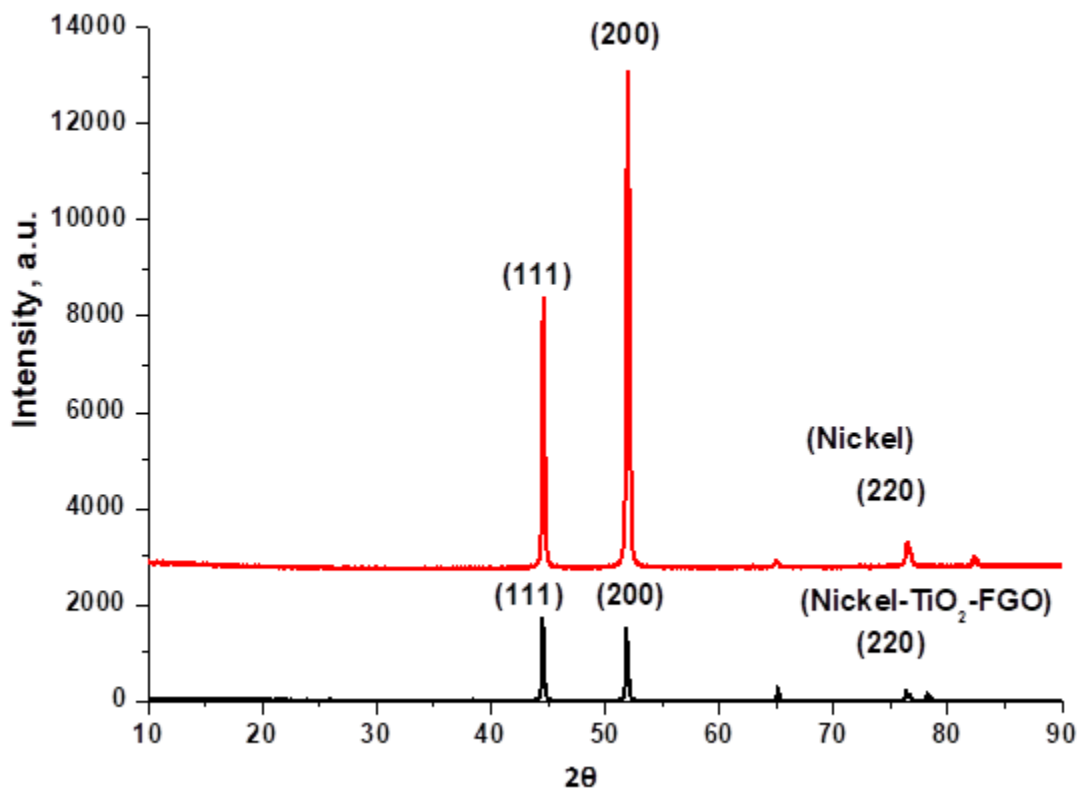


Fig .2. XRD Plots of Ni and Ni-TiO₂-FGO Coating

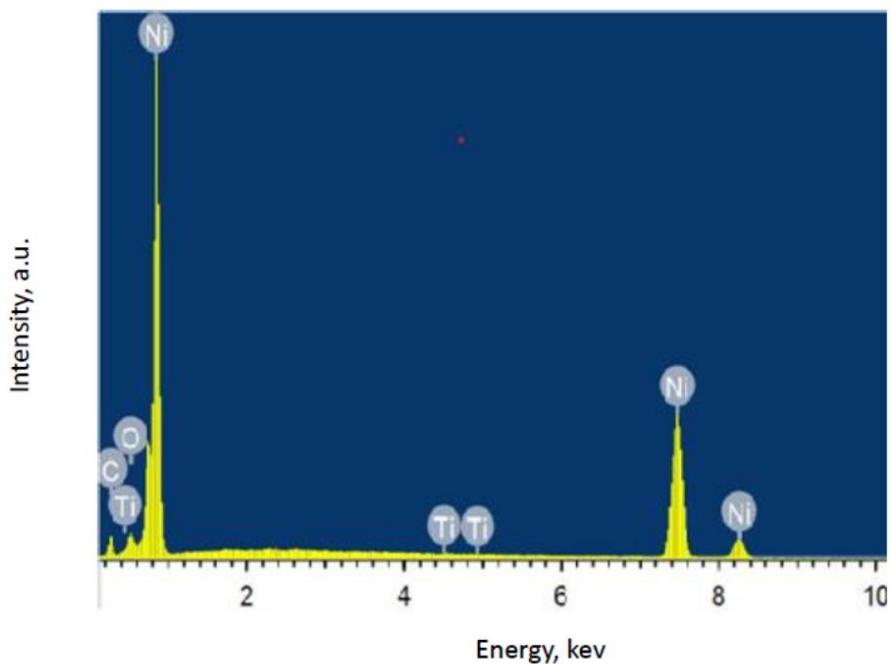


Fig .3. EDAX analysis of Ni-TiO₂-FGO Coating

B. Corrosion Testing

This study was conducted in a conventional glass container containing a corrosive solution (3.5 % NaCl). Uncoated pure Ni and Ni-TiO₂-FGO steel substrates, each of 1 cm² surface area, were the working electrodes and left to soak in the corrosive liquid for five minutes. Steel corrosion is caused by the chloride ion, one of its constituent ions. A free chloride ion can cause serious corrosion to steel bars by damaging the passive film. In most of the current research, steel bars are directly inserted into sodium chloride solutions to study their electrochemical characteristics [43]. Saturated calomel electrodes were used as a reference electrode, with platinum wire as a supplementary counter electrode. A combination of Tafel extrapolation and impedance spectroscopy was used to evaluate corrosion resistance [7].

The Tafel plot of the Ni-TiO₂-FGO coating (1 g/L) in Figure 5 shows lower I_{corr} value for the maximum applied voltage. From Table 2, addition of TiO₂-FGO particles into the solution increased the inhibition efficiency of the Ni coating, which increased from 27.41% to 76.67 % (Table 2).

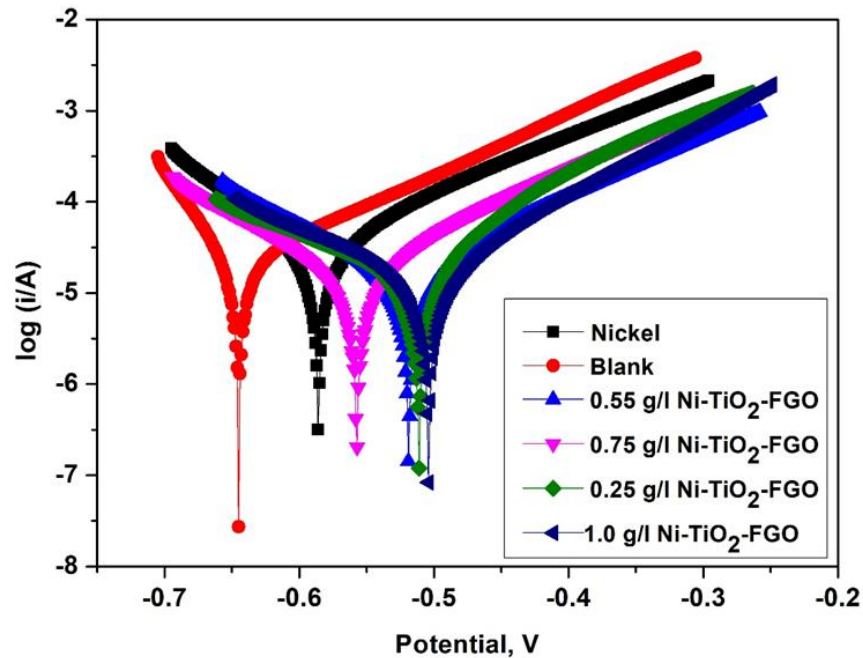


Fig .5. Polarization curves of Nickel and Ni-TiO₂-FGO coating

Table .2. Tafel extrapolation data for 3.5% NaCl corrosive media

Samples	$-E_{corr}/V$ vs. SCE	$I_{corr}/A\text{ cm}^{-2}$	Corrosion rate (mm year ⁻¹)	% Inhibitor Efficiency (η_p)
Steel	0.645	8.123	3.624	----
Nickel	0.593	5.896	3.489	27.41
Ni- TiO ₂ -FGO (0.25 g l ⁻¹)	0.578	5.105	2.796	37.15
Ni- TiO ₂ -FGO (0.50 g l ⁻¹)	0.585	4.426	2.256	45.51
Ni-TiO ₂ -FGO (0.75 g l ⁻¹)	0.550	3.002	2.005	63.04

Ni-TiO ₂ -FGO (1.00 g/l ⁻¹)	0.532	1.895	1.783	76.67
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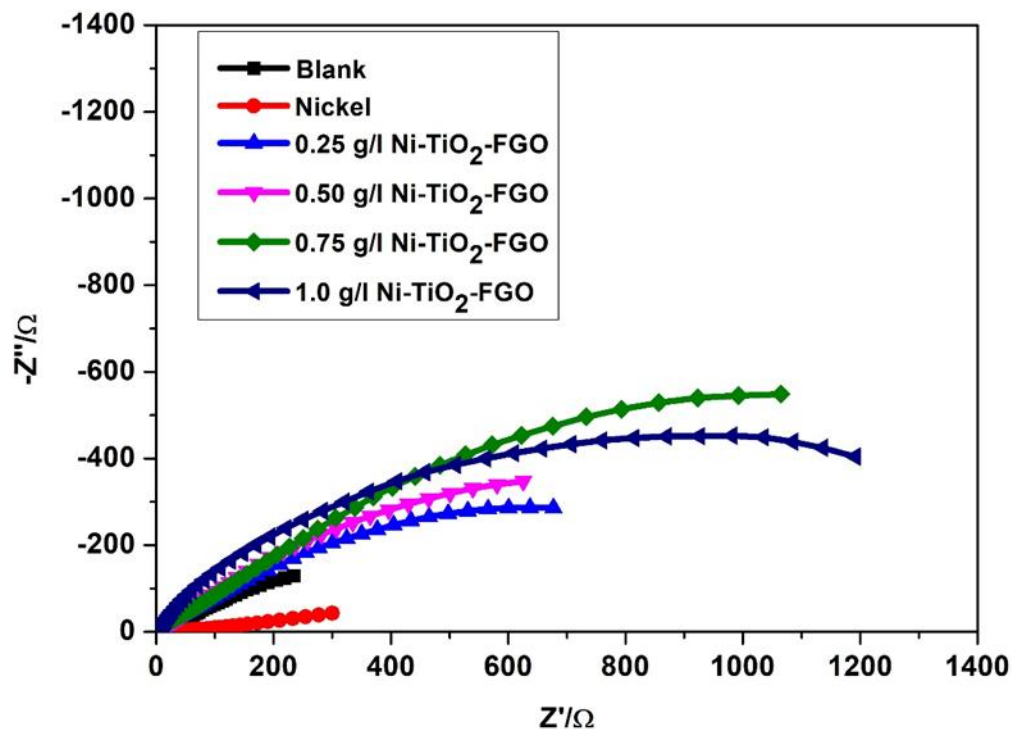
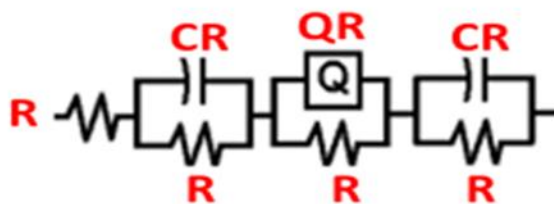


Fig .7. Nyquist plots for Nickel and Ni-TiO₂-FGO coating



R: Resistance

CR: Current Rectifier

QR: Quick Response

Table .3.Impedance Spectroscopy Data for 3.5% Nacl corrosive media

g/l	R _p (Ωcm ²)	Cdl (μF/cm ²)	%Inhibitor Efficiency (η _i)
Steel	244.3	495	----
Nickel	322.8	213	24.3

Ni- TiO ₂ -FGO (0.25 gl ⁻¹)	608.7	192	59.8
Ni- TiO ₂ -FGO (0.50 gl ⁻¹)	690.3	175	64.6
Ni-TiO ₂ -FGO (0.75 gl ⁻¹)	1024.8	150	76.1
Ni-TiO ₂ -FGO (1.00 gl ⁻¹)	1197.0	139	79.5

Figure 8 shows an electrical equivalent circuit that is fitted to the impedance parameters. Table 3 shows the related impedance data. In Figure 7, impedance diagrams of steel samples coated with Ni-TiO₂-FGO are shown. Semicircles for Steel samples are smaller than those for Ni -TiO₂-FGO coated samples. There is a maximum semicircle diameter for Ni-TiO₂-FGO (1 gl⁻¹) sample. Polarization resistance values are also increased from the steel sample to Ni-TiO₂-FGO (1 gl⁻¹) sample. Double layer capacitance values are decreased from steel to Ni-TiO₂-FGO (1 gl⁻¹) sample. According to these values, steel coated with Ni-TiO₂-FGO exhibits greater corrosion resistance than steel samples without the coating. Further, Steel coated with Ni-TiO₂-FGO (1 gl⁻¹) exhibited maximum corrosion resistance than other samples. The TiO₂-FGO have hindered the flow of current and reduced corrosion [41]. The reduction in current flow in Ni-TiO₂-FGO samples is also attributed by the adsorption of nickel ions to the anodic sites that interferes with the dissolution of nickel in the alloy coating [44, 45]. TiO₂-FGO at a higher concentration was also tried but showed similar results to TiO₂-FGO (1gl⁻¹).

C. Microhardness

For microhardness measurement, a Vicker's diamond indenter with two load segments of 50 g and 100 g and a sustain duration of 5 seconds was used. This graph (Fig. 10) shows the microhardness of nickel with and without Ni-TiO₂-FGO coating at a concentration of 1 gl⁻¹.

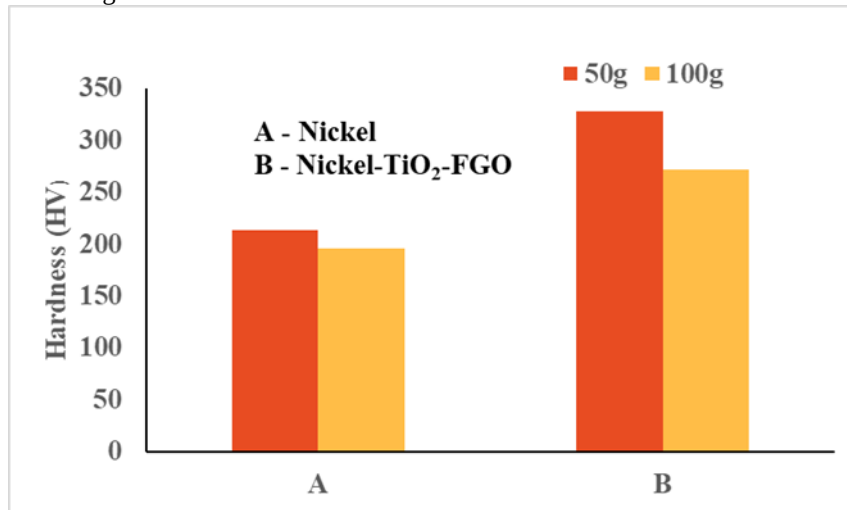
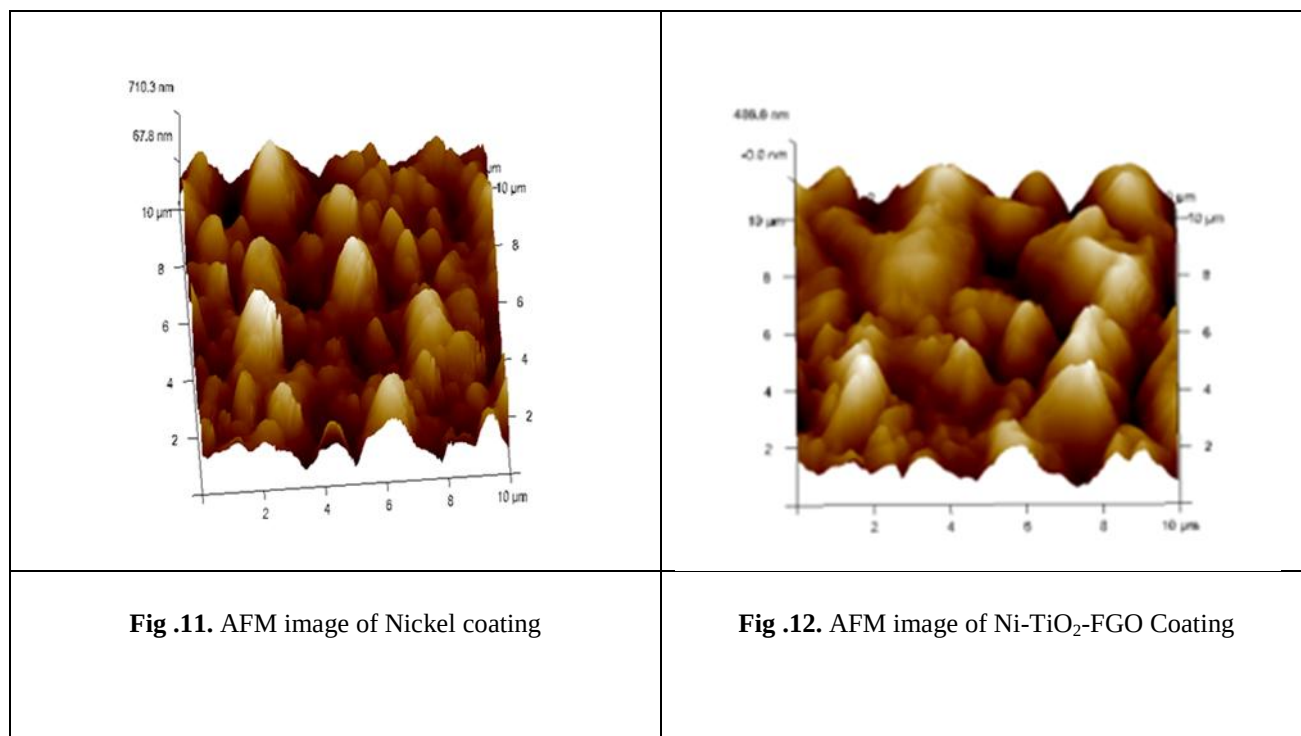


Fig .10. Microhardness of Ni and Ni-TiO₂-FGO coating

The Ni-TiO₂-FGO coating observed a hardness value of 328.12 hv for the 50g load segment and 271.5 hv for the 100g load segment respectively, compared to the nickel counterpart of 213.6 hv and 195.78 hv. Ni-TiO₂-FGO composite coatings have a much higher microhardness than pure nickel coatings. By refining grains and strengthening dispersion, TiO₂ nanoparticles within the Ni matrix suppress grain growth while minimizing plastic deformation. [46, 47]. Deformation caused by Graphene Oxide particles as well as its high hardness are both responsible for coatings microhardness increase. [48].

D. Atomic Force Microscopy



The granular surface morphology of TiO₂-FGO particles is depicted in Figures 11 and 12. It is also equitably distributed within the Ni matrix. The deposition of TiO₂ -FGO (1gl⁻¹) resulted in a more compact structure with clustered granules with a decreased grain size. As a result of nanoparticles and increasing cathode surface area, nickel growth begins to be inhibited [49]. Graphene in the coating is said to increase nucleation sites and decrease grain size, leading to greater coating roughness [50]. As a result, as shown in Table 4, embedding TiO₂-FGO nanoparticles in nickel coatings improved crystallisation and increased surface roughness.

Table .4. Roughness data

Coating	Ra (nm)	Rq(nm)
Nickel	78.8	102
Ni-TiO ₂ -FGO	128	161

CONCLUSION

- Ni-TiO₂-FGO was electrodeposited on mild steel with distinct TiO₂-FGO proportions of 0.25, 0.50, 0.75, and 1gl⁻¹. The following conclusions were drawn from the work:
- The composite coating has proved to be an efficient barrier against corrosion. Ni-TiO₂-FGO (1 gl⁻¹) nanoparticles in a nickel matrix yielded maximum inhibitor efficiencies of 76.7% (Tafel) and 79.5% (EIS).
- The microhardness increased by 54% and 39% respectively in Ni-TiO₂-FGO coating (1g/l) for applied load segments of 50g and 100g.
- TiO₂-FGO particles improved the roughness value of the coating by 60%.
- These properties make Ni-TiO₂-FGO composite coating a promising alternative for industrial protective coating.

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