STM JOURNALS

Journal of
Polymer & Composites

ISSN: 2321-2810 (Online)
ISSN: 2321-8525 (Print)
Volume 13, Special Issue 4, 2025
DOI (Journal): 10.37591/JoPC

https://journals.stmjournals.com/jopc

Research JoPC

Electrodeposition and Magnetron Sputtering of Rhenium Coatings on Carbon-Carbon Composites for Enhanced Corrosion Resistance and Structural Integrity

Sai Prasanna Kumar.J.V[1,*], Sundharesan R[2]

Abstract

It is well known that tailored materials are gaining in-roads to aviation and other manufacturing industries where stringent requirements are to be met, due to high temperature erosion, which is a problem. In order to counter these issues, materials which can resist a very high temperature are to be sought. One of the several methods to combat this high temperature issue is to provide a metallic refractory material coating on the carbon-carbon substrates. Rhenium is one of the heaviest known materials with a high melting point and could be used in Combustor Liners and Turbine Blades, Throat Inserts, Nozzle Extensions, and Exit Cones in aircraft and rocket engines. In this present work 3D Carbon-carbon composite with 0/90 combination were used an experimental substrate on which Rhenium coating was provided. Rhenium was deposited electrophoretically and also by DC Magnetron Sputtering. The DC Magnetron sputtering was accomplished. CC pattern was revealed by X-ray Diffraction. Further the samples were immersed in 3.5% sodium chloride solution using three cell electrochemical works station the corrosion rate was estimated from the Tafel plots and it was found to be 1.33 x 10^{-3}mm/ year and the electron transfers during the coating process was studied by the Nyquist Plots. A Rhenium Electrolytic solution was developed comprising of precursor Rhenium salts, and thin layer of Ni was given for better adherence. A thick coating of 30 microns was obtained from electrochemical deposition and with magnetron sputtering 3 microns thick coating was obtained.

Keywords: Rhenium, X-ray diffraction, corrosion, microstructure, Tafel & Nyquist plots

INTRODUCTION

The relevance of refractory metals in high-temperature, high-stress applications like aerospace and propulsion is highlighted by the extensive research on coatings and deposition methods for these metals, with a major focus on rhenium (Re) and iridium (Ir). Because of their exceptional oxidation resistance and longevity at temperatures that can reach 2000°C, iridium coatings on rhenium substrates have been the subject of extensive investigation. To increase the adhesion, microstructural integrity, and performance of these coatings, a number of deposition techniques have been studied, including chemical vapor deposition (CVD), electrodeposition, magnetron sputtering, and plasma processes.The influence of deposition temperature and heat treatment on iridium coatings applied to carbon-carbon composites and isotropic graphite using R.F. magnetron sputtering. Coatings formed at ambient temperature have extremely fine-grained, columnar formations, whereas those coated at 1073 K have denser, bigger grain structures that are prone to cracking. Heat treatment of iridium coatings at high temperatures leads in equiaxed grains with reduced or eliminated porosity, especially for coatings placed at higher substrate temperatures [1]. Iridium coatings were electrodeposited in molten salt on substrates made

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Received Date: November 15, 2024
Accepted Date: February 17, 2025
Published Date: May 10, 2025

Citation: Sai Prasanna Kumar J. V, Sundharesan R. Electrodeposition and Magnetron Sputtering of Rhenium Coatings on Carbon-Carbon Composites for Enhanced Corrosion Resistance and Structural Integrity. Journal of Polymer & Composites. 2025; 13(Special Issue 4): S35–S44p.

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of molybdenum, rhenium, and carbon-carbon (C/C). The coatings on molybdenum and C/C composites had weak adherence and surface flaws, but the rhenium coating was compact, defect-free, and adhered strongly. The discrepancies in adhesion and surface quality are attributable to the varying corrosion behavior of the substrates in the molten salt and their surface properties [2]. Iridium coatings were successfully electrodeposited on rhenium substrates employing molten salts at 600°C in an air environment. The iridium coating achieved good adhesion, a flat surface, and a microhardness of 442 kg/mm². The binding strength between the iridium coating and the rhenium substrate exceeded 16 MPa, and the coating remained intact and crack-free after 114 cycles of thermal shock testing [3]. Electrodeposited iridium (Ir) on rhenium (Re) substrates exhibits high-temperature diffusion behavior. The diffusion coefficients and activation energy of Re diffusing into Ir were examined, demonstrating that electrodeposited Re has much greater diffusion rates than chemical vapor deposited Re due to its increased defect concentration and unstable grain structure. The results reveal that the diffusion behavior is impacted by the microstructural variations, which affect the material's performance and lifespan in high-temperature applications [4]. The morphology and mechanical properties of monolayer and multilayer iridium (Ir) coatings on tungsten carbide substrates formed utilizing a double glow plasma technique. Both coatings had robust adhesion and no delamination, a polycrystalline phase with (110) structure, and compressive residual stresses (-1.6 GPa for monolayer and -1.1 GPa for multilayer). The high-temperature deposition procedure led to the discovery of a new WIr phase, which improved the coating's resistance to oxidation and wear [5].The influence of pH and temperature on the electrodeposition of Re-Ir-Ni alloy coatings from aqueous solutions. It was discovered that increasing the pH from 2.0 to 8.0 enhanced the metals' faradaic efficiency, deposition rate, and partial current densities, with pH 2.5 yielding the maximum rhenium concentration (82%). Bath temperature also affected the surface morphology, fracture density, and grain size of the coatings, with higher temperatures leading to the production of crystalline phases and greater iridium concentration [6].The features of iridium oxide (IrO2) thin films produced by spray pyrolysis method (SPT) on glass substrates that are dependent on thickness. Differentially thick films (700–2250 Å) were examined for their thermal, electrical, and structural properties. It was discovered that increasing layer thickness increases crystallinity and decreases electrical resistance at ambient temperature. After annealing at 600°C, heavily doped semiconductor behavior in the as-deposited films changes to metallic behavior [7]Vacuum plasma spraying (VPS), electroformed deposition (ED), and diffusion bonding are the three fabrication techniques used to create iridium-coated rhenium (Ir/Re) rocket chambers. Gaseous hydrogen and oxygen propellants were used to test the 22-N class chambers; the VPS and ED chambers were able to reliably withstand testing for about ten hours. Two hours of testing revealed no deterioration of the iridium lining or its attachment to the rhenium substrate, but the wrought chamber needed to be reworked [8]. The development and operation of iridium/rhenium (Ir/Re) rocket engines, emphasizing how much better they are at high temperatures—up to 2200°C—than with conventional materials. In addition to addressing technical concerns including mechanical qualities, connecting techniques, and life-limiting mechanisms, it talks about a variety of manufacturing techniques, including chemical vapor deposition (CVD). The excellent performance and long-life potential of Ir/Re engines for satellite and spacecraft propulsion systems have been confirmed by their successful development and in-orbit testing [9]. An oxide-layered iridium/rhenium (Ir/Re) structure is the main focus of the study, which assesses novel materials for high-performance, high-temperature combustion chambers. Several oxide topologies and thicknesses were investigated in these chambers to improve their endurance and resistance to oxidation. Extensive testing at high combination ratios and temperatures over a period of 10 hours demonstrated the potential for extended usage of oxide-protected Ir/Re chambers in propulsion systems, as the results demonstrated a considerable improvement in lifespan under harsh conditions [10,11]. Improve adhesion and reduce thermal stress by utilizing rhenium (Re) as an interlayer between iridium (Ir) coatings and carbon-carbon (C/C) composites. The bond strength of as-deposited Ir/Re/C/C increased to 7.9 MPa, compared to 3.2 MPa for Ir/C/C, indicating that Re greatly strengthens the bond strength of Ir coatings, according to the results." As a result of molten salt seeping into the C/C substrate, binding strength dropped by roughly 50% following high-temperature annealing [12]. Carbon-carbon (C/C) composites can benefit from the application of ZrB2-SiC-ZrC ultra-high temperature ceramic coatings to increase ablation resistance over 2000 K.

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Improved compactness and interface compatibility were achieved by applying the coatings using a mix of reaction melt infiltration and supersonic atmospheric plasma spraying (SAPS). After being exposed to high temperatures, the coated C/C composites showed outstanding ablation resistance, with little mass and linear ablation rates, preserving structural integrity without cracking [13].The behavior of rhenium ions in molten KF-KBF4-B2O3-KReO4, with an emphasis on rhenium electrodeposition and reduction, electrochemical nature. Through the use of cyclic voltammetry, it was possible to determine that the reduction of rhenium ions involved two different forms of complex rhenium ions and was a quasi-reversible activity under diffusion control. Potentiatiodynamic electrolysis has shown promise for producing compact and pure rhenium coatings by effectively producing high-purity (99.98%) rhenium nanofibers on a glassy carbon substrate [14]. The iridium (Ir) coatings' long-term high-temperature oxidation resistance on rhenium (Re) substrates, achieved by electrical resistance heating at 2000°C in air. After oxidation, the Ir coating showed outstanding adhesion and maintained its density and smooth surface, however oxidation and diffusion processes caused failure after 183 minutes. Following oxidation, the Ir coating's preferred orientation shifted from (220) to (111), and the degradation was mostly caused by the interaction of direct Ir oxidation with rapid Re diffusion into the Ir coating[15]The effective use of chemical vapor deposition (CVD) on molybdenum substrates, which were subsequently etched away, to produce thin-wall rhenium tubes. The CVD rhenium showed a columnar grain structure with preferential development along the c-axis, as well as a facetted surface. At high temperatures (2000 K), significant grain expansion took place, producing a recrystallized structure with big grains and smooth transverse borders. [16].

MATERIALS AND METHODS

The substrates carbon-carbon composites were fabricated using CVI technique and the ensuing sample had a 0/90 configuration and reinforced in the z direction. A total of 10 samples were fabricated and were machines to the suitable size of 25 mm x25 mm x5 mm. All the samples were cleaned with isopropanol to remove any containments. The precursor material source of rhenium both Ammonium Perrhenate and the Sputtering target was obtained from Intelligent materials, Haryana and Nano Research Elements, Bangalore respectively. While the other chemicals necessary to prepare the Rhenium electrolyte was sourced locally.

Preparation of Rhenium Electrolyte

The electrolyte is prepared for coating Rhenium at room temperature by the use of three cellelectrochemical works station. Rhenium source of concentration of 0.1M was added to deionized water in 500 ml beaker and was sonicated magnetically till the precursor powder dissolved. To this solution about 0.05M of Ni sulphate was added to improve the adhesion, then slowly Sulphuric acid of concentration of 0.1 M in a dropwise fashion. Acetic acid of strength 0.05 M to improve the pH value and was maintained at 3. Critic acid was also added to improve the deposition of Rhenium on the substrate. The solution was stirred for an hour and was aged for 48 hours in a cool dark place. The electrolyte is ready for deposition on the substrate. The coating process performed on the traditional three cell potentiostat with carbon-carbon substrate as the working electrode while the Ag/AgCl and Platinum wires are the other electrodes on which a potential of 1 mA/cm2 and scan rate of 100 mv/sec. As time progressed a thick coating ensued on the sample, figure 1. Thick coating ensued on the sample, Figure 1.

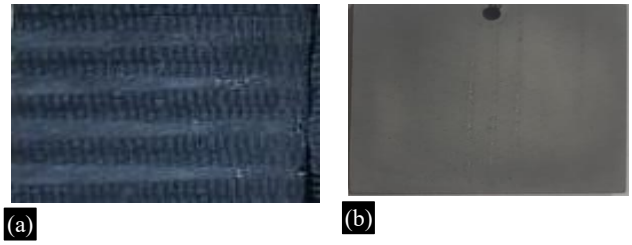

Figure 1. (a) Carbon-carbon substrate, (b) rhenium deposited sample.

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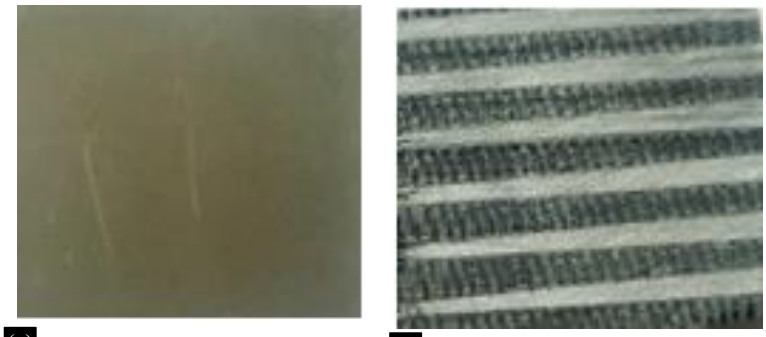

Figure 2. (a) Glassy plate and (b) Carbo-carbon substrate.

Magnetron Sputtering of Carbon Composites

The sputtering target of 99.99% pure was placed in the Vacuum chamber of the sputtering device. At first the chamber is degassed and the substrate was placed in the holder. The machine operated at 1KV and 1KW. The vacuum pressure was maintained at 5 $x10^{-7}$. Argon was the purging gas, while the substrate temperature was maintained at 200°C. The powerful source of plasma bombarded the target material making it to diffuse and the gaseous elements reaches the substrate which was placed at 10 cm away from the target material. Upon cooling this gaseous discharge condensed and formed layer of rhenium on the substrate, Figure 2.

It can be observed that on the glassy plate, Figure 2a. the Sputtering of the rhenium target was largely uniform but, on the carbon-carbon substrate the coating appeared thick, Figure 2b. This could because the carbon-carbon substrate had a porous surface and hence the coating appears not uninform, but micrograph would reveal otherwise.

Coating Characterization

The dense coating was characterized for its Microstructure was performed on Carl Zeiss Field Emission Electron microscope, while XRD characterization was performed at room temperature with a scan rate of 40 per min between 0 and 120 degrees. The hardness of the coating was measured with cantilever type of stylus and the corrosion study was established on a conventional three cell electrochemical works station with 3.5 % Aqueous sodium chloride solution. The Tafel and Nyquist plots assessed the corrosion characteristics of the coating.

RESULTS AND DISCUSSION

Microstructure Characterization

To assess the microstructure of the coated samples, after coating the samples were machined to a dimension of 10 mm x 10 mm to accommodate into the FESEM specimen holder. First the samples which were electrodeposited were scanned, figure 3(a-c) and figure4 (a & b) with the Scanning electron microscope and then later on with the DC sputtered samples were scanned, Figure 5(a-f).

Table 1. Elemental mapping of Rhenium.

Map sum spectrum				
Element	***Line type***	***Weight %***	***Weight % sigma***	***Atomic %***
C	K series	16.7	0.21	54.62
O	K series	9.21	0.1	22.62
Ni	K series	15.55	0.11	10.4
Re	L series	58.54	0.22	12.35
Total		100		100

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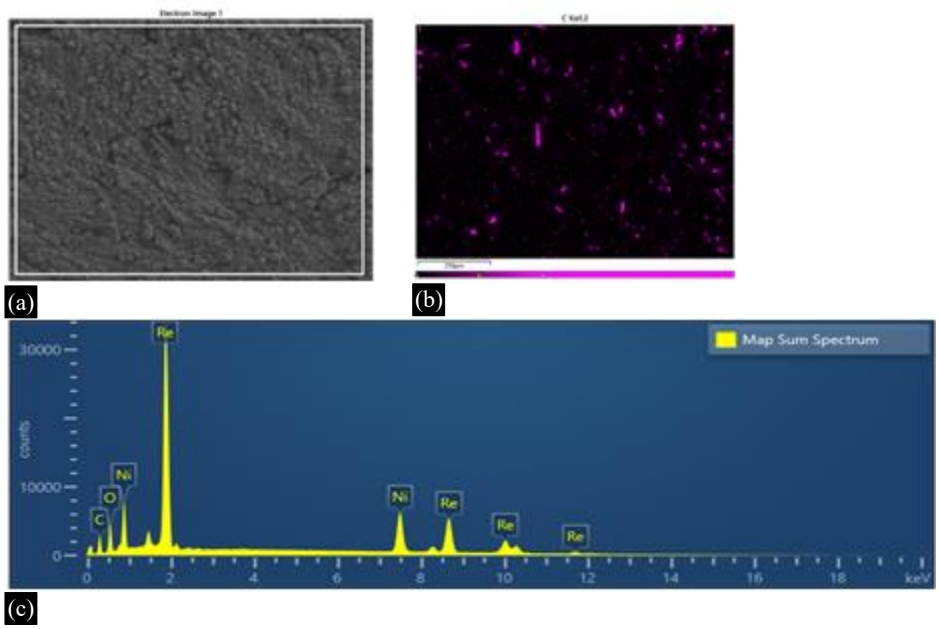

Figure 3. Microstructure of electro deposited Re-Ni coating, (a) Sem Image at 25 μm, (b) Elemental mapping, (c) Map spectrum of Re-Ni coating

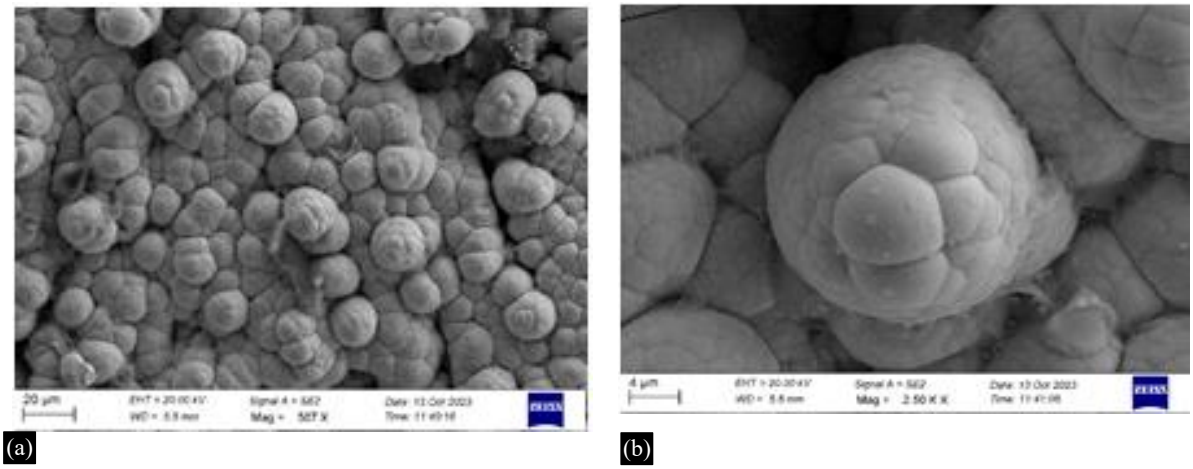

Figure 4. Microstructure of Re-Ni coated samples, (a) Micrograph at 20 microns, (b) Micrograph at 4 microns.

Figure 3a represents the microstructure of the electrodeposited sample, it can be seen that the Rhenium adhered to the substrate and it was rather smooth. It had a grainy architecture without any exclusions and this coating was scanned at 250 microns. While in Figure 3b Rhenium was mapped in violet and in elemental mapping in Figure 3c the percent of Rhenium in the L series about 58.54 % evidenced by table 1. Small amounts of C, O, Ni was present and its is expected due to the various ingredients present in the electrolyte, which was expected.

In Figure 4a the grains of rhenium were deposited perpendicular to the surface and it was scanned at 20 microns while in Figure 4b the Rhenium deposition had similar architecture the micrograph was obtained at 4 microns, and at this depth of coating cracks, or voids are not visible. The magnetron sputtering occurred by pulverizing the Sputter target and Rhenium got deposited on the substrate layer by layer, Figures 5 (a-f). This technique is a line-of-sight operation; hence this method is not ideal for complex geometries, while the limitation of the electrochemical deposition is limited by the target size and not by its complexity.

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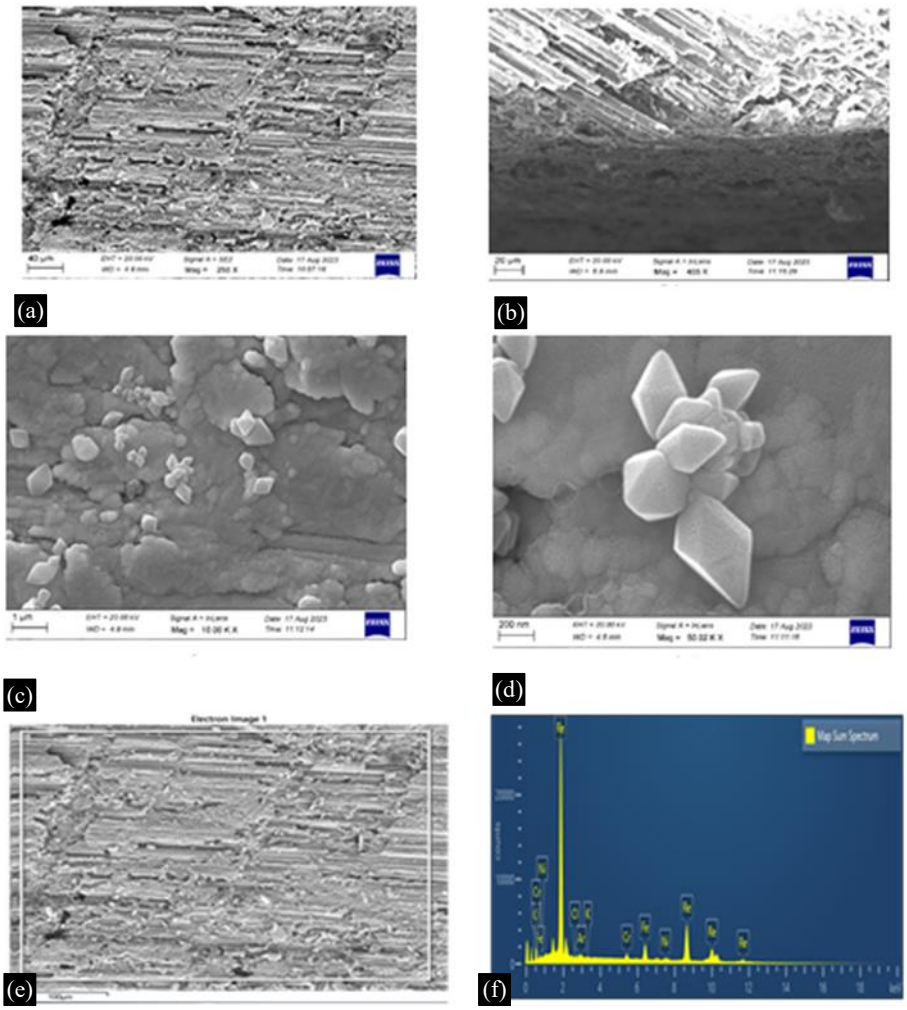

Figure 5. Microstructure characterization: (a) SEM image at 40 microns, (b) Microstructure at 20 microns, (c) Image at 1 micron, (d) Image at 200 nm , (e) Micrograph at 100 microns , (f) Elemental mapping.

Table 2. Elemental mapping and it's percent.

Map sum spectrum				
Element	***Line type***	***Weight %***	***Weight % sigma***	***Atomic %***
O	K series	9.52	0.16	49.3
Cl	K series	0.51	0.04	1.2
Ar	K series	0	0.05	0
K	K series	0.25	0.04	0.53
Cr	K series	1.53	0.06	2.44
Fe	K series	6.35	0.09	9.43
Ni	K series	0.7	0.08	0.99
Re	L series	81.13	0.21	36.11
Total		100		100

The samples after the deposition from the DC magnetron, smaller pieces were cut to f accommodate them at the specimen holder of FESEM. The coating was scanned at depths of 40 microns figure a, 20 microns as in Figure b, 1 micrometer and 200 nm in Figures c and d. It can be seen that the coating adhered to the substrate in a layer wise fashion, the grainy structure was not evident in all Figures except in Figures 4c and 4d. There was a peculiarity in the coating of the images 4c and in d they had a Spike

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like appearance, nevertheless the coating deposited perpendicular to the substrate, possibly the coating oxidized. The grainy architecture is visible in Figure 4c which is similar to Figure 3 d. The Elemental mapping was considered for the sputtered target at 100 microns, it can be seen in Figure 4e Rhenium peaks, figure 4f, were obtained at L series about 81 %, table 2 while other elements such as Fe, C, Cr, Ni, O were present in small percent. The presence of Argon is because of the purging gas.

XRD ANALYSIS

The samples were characterized for its crystal structure and they had the similar crystal structure without any large deviations The diffractograms were constructed at room temperature. It can be seen that there wasn't any preferential orientation was and it had FCC structure. The crystalline structures were characterized by the peaks and gradually the intensity of the peaks diminished. The composition of the substrate influenced the crystalline structure, figure 6, according to JCPDS no: 05-0702 powder diffraction data.

MICRO HARDNESS OF THE COATING

Rhenium coating was administered by means of electrodeposition and as well aby DC magnetron sputtering. ASTM E 384 prescribes the procedure to test the coating hardness. Both the coatings had improved the hardness due to the coating and it was found to be more than that of Rhenium and it was about 170 HV while the porosity was about 1 % with its surface roughness of about 6 microns. This property has an effect on corrosion behavior of the sample.

CORROSION

Crucial details on the corrosion properties of the carbon-carbon composite with rhenium coating are provided by the Figure 7 and table 3. The material appears to have a modest tendency to corrode in the given environment, based on the corrosion potential (E_{corr}) of -0.332 mV. The low value of the corrosion current density (I_{corr}) of 0.244 μA/cm² suggests that the rhenium layer provides excellent protection against corrosion. The anodic and cathodic reactions continue at different rates, as indicated by the anodic Tafel slope (βa) of 0.0891 V/decade and the cathodic Tafel slope (βc) of 0.07105 V/decade. These results indicate that the pace of electrochemical reactions on the surface is slowed down by the stable and efficient barrier that the rhenium coating provides. As a result, it is determined that the overall corrosion rate is 1.33×10^{-3} mm/year, indicating that the rhenium coating is useful in improving the resilience and lifetime of the carbon-carbon composite material in corrosive conditions.

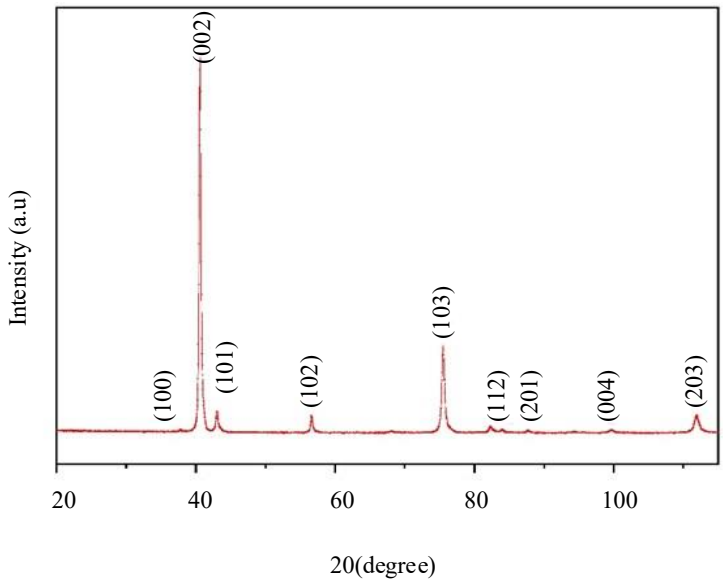

Figure 6. XRD diffractogram of sputtered Rhenium on C/C composites.

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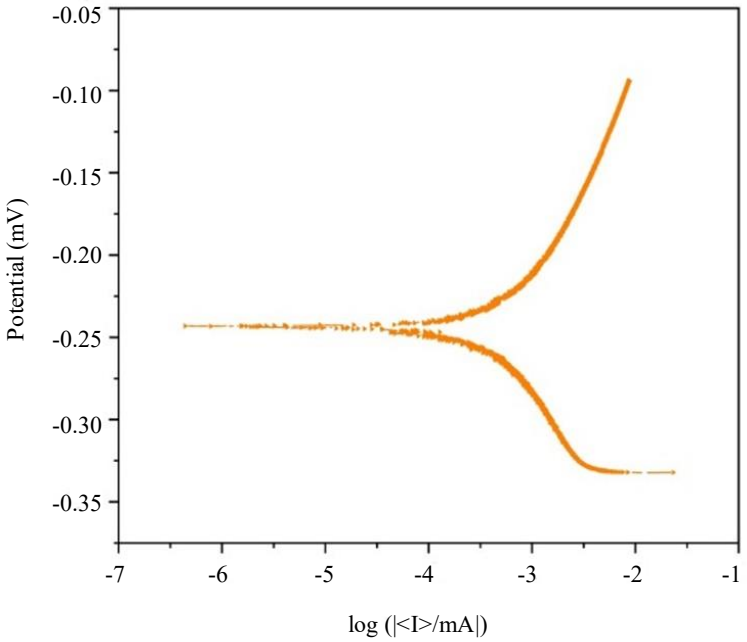

Figure 7. Tafel Plot for coating of rhenium on carbon-carbon composite.

Table 3. Corrosion Parameters for Coating of Rhenium on Carbon-Carbon Composite.

Material	E_{corr} (mV)	I_{corr} (μA)	βc (mV)	βa (mV)	Corrosion rate mm/year
C-C Composite	-0.332	0.244	0.07105	0.0891	1.33×10^{-3}

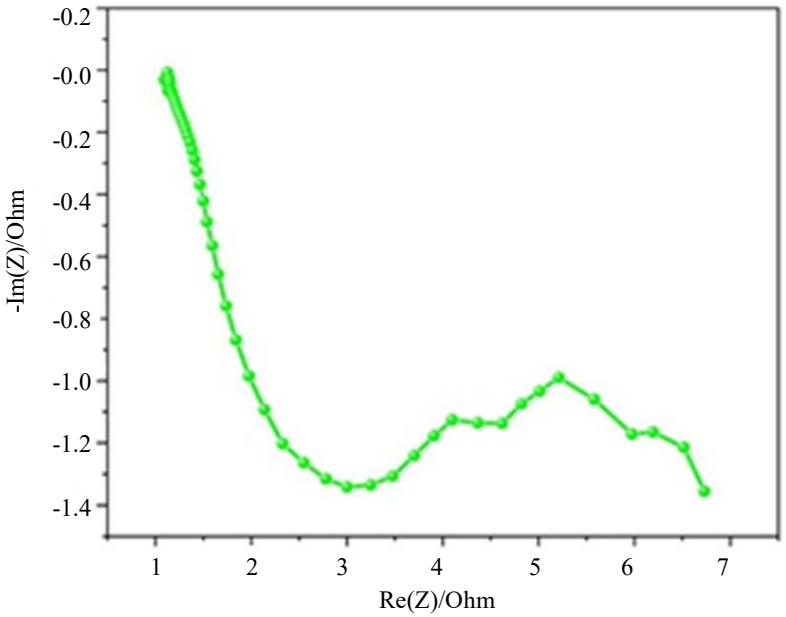

Figure 8. Nyquist plot of rhenium-coated carbon-carbon composite.

An essential instrument in electrochemical impedance spectroscopy (EIS) for examining the impedance properties of materials is the Figure 8, Nyquist plot. Plotting begins on the left at the high frequency end and moves rightward to lower frequencies. The first semicircle-like arc shows normal electrochemical activity incorporating both resistive and capacitive elements. It extends from around Z_{re}=1Ω to 3 Ω. This semicircle's diameter, which represents the resistance to electron transfer at the interface between the carbon-carbon composite and the rhenium coating, is equivalent to the charge transfer resistance (R_{ct}). A distinct semicircle indicates a homogenous, stable coating that has a high

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degree of charge transfer resistance, which is advantageous for preventing corrosion. Plotting in the lower frequency range reveals a tail in the data that deviates from the semicircle; this tail is commonly ascribed to diffusion-controlled processes (Warburg impedance). This tail suggests that mass movement of ions or molecules, through the pores in the coating or the composite material itself, may affect the impedance behavior at lower frequencies. The emergence of this tail indicates that the impedance response is dominated by diffusion processes. The prominent semicircle in this Nyquist figure indicates that the rhenium coating on the carbon-carbon composite offers strong resistance to charge transfer. Moreover, the diffusion-controlled phenomena observed at reduced frequencies emphasize the intricate character of the electrochemical reactions occurring within the substance.

CONCLUSION

This present study was undertaken to deposit a thicker coating on the carbon-carbon substrate, by different methods namely by electrochemical and DC magnetron sputtering. This was achieved by preparing an electrolyte comprising of Ammonium Perrhenate, small amounts of Nickel sulphate to improve the adhesion, while other chemicals aided in maintain the pH and deposition powers. While a Sputtering Rhenium target material consisting 99.99% purity was utilized in depositing metallic rhenium on carbon-carbon composites. Further, the hardness of the coating and the corrosion rate of the electrochemical deposits was established by means Tafel and Nyquist extrapolations The following conclusions can be drawn from this study:

1. Complex geometries can be coated with good thickness using electrochemical deposition, for the present study the thickness was about 30 microns was obtained, even larger thickness is possible. Upscaling is possible.
2. The DC Magnetron sputtering process is efficient and it is a line-of-sight process indicating that complex geometries can't be attempted. It's limited by the power build up and its eventual harness to pulverize the target material. A 3-micron thick coating ensued.
3. The microstructure of the electrodeposited coating was grainy and Ni coating was given to improve the adhesion of coating to the substrate and the coating was uniform without any micro voids and or cracks. The coatings run perpendicular to the surface. Small amounts of Carbon, oxygen was present in the EDX mapping and this was due to the various agents added in developing the electrolyte for the deposition.
4. The electrolyte was aged for 48 hours and there was no evidence of the electrolyte deterioration, and it can be understood that it can have a shelf life of at least six months.
5. The microstructure of the sputtered samples had similar architecture of grainy architecture and uniform coating but at about 200 nm the it was observed a spike like coating appeared on the substrate, perhaps, it could be the particles Rhenium solidified independently and faster than that of the other layered deposits.
6. The micro hardness improved with respect to the target materials, while the surface roughness and its porosity was within the acceptable limits, indicating that better corrosion rates are possible.
7. EIS are useful in understanding the corrosion behavior of the coated samples. Both Tafel and Nyquist plots were constructed for the electrodeposited samples and the corrosion rate was found to be 1.33×10^{-3} per year.
8. Thicker coating up to 70-80 microns can be obtained from electrochemical deposition and the deposition was performed at room temperature, although use of molten salts and ionic liquids were not attempted in this study.

Acknowledgements

The authors would like to thank the management for their financial support in conducting this study vide the slip number: 0012334/08.06.2023. The authors would like to like to thank Dean [R &D], Dean, SOMC and the Head Aeronautical Engineering Department, Vel Tech Rangarajan Dr. Sagunthala R&D Institute of Science and Technology, Avadi.

Conflict of Competing Interest

The authors declare that there was no competing interest in conducting this study.

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