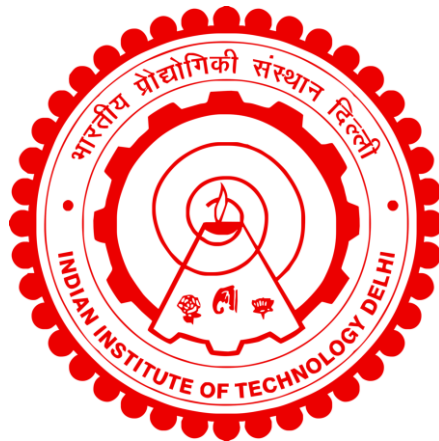


# **DEVELOPMENT AND CHARACTERIZATION OF METAL BASED COMPOSITE COATINGS**

**RAKESH MOHARANA**



**DEPARTMENT OF MECHANICAL ENGINEERING  
INDIAN INSTITUTE OF TECHNOLOGY DELHI  
MARCH 2026**

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**DEVELOPMENT AND CHARACTERIZATION OF METAL BASED  
COMPOSITE COATINGS**

by

**Rakesh Moharana**

Department of Mechanical Engineering

Submitted

in fulfilment of the requirements of the degree of Doctor of Philosophy

to the



**INDIAN INSTITUTE OF TECHNOLOGY DELHI**

**NEW DELHI-110016, INDIA**

**March 2026**

**Dedicated to**

*My teachers*

## CERTIFICATE

This is to certify that the thesis entitled “**Development and characterization of metal based composite coatings**” being submitted by **Mr. Rakesh Moharana** to the **Indian Institute of Technology Delhi** for the award of the degree of **Doctor of Philosophy** in the Department of Mechanical Engineering is a bonafide research work carried out by him under our supervision and guidance. To the best of our knowledge, the thesis has reached the requisite standard. The research reports and the results presented in this thesis have not been submitted in parts or in full to any other University or Institute for the award of any degree or diploma.

**Prof. S. Aravindan**

Professor

Mechanical Engineering Department

IIT Delhi

Delhi-110016

**Prof. Sudarsan Ghosh**

Professor

Mechanical Engineering Department

IIT Delhi

Delhi-110016

## ACKNOWLEDGEMENTS

I would like to express my heartfelt gratitude and esteem to both of my supervisors, **Prof. S. Aravindan** and **Prof. Sudarsan Ghosh**, for all their assistance in completing my thesis. Their guidance has been the foundation of my academic and research endeavours. They consistently provided direction, support, and intellectual insight, which significantly enhanced the depth and breadth of my studies. Their dedication to promoting intellectual progress has left a lasting impression on my academic career.

I extend my heartfelt gratitude to the eminent members of my research committee, **Prof. S. K. Sinha**, **Prof. D Ravi Kumar**, and **Prof. Deepak Kumar**. The thorough comments and constructive feedback received during several interactions not only polished the contours of this thesis but also widened my intellectual perspective. I am exceedingly grateful to **Dr. N. Rajasekharan** (CECRI, Karaikudi) for their invaluable assistance in doing my experimental work investigation. The tremendous assistance I received from **Prof. Praveen Ingole** (Chemistry Department, IIT Delhi) while conducting my experimental work is something for which I am very grateful. I am thankful to all the staff members of the Central Research Facility and Central Workshop for giving me unwavering support during my experimental and characterization works.

Finally, I have no words to express my sincere gratitude to my family: my father, Mr. Akshaya Maharana, my mother, Mrs. Mamata Maharana, and my brother, Mr. Saroj Maharana, without their support, I could not have completed my thesis. This thesis represents not only a personal accomplishment but also a collective triumph, crafted through the support and contributions of these distinguished individuals.

**Rakesh Moharana**

## ABSTRACT

Aluminum coatings are widely used for corrosion resistance, high-temperature oxidation resistance, and a bright aesthetic appearance. The aluminum electrodeposition involving an ionic liquid-based electrolyte is preferred because of its extensive electrochemical window, minimal vapor pressure, non-flammability and a greener alternative to the existing organic solvent system. Aluminum deposition employing an ionic liquid-based electrolyte on mild steel has been used as a protective coating for enhancing corrosion resistance. Nevertheless, the adherence of aluminum to the mild steel has been a concern since the iron oxide layer forms on its surface before the deposition. Conventional in-situ electrochemical etching prior to electrodeposition improves adhesion, but it leads to electrolyte contamination from substrate dissolution. This leads to electrolyte degradation and the formation of an impure coating. To overcome these shortcomings, plasma treatment is proposed, and its feasibility as a surface modification technique is investigated. The effects of surface treatment, both plasma and electrochemical, on deposit morphology, roughness, crystal orientation, micro-hardness, and corrosion resistance are discussed. Plasma modification has led to the development of a finer, denser, and smoother aluminum coating. The aluminum deposition on plasma-modified steel exhibited higher micro-hardness with an increase of 32.4 % and lower corrosion current with a reduction of 65.3 % in comparison to that on the electrochemically etched sample. These improved performances establish plasma modification as the superior alternative to the electrochemical etching for aluminum electrodeposition on steel.

Plasma modification of the steel substrate enabled the deposition of a well-adherent aluminum coating. Since aluminum electrodeposition from the chloroaluminate solution is complex, careful optimization of process parameters is necessary to ensure the development of high-quality aluminum deposition in the form of dense, uniform, and compact polyhedral growth with consistent excellent surface coverage. Among the process variables, applied potential,

stirring speed and temperature of the electrolyte were chosen as the critical factors due to their dominating role in governing the nucleation, growth, kinetics of ions, and transport of ions, particularly in the context of aluminum electrodeposition from decane-shielded chloroaluminate electrolyte. After conducting a series of experiments, the optimal values of these parameters were found. The optimal value of applied potential, stirring speed, and electrolyte temperature are -400 mV, 250 rpm, and 25 °C, respectively. With these process parameters, a high-quality aluminum coating was developed.

Two major shortcomings of the pure aluminum coating are its reactivity towards the chlorinated aqueous solution and its low hardness, which are addressed by incorporating functionalized graphene nanoplatelets into the matrix of aluminum coating. The performance of the developed coating was assessed in terms of coating morphology, topography, thickness, crystal orientation, micro-hardness, and corrosion, and compared with that of pure aluminum coating. The composite coating exhibited an increase of 38.18 % in micro-hardness and an increase of 141.7 % in polarization resistance as compared to the pure aluminum coating. The reinforcement successfully shifted the corrosion potential to a more positive potential from -824 mV to -767 mV. These improvements highlight that the aluminum-based composite coating can be a strong alternative to pure aluminum coating on steel.

The development and characterization of pure and composite coatings were done successfully on the flat mild steel sample. For broadening the horizon of its real-world applications, the optimized parameters were effectively transferred to the wire geometry, aimed at potential deployment in aerospace fasteners, industrial fittings, and welding wire. Continuous and adequately adhered aluminum-based coatings were successfully deposited on the mild steel welding wire. This successful transfer suggests the suitability of this process for real-world industrial environments necessitating continuous coating along an extended wire.

**Keywords:** Electrodeposition, Composite coating, Graphene nanoplatelets, Corrosion

## सार

एल्यूमीनियम कलई का व्यापक रूप से संक्षारण प्रतिरोध, उच्च तापमान ऑक्सीकरण प्रतिरोध, और उज्ज्वल सौंदर्य उपस्थिति के लिए उपयोग किया जाता है। आयनिक तरल-आधारित इलेक्ट्रोलाइट से युक्त एल्यूमीनियम वैद्युत निक्षेपण को इसकी व्यापक विद्युत रासायनिक खिड़की, न्यूनतम वाष्प दबाव, गैर-ज्वलन शीलता और मौजूदा कार्बनिक विलायक प्रणाली के लिए एक हरित विकल्प के कारण पसंद किया जाता है। हल्के स्टील पर एक आयनिक तरल-आधारित इलेक्ट्रोलाइट को नियोजित करने वाले एल्यूमीनियम निक्षेपण का उपयोग संक्षारण प्रतिरोध को बढ़ाने के लिए एक सुरक्षात्मक कलई के रूप में किया गया है। फिर भी, मृदु इस्पात के लिए एल्यूमीनियम का आसंजन एक चिंता का विषय रहा है क्योंकि निक्षेपण से पहले इसकी सतह पर लोहे की ऑक्साइड परत बनती है। से पहले पारंपरिक यथावत् वैद्युत-रासायनिक निक्षारण आसंजन में सुधार करती है, लेकिन यह क्रियाधार विघटन से इलेक्ट्रोलाइट संदूषण की ओर ले जाती है। इससे इलेक्ट्रोलाइट का क्षरण होता है और अशुद्ध परत बनती है। इन कमियों को दूर करने के लिए, प्लाज्मा उपचार प्रस्तावित किया जाता है, और सतह संशोधन तकनीक के रूप में इसकी व्यवहार्यता की जांच की जाती है। जमा आकृति विज्ञान, खुरदरापन, क्रिस्टल अभिविन्यास, सूक्ष्म-कठोरता और संक्षारण प्रतिरोध पर सतह उपचार, प्लाज्मा और विद्युत रासायनिक दोनों के प्रभावों पर चर्चा की गई है। प्लाज्मा संशोधन ने एक महीन, सघन और चिकनी एल्यूमीनियम परत का विकास किया है। प्लाज्मा-संशोधित स्टील पर एल्यूमीनियम निक्षेपण ने वैद्युत-रासायनिक निक्षारित नमूने की तुलना में 32.4% की सूक्ष्म-कठोरता वृद्धि, और 65.3% की कमी के साथ कम संक्षारण धारा का प्रदर्शन किया। ये बेहतर प्रदर्शन, स्टील पर एल्यूमीनियम वैद्युत निक्षेपण के लिए वैद्युत-रासायनिक निक्षारण के बेहतर विकल्प के रूप में, प्लाज्मा संशोधन को स्थापित करते हैं।

स्टील क्रियाधार पर प्लाज्मा संशोधन ने एक अच्छी तरह से सुसंगत एल्यूमीनियम कलई के निक्षेपण को सक्षम किया। चूंकि क्लोरोएल्यूमिनेट समाधान से एल्यूमीनियम वैद्युत निक्षेपण

जटिल है, इसलिए लगातार उत्कृष्ट सतह कवरेज के साथ घने, समान और कॉम्पैक्ट पॉलीहेड्रल विकास के रूप में उच्च गुणवत्ता वाले एल्यूमीनियम जमाव के विकास को सुनिश्चित करने के लिए प्रक्रिया मापदंडों का सावधानीपूर्वक अनुकूलन आवश्यक है। विशेष रूप से डकेन-शील्डेड क्लोरोएल्यूमिनेट इलेक्ट्रोलाइट से एल्यूमीनियम वैद्युत निक्षेपण के संदर्भ में प्रक्रिया चरों में, अनुप्रयुक्त विभव, विलोडन गति, और इलेक्ट्रोलाइट के तापमान को महत्वपूर्ण कारकों के रूप में चुना गया था क्योंकि नाभिकन, विकास, आयनों की गतिकी, और आयनों के परिवहन को नियंत्रित करने में उनकी प्रमुख भूमिका थी। प्रयोगों की एक श्रृंखला आयोजित करने के बाद, इन मापदंडों के इष्टतम मान पाए गए। अनुप्रयुक्त विभव, विलोडन गति और इलेक्ट्रोलाइट तापमान के इष्टतम मान क्रमशः-400 mV, 250 rpm और 25°C हैं। इन प्रक्रिया मापदंडों के साथ, एक उच्च गुणवत्ता वाली एल्यूमीनियम कलई विकसित की गई थी।

शुद्ध एल्यूमीनियम कलई की दो प्रमुख कमियां क्लोरिनेटेड जलीय घोल के प्रति इसकी प्रतिक्रियाशीलता और इसकी कम कठोरता हैं, जिन्हें एल्यूमीनियम कलई के सांचे में कार्यात्मक ग्राफीन नैनो प्लेटलेट्स को शामिल करके घटाया जाता है। विकसित कलई के प्रदर्शन का मूल्यांकन कलई रूप प्रक्रिया, स्थलाकृति, मोटाई, क्रिस्टल अभिविन्यास, सूक्ष्म-कठोरता और संक्षारण के संदर्भ में किया गया था और इसकी तुलना शुद्ध एल्यूमीनियम कलई से की गई थी। समग्र कलई ने शुद्ध एल्यूमीनियम कलई की तुलना में सूक्ष्म-कठोरता में 38.18% की वृद्धि और धुवीकरण प्रतिरोध में 141.7% की वृद्धि का प्रदर्शन किया। कार्यात्मक ग्राफीन नैनोप्लेटलेट्स के सुदृढीकरण ने संक्षारण क्षमता को 824 mV से 767 mV तक अधिक सकारात्मक क्षमता में सफलतापूर्वक स्थानांतरित कर दिया। ये सुधार इस बात पर प्रकाश डालते हैं कि एल्यूमीनियम आधारित समग्र कलई स्टील पर शुद्ध एल्यूमीनियम कलई का एक मजबूत विकल्प हो सकता है।

शुद्ध और मिश्रित कलईयों के विकास और लक्षण वर्णन समतल इस्पात के नमूनों पर सफलतापूर्वक किये गये थे। इसके वास्तविक दुनिया के अनुप्रयोगों के क्षितिज को व्यापक बनाने के लिए, अनुकूलित मापदंडों को प्रभावी रूप से तार ज्यामिति में स्थानांतरित कर दिया गया था, जिसका उद्देश्य अंतरिक्ष बंधकों, औद्योगिक पुर्जे और वेल्डिंग तार में संभावित परिनियोजन था। अनवरत एवं पर्याप्त रूप से चिपके हुए एल्यूमीनियम-आधारित कलईयों को मृदु इस्पात की वेल्डिंग तार पर सफलतापूर्वक जमा किया गया था। यह सफल स्थानांतरण वास्तविक दुनिया के औद्योगिक वातावरण के लिए इस प्रक्रिया की उपयुक्तता का सुझाव देता है, जिसके लिए एक विस्तारित तार के साथ निरंतर कलई की आवश्यकता होती है।

मुख्य शब्द: वैद्युत निक्षेपण, मिश्र कलई, ग्राफीन नैनोपलेटलेट्स, संक्षारण

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