

**CHARACTERISATION AND CORROSION  
EVALUATION OF SOL GEL ALUMINA-GNP  
COATING ON NICKEL SUBSTRATE**

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We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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## **ABSTRAK**

Alumina ( $\text{Al}_2\text{O}_3$ ) ialah salah satu bahan seramik yang penting dan mempunyai ciri-ciri mekanik dan haba yang cemerlang dan berguna untuk aplikasi pembuatan di pelbagai tujuan industri terutamanya sebagai salutan seramik. Salutan seramik alumina berfungsi sebagai perlindungan terhadap karatan dengan menghalang lapisan dibawahnya daripada berinteraksi dengan persekitaran yang menghakis. Nikel biasanya dikenali kerana mempunyai ketahanan terhadap karatan yang tinggi terhadap larutan kaustik yang juga merupakan tempat dimana nikel selalu dijumpai. Walau bagaimanapun, nikel boleh berkarat dengan cara yang tidak dijangka, yang boleh menjelaskan fungsinya terutama dengan hadirnya ion klorida. Meskipun dilindungi oleh lapisan pasif, namun permukaan nikel masih terdedah kepada karatan setempat. Oleh itu, salutan alumina boleh digabungkan dengan grafin untuk menambahbaik matriks seramik supaya dapat memberikan perlindungan daripada karatan. Kajian ini bertujuan untuk menjalankan proses sintesis alumina-grafin platelet nano ( $\text{Al}_2\text{O}_3$ -GNPs) yang dihasilkan melalui kaedah sol gel, mengkaji pengaruh kepekatan alumina dan rawatan haba terhadap salutan, selain menilai kelakuan karatan salutan sol gel  $\text{Al}_2\text{O}_3$ -GNPs pada substrat nikel. Sintesis  $\text{Al}_2\text{O}_3$ -GNPs telah berjaya dijalankan untuk mengkaji kesan kepekatan alumina dan rawatan haba yang pelbagai terhadap tingkah laku karatan pada salutan. Untuk pencirian salutan, mikroskopi elektron imbasan pancaran medan (FESEM), mikroskopi elektron imbasan (SEM), pembelauan sinar-X (XRD) dan termogravimetrik (TG) telah digunakan. Tingkah laku karatan sampel bersalut dikaji menggunakan spektroskopi impedans elektrokimia (EIS) dan ujian pengutuban dinamik upaya (PDP). Analisis melalui FESEM, menunjukkan zarah bersaiz nano muncul dibeberapa bahagian pada permukaan salutan disebabkan kesan kepekatan alumina yang boleh dilihat daripada keliangan dan penggumpalan kerana pemecahan kelikatan sol gel pada permukaan salutan. Oleh itu, kehadiran grafin boleh berupaya memangkin tindak balas pengawetan dan berperanan sebagai penstabil haba. Plot Nyquist dari EIS mendedahkan lengkung separuh bulatan untuk semua sampel. Lengkung jenis ini menunjukkan pemindahan cas aktif antara salutan dan larutan sodium klorida ( $\text{NaCl}$ ). Rintangan pemindahan cas tertinggi bagi salutan  $\text{Al}_2\text{O}_3$ -GNPs diperolehi pada kepekatan 0.01 M melalui rawatan haba pada  $900^\circ\text{C}$  akibat daripada penggumpalan kecil dan pengasingan zarah nano pada permukaan salutan. Analisis PDP mendapat kepekatan alumina pada 0.1 M dengan suhu rawatan haba  $900^\circ\text{C}$  menunjukkan kadar karatan terendah iaitu  $4.31 \times 10^{-7}$  mm setahun. Jadi, nilai rendah ketumpatan arus karatan sampel ini menunjukkan tingkah laku kepasifan. Oleh itu, penggabungan grafin dengan salutan seramik alumina menggunakan teknik sol gel meningkatkan rintangan karatan pada substratum nikel. Melihat potensi  $\text{Al}_2\text{O}_3$ -GNPs, salutan ini boleh digunakan untuk pelbagai aplikasi dalam industri kejuruteraan khususnya dalam sektor pembuatan dan elektronik dimana nikel digunakan secara meluas.

## ABSTRACT

Alumina ( $\text{Al}_2\text{O}_3$ ) is one of the most significant ceramic materials that is well known of its outstanding mechanical and thermal properties and useful for industrial purposes, especially as ceramic coating. Alumina coating served as a protection against corrosion by preventing the underneath layer from interacting with corrosive environment. Nickel is usually known for having high corrosion resistance towards caustic solution which is also where the usage of nickel is commonly found. However, nickel can corrode in an unusual way that jeopardise its function especially in the presence of chloride ion. Even when shielded by passive layers, nickel surfaces are still prone to localised corrosion. Therefore, the alumina coatings can be incorporated with graphene to improve the ceramic matrix to provide further protection against corrosion. This study aims to synthesise alumina-graphene nanoplatelets ( $\text{Al}_2\text{O}_3$ -GNPs) produced by sol gel method, to investigate the influence of the concentration of alumina and heat treatment on the coating and to evaluate the corrosion behaviour of the  $\text{Al}_2\text{O}_3$ -GNPs sol gel coated nickel substrate. The synthesis of  $\text{Al}_2\text{O}_3$ -GNPs has been successfully carried out to study the effect of different concentration of alumina and heat treatment on the corrosion behaviour on the coating. To characterise the coating, field emission-scanning electron microscope (FESEM), SEM, X-ray diffraction (XRD) and thermogravimetric (TG) tests were used. The corrosion behaviour of the coated samples is studied using electrochemical impedance spectroscopy (EIS) and potentiodynamic polarisation (PDP). The analysis through FESEM, showed that the nanoparticles appeared at some part on the surface of the coating due to the effect of concentration of alumina which can be seen from the porosity and agglomeration causes by the abruptness of the viscosity of sol gel on the surface of the coating. Taking that into account, the presence of graphene can be interpreted in terms of its capability to catalyse the curing reaction and its role as a thermal stabiliser. The Nyquist plot from EIS revealed the half semi-circle curve for all samples. This kind of curve indicating the active charge transfer between the coating and the sodium chloride ( $\text{NaCl}$ ) solution. The highest charge transfer resistance of  $\text{Al}_2\text{O}_3$ -GNPs coating at concentration of 0.01M at 900 °C as a consequence of minor agglomeration and segregation of nanoparticles on the coating surface. The PDP analysis found that the concentration of alumina-GNPs at 0.1 M with heat treatment temperature of 900 °C exhibited the lowest corrosion rate at  $4.31 \times 10^{-7}$  mm/year and the sample indicated a brief passivation behaviour. Thus, the incorporation of graphene with alumina ceramic coating using sol gel technique increases the corrosion resistance on the nickel substrate. Considering the potential of  $\text{Al}_2\text{O}_3$ -GNPs, it is possible to apply this coating on various application in the engineering industry specifically in manufacturing and electronics where the nickel is widely used.

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