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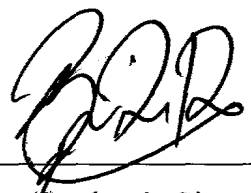
I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.



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SYNTHESIS AND CHARACTERISATION OF GRAPHENE AND ITS CORROSION RESISTANCE ON MAGNESIUM ALLOYS

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ABSTRAK

Magnesium sebagai salah satu logam struktur paling ringan amat digunakan dalam banyak aplikasi. Sifat biodegradasinya yang baik dan sifat mekanikalnya, aloi magnesium dianggap sebagai bahan yang ideal untuk stent kardiovaskular. Kemerosotan pesat dalam persekitaran sekeliling manusia dan bio-kompatibiliti yang lemah telah mengehadkan penggunaannya untuk biomaterial tersebut. Struktur magnesium yang padat dengan bentuk heksagonnya telah menurunkan kemulurannya dan perlindungan karat yang rendah dalam elektrolit akueus. Bagi memaksimumkan perlindungan karat magnesium dan biokompatibilitinya, terdapat kaedah celupan telah dilakukan ke atasnya dengan membina lapisan multilayer. Kaedah perlindungan lapisan karat ini merupakan suatu kaedah yang mudah dan sangat menguntungkan. Pada masa kini, grafin telah muncul dan dikenali sebagai salah satu bahan terkuat, nipis, dan paling ringan di dunia yang sangat sesuai digunakan sebagai bahan lapisan untuk meningkatkan ketahanan karat. Terdapat banyak kaedah yang boleh digunakan dalam menghasilkan grafin tetapi tidak semua kaedah tersebut mampu menghasilkan grafin dalam kuantiti yang banyak. Kaedah yang digunakan untuk pengeluaran skala besar grafin berkualiti biasanya memerlukan kos yang tinggi, penyediaan yang merbahaya dan sukar dan memerlukan tenaga yang tinggi. Pengelupasan elektrokimia dipercayai salah satu kaedah yang berpotensi untuk mengatasi masalah ini kerana ia mudah dilakukan dengan kos rendah dan tidak merbahaya. Tujuan kajian ini adalah untuk menilai kesan pelarut yang berlainan dalam proses sonikasi dalam pembentukan grafin dari pengelupasan grafit elektrokimia. Secara ringkasnya, rod grafit diceraikan menjadi serpihan kecil oleh proses elektrokimia dengan amonium sulfat sebagai elektrolit garam. Serbuk tersebut kemudian diletakkan di bawah dua pelarut yang berbeza, iaitu dimethylformamide (DMF) dan larutan berair untuk membentuk grafin. Keputusan mendapati bahawa struktur grafin ini serupa diperolehi daripada kedua-dua jenis pelarut. Struktur mikro yang terkelupas telah dilihat secara teliti di bawah pencirian Mikroskop Imbasan Elektron (FESEM) dan Pencirian Mikroskopi Elektron Transmisi (TEM). Grafin yang berjaya dihasilkan kemudiannya melapisi magnesium dengan menggunakan kaedah celupan bagi menguji sifat-sifat karat. Walau bagaimanapun, jumlah grafin yang dihasilkan berbeza kerana perbezaan dalam kestabilan grafin. Grafin lebih stabil dalam pelarut DMF berbanding larutan berair yang didapati dalam ujian spektroskop UV-Vis. Larutan akues dapat membolehkan tindak balas yang dapat diubah yang dapat mengubah grafit oksida dari grafin akibat adanya komponen hidroksil dalam larutan berair. Pelarut DMF digunakan untuk membantu dalam mencapai objektif kedua dengan mengkaji kesan salutan grafin terhadap sifat karatan magnesium. Lengkung pembelauan potensiodinamik potensiometer menunjukkan bahawa lapisan grafin telah meningkatkan ketahanan karat pada aloi magnesium. 7 lapisan grafin memberikan hasil kadar ketahanan karat yang ideal dengan jumlah 0.2944 mA/cm^2, sementara 5 lapisan dan 9 lapisan masing-masing menunjukkan 2.234 mA/cm^2 dan 0.9940 mA/cm^2. Kesimpulannya, hasil penyelidikan ini dapat membuktikan bahawa lapisan grafin pada aloi magnesium AZ31B mampu meningkatkan lapisan dan kakisan magnesium itu sendiri.

ABSTRACT

Magnesium is one of the lightest structural metal is greatly used in many applications. Due to its good biodegradability and mechanical properties, magnesium alloys are considered as the ideal candidate for the cardiovascular stents. The rapid degradation in human physiological environment and the poor biocompatibility seriously limit its application for biomaterials. Its hexagonal close packed structure of magnesium has lowered the ductility of it and it is poor corrosion resistance in aqueous electrolyte. In order to maximize the corrosion resistance of magnesium, some research had be done on it as multilayer coating has carry out by dip coating method. Coating a layer of high corrosion resistance material on it is one of the most simple and economic way. Graphene has emerged and known as one of the strongest, thinnest, and lightest material in the world which is very suitable used as a coating material to improve corrosion resistance of magnesium. There are many methods can be used in synthesising graphene but not all could be used in producing it in bulk quantities. The method used for huge scale of high quality graphene production usually demands high costing, high-skilled and requires high energy. Electrochemical exfoliation is believed to be one of the potential method to overcome this problem as it is easy to conduct at low. The aim of this study is to evaluate the effect of different solvents in sonication process on the formation of graphene from the electrochemical exfoliation of graphite. Graphite rod was exfoliated by electrochemical process with ammonium sulfate as salt-electrolyte. The exfoliated powder then sonicated under two different solvents, which are dimethylformamide (DMF) and aqueous solution to form graphene. Both type of solvent produced similar structure of graphene. The microstructures of exfoliated graphene were carefully characterized under field emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM) characterization. The successful graphene produced was then coated by using dip-coating method to test the corrosion properties of magnesium. The amount of graphene produced was different due to the differences in graphene stability. Graphene is more stable in DMF solvents compared to the aqueous solution as resulted in UV-Vis Spectroscope. The aqueous solution can allow the reversible reaction that can reform graphite oxide from graphene due to the existence of hydroxyl component in the aqueous solution. The DMF solvents used to help in achieving the second objective by study the effect of graphene coating on corrosion properties of magnesium. The potentiodynamic polarization curve showed that the coating of graphene had improved the corrosion resistance of magnesium alloy. The 7 layers of coating presents an ideal result of corrosion rate with 0.2944 mA/cm^2, while 5 layers and 9 layers shows 2.234 mA/cm^2 and 0.9940 mA/cm^2 respectively. In conclusion, this research was able to prove that the graphene coating on the magnesium AZ31B alloy was able to increase the graphene coating and corrosion properties of the magnesium itself.

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