UNIVERSITI PUTRA MALAYSIA

INVESTIGATION OF FLOATING CATALYST CVD FOR DEVELOPMENT OF CNT-COATED CARBON FIBRE REINFORCED COMPOSITE

SITI NORAZIAN BINTI ISMAIL

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By

SITI NORAZIAN BINTI ISMAIL

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

April 2010

DEDICATION

With gratitude for their love, support, and guidance:
I dedicate this thesis to my beloved family and friends
with love.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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Chair : Dr. Suraya binti Abdul Rashid

Faculty : Engineering

Composites produced from untreated carbon fibres result in poor mechanical properties. To increase the bonding between the carbon fibres and the matrix, the surface of the carbon fibres has been coated by carbon nanotubes (CNTs) through CNT-coated carbon fibre treatment process using a floating catalyst chemical vapour deposition (FCCVD) technique. This treatment was carried out with the presence of ferrocene and benzene as the catalyst and carbon precursor. By varying the technique of floating catalyst used to introduce ferrocene into the CVD furnace, it will influence the growth of CNT on the carbon fibre surface and hence influence the strength of the resulting carbon fibre composite. In the present investigation, two types of floating catalyst techniques were explored with different approaches in the way in which the ferrocene was introduced inside the furnace. Each technique was applied by varying two selected parameters which were the reaction temperature and the amount of ferrocene concentration. The reaction temperature was fixed at 700 and 800^{o}C meanwhile the amount of ferrocene was varied between 0.2, 0.5 and 1.0g. Technique 1 was to introduce the vapourized ferrocene and benzene into the furnace by the hydrogen as the carrier gas. Through this, only less CNTs were grown with

the presence of carbonaceous products. Meanwhile, in technique 2, the ferrocene was dissolved in liquid benzene before the mist of solution being introduced from outside of the furnace using hydrogen gas. The densely packed pure CNT grown by technique 2 was totally covered the structures of the carbon fibres. This directly affected the increase in surface area of the carbon fibre itself which gave an indication of good adhesion between the carbon fibre and the polypropylene (PP) matrix during the fabrication of composite. The morphology of the carbon fibres after the treatment was analysed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) before being incorporated into the composites. Meanwhile, the energy diffraction x-ray (EDX) characterisation was used to analyze the chemical composition on CNT grown by both techniques. Tensile tests were performed to evaluate the effectiveness of both techniques on the mechanical properties of the composites. The tensile strength and modulus from technique 2 improved by 64% and 109% respectively, compared to the composite made using the untreated carbon fibres. These values were two times higher than tensile properties obtained through composites made from CNT-coated carbon fibre synthesized using technique 1 which recorded 33 and 44% increment in tensile strength and modulus properties.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Sarjana Sains

KAJIAN PEMANGKIN APUNGAN CVD DALAM PEMBANGUNAN KOMPOSIT TETULANG GENTIAN KARBON BERSALUT KARBON TIUB NANO (CNT)

Oleh

SITI NORAZIAN BINTI ISMAIL

April 2010

Pengerusi : Dr. Suraya binti Abdul Rashid

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Komposit yang diperbuat daripada gentian karbon tidak dirawat memberikan sifat-sifat mekanikal yang rendah. Bagi meningkatkan ikatan antara gentian karbon dan matriks, permukaan gentian karbon telah disalut dengan karbon tiub nano (CNT) melalui proses rawatan gentian karbon bersalut CNT dengan menggunakan teknik pemangkin apungan pengendapan wap kimia (FCCVD). Rawatan ini dijalankan dengan kehadiran ferosena dan benzena sebagai pelopor pemangkin dan karbon. Dengan mengubah teknik pemangkin apungan yang digunakan untuk memperkenalkan ferosena ke dalam relau CVD, ini akan mempengaruhi pertumbuhan CNT pada permukaan gentian karbon dan oleh itu mempengaruhi pula kekuatan komposit gentian karbon. Dalam kajian ini, dua teknik pemangkin apungan telah diterokai dengan pendekatan yang berbeza iaitu cara di mana ferosena diperkenalkan ke dalam relau. Rawatan ini dilakukan dengan mempelbagaikan dua parameter yang terpilih iaitu suhu tindak balas dan jumlah kepekatan ferosena. Suhu tindak balas ditetapkan pada 700 dan 800^{o}C manakala jumlah ferosena diubah antara 0.2, 0.5 dan 1.0g. Teknik 1 adalah dengan memperkenalkan ferosena dan benzena terwap ke dalam CVD melalui gas hidrogen sebagai gas pembawa. Melalui teknik

ini, hanya sedikit CNT telah tumbuh dengan kehadiran produk berkarbon. Manakala, teknik 2 memerlukan ferosena dilarutkan ke dalam cecair benzena sebelum kabus larutan ini diperkenalkan dari luar relau menggunakan gas hidrogen. CNT yang tumbuh dengan bersih dan padat melalui teknik 2 telah menyaluti seluruh struktur gentian karbon secara total. Ini memberi kesan secara langsung kepada peningkatan luas permukaan gentian karbon itu sendiri dengan memberi indikasi lekatan yang baik antara gentian karbon dengan matriks polipropilena (PP) semasa fabrikasi komposit. Morfologi gentian karbon selepas rawatan telah dianalisa menggunakan mikroskop imbasan elektron (SEM) dan mikroskop penghantaran elektron (TEM) sebelum difabrikasi ke dalam bentuk komposit. Manakala, pencirian tenaga sebaran x-ray (EDX) digunakan untuk menganalisa komposisi kimia CNT yang tumbuh melalui kedua-dua teknik. Ujian tegangan dilakukan untuk menilai keberkesanan kedua-dua teknik terhadap sifat-sifat mekanikal bahan komposit. Kekuatan tegangan dan modulus daripada teknik 2 telah meningkat sebanyak 64% dan 109% masing-masing, berbanding dengan komposit yang diperbuat menggunakan gentian karbon tidak dirawat. Nilai ini adalah dua kali lebih tinggi berbanding sifat-sifat tegangan yang diperolehi melalui bahan komposit diperbuat daripada gentian karbon disalut CNT yang disintesis menggunakan teknik 1 yang hanya merekodkan 33 dan 44% peningkatan dalam sifat kekuatan tegangan dan modulus.

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I certify that a Thesis Examination Committee has met on 23rd April 2010 to conduct the final examination of Siti Norazian binti Ismail on her thesis entitled "Investigation of Floating Catalyst CVD for Development of CNT-Coated Carbon Fibre Reinforced Composite" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science degree.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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Date: 15 July 2010

DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

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SITI NORAZIAN BINTI ISMAIL

Date:

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