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REINFORCEMENT OF ALUMINUM MATRIX BY CARBON NANO TUBES AND NANO ALUMINA USING POWDER METALLURGY METHOD

FARHAD OSTOVAN

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By

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

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Dedicated to

The soul of my Mother, my Father and my Brother

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

REINFORCEMENT OF ALUMINUM MATRIX BY CARBON NANO TUBES AND NANO ALUMINA USING POWDER METALLURGY METHOD

By

FARHAD OSTOVAN

June 2015

Chairman : Assoc. Prof Khamirul Amin Matori, PhD
Faculty : Institute of Advanced Technology

For the past few years, particulate reinforced aluminum matrix composites (AMCs) have attracted more attention in aerospace, automotive and military industries due to their outstanding mechanical properties including high strength to weight ratio, good wear resistance, good environmental resistance as well as lower costs of production. Besides, AMCs are attractive in engineering applications for weight critical applications which may assist in energy saving along with reduction in the cost. With its low density, aluminum (Al) is a candidate of interest, but further strengthening is needed. The strengthening and development of aluminum has become a critical issue, because only a few materials have been proposed to the industry to strengthen the aluminum matrix. For those proposed materials, fabrication methods and their parameters in order to disperse reinforcements, especially nano reinforcements, within aluminum matrix are still under close scrutiny by researchers all around the world. Because of tangled nature of nano reinforcements, the dispersion of these reinforcements in aluminum matrix is a difficult task and a proper technique to fabricate nano AMCs is needed.

In this work, the benefits and limitations of adding Carbon Nano Tubes (CNTs) and Nano Alumina (n-Al_2O_3) to strengthen the aluminum matrix have been investigated with an emphasis on mechanical milling process and its effects on both nano reinforcement and aluminum matrix. Mechanical milling for different times of 0.5, 2, 5, 8 and 12 h was used to do mechanical alloying of different nano reinforcement contents with aluminum powder. Composite powders were then compacted and sintered at 530 °C. Micro-structural characterization of powders, grain refining analysis through XRD analysis, interfacial bonding assessment through micro structural observation of polished surface of sintered composites, the changes in the density and dimensions of sintered compacts as well as mechanical properties of Al-CNT and Al-Al_2O_3 composites were measured and compared.

Micro structural characterizations showed that, mechanical alloying via ball milling could homogenously disperse CNTs within aluminum matrix. Dispersion uniformity decreased with an increase in the amount of CNTs from 2 wt% up to 5 wt% and 10 wt%. However, further increasing the time of milling caused damage to the CNTs structure and formation of Al_4C_3 phase. On the other hand, n-Al_2O_3 dispersed homogenously within aluminum matrix even after adding up high amount of this nano

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reinforcement to the matrix. Furthermore, mechanical milling offered the advantages of aluminum strengthening through grain refinement and strain hardening.

As it was expected for samples fabricated by powder metallurgy processing, nano composite compression and sintering have presented several challenges as it resulted in porous structures, dimension growth and lack of bonding due to the sintering process. Results showed that nano composites which were milled for longer times had higher density after sintering. A minimum densification of 89% was achieved for all specimens. However, attempts to produce dense parts of Al-10CNTs failed due to excessive increase in the dimensions in the presence of CNTs clusters and agglomerations.

Micro and nano hardness and Young's modulus of Al-CNTs and Al-Al_2O_3 nano composites as well as compression properties of Al-Al_2O_3 were measured. Comparison of the results with the previous studies indicated that higher hardness and Young's modulus were obtained from the addition of CNTs versus n-Al_2O_3. On the other hand, in the case of compressive strength of nano composites, an increase in the n-Al_2O_3 resulted in an increase in compressive stress at break point of 689 MPa, where a homogenous dispersion of nano reinforcement was observed.

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Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

MENGUKUHKAN MATRIKS ALUMINIUM DENGAN CARBON NANO TUBES DAN NANO ALUMINA MENGGUNAKAN KAEDAH METALURGI SERBUK

By

FARHAD OSTOVAN

Jun 2015

Pengerusi : Prof Madya Khamirul Amin Matori, PhD
Fakulti : Institut Teknologi Maju

Untuk beberapa tahun kebelakangan ini, komposit matriks aluminium (AMCs) yang diperkukuh zarah telah menarik perhatian lebih dalam aeroangkasa, automotif dan industri ketenteraan kerana sifat mekaniknya yang cemerlang termasuk kekuatan yang tinggi berbanding nisbah beratnya, rintangan haus yang baik, rintangan alam sekitar yang baik serta kos pengeluarannya yang lebih rendah. Selain itu, AMCs mempunyai aplikasi kejuruteraan yang menarik untuk aplikasi berat yang kritikal di mana ia mungkin dapat membantu dalam penjimatan tenaga bersama-sama dengan pengurangan kos. Dengan ketumpatannya yang rendah, aluminium (Al) adalah calon yang menarik, tetapi pengukuhan lanjut adalah diperlukan. Pengukuhan dan pembangunan aluminium telah menjadi satu isu yang kritikal, kerana hanya beberapa bahan yang telah dicadangkan kepada industri untuk pengukuhan matriks aluminium. Untuk bahan-bahan yang dicadangkan, kaedah fabrikasi dan parameter bagi penyerakan/penyebaran bahan pengukuhan, terutama nano bahan pengukuhan, dalam matriks aluminium masih dalam penelitian rapi oleh penyelidik di seluruh dunia. Ini kerana sifat kusut nano bahan pengukuhan, penyebaran bahan pengukuhan ini dalam matriks aluminium adalah tugas yang sukar dan teknik yang betul untuk menghasilkan nano AMCs adalah diperlukan.

Dalam karya ini, manfaat dan had penambahan Carbon Nano Tubes (CNTs) dan Nano Alumina (n-Al_2O_3) untuk mengukuhkan matriks aluminium telah dikaji dengan penekanan kepada proses pengilangan mekanik dan kesannya ke atas kedua-dua bahan pengukuhan nano dan matriks aluminium. Masa yang berlainan untuk pengilangan mekanik 0.5, 2, 5, 8 dan 12 jam telah digunakan untuk melakukan pengaloian mekanik yang berbeza kandungan nano pengukuhan dengan serbuk aluminium. Serbuk komposit tersebut kemudiannya dipadatkan dan dibakar pada suhu 530 °C. Ciri-ciri mikro-struktur serbuk, penganalisisan saiz butiran melalui kaedah XRD, penilaian ikatan antara muka melalui pemerhatian struktur mikro permukaan komposit tersinter yang digilap, perubahan ketumpatan dan dimensi bahan tersinter serta sifat mekanik Al-CNT dan Al-Al_2O_3 komposit diukur dan dibandingkan.

Pencirian struktur mikro menunjukkan bahawa, pengaloian mekanik melalui bola pengilangan boleh menyebarkan CNTs secara seragam dalam matriks aluminium. Dan ia menurun dengan peningkatan dalam jumlah CNTs dari 2 wt% seterusnya 5 wt% dan

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10 wt%. Walau bagaimanapun dengan meningkatkan lagi masa pensilangan akan menyebabkan kerosakan kepada struktur CNTs tersebut dan pembentukan fasa Al_4C_3. Sebaliknya, n-Al_2O_3 tersebar secara seragam dalam matriks aluminium walaupun dengan peningkatan yang tinggi bahan pengukuhan nano ini pada matriks. Tambahan pula, pengilangan mekanik memberikan kelebihan bagi pengukuhan aluminium melalui penghalusan butiran dan pengerasan terikan.

Seperti yang telah dijangkakan, untuk sampel dihasilkan melalui proses serbuk metalurgi, mampatan nano komposit dan pensinteran telah memberikan beberapa cabaran kerana ia menyebabkan struktur berliang, pertumbuhan dimensi dan kekurangan ikatan kerana proses pensinteran. Keputusan menunjukkan bahawa komposit nano yang telah dikisar untuk jangka masa yang lebih lama mempunyai ketumpatan yang lebih tinggi selepas pensinteran. Ketumpatan minimum sebanyak 89% telah dicapai untuk semua spesimen. Walau bagaimanapun, percubaan untuk menghasilkan bahagian berketumpatan tinggi Al-10CNTs telah gagal disebabkan oleh peningkatan yang berlebihan dalam peningkatan dimensi dengan kehadiran kelompok CNTs dan gumpalan.

Kekerasan mikro dan nano dan modulus Young komposit nano Al-CNTs dan Al-Al_2O_3 serta sifat mampatan Al-Al_2O_3 telah diukur. Perbandingan hasil kajian ini dengan keputusan hasil kajian yang lain menunjukkan bahawa kekerasan yang lebih tinggi dan modulus Young diperoleh daripada penambahan CNTs berbanding dengan n-Al_2O_3. Sebaliknya, dalam hal kekuatan mampatan komposit nano, peningkatan n-Al_2O_3 menyebabkan kekuatan mampatan yang lebih tinggi di mana penyebaran homogen bahan nano pengukuhan diperoleh.

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I certify that a Thesis Examination Committee has met on 01 June 2015 to conduct the final examination of FARHAD OSTOVAN on his thesis entitled “Reinforcement of aluminum matrix by Carbon nano tubes and nano Alumina using powder metallurgy method” in accordance with the Universities and Universiti College 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 doctor of philosophy.

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

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LIST OF ABBRAVIATIONS

Al	Aluminum
CNTs	Carbon Nano Tubes
MWCNTs	Multi Wall Carbon Nano Tubes
SWCNTs	Single Wall Nano Tubes
EDX	Energy-Dispersive X-ray Spectrometer
n-Al_2O_3	Nano Alumina
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
XRD	X-Ray Diffraction
FESEM	Field Emission Scanning Electron Microscopy
PDC	Physical Dimensional Changes

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CHAPTER ONE

INTRODUCTION

1.1 Introduction

Nano materials have been scrutinized by many researchers since their discovery. It has opened vast zones of exploration which also incorporate nano scale reinforcement in composites in order to enhance their mechanical, thermal and even electrical properties. Although most research done on nanotubes is focused on polymer based composites (Matthews et al., 1999; Suryanarayana and Nasser, 2013), the unique properties of carbon nanotubes (CNTs) and nano alumina (n-Al_2O_3) can also be exploited in metal matrix composite (MMC) like aluminum matrix. But even after the discovery of CNTs and nano alumina, their full potential in the field of reinforcement of a metal matrix has not been realized yet which request more research in this field.

Reinforcing of a ceramic or a metal matrix by nano reinforcements can create composites possessing high strength, toughness, hardness and high creep resistance (Shankar et al., 2013; Knowles et al., 2014). Only a small amount of these nano reinforcements can increase mechanical, thermal and electrical conductivity of the composites which are vastly used in the applications like structural applications, aerospace and auto motive parts, electrical igniters and thermal shock resistance elements (Knowles et al., 2014; Chen et al., 2013).

Today, aluminum is the most widely used nonferrous metal. The main properties contributing to its widespread use are lightness, resistance to corrosion, low toxicity, form ability and electrical and heat conductivity. Among those, its low density has attracted the aerospace and automotive industries use in lightweight vehicles. The transportation industry is now the largest user of aluminum (Estruga et al., 2013). Transport structural applications, aerospace and automotive industries require materials with high specific strength and stiffness. With its low density, aluminum is a candidate of interest, but further strengthening is useful. Particulate reinforced aluminum composites are more marketable due to moderate fabrication cost, easier manufacturing and convenience to use standards on metal working methods (Shankar et al., 2013).
On the other hand, a range of processes is available to fabricate Metal-Matrix Composites (MMCs). These processes typically involve liquid or solid state manufacturing processes. Liquid state techniques include squeeze casting, spray deposition, slurry casting and reactive processing. The presence of liquid raises the issues of possible interfacial reaction layer formation, reinforcement wettability and reinforcement agglomeration. The solid state manufacturing processes mainly involve the powder metallurgy route. While being typically more complex and expensive, powder metallurgy can prevent the problems associated with liquid state techniques (Kainer, 2006).

The most problematic part of these methods is the blending part. The objective is to disperse homogeneously the reinforcement in the matrix powder and to remove all clustering. Clustering of reinforcement is to be avoided because it leads to a greater

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plastic strain in the region of clusters and promotes the formation of voids, thus reducing the resistance to fracture and fatigue properties (Knowles et al., 2014). Mechanical alloying of reinforcement and matrix is proposed to be a suitable dealing with blending part. In this area, ball milling can hinder the problems associated with nano scale reinforcements such as clustering.

There is a necessity of exchange techniques for manufacturing the composites due to the damage of nanotubes because of the existence of a high temperature and a very high reactive environment in the fabrication of MMCs by conventional methods. The interface between reinforcements and metal matrix should be also controlled. Interfacial bonding of them can be favored in order to increase the mechanical properties of the composite (Knowles et al., 2014). On the other hand, controlling the powder metallurgy parameters such as sintering temperature, furnace atmosphere, compression process and further heat treatment of parts can affect the whole mechanical properties of aluminum composite reinforced by CNTs or nano alumina. At the same time, the examination is still in the primary stage and there should be more tests to be done before it is prepared for industrial utilization.

This research investigates the effects of addition of CNTs and nano alumina on mechanical properties of an aluminum matrix. Ball milling process is used in order to disperse these nano reinforcements within Al matrix. Characterization of obtained samples is extensively done to have better understanding of dispersion. All possible parameters involved in powder metallurgy like dimensional changes, density and hardness have been considered and reported.

1.2 Problem statement

The strengthening and development of aluminum has become a critical issue, because only a few materials have been proposed to the industry to strengthen the aluminum matrix. For those proposed materials, fabrication methods and their parameters in order to disperse reinforcements, especially nano reinforcements, within aluminum matrix are still under close scrutiny by researchers all around the world. Because of tangled nature of nano reinforcements, the dispersion of these reinforcements in aluminum matrix (especially in the case of high weight fraction of nano reinforcement) is a difficult task (Knowles, 2014; Estruga et al., 2013). It may even cause a decrease in mechanical properties of aluminum (Estruga et al., 2013; Suryanarayana and Nasser, 2013). Clustering of reinforcements is a challenging issue in aluminum composites. The dispersion method can deeply affect final properties and this requires a research dealing with it.

Examples of available reinforcements for aluminum are silicon carbide (SiC), graphite and alumina (Al_2O_3). This is due to high availability, low cost and overall good properties. Based on previous research, carbon nano tubes (CNTs) and nano alumina (n-Al_2O_3) could be two important and suitable alternatives reinforcements identified by several researchers for aluminum (Esawi et al., 2008; Aghajanian et al., 1993). Studying the results from previous research indicates that there are several parameters, involving in reinforcement of aluminum matrix via CNTs and nano alumina using the powder metallurgy route, which demands a more elaborated research dealing with it. However, by using mechanical alloying as a dispersion method, the dispersion uniformity of CNTs and nano alumina in the aluminum matrix in the case of high weight fraction of nano reinforcement is not well discussed. On the other hand, a

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controlled powder metallurgy process can produce final parts with very accurate dimensions. Since the shape of reinforcements after ball milling can affect the density, hardness and even dimensions, a profound study of characterization and accurate analysis of powder metallurgy conditions is needed. In this scope, data collection about carbon nanotubes and nano alumina and their effects on the mechanical properties requires a deep study at the microstructure of every single stage of experiment.

Hypothesis may include the following:

- Doing ball milling could affect the dispersion of a non-metal reinforcement in a metal matrix.
- An adjusted ball milling process could do mechanical alloying.
- Initial powder size does not affect the milling.
- Addition of CNTs to the Al matrix increases the amount of hardness, Young modulus and yield strength.
- Addition of n-Al_2O_3 to the Al matrix increases the amount of hardness, Young modulus and yield strength.
- Powder metallurgy is a suitable method to produce Al-CNT and Al- n Al_2O_3 alloys.
- A homogenous microstructure of a well dispersed reinforcement in Al matrix will help the densification during sintering.

1.3 Objectives

This study embarks on the following objectives:

- To evaluate morphology changes of Al-CNT and Al-Al_2O_3 mixture composites as a function of milling process.
- To evaluate dispersion of CNTs and nano alumina, with an emphasis on mechanical milling process and its effect on both nano reinforcement and aluminum matrix.
- To evaluate powder metallurgy variations like density and physical dimensional changes of Al-CNT and Al-Al_2O_3 composites.
- To investigate the effect of CNTs and nano alumina on the mechanical properties of aluminum matrix.

The structure of this thesis is provided as following chapters:

Chapter 1 gives the introduction to the processes used, hypothesis and objectives of this project. Glancing through the ball milling as a dispersion method and an overview of powder metallurgy as a manufacturing method is included in the first chapter.

Chapter 2 discusses the literature review on the works done related to this thesis and includes the describing composites, aluminum composite, CNTs, n-Al_2O_3, ball milling, powder metallurgy and the latest work done related to this research work.

Materials and methods/methodology are brought in Chapter 3. Materials, machines, standards, the amount of each single sample, tools and method of characterization is described in this chapter. Theoretical approaches and experimental designs are included in Chapter 3.

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Result and discussion are included in Chapter 4. All findings about dispersion of CNTs and nano alumina in aluminum are separately elaborated. Powder metallurgy conditions are analyzed comprehensively in order to show their effects on mechanical properties of parts. Results about CNTs/Al and nano alumina/Al composites are compared in this chapter. Chapter 5 contains conclusions and recommendations for future studies.

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Relevant Publication to Thesis:

Farhad Ostovan, Khamirul Amin Matori, Meysam Toozandehjani, Way Foong Lim, Arshin Oskoueian, Hamdan Mohamed Yusoff, Robiah Yunus, Azmah Hanim Mohamed Ariff. Micro-structural Evaluation of Ball Milled Nano Al_2O_3 Particulate-Reinforced Aluminum Matrix Composite Powders, International Journal of Material Research (accepted).

Farhad Ostovan, Khamirul Amin Matori, Hock Jin Quah, Way Foong Lim, Meysam Toozandehjani, Arshin Oskoueian, Hamdan Mohamed Yusoff, Robiah Yunus, Azmah Hanim Mohamed Ariff. Effects of CNTs Content and Milling Time on Mechanical Behavior of MWCNT-Reinforced Aluminum Nanocomposites, Journal of Material Chemistry and Physics (Under review).

Farhad Ostovan, Khamirul Amin Matori, Hamdan Mohamed Yusoff, Robiah Yunus, Azmah Hanim Mohamed Ariff, Meysam Toozandehjani. Different processing methods to reinforce aluminum matrix by using carbon nanotubes -A review on basics and literature, Transcation of Indian Institute of Metals (under review).

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Non-relevant Publication to Thesis:

Farhad Ostovan, Khamirul Amin Matori, Hamdan Mohamed Yusoff, Robiah Yunus, Azmah Hanim Mohamed Ariff, Meysam Toozandehjani, Mohammad Meschian. Occurrence of pattern formation of microstructural, physical and mechanical properties of sintered PM steels containing pre-alloyed Astaloy E powder, Transcation of Indian Institute of Metal; DOI 10.1007/s12666-014-0411-x (IF:0.427).

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