Direct Nitridation of Aluminium and Fabrication of Aluminium-Aluminium Nitride Composite using Powder Metallurgy Route

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LIST OF ABBREVIATIONS

Abbreviations	Meaning
AL	Aluminium
AlN	Aluminium nitride
AMC	Aluminum Matrix Composite
CTE	Coefficient of Thermal Expansion
CVD	Chemical Vapour Deposition
EDS	Energy Dispersive Spectroscopy
MMC	Metal Matrix Composite
OFN	Oxygen Free Nitrogen
SEM	Scanning Electron Microscope
SHS	Self-propagating High-Temperature Synthesis
S-Q	Semi-quantitative analysis
TEM	Transmission Electron Microscope
XRD	X-ray Diffraction

Proses Nitridisasi Langsung Aluminium dan Fabrikasi Komposit Aluminium-Aluminium Nitrida menggunakan kaedah Metalurgi Serbuk

Abstrak

Sifat-sifat aluminium nitrida (AlN) yang istimewa seperti; takat lebur yang tinggi, rintangan haus dan kakisan yang baik, sela jurang jalur luas yang serupa dengan silikon, rintangan tinggi dan pekali pengembangan haba yang rendah terus menarik perhatian penyelidikan dalam beberapa dekad yang lalu. Penitradan langsung aluminium adalah salah satu kaedah perindustrian untuk mensintesis seramik ini tetapi kesukaran untuk mencapai 100% penukaran aluminium kepada aluminium nitrida (AlN) telah menjadi pemasalahan utama. Untuk mencari penyelesaian kepada masalah ini, serbuk aluminium dipanaskan pada kadar 20^{o}C/min dalam relau tiub keluli tahan karat pada suhu antara 650 hingga 950^{o}C dalam nitrogen mengalir pada kadar 1.0L/min untuk 4-6 jam. 5.0 dan 10.0wt% magnesium ditambah untuk memudahkan proses manakala gas hidrogen yang mengalir ke relau pada kadar 0.2L/min digunakan untuk mengekalkan tekanan oksigen yang rendah. Mikroskop imbasan elektron (SEM) dan Pembelauan sinar-X (XRD) digunakan untuk menganalisis serbuk nitridasi. Masalah eksudasi bengkak dan cecair semasa penghasilan komposit Al-AlN menggunakan laluan metalurgi serbuk juga dilaporkan. Untuk mencari punca dan penyelesaian kepada masalah ini, komposit Al-AlN yang terdiri daripada tetulang 0 - 16.25% AlN dan 0 - 2.0% Mg dipadatkan menggunakan beban 3.8 - 5.0 Mpa. Padatan anum disinter menggunakan teknik pensinteran dua langkah biasa dalam atmosfera nitrogen dan hidrogen pada suhu antara 600 dan 650^{o}C selama 30 - 90 minit. Padatan Al-AlN yang dihasilkan diuji untuk pelbagai ujian. Keberintangan / kekonduksion elektrik diukur dengan menggunakan kuar empat titik; manakala pekali pengembangan haba (CTE) diukur dengan menggunakan dilatometer pada suhu 30 hingga 450^{o}C pada kadar pemanasan 10^{o}C/min dalam suasana nitrogen. Kadar kakisan Al-AlN ditentukan dengan rendaman dalam 0.05M NaOH selama 24 jam. Keputusan XRD menunjukkan bahawa serbuk aluminium yang mengandungi 10% Mg memberikan penukaran 100% dari Al ke AlN pada suhu 950^{o}C selepas 5 jam proses nitridisasi. Penyebab utama bengkak dalam padat adalah disebabkan oleh proses pemeluwapan yang telah dihapuskan oleh pensinteran dua langkah. Faktor-faktor yang menggalakkan eksudasi cecair semasa pensinteran dikenal pasti sebagai: kehadiran magnesium dan cecair eutektik suhu rendah, suhu sintering yang tinggi, masa sintering yang panjang dan penggunaan atmosfera hidrogen (tidak aktif) semasa sintering. Nilai konduktiviti elektrik meningkat dengan pecahan berat AlN dan magnesium ditambah. Nilai konduktiviti elektrik tertinggi yang diperolehi ialah 131.326$(\Omega m)^{-1}$ dalam padatan yang mengandungi Al-16.25wt%AlN-1.0wt%Mg. Keputusan CTE menunjukkan bahawa Al-6.5wt%AlN-0.5wt%Mg memberikan nilai terendah (terbaik) bersamaan dengan 10.60x10^{-6}K^{-1} pada suhu 50^{o}C dan 20.91x10^{-6}K^{-1} pada 450^{o}C. Sampel terkakis menunjukkan bahawa produk kakisan utama adalah aluminium hidroksida, $Al(OH)_3$. Aluminium tersinter yang tidak diperkuat terhakis lebih daripada diperkuatkan dengan AlN. Padatan Al-16.25wt%AlN-1.0wt%Mg menawarkan rintangan kakisan terbaik dengan kadar terendah karat yang bersamaan dengan 22.74 mm/y.

Direct Nitridation of Aluminium and Fabrication of Aluminium-Aluminium Nitride Composite using Powder Metallurgy route

Abstract

The enviable attributes of aluminium nitride (AlN) such as; high melting point, good wear and corrosion resistance, wide band gap similar to that of silicon, high resistivity and low coefficient of thermal expansion have continued to attract research attention in the last few decades. Direct nitridation of aluminium is one of the industrial methods of synthesizing this ceramic but difficulty in achieving 100% conversion of aluminium to aluminium nitride (AlN) has been the major setback. In order to find solution to this problem, aluminium powder was heated at the rate of 20^oC/min in a stainless steel tube furnace at temperatures ranging from 650 to 950^0C in flowing nitrogen at the rate of 1.0L/min for 4-6 hours. 5.0 and 10.0wt% magnesium were added to facilitate the process while hydrogen gas flowing into the furnace at the rate of 0.2L/min was used to maintain low oxygen pressure. SEM and XRD were used to analyze the nitrided powders. Problems of swelling and liquid exudation during production of Al-AlN composite using powder metallurgy route have also been reported. In order to find the causes and remedies to these problems, Al-AlN composite consisting of 0 – 16.25%AlN reinforcement and 0 - 2.0%Mg were compacted using load of 3.8 - 5.0 tons. The green compacts were sintered using normal and two-step sintering techniques in nitrogen and hydrogen atmospheres at temperatures between 600 and 650^oC for 30 – 90 minutes. The Al-AlN compacts produced were used for various tests. The electrical resistivity/conductivity was measured using four point probes; while coefficient of thermal expansion (CTE) was measured using dilatometer at temperatures from 30 to 450^oC at heating rate of 10^oC/min in nitrogen atmosphere. Corrosion rate of Al-AlN compact was determined by immersion of the compacts in 0.05M NaOH for 24 hours. XRD results show that aluminium powder containing 10%Mg gave 100% conversion from Al to AlN at temperature of 950^oC after 5 hours nitridation. The main cause of swelling in the compacts was due to presence of volatiles which were eliminated by two-step sintering. The factors that promote liquid exudation during sintering were identified as: presence of magnesium and low temperature eutectic liquid, high sintering temperature, long sintering time and use of hydrogen (inactive) atmosphere during sintering. The values of electrical conductivities of the compacts increase with weight fractions of AlN and magnesium added. The highest value of electrical conductivity obtained was $131.326(\Omega m)^{-1}$ in compact containing Al-16.25wt%AlN-1.0wt%Mg. CTE results show that Al-6.5wt%AlN-0.5wt%Mg gave the lowest (best) values equivalent to $10.60 \times 10^{-6}K^{-1}$ at temperature of 50^oC and $20.91 \times 10^{-6}K^{-1}$ at 450^oC. Corroded samples show that the main corrosion product on the compacts was aluminium hydroxide, $Al(OH)_3$. Unreinforced sintered aluminium corroded more than compacts reinforced with AlN. Compact containing Al-16.25wt%AlN-1.0wt%Mg offered the best corrosion resistance with lowest value of corrosion rate equivalent to 22.74 mm/y.

CHAPTER 1: INTRODUCTION

1.0 Overview

Aluminium nitride, AlN, a non-oxide ceramic, was first discovered by Briegler and Geuther in 1862, but was first synthesize fifteen years later by J. W. Mallets (Mussler, Venigalla, Johnson, & Rudolph, 2000). It is an important ceramic material with high thermal conductivity, high melting point of about 2800^0C, low dielectric constant, low coefficient of thermal expansion, large band gap similar to that of silicon, low density, high intrinsic thermal conductivity, high electrical resistivity, low compressibility, high resistance to wear and corrosion, non-toxic, high resistance to chemical attack and thermal shock and good mechanical strength (Angappana, Jeneaferb, Visuvasama, & Berchmansa, 2013; Baik & Drew, 1996; Kim et al., 2015; Qiu & Gao, 2003; Smolen et al., 2013).

The attributes of AlN make it useful for various applications: The high melting temperature makes it thermally and chemically stable at higher temperatures that is suitable for use as refractory materials (e.g crucibles) since it is not affected by molten metal up to 2000^0C (Liu et al., 2016; Qadri, Gorzkowski, Rath, Feng, & Amarasinghe, 2017b; Vlado, 1964). It is used for fabrication of car engines through infiltration of porous performs and for fabrication of cutting tools (Abbasi, Shariat, & Javadpour, 2013; Baik & Drew, 1996; Hou, Mutharasan, & Koczak, 1995). Its wide band gap and good thermal properties make it applicable in the electronic industry as semi-conductor material especially as a substrate material for used in advanced circuit materials; it has also provided solution to problem of thermal matching between the substrate and semi-conductor material (La Spina et al., 2008; Yamakawa, Tatami, Komeya, & Meguro, 2006). The high thermal conductivity of AlN, which is said to be higher than that of copper above 200^{o}C, makes it suitable for use as heat sink for quick heat dissipation in electronic components, while the low coefficient of thermal expansion of aluminium nitride provides for dimensional stability of components used at elevated temperatures (Couturier, Ducret, Merle, Disson, & Joubert, 1997).

There are different methods of synthesizing AlN ceramic, but each of the methods has its advantages and shortcomings. For example, use of Si_3N_4 as source of nitrogen introduces Si into the product; while carbothermal method often introduces impurities such as carbon and cyanide (CN) which need to be removed to improve the purity of AlN synthesized; while direct nitridation is associated with untransformed aluminium (Baik & Drew, 1996; Xie, Cheng, & Huang, 2003). In this work, AlN ceramic has been produced using direct nitridation of aluminium powder; the difficulty in achieving 100% conversion from aluminium to aluminium nitride (Baik & Drew, 1996) has been addressed by introduction of magnesium into aluminium powder prior to nitridation. The presence of Mg in the powder creates pores in the powder mass as it vaporizes; hence enhancing diffusion of nitrogen into the aluminium powder mass and complete nitridation. The AlN obtained was used for fabrication of Al-AlN composites using powder metallurgy route.

Composite materials comprising of the matrix and strengthening or reinforcing phases such as ceramic particulates, whiskers, fibers or the like, show great promise for a variety of applications because they combine the strength and hardness of the strengthening phase with the ductility and toughness of the matrix to enhance their mechanical properties (Sharma, 2000; White, Urquhart, Aghajanian, & Creber, 1989).

The properties of composites depend on three factors (Biron, 2013): the matrix, reinforcement and the quality of bonding between the two components. Aluminum matrix composite (AMC), a form of metal matrix composite (MMC), is characterized by high specific strength and stiffness for engine parts and can contribute significantly to the reduction of the entire weight and fuel consumption of automobiles and aircrafts (Miracle, 2005; Surappa, 2003). When compared with unreinforced or monolithic aluminium metal, AMCs have high wear resistance, high dimensional and chemical stability and high temperature properties; and these properties could be streamlined to meet certain requirements (Aigbodion & Hassan, 2007).

Aluminium Matrix Composites are classified into: Particle-reinforced AMCs (PAMCs), Continuous Fibre-reinforced (CFAMCs), Whisker or short fibre-reinforced (SFAMCs) and Mono filament-reinforced (MFAMCs) (Surappa, 2003). Al-AlN composite, which is one of the main interests of this research, is an example of particle reinforced aluminium matrix composite (PAMCs). AlN reinforcement in Al matrix, however, has been found to offer working temperatures of Al-AlN composite up to the temperature of 350^0C compared with other ceramics such as SiC & Al_2O_3 in Al matrix whose working temperatures cannot be guaranteed above 250^0C due to their relatively lower values of coefficient of thermal expansions (Amosov, Titova, Timoshkin, & Kuzina, 2016). Problems of swelling and liquid exudation have been reported during fabrication of Al-AN compacts using powder metallurgy route. Swelling reduces the chances of achieving full densification during sintering while liquid exudation brings about loss in weight and excessive shrinkage, which affects the dimensional stability of the final sinter. These problems are being addresses in this work.

1.1 Background of the Study

Due to the relatively high cost of ceramic particles used as reinforcements, researchers' interests were, at one time, tilted toward the used of cheaper alternatives such as; ashes obtained from the controlled burning of agro-wastes such as sugarcane baggase, rice husk, coconut shell and bamboo leaf, all of which, contain Al_2O_3, SiO_2 and Fe_2O_3 (Aigbodion, Hassan, Dauda, & Mohammed, 2011; Aku, Yawas, & Adokma, 2013). These cheap alternatives have their own limitations and cannot replace the use of certain ceramic reinforcements no matter their costs. A good example is AlN ceramic which possesses unique properties that have continued to attract research attention in the last few decades.

With advancement in technology and new inventions coming up every day, it is now more challenging sourcing for new materials to meet new demands. With the discovery of AlN, many researchers have tried to take advantage of cheap and natural abundance of nitrogen in the air for nitridation of aluminium powder in order to produce this ceramic material. The nitrogen sources that have been used by different researchers for the purpose of nitridation of aluminium include gaseous sources: oxygen-free nitrogen (OFN) or pure nitrogen and ammonia gas (Jung & Ahn, 2000; Takeda et al., 2015; Yamakawa et al., 2006); and solid sources of nitrogen such as Si_3N_4 (Abbasi et al., 2013; Yeh & Liu, 2007), Mg_3N_2 (Guojun, Guangde, & Huiming, 2008) and Na_3N (Yamane, Shimada, & Disalvo, 1998). In this research, oxygen-free nitrogen (OFN) was used to produce AlN and as sintering atmosphere for fabrication of Al-AlN compacts.

Fabrication of Al-AlN composite using liquid-gas in-situ method has great potential to produce clean and high purity Al-AlN composite (Hou et al., 1995), but its high energy consumption coupled with low conversion of Al to AlN during this process and non-uniform distribution of the small amount of AlN formed in aluminium matrix are major concern. The use of powder metallurgy route to produce particles reinforced metal matrix composite is well known for its popularity. With this method it is possible to produce composite materials with more compositional flexibility than it is possible using the conventional melting and casting. Two major challenges associated with powder metallurgy route of fabricating Al-AlN compacts, as already mentioned, are problems of swellings and liquid exudation during sintering and are being addressed in this study. Details on swellings and liquid exudation during sintering have been elucidated in this study.

1.2 Problem Statement

One major problem of concern in this work is the in ability to achieve 100% conversion of aluminium to aluminium nitride during direct nitridation method of synthesizing AlN as reported by Kurokawa et al. (1985), Baik and Drew (1996), Nasery et al. (2011) and Abbasi et al. (2013). In this work, a means of achieving 100% conversion of Al to AlN has been employed by addition of magnesium powder into aluminium powder prior to nitridation. The magnesium added is expected to reduce any aluminium oxides in the powder. The excess magnesium is expected vapourise; hence creating pores in the aluminium powder mass through which nitrogen gas can gain access into the mass to ensure complete nitridation. This study will encourage local production of AlN ceramic by researchers in Malaysia rather than continuous importation of this same ceramic. A web search in Google scholar revealed that few of the authors who have used AlN powder for research in Malaysian Universities all imported AlN powder from Sigma Aldrich (Anithambigai, Mutharasu, Huong, Zahner, & Lacey, 2014; Daud & Wahab, 2012; N. Hadi et al., 2016; Ramli, Sulong, Arifin, Muchtar, & Muhamad, 2012; Shamsul & Zamri, 2011; Wahab, Daud, & Ghazali, 2009). In this work, direct nitridation of aluminium powder has been carried out not only to solve the perennial problem of incomplete nitridation of aluminium during the process, but also to encourage students to produce the ceramic locally instead of importation which usually takes two to three months for the order to arrive.

Problem of swellings of sintered compacts during sintering of Al-AlN compacts and other aluminium matrix composites have been reported (Schaffer et al., 2008; Showaiter & Youseffi, 2008; Tiwari, Rajput, & Srivastava, 2012). Some of the causes of swellings during sintering have been attributed to; presence of volatiles, too much pressure during densification, pore expansion, precipitation of intermetallic phases and due to thermal expansion (Coovattanachai et al., 2017; German, 2013; Molisani, Goldenstein, & Yoshimura, 2017; O'Flynn & Corbin, 2017; Soyama, Oehring, Ebel, Kainer, & Pyczak, 2017). Pre-heating of compact prior to densification as means of controlling swellings caused by entrapped gases and volatiles has been suggested (Tiwari et al., 2012). During pre-heating of powders, however, the initial characteristics of powders could be changed. In this work, two-step sintering has been employed to control swelling in addition to other sintering variables.

Liquid exudation is a phenomenon associated with powder metallurgy method of fabrication where by liquids are squeezed out and solidified on the surface of compact during sintering. This phenomenon has been reported during sintering of Al-AlN compacts using tin as an activator (Schaffer & Hall, 2002). During sintering of other particle reinforced metal matrix composites, liquid exudations have been reported

(Lange, 1982; Sautereau & Mocellin, 1974; Shu-dong et al., 2009; Xu, Upadhyaya, German, & Iacocca, 1999). Schaffer and Hall (2008) reported that poor wetting of reinforcement by liquid tin is the cause of liquid exudation. In order to eliminate this menace, magnesium was placed inside the furnace during the sintering process in order to free aluminium of its oxide and to increase wettability. The role of liquid phase sintering (LPS), which enhances full densification during sintering of compacts, has been reported (German, 2013; German, Suri, & Park, 2009; Schaffer, Sercombe, & Lumley, 2001). This means that the presence of liquid during sintering is an advantage, but when the liquid is allowed to accumulate and exude from the compact during sintering, then it becomes a problem. In this study, the factors that promote liquid exudation during sintering of Al-AlN containing magnesium have been investigate with the view to control sintering variables in the furnace and compositions of compacts in order to avoid liquid exudation.

Al-AlN compacts fabricated in this work have been used to investigate the mechanical and physical properties of these compacts with the view to determining the effect of weight fractions of reinforcement and magnesium on these properties. This will also provide literature fur other researchers as there are but a few literatures on mechanical and physical properties of Al-AlN composite produced using powder metallurgy route.

1.3 Objectives of the Research

a) To locally produce AlN by direct nitridation of aluminium powder in nitrogen atmosphere with the sole aim of achieving 100% conversion from aluminium to aluminium nitride using a novel approach. In this approach magnesium is added in to aluminium powder so that during the nitridation process, magnesium vaporizes and creates pores where nitrogen can easily diffuse into the powder to enhance complete conversion from aluminium to aluminium nitride.

b) To determine the possible causes of swelling during sintering of Al-AlN compacts and to find ways of preventing or minimizing it. This was achieved by controlling the sintering variables involved such as sintering atmosphere, sintering temperature and time and use of two-step sintering technique.

c) To investigate the factors promoting liquid exudation during sintering of Al-AlN compacts and to find the ways of controlling this menace by modifying sintering parameters. Liquid phase sintering is known for its use to enhance densification, but when liquid accumulates and start coming out the desired result cannot be guaranteed. The aim here is to control the sintering parameters in order to make the presence of liquid phase and advantage rather than a disadvantage.

d) To characterize composites produced using SEM and XRD and to test and examine the hardness and wear resistance of Al-AlN compacts produced and how these properties are affected by variations in weight fractions of AlN reinforcement whose attributes have shown to be promising in many areas of applications.

e) To investigate the influence of AlN ceramic on coefficient of thermal expansion and electrical conductivities of Al-AlN sinters. Also, to investigate the effect of corrosion resistant AlN ceramic on corrosion behavior of Al-AlN sinters in 0.5M NaOH. Since AlN is known to have low CTE, high electrical resistivity and