EFFECT OF SURFACE FUNCTIONALIZATION BY NaOH ON ELECTROPHORETIC DEPOSITION ON H-BN PARTICLES

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This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials) (Hons.)

by

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DECLARATION

I hereby, declared this report entitled "EFFECT OF SURFACE FUNCTIONALIZATION BY NaOH ON ELECTROPHORETIC DEPOSITION OF H-BN PARTICLES" is the results of my own research except as cited in references.

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfilment of the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Materials) (Hons.). The member of the supervisory committee is as follow:

……………………………………………………………………

(DR. LAU KOK TEE)

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TAJUK: **EFFECT OF SURFACE FUNCTIONALIZATION BY NaOH ON ELECTROPHORETIC DEPOSITION OF H-BN PARTICLES**

SESI PENGAJIAN: 2015/16 Semester 11

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ABSTRACT

H-BN has no functional groups such as amine and hydroxyl groups that can be used for bonding or interactions. For effective use of h-BN particles, surface functionalization is necessary for h-BN to functionalize h-BN by adding functional groups to its surface. The effectiveness of the surface functionalization is greatly depended on the processing parameters used during the surface functionalization of the particles. Thus, the optimum processing parameters such as treatment time is needed to be determined and to be refined in order to obtain functionalized h-BN particles which suitable for electrophoretic deposition (EPD) method.

Functionalization of hexagonal boron nitride (h-BN) particles was successfully achieved by treating raw h-BN particles with sodium hydroxide (NaOH) solution. The particles were treated with different period of time (6 hours, 12 hours, 24 hours) to study the effect of time on the functionalization of h-BN particles. Sedimentation tests was used to evaluate the effectiveness of the functionalization process of h-BN particle. Results clearly show that h-BN (Nova Scientific) suspensions have similar stability for the period of time studied where as h-BN (Showa Denko) displayed much lower stability. The functionalized h-BN particles were successfully deposited on targeted metallic substrate by using electrophoretic deposition (EPD) method. Different potential difference (160 V, 190 V, 220 V) were applied across the EPD electrodes. Optimum voltage which produces the highest yield coating and the functionality of poly(diallyldimethylammonium chloride) in this coating system.

ABSTRAK

H-BN tidak mempunyai kumpulan berfungsi seperti kumpulan amina dan hidroksil yang boleh digunakan untuk interaksi. Untuk penggunaan berkesan zarah h-BN, functionalisasi permukaan adalah penting bagi h-BN untuk memfungsikan h-BN dengan menambah kumpulan berfungsi pada permukaannya. Keberkesanan functionalisasi permukaan amat bergantung kepada parameter pemprosesan yang digunakan semasa functionalisasi permukaan zarah. Oleh itu, parameter pemprosesan optimum seperti masa rawatan akan ditentukan dan perlu diperhalusi untuk mendapatkan zarah h-BN berfungsi yang sesuai untuk kaedah pemendapan electrophoretic (EPD).

Functionalisasi zarah h-BN telah berjaya dicapai dengan merawat zarah h-BN mentah dengan NaOH proses. Zarah telah dirawat dengan tempoh masa yang berbeza (6 jam, 12 jam, 24 jam) untuk mengkaji kesan masa di functionalisasi zarah h-BN. Ujian pemendapan telah digunakan untuk menilai keberkesanan proses functionalisasi atas zarah h-BN, keputusan jelas menunjukkan bahawa h-BN (Nova Scientific) penggantungan mempunyai tidak berbeza dalam kestabilan bagi tempoh masa belajar di mana h-BN (Showa Denko) yang dipaparkan banyak kestabilan rendah berbanding dengan penggantungan h-BN (Nova Scientific). Zarah h-BN functionalisasi telah berjaya disimpan di logam substrat yang disasarkan dengan menggunakan EPD. Nilai-nilai beza keupayaan (160 V, 190 V, 220 V) telah digunakan di seluruh elektrod EPD untuk mencapai tujuan pengoptimuman. Dapatan kajian ini adalah voltan yang optimum yang boleh menghasilkan lapisan yang paling tinggi hasil dan fungsi PDADMAC dalam sistem salutan ini.

DEDICATION

I hereby dedicate this to

my beloved father, Lai Yoon On

my dearest mother, Lee Poh Yoke

and my two lovely sisters Lai Shuw Wei and Lai Shuw Kuan

last but not least my coolest brother Lai Chii Hou .

Thank you for giving me support and courage to finish years of education.

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Finally, I want to show great gratitude to my family for providing such warmth upbringing before stepping into society.

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LIST OF ABBREVIATIONS

BJT	-	Bipolar Junction Transistor
FET	-	Field-effect Transistor
BN	-	Boron Nitride
h-BN	-	Hexagonal Boron Nitride
r-BN	-	Rhombohedral Boron Nitride
SiO_2	-	Silicon dioxide
UV	-	Ultraviolet
APCVD	-	Atmospheric Pressure Chemical Vapour Deposition
PLD	-	Pulse Layer Deposition
B_2O_3	-	Boron Oxide
EPD	-	Electrophoretic Deposition
NaOH	-	Sodium Hydroxide
FTIR	-	Fourier Transform Infrared
SEM	-	Scanning Electron Microscopy
Al_2O_3	-	Alumina
TEOS	-	Tetraethyl Orthosilicate
DI	-	Deionized
HCL	-	Hydrochloric Acid
ELD	-	Electrolytic Deposition
CaO	-	Calcium Oxide
C	-	Carbon
KBr	-	Potassium Bromide
PDADMAC	-	Poly(diallyldimethylammonium chloride)

LIST OF SYMBOLS

h	-	Hours
g	-	Gram
V	-	Voltage
eV	-	Electron volt
W/(m.K)	-	Watt per meter kelvin
^{o}C	-	Degree Celcius
%	-	Percentage
g/cm^3	-	Bulk density
cm^{-1}	-	Wavelength
µm/min	-	micro-metre per minutes
mm	-	millimetre
E	-	Electric field strength
A	-	Surface area
t	-	Time
s	-	Second
pH	-	Acidity
vol%	-	Volume percentage
mA/cm^2	-	Milli-ampere per square centimetre
m^2/g	-	Specific surface area
ml	-	Millilitre

CHAPTER 1
INTRODUCTION

1.1 Research Background

Materials can be divided into three main categories: insulators, semiconductors and conductors. They can be differentiated by considering their electrical properties, specifically electrical conductivity. A semiconductor material is a material in which its electrical conductivity value fall between insulators and conductors.

A transistor package is an electronic component that formed by joining two types of semiconductor, namely p-type semiconductor and n-type semiconductor. Basically, transistors are used to switch or amplify electronic signal. There are several types of transistors which serves for different purposes in the market and they can be categorized into two major families which are Bipolar Junction Transistors (BJTs) and Field-effect Transistors (FETs).

All electronic components dissipate heat including power transistor. The internal heat transfer due to the flow of current is one of the limiting factor in the operation of a power transistor. Power transistors are intended to carry large current loads, thus it is important to take internal heating generated within transistor into account during design consideration. Also it comes in larger package. In many cases, component life is govern by the thermal management within or of the package (Gilmore *et al.*, 2000). The failure mechanism due to thermal issue of the transistor called thermal fatigue. In order to prevent thermal failure of component, it is necessary to gain in depth knowledge about thermal management of transistors. An example of transistor is shown in Figure 1.1.

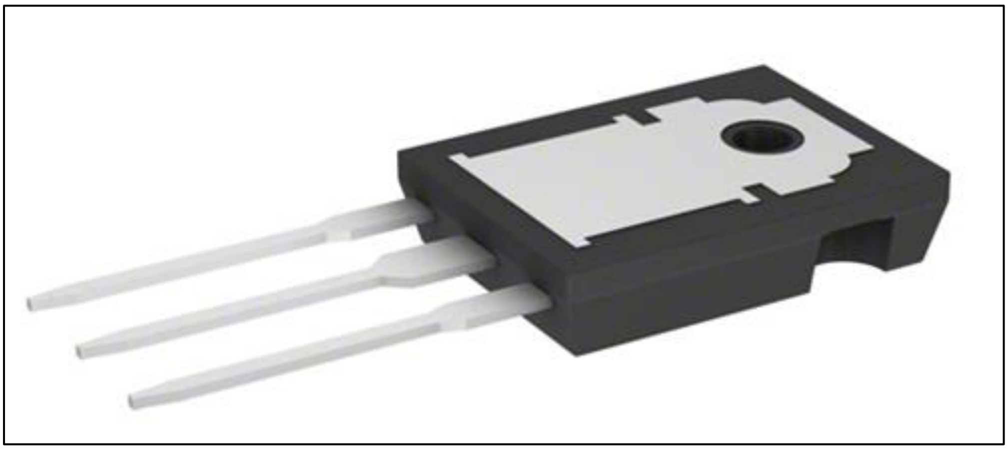

Figure 1.1: Rear side of a transistor (Infineon Technologies A.G., 2012)

Most of the heat is generated in the high resistivity region near base-collector junction. It is crucial to dissipate heat generated from the component as swiftly as possible in order to keep the temperature of the components or system at a desired level (Sim *et al.*, 2005). Therefore, a heat sink is necessary for power transistor and the heat sink should attached to the collector tab of the transistor. According to the heat transfer principle, heat will always flow from high temperature medium to low temperature medium. Also means that, heat sink conducts heat away from the transistors (Patrick *et al.*, 2005). However, it is important to note that isolation should be provided between uninsulated live part and uninsulated heat sink in order to comply with international safety standards. Also without good thermal contacts, interfacial thermal resistance will be existed due to non-surface flatness and surface roughness of either heat sink or components (Sim *et al.*, 2005). Interstitial air trapped between the surfaces will reduces the efficiency of the heat sink since air is not a good thermal conductor (k_{air} = 0.026 $Wm^{-1}K^{-1}$) (Sim *et al.*, 2005). One common method to eliminate thermal contact resistance and provides isolation is to include a material between heat sink and heat spreader, usually termed as interface materials. Interface materials not only provides isolation but also helps to enhance the performance of heat sink. Interface materials are subject to challenging requirements: the ability to reduce thermal stress between regions with difference in thermal expansion coefficients, can be reworked, low viscosity at application, electrically insulating, and highly thermal conductive (Patrick *et al.*, 2005). Figure 1.2 shows that an isolation layer is placed between transistor and heat sink.

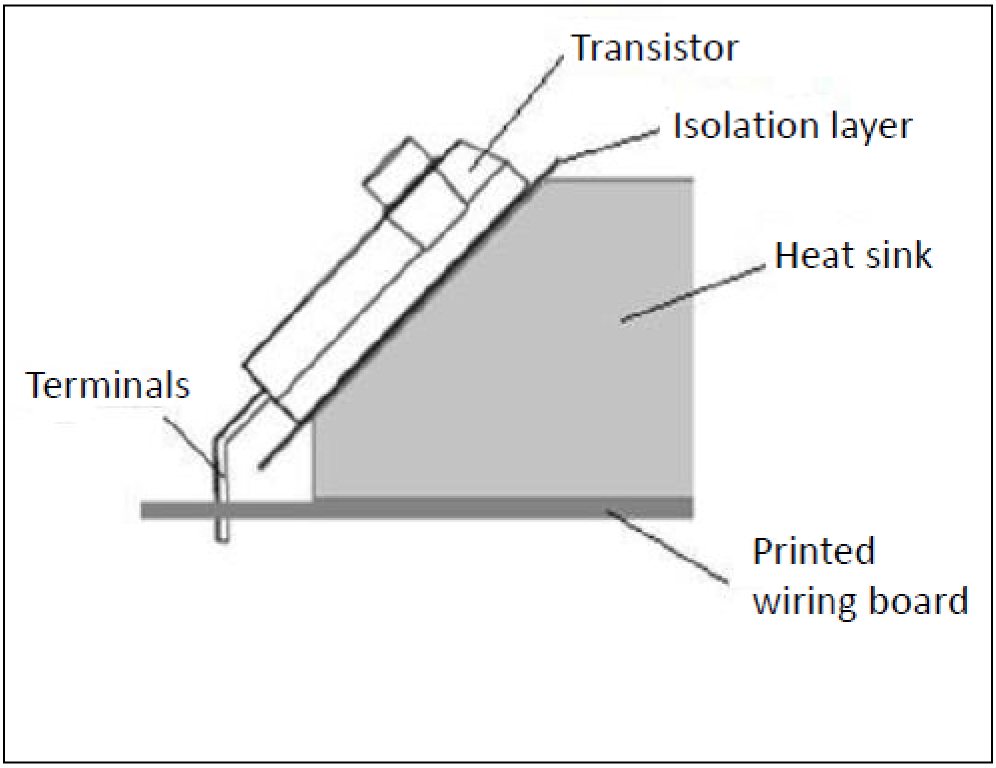

Figure 1.2: Example of a transistor with bending of terminals and heatsink (Infineon Technologies A.G., 2012).

At present, there are several interface materials available in the market. Interface materials can be classified into elastomeric thermal pads, thermal greases, solders, inorganic insulator sheets and phase change materials (Sim *et al.*, 2005). However, adding interface materials require extra materials and assembly, indirectly providing the chance for workers to make mistakes during the assembly process that would further increases burden in cost.

Boron nitride (BN) is the less investigated III-nitride materials, now it has gathers researchers' interest due to its interesting properties and close similarities with carbon (Chubarov *et al.*, 2013). BN can be existed in various forms, it can form crystals with either sp3-hybridized (cubic (c-BN) or wurtzite (w-BN) phase) or sp2-hybridized (hexagonal (h-BN) or rhombohedral (r-BN) phase) bonds (Chubarov *et* *al.*, 2013). Hexagonal boron nitride (h-BN) has similarity to graphene in structure (Al-Hamdani *et al.*, 2015). Therefore, graphene can be aligned much more closely on h-BN substrate during transistor fabrication than graphene on silicon dioxide (SiO_2) substrate, and graphene/h-BN devices also shown reduced surface roughness (Dean *et al.*, 2010).

H-BN is anticipated to play vital roles as thin dielectric layers (nano-thickness) in graphene field effect transistor (FET) (Hibino *et al.*, 2015, Kim *et al*., 2012). Researchers have shown that hexagonal boron nitride films (nano-thickness) are impervious to oxygen and can serve as high-temperature oxidation-resistant coatings for nickel up to 1100^{o}C in oxidizing atmospheres (Liu *et al.,* 2013). H-BN has potential in several applications such as mechanical and thermal coatings, deep UV optoelectronic devices, and for nanoelectronic devices and for fundamental studies of the basic properties of this materials (Ismach *et al*., 2012). Synthesis of single or few-layer h-BN (nano-thickness) can be achieved by using atmospheric pressure chemical vapour deposition (APCVD) and low-pressure chemical vapour deposition (LPCVD) (Ismach *et al*., 2012). Liu *et al*. (2013) also reported that hexagonal boron nitride coatings can also be synthesized via scalable chemical vapour deposition method. Other than that, pulse laser deposition (PLD) also has been deployed as a technique for synthesis of ultra-thin, few layer h-BN thin films (nano-thickness) on graphite and sapphire substrates (Glavin *et al.*, 2014).

1.2 Problem Statement

It is known that h-BN particle has a plate-like shape with flat surfaces corresponding to the basal plane of hexagonal crystal structure (Kim *et al*., 2014). In detail, h-BN has molecularly smooth basal plane, and there are no functional groups such as amine and hydroxyl groups that can be used for bonding or interactions (Jin *et al*., 2013). But, the edge planes of the platelets have functional groups which will allow h-BN chemically bond with other molecules (Kim *et al*., 2014). However, large h-BN particles are considerably decreased in the edge plane areas, results in difficulty of forming chemical bonds (Kim *et al*., 2014). Therefore, smaller h-BN particles (sub-micron) are preferred over large h-BN particles during reaction.

For effective use of micro-h-BN particles, surface functionalization is necessary for h-BN to functionalize h-BN by adding/attaching functional groups to its surface. (Kim *et al*., 2014). Researchers studied the mechanism of functionalizing the surface of h-BN by using silane-based coupling agent, and concluded that silane coupling agent can react with the boron oxide (B_2O_3) layer covering the h-BN particles and create covalent bonds (Kochetov *et al*., 2009). Bhattacharya *et al*. (2012) have reported the functionalization of h-BN with various groups: OH, CH_3, CHO, CN, NH_2, etc. Kim *et al*. (2014) also have indicated the surface functionalization of h-BN can be achieved by reacting h-BN particles with NaOH solution. Nazarov *et al*. (2012) have used inorganic reagent such as hydrazine, hydrogen peroxide, nitric/sulphuric acid mixture, or oleum to functionalize h-BN. Nevertheless, note that surface functionalization of h-BN is always difficult due to its special structure (Jin *et al*., 2013). The effectiveness of the surface functionalizing agent for untreated h-BN particles is greatly depended on the processing parameters used during the surface functionalization of the particles. Thus, the optimum processing parameters such as temperature and time are needed to be determined and to be refined in order to obtain functionalized h-BN particles which suitable for electrophoretic deposition (EPD) method. Efficient EPD process required a stable suspension. EPD is one of the colloidal processes for production of ceramic, it has benefits of short formation time, simple setup, and less restriction of the shape of substrate and require no binder burnout (Besra *et al*., 2007).

1.3 Objectives

The objectives of this research are as follows:

(i) To prepare and characterize the functionalized hexagonal boron nitride particles (using NaOH solution) by varying the treatment time.

(ii) To deposit and characterize functionalized boron nitride micro-thickness coatings on metallic substrate.

1.4 Scope of Research

In achieving the objectives, this study focuses on the following scope:

For objective 1:

a) Prepare functionalized h-BN particles for fixed loading (10 gram) of h-BN particles with fixed concentration of NaOH solution at varied treatment time (6 hours, 12 hours, and 24 hours).
b) Evaluate the effectiveness of surface functionalization on h-BN by using sedimentation test.
c) Determine the existence of hydroxyl functional groups in the functionalized h-BN particles by using Fourier Transform Infrared (FTIR) spectroscopy.

For objective 2:

a) Deposit the functionalized h-BN particles on metallic substrate by using electrophoretic deposition (EPD) method at varied voltage (160 V, 190 V, and 220 V), deposition time and electrode separation.
b) Determine the yield of functionalized h-BN coatings by using weight gain method.