UNIVERSITI PUTRA MALAYSIA

EFFECTS OF SINTERING TEMPERATURE ON THERMAL, MECHANICAL AND DIELECTRIC PROPERTIES OF SiC/Si3N4 NANOPARTICLES-INSERTED KAOLINITE–MULLITE

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By

ALEX SEE

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

March 2016

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DEDICATION

In memory of Associate Professor Dr Mansor Hashim,

Who taught me there is a purpose behind every material, no matter whether the material exhibits good or bad physical properties.

"Engineering is quite different from science. Scientists try to understand nature. Engineers try to make things that do not exist in nature. Engineers stress invention. To embody an invention the engineer must put his idea in concrete terms, and design something that people can use. That something can be a device, a gadget, a material, a method, a computing program, an innovative experiment, a new solution to a problem, or an improvement on what is existing. Since a design has to be concrete, it must have its geometry, dimensions, and characteristic numbers. Almost all engineers working on new designs find that they do not have all the needed information. Most often, they are limited by insufficient scientific knowledge. Thus they study mathematics, physics, chemistry, biology and mechanics. Often they have to add to the sciences relevant to their profession. Thus engineering sciences are born."

Yung Cheng Fung, Classical and Computational Mechanics, (2001)

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

EFFECTS OF SINTERING TEMPERATURE ON THERMAL, MECHANICAL AND DIELECTRIC PROPERTIES OF SiC/Si_3N_4 NANOPARTICLES-INSERTED KAOLINITE –MULLITE

By

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March 2016

Chairman: Associate Professor Jumiah Hassan, PhD
Faculty : Institute of Advanced Technology

Malaysian kaolinite production and exports have declined despite increasing global consumption of kaolinite material in paper and whiteware industries. New applications for Malaysian kaolinite are necessary to improve its competitiveness in the global environment. This research was designed to test the hypothesis that insertion of nanoparticles within a micron grain-size matrix will form a composite material with enhanced values in thermal, mechanical and electrical properties. The matrix chosen was a kaolinite-mullite matrix and the nanoparticles selected were β-Silicon Carbide (SiC) and amorphous Silicon Nitride (SN). Both Silicon Carbide-Mullite (SC-M) and Silicon Nitride-Mullite (SN-M) composite powders were prepared with a mixed solution followed by conventional sintering in an argon environment at 1000 °C, 1100 °C and 1200 °C temperatures.

XRD data of the SC-M composites yielded kaolinite-mullite products with embedded SiC presence. The SN-M composites however, yield the presence of α- and β-Silicon Nitride phases as the amorphous nanoparticles were detected to be crystalline by the XRD data. Densities of the composites were lower than the true density of the mullite matrix as the composites weights were influenced by the bulk densities of the nanopowders. Thermal diffusivity of the SC-M and SN-M composites yielded lower values as compared to the thermal diffusivity of the Kaolinite matrix itself. The thermal diffusivity values of both SC-M and SN-M were dependent on the presence of mullite concentration within the matrix.

Mechanical measurements of the SC-M and SN-M composites yielded significantly higher compressive strength as compared to those of the matrix samples. Kaolinite-mullite samples presented layered fractures under the compression test with a maximum value of 21.3 MPa. The 5% SC-M sample yielded compression strength of 54.2 MPa. SiC additions higher than 5% have lower, linear compression relation with a sharp break at maximum. SN-M composites exhibited typical ceramic compression strength at low weight additions; higher SN additions displayed a constant

compression loading effect followed by ceramic loading behavior with 30% SN-M displaying ultimate compressive strength of 110 MPa.

Dielectric permittivity of the kaolinite-mullite matrix samples has low relaxation behavior with ε_r' values marked at 9.3 to 19.3 units (at 1 MHz) for room temperature measurement. The insertion of SiC nanoparticles has elevated the real dielectric permittivity range from 5.7 units to 17.1 at 1 MHz. Insertion of the SN nanoparticles exhibited dielectric suppression as the relative dielectric permittivity values were lower than those of the matrix itself, from 4.1 to 12.2 units at 1 MHz.

The insertion of nanoparticles within the kaolinite-mullite matrix is fruitful as different properties can be examined in detail. Both SiC and SN nanoparticles yielded different degrees of enhancements in thermal, mechanical and electrical properties. The nanoparticles insertions were beneficial to thermal and electrical insulating behavior as well as mechanical compression strength.

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

KESAN SUHU PENSINTERAN KE ATAS SIFAT TERMA, MEKANIKAL DAN DIELEKTRIK KAOLINIT-MULLITE YANG DISISIPKAN NANOPARTIKEL SiC/Si_3N_4

Oleh

ALEX SEE

Mac 2016

Pengerusi: Profesor Madya Jumiah Hassan, PhD
Faculti : Institut Teknologi Maju

Penghasilan dan ekspot kaolinit Malaysia telah menurun walaupun terdapat peningkatan dalam penggunaan global dalam industri kertas dan tembikar putih. Aplikasi baru untuk kaolinit Malaysia adalah diperlukan untuk meningkatkan daya saing bahan tersebut dalam persekitaran global. Penyelidikan ini bertujuan untuk menguji hipotesis bahawa penyuntikan nanopartikel dalam butiran matriks bersaiz mikron akan membentuk bahan komposit dengan peningkatan di dalam dalam nilai-nilai sifat terma, mekanikal dan elektrik. Matriks yang terpilih adalah matriks kaolinit-mullite serta nanopartikel yang terpilih ialah β-Silikon Karbida (SiC) dan Silicon Nitrida beramorfus (SN). Kedua-dua serbuk komposit Silikon Karbida-Mullite (SC-M) dan Silikon Nitrida-Mullite (SN-M) telah disediakan dengan larutan campuran diikuti oleh pensinteran konvensional dalam persekitaran argon pada suhu 1000 °C, 1100 °C dan 1200 °C.

Data XRD bagi komposit SC-M telah menunjukkan sifat SiC tebenam dalam produk kaolinit-mullit. Walau bagaimanapun, komposit SN-M menunjukkan kehadiran fasa α- dan β-Silikon Nitrida setelah nanopartikel amorfus dikesan berada dalam keadaan kristal oleh data XRD. Ketumpatan bahan-bahan komposit adalah lebih rendah daripada ketumpatan sebenar matriks mullite kerana berat komposit dipengaruhi oleh ketumpatan pukal serbuk nano. Sifat kemeresapan terma komposit SC-M dan SN-M telah menghasilkan nilai-nilai yang lebih rendah berbanding dengan data kemeresapan terma matriks kaolinit sendiri. Nilai-nilai kemeresapan terma kedua-dua SC-M dan SN-M bergantung kepada kehadiran konsentrasi matriks mullite dalam matriks tersebut.

Kajiuji sifat mekanikal komposit SC-M dan SN-M telah menghasilkan kekuatan mampatan yang lebih tinggi berbanding dengan sifat sampel matriks. Sampel kaolinit-mullite menunjukkan sifat pematahan secara berlapis di bawah ujian mampatan dengan nilai maksimum sebanyak 21.3 MPa. Sampel 5% SC-M menghasilkan kekuatan mampatan sebanyak 54.2 MPa. Penambahan SiC melebihi 5%

mempunyai sifat yang lebih rendah bersehubungan dengan mampatan linear serta patahan mendadak pada takat maksimum. Komposit SN-M yang dipamerkan menunjukkan kekuatan mampatan seramik pada konsentrasi SN yang rendah; Penambahan SN yang lebih tinggi memaparkan kesan muatan mampatan yang malar diikuti dengan sifat muatan seramik engan 30% SN-M memaparkan kekuatan mampatan muktamad 110 MPa.

Sifat ketelusan dielektrik sampel matriks kaolinit-mullite mempunyai sifat santaian yang rendah dengan nilai-nilai ε_r' di antara 9.3 – 19.3 unit (pada 1 MHz) dalam pengukaran pada suhu bilik. Penyuntikan nanopartikel SiC telah meningkatkan sifat ketelusan dielektrik sebenar daripada 5.7 kepada 17.1 pada 1 MHz. Penyuntikan nanopartikel SN memaparkan sifat penyekatan dielektrik dengan nilai-nilai ketelusan dielektrik relatif yang lebih rendah berbanding sifat matriks itu sendiri, dari 4.1 kepada 12.2 unit pada 1 MHz.

Penyisipan nanopartikel dalam matriks kaolinit-mullite membawa manfaat apabila sifat yang berbeza boleh dikaji secara terperinci. Kedua-dua nanopartikel SiC dan SN telah menghasilkan takat penambahbaikan yang berbeza dalam sifat terma, mekanikal dan elektrik. Penyisipan nanopartikel telah memberi manfaat kepada sifat penebat terma dan elektrik serta kekuatan mampatan mekanikal.

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I would like say thanks to my supervisors, Associate Professor Dr. Jumiah Hassan, the late Associate Professor Dr. Mansor Hashim and Associate Professor Dr. Zaidan Abdul Wahab for their patience and assistance in guiding me throughout the entire research process.

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"Engineering is the art or science of making practical."

Samuel Charles Florman, The Existential Pleasures of Engineering (1976)

"Science is concerned with <u>what</u> is possible while engineering is concerned with <u>choosing</u>, from among the many possible ways, <u>one</u> that meets a number of often poorly stated economic and practical objectives."

Richard Hamming, Turing Award lecture (1968), 'One Man's View of Computer Science', collected in ACM Turing Award Lectures: The First Twenty Years, 1966 to 1985 (1987), 209.

I certify that a Thesis Examination Committee has met on 1 March 2016 to conduct the final examination of Alex See on his thesis entitled "Effects of Sintering Temperature on Thermal, Mechanical and Dielectric Properties of SiC/Si_3N_4 Nanoparticles-Inserted Kaolinite-Mullite " 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 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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Declaration by the Members of the Supervisory Committee

This is to confirm that:

- The research conducted and the writing of this thesis was under our supervision;
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LIST OF ABBRIEVATIONS

α	Thermal Diffusivity
α-SiC	alpha Silicon Carbide (hexagonal)
α-SN	alpha Silicon Nitride (trigonal)
ASTM	American Society for Testing and Materials
β-SiC	beta Silicon Carbide (cubic)
β-SN	beta Silicon Nitride (hexagonal)
C_H	Specific Heat Capacity of the material
C_P	Capacitance in Parallel Mode (Chapter 3)
ε_o	Permittivity at Vacuum
ε_r`	Relative Dielectric Permittivity
ε_s	Static Dielectric Permittivity
ε_∞	Infinite Dielectric Permittvity
σ	Standard Deviation of UCS
f	Frequency
AC	Alternating Current
EDX	Energy Dispersive X-Rays
FESEM	Field Emission Scanning Electron Microscope
G	Conductance
HFH	High Frequency Hopping
λ	Thermal Conductivity
LFD	Low Frequency Dispersion
Ln/ln	Exponential Logarithm
ρ	Density of material (gcm^{-3})
ρ_c	Density of a Composite Material
ρ_f	Density of the Filler Material
ρ_m	Density of the Matrix Material
Ø	Diameter

psi	Pounds per square inch
QDC	Quasi Direct current Conductivity
R	Resistance
rpm	Revolutions per Minute
SC-M	Silicon Carbide-Mullite Ceramic
SiC	Silicon Carbide
SN	Silicon Nitride
SN-M	Silicon Nitride-Mullite Ceramic
UCS	Ultimate Compressive Strength
UCS/m	Ultimate Compressive Strength to mass ratio
UCS/ρ	Ultimate Compressive Strength to density ratio
UCS/v	Ultimate Compressive Strength to volume ratio
UTS	Universal Testing System
V	Voltage
$<\bar{\upsilon}>$	Mean Average Electron Velocity
vol.%	Volume %
WEI	Work Efficiency Index
wt.%	Weight %
XRD	X-ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Introduction

When Prof. Richard P. Feynman gave the famous lecture entitled "There's plenty of room at the bottom" [1], the emergence of nano-technology has begun in the ceramic material research. This paradigm has advanced beyond the conventional ceramic materials prevalent in current refractory ceramic industries. Since then, the research into material has delved into making smaller size materials and the characterizations of the resultant materials yielded surprisingly better properties compared to the micron sized ceramics [2].

In conventional refractory ceramics, there was no need to pursue the miniaturization of the material grain size as the industrial demands and cost of the raw material used do not allow it. Hence, the expansion of nanotechnology in this small field related to clays and aluminosilicates was restricted to novel research or thermal insulation panels where higher prices could be negotiated.

In the 1990s, thanks to Schneider et al. [3], the importance of pursuing research of mullite came back as the material provides good structural and functional electrical properties. Mullite has low thermal expansion, low electrical conductivity and high creep resistance, which provides good electrical and thermal insulation for high temperature electronics [4]. Since then, mullite based material have served as a potential high temperature gas filters [5] and heat exchangers [6].

However, the advancements of mullite in new applications have been restricted to process modification like mullite thin films [7], mullite coatings [8], mullite whiskers [9], or mullite fibers [10]. Therefore, the focus has been on reducing and reusing the mullite through size reduction of mullite. However, emphasis should be placed on exploiting the advantages of mullite in high temperature operations as matrix composite. The mullite matrix composites with platelet and particle fillers have been explored by Schneider and Okada [11]. The major focus has been directed at zirconium and silica as the main filler components [12] with application in mechanical and thermal properties.

As such, this research is directed towards the utilization of the Malaysian kaolinite as the starting precursor for the mullite matrix. The fillers used were silicon carbide (SiC) and silicon nitride (SN) nanopowders procured from the industry to ensure repeatable quality.

1.2 Research Background

Kaolinite has been a major constituent for ball clay/china clay for the making of porcelain and refractory materials [13]. It is used for fine china and commercial porcelain wares. Modern products that use kaolinite clay include glossy paper, toothpaste and geo-polymers fibers [14]. The consumption of kaolinite in United States of America by United States Geology Survey (USGS) is estimated at 5830 metric tons per year (refer to Figure 1.1) [14]. The current price per ton for kaolinite clay export is around USD 147-150 [14]. The Malaysian Geology department estimates the Malaysian kaolinite production at 35 metric tons per year [15]. Hence, kaolinite is a major ceramic raw material for modern productions.

	Mine production					
	Bentonite		Fuller's earth		Kaolin	
	2013	2014[e]	2013	2014[e]	2013	2014[e]
United States (sales)	4,350	4,660	[2]1,990	[2]2,070	5,950	5,830
Brazil (beneficiated)	513	500	—	—	2,200	1,800
Czech Republic (crude)	226	230	—	—	3,110	3,110
Germany (sales)	375	350	—	—	4,900	4,500
Greece (crude)	1,000	1,000	—	—	—	—
Italy	110	100	3	3	640	640
Mexico	618	620	108	100	163	160
Spain	115	100	593	590	303	250
Turkey	1,100	1,100	—	—	3,800	3,800
Ukraine (crude)	210	180	—	—	1,100	1,100
United Kingdom (sales)	—	—	—	—	900	900
Uzbekistan (crude)	25	25	—	—	7,500	7,000
Other countries	3,360	3,300	306	270	9,730	11,000
World total (rounded)	12,000	12,200	[2]3,000	[2]3,000	40,300	41,000

Figure 1.1: Global Consumption of Kaolin Clay [14].

However, there is a major shortcoming with kaolinite material. As kaolinite is mined geographically by each country, the kaolinite material differs in alumina-silica concentration and impurities depending on the mining source. Hence, kaolinite material from Georgia, USA will differ from Indian kaolinite and Malaysian kaolinite both in material composition and resulting products.

1.3 Problem Statement

Despite kaolinite being one of Malaysia's clay exports (with majority of them produced from Bidor-Tapah area), the production of Malaysian kaolinite has decreased by 10% in 2013 compared to 2012 values at 442.5 metric tons [15]. The slow decline in kaolinite production may be related to the market forces, but the Malaysian kaolinite is currently exported as a raw material with little processing rather as a finished product. Hence, a paradigm shift is necessary in adding value to the Malaysian kaolinite products industry.

The current advances in pushing materials towards nano-size levels and mono-layered materials like graphene, has pushed them towards composite formation of micron matrix with nanoparticle fillers, which are rare in the material literature. So far, the literature in this aspect has been on by SiC-mullite composition with

emphasis on thermal and mechanical applications (refer to section 2.5 and 2.6 of Chapter 2).

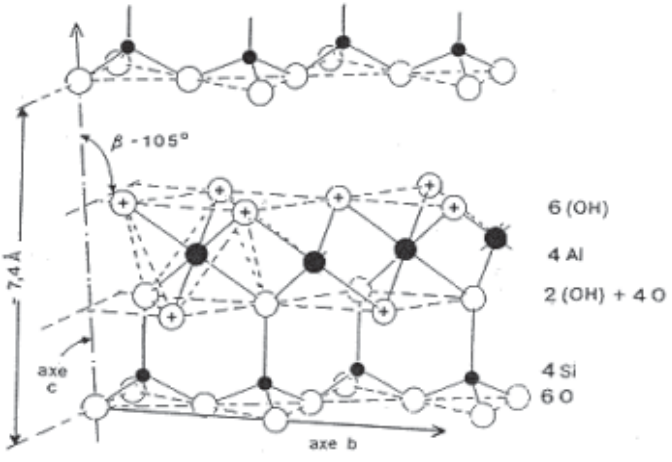
This research hopes to replicate part of the SiC-mullite with industry based standard materials obtained from kaolinite and SiC nanopowders. The insertion of foreign particles into mullite materials (sintered from kaolinite at 1000 °C to 1200 °C) is not well researched as the particle distributions and mullite growth mechanism (in response to particles) in this region affects the final composition morphology obtained by acicular mullite at 1500 °C to 1700 °C.

The research will also explore the application of inserting SN nanoparticles in the mullite matrix phase with similar methodology. This composition mix has not been tested in the research literature, especially in the nanopowder region. This pilot project will characterize the electrical, thermal and mechanical (uniaxial compression) properties for the SiC-mullite and SN-mullite composites.

1.4 Matrix Information

Kaolinite is a common aluminosilicate clay material formed from the weathering process of basalt rocks. It was named after the word 'kaoling' in China [16]. It is a main ingredient in the process of making fine china or bone china ceramics. According to USGS Clay summary report [14], the usage of kaolinite material in US domestic market is mainly in paper manufacturing. Other uses of kaolinite raw powder include: filler and extender for paint, plastic and rubber products.

The kaolinite structure is unique in nature as the water molecules are sandwiched between the alumina and silica layers [17]. The sandwich water layer is a result of the natural rock weathering process due to slow absorption of the water molecule into the rock and/or minerals through centuries. So far, this process could not be replicated synthetically. However, the sandwich water removal during the sintering will deconstruct the aluminosilicate structure [17]. Figure 1.2 compares the same monolayer of the alumina-silica composition as a result of the water removal [18].



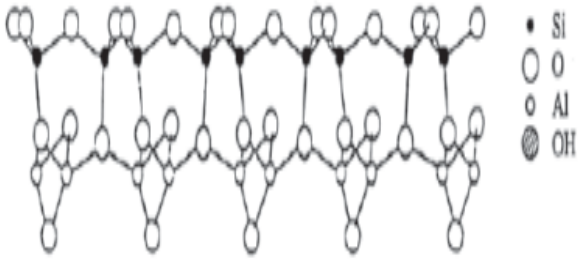


Figure 1.2: Kaolinite and Mullite Crystal Structure [18].

The sintering of kaolinite will produce different aluminosilicate products depending on the soaking temperature used. Details of the aluminosilicate process are explained in section 4.2 of Chapter 4, with the XRD data of the pure kaolinite-mullite matrix. The products of the kaolinite conversion are metakaolin, γ-alumina (also known as spinel aluminum-silica) and mullite. Each product depends on the reaction temperature of the kaolinite-mullite transformation (Figure 1.3).

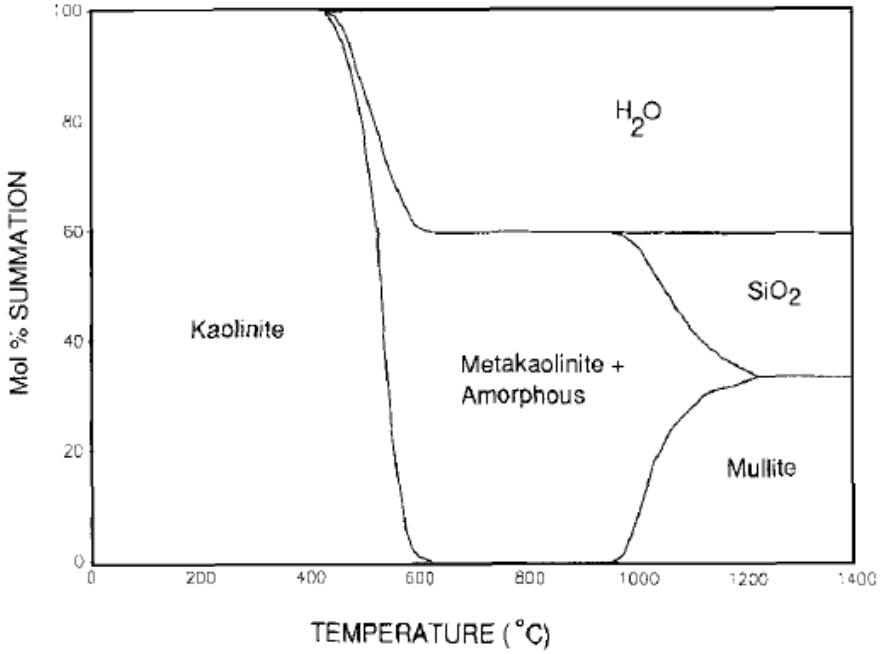


Figure 1.3: Phase Diagram for the Kaolinite to Mullite Transformation [19]

1.5 Filler Information

1.5.1 Silicon Carbide (SiC)

Silicon carbide (SiC) is a synthetic product produced from silicon and carbon. There are natural deposits of SiC called Moissanite, which are rare and found mostly in crater sites resulting from meteorites crash sites. The bonding of silicon and carbon requires intense heat and pressure to create covalent bonding between the silicon and carbon atoms [20].

SiC materials usually result in polytypes as variations of the Si-C-Si-C stacking order in layers [21]. There are three common polytypes stacking orders: namely 3c-SiC (also known as β-SiC), 4h-SiC and 6h-SiC (known as α-SiC), which are illustrated in Figure 1.4. The differences between them are the resultant crystal form and the number of repeating stacking order: c and h denotes cubic and hexagonal crystal form whereas the 3, 4 and 6 denote the layers per stacking sequence.

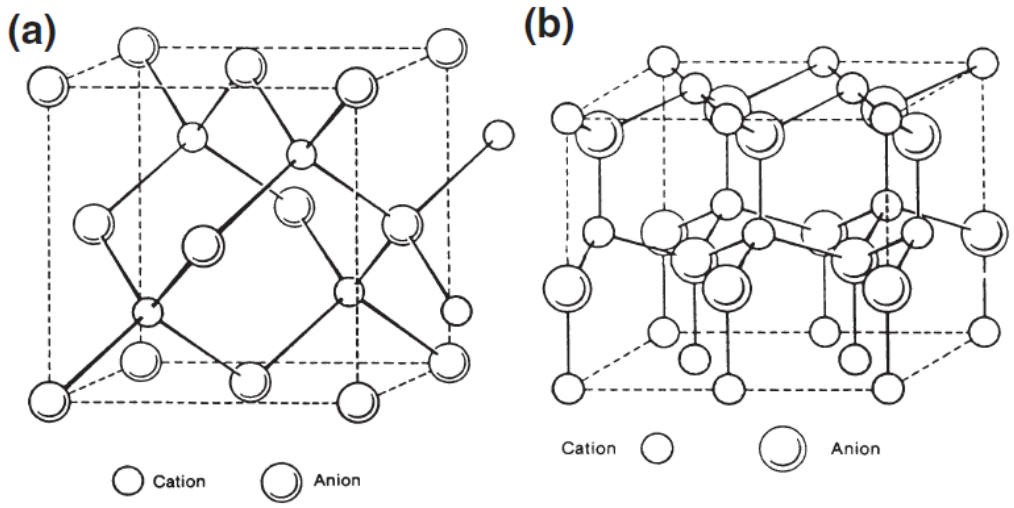


Figure 1.4: (a) β-SiC and (b) α-SiC Crystal Structure [22].

Both α and β polytypes are the most commonly produced SiC crystals. The α-SiC are formed above 1700 °C. Temperatures below the 1700 °C temperature mark usually result in the β-SiC formation [23]. The SiC does not melt at high temperature of 2700 °C; instead it decomposes into Si (liquid) and C (gas). This process is also known as sublimation. This makes SiC a useful material for high temperature applications.

Synthetic pure SiC crystals are colorless and transparent, whereas the industrial products can vary from black, grey to green in color depending on the impurities present. The production of SiC results from the pressurized, vacuum sintering of silicon and carbon. The presence or vacuum or pressurized inert gases are needed because the carbon has higher affinity to oxygen compared to the silicon. Therefore, there is a possibility of minute silica (SiO_2) crystals in between the SiC crystals.

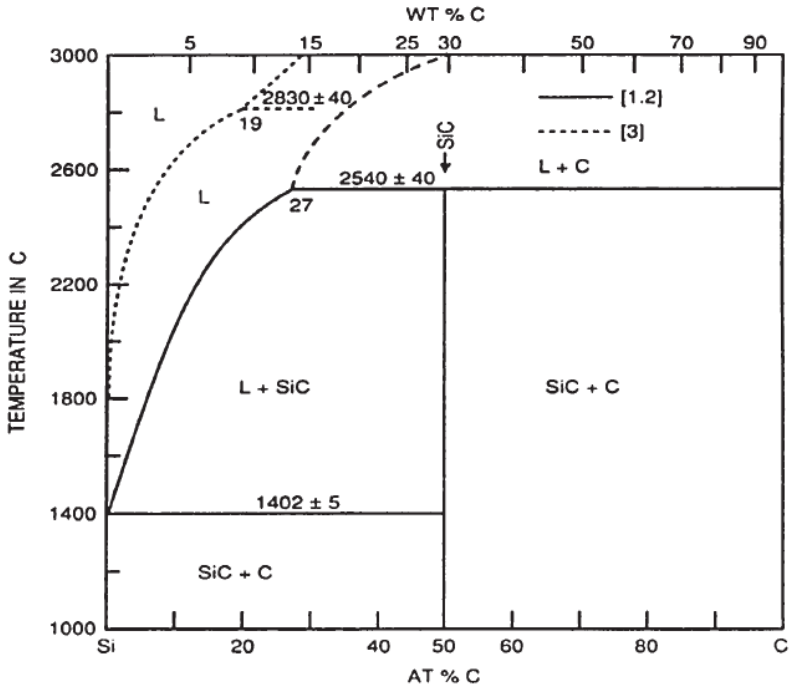


Figure 1.5: Phase Diagram for the Formation of SiC [24]

Conventional process include the Acheson and Lely methods of producing micron size SiC crystals [25]. Referring to Figure 1.5, the temperatures needed to sustain Acheson reaction is above 1800 °C for 30 atomic % to 40 atomic % of carbon present. Hence, modern nano-materials processing technology developed lower temperature reactions such as plasma enhanced chemical vapor deposition (PECVD) to mass produce SiC nanopowders in large quantities with controlled grain sizes and polytypes orders [26].

Applications of the SiC include modern abrasives and cutting tools for metalwork; electronic components such as MOSFETS and transistors [27], diodes and LEDs [28]. It is also used for mirrors for radiation sensors [29], nuclear fuel particles and cladding systems [30]. Current research also focuses on its ability as a catalyst and possible graphene starter material [31].

1.5.2 Silicon Nitride (SN)

Silicon nitride (SN) consists of three Silicon atoms bonded covalently to 4 Nitrogen atoms. It is a man made compounds as the natural mineral sources of SN compounds are not found underground except for Nierite mineral, which is found in meteorite craters [32]. The phase diagram for the synthesis of SN is shown in Figure 1.6.

Common SN materials have three different crystal structures, namely, trigonal, hexagonal and cubic structure. Both trigonal and hexagonal SN structures are known as α- and β-SN. Both materials displayed stacking layers as ABCD-ABCD layers (in α-SN) AB-AB layers (in β) [33]; both structures are shown in Figure 1.7. The last

structure is known γ-SN, which has a spinel cubic crystal structure similar to boron nitride [33]. Of the three crystal structures, β-SN is the most stable and major form of SN in ceramic production.

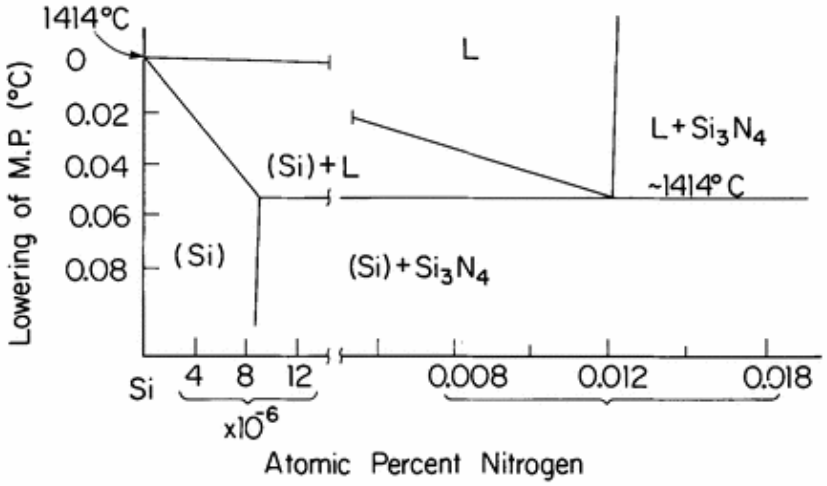


Figure 1.6: Phase Diagram for the Formation of SN Materials [34]

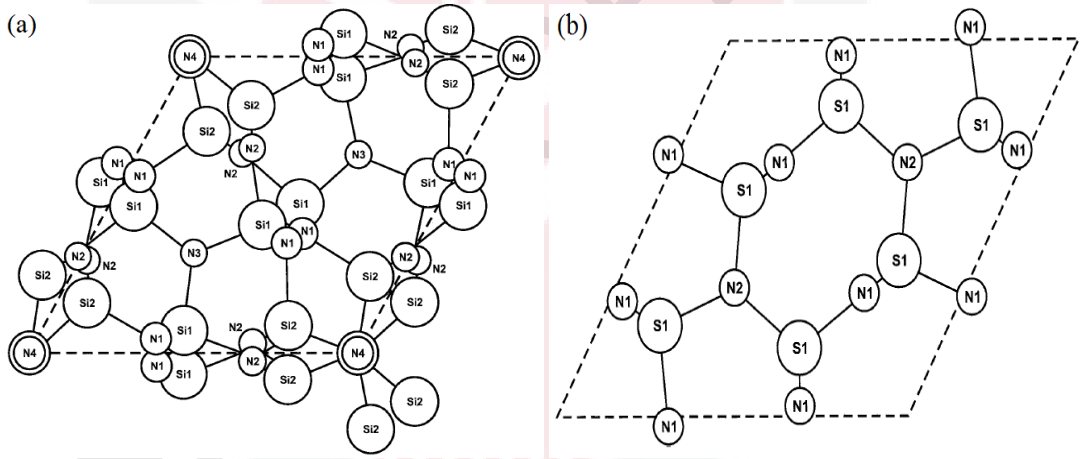


Figure 1.7: (a) α-SN and (b) β-SN Crystal Structure [35]

Applications of the SN ceramics materials are wear resistance materials such as bearings and engine components [36]; high temperature material such as rocket thrusters [37]; cuttings tools [38]; electronics (but not as a semiconductor like SiC, but as insulating masks) [39].

1.6 Theoretical Framework

SiC and SN nano-materials have been well researched as composite materials for applications into thermal, mechanical and electrical improvements [40 - 44]. The reduction in grain sizes compared to their micron counterparts has increased the electrical and mechanical properties of the materials as the miniaturization allows for

larger surface to volume ratio. This in turn affects the electronic transport and chemical bonding properties significantly and enhances the overall material.

However, the fore-mentioned literature examines the single phase aspect of the nano-material enhancement. The improvement in electrical thermal and mechanical properties proven by the nanomaterials should function well as an intermediate material between the micron size materials. Therefore, the mullite derived from the industry sector would be elevated by the addition of the nanoparticles during the sintering. The selection of mullite, SiC and SN are carefully considered in both chemical reactivity and thermal expansion in relation to each other during the sintering environment. Following the theory of concrete application, the smaller sizes of the nanomaterials will fill in the voids of the micron grain gaps and provide higher mechanical strength in uniaxial compression tests.

Similarly, thermal diffusivity of the composite material will increase due to the fused contact between the nanomaterials and micron materials. The heat transfer across the material will be easier as the radiation propagation is bypassed and the heat is transferred via the phonon and vibration across the medium. For steady state systems, thermal conductivity (α) represents the temperature gradient changes governed by Fourier's Law (refer to equation 1.1) [45]. For composites materials with random filler orientation, the temperature gradient becomes a transient state system, which dependent on variations in heat capacity and density at different temperatures [45].

$$\frac{dT}{dt} = a \cdot \nabla^2 T \tag{1.1}$$

Where dT/dt represents the temperature gradient across the material, *a* is the thermal conductivity (steady state systems); thermal diffusivity (transient systems) and T is the measuring temperature.

In this research, SiC and SN nanoparticles functions as porosity control within the mullite structure, which dictates a possible lower and transient temperature gradient based on the random distribution of the nanoparticles. The composite system would also contain trapped air, which will reduce the overall thermal diffusivity of the composite system [45].

1.7 Conceptual Framework

The determination of the nanomaterials' enhancement will be tested against the properties of the original matrix itself. The index for the comparison will form the basis of whether the properties could be enhanced through physical means. A comparison index is defined as

$$P_{index} = \frac{P_{After\ Addition} - P_{Before\ Addition}}{P_{Before\ Addition}} \times 100\% \tag{1.2}$$

Where P can be defined as the tested property such as thermal diffusivity, Compression strength and relative dielectric permittivity. The resultant values would be in positive or negative numerical values. A positive would indicate enhancement; the negative value would show a suppressive element by the nanoparticles in the micron matrix material.

Of course, in order to safeguard the consistency of the experiments, both the matrix material and subsequent nanoparticle added composites would have to be processed and sintered according to the same methodology, which includes the delicate settings of the argon assisted sintering process. The testing protocol will be conducted in a similar fashion.

The research material in this project was selected from industry sources to ensure repeatability for the property testing. The research discourages its own production of the nanoparticles under testing to discourage any possible 'black swan' enhancement argument. All materials are bought from reputable manufacturers with specific production batches for quality and quantity control in cases of repeatability argument.

1.8 Research Objectives

Based on the problem statements section, this research project is focused on the following main research objectives:

1. To obtain a homogenous ceramic mix of the micron matrix and nanopowder in specific mix ratios will be produced.
2. To study both SC-M and SN-M series for structural, thermal (thermal diffusivity), mechanical (uniaxial compression) and electrical (low frequency dielectrics) properties.
3. To study and assess the level of enhancement between the properties of the mullite matrix against nanopowder filer in the mullite matrix in the characterization data collected in the second objective.

1.9 Research Limitations

In this research project, there are some limitations to the scope of research carried out in the investigation of the SC-M and SN-M ceramic composites. Some of the limitations are listed below:

1. The research matrix material is restricted to Malaysian sourced kaolinite as the purpose is to determine the properties in the kaolinite as starting matrix material for nanopowder enhancement.
2. The nanopowders used in this research are procured from an established nanopowder manufacturer (US Research Nanopowders, Inc.). This is to

ensure that there are no discrepancies or random deviations of the nano-materials.

3. Certain aspects of material process technology have to be designed specifically as there is no resource for the material fabrications. Hence, there are possible errors related to the design specification and safety use.
4. The process technologies of the composite are controlled to prevent the nanoparticles from oxidation attack. Therefore, the sintering process is restricted to the available controlled environment furnaces, which have a maximum operating temperature of 1200 °C in argon gas. This forms the upper limit for the sintering process in this research.
5. Not all data types of the characterization test are explored in this research. High frequency dielectric measurements, different operating temperatures, tensile and bending tests will be explored in the future work.

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