

Effect of Heating Duration on the Synthesis of Silicon Carbide Nanotubes by Microwave Heating of MWCNTs and Silica

(Kesan Tempoh Pemanasan terhadap Sintesis Tiub Nano Silikon Karbida oleh Pemanasan Gelombang Mikro MWCNTs dan Silika)

TONY VOO CHUNG SUNG, VOON CHUN HONG*, LEE CHANG CHUAN, SUBASH C.B. GOPINATH, LIM BEE YING, MOHD KHAIRUDDIN MOHD ARSHAD, FOO KAI LOONG, UDA HASHIM, RUSLINDA A. RAHIM, MOHD NASHA AIN BIN NORDIN & NOR AZIZAH PARMIN

ABSTRACT

In this article, the effect of heating duration on the synthesis of silicon carbide nanotubes (SiCNTs) was reported. SiCNTs were synthesized from blend of silicon dioxide (SiO₂) and multi-walled carbon nanotubes (MWCNTs) in the ratio of 1:3 by using the microwave heating at 1400°C and maintained at duration of 20, 40 and 60 min, respectively. SiCNTs synthesized at heating duration of 40 and 60 min showed the presence of single phase β-SiC in X-ray diffraction patterns. Meanwhile, field emission scanning electron microscope images showed that SiCNTs were formed and no residual of SiO₂ and MWCNTs was observed for SiCNTs formed at heating duration of 40 and 60 min. Transmission electron microscopy images showed the SiCNTs have inter-planar spacing of 0.263 nm and tubular structure of nanotube were retained. The peak corresponded to β-SiC was observed at wavelength of 465 nm from the photoluminescence spectroscopy and associated with energy band gap of 2.67 eV. Absorption bands of Si-C bond were detected at 806.23 cm⁻¹ from the Fourier transform infrared spectra. High purity SiCNTs was obtained at 40 and 60 min as indicated by low weight loss by thermo-gravimetric analysis. 40 min is the most suitable heating duration for the synthesis of single phase β-SiCNTs.

Keywords: Microwave processing; multi-walled carbon nanotube; silicon carbide nanotube; silicon dioxide; sintering

ABSTRAK

Dalam kertas ini, kesan jangka masa pemanasan terhadap sintesis tiub nano silikon karbida (SiCNTs) telah dilaporkan. SiCNTs telah disintesis daripada campuran silikon dioksida (SiO₂) dan tiub nano karbon berbilang dinding (MWCNTs) dalam nisbah 1:3 dengan menggunakan pemanasan gelombang mikro pada 1400°C dan dikekalkan pada jangka masa 20, 40 dan 60 min. SiCNTs yang disintesis dengan tempoh pemanasan 40 dan 60 min telah menunjukkan kehadiran β-SiC fasa tunggal dalam pola pembelauan sinar-X. Sementara itu, imej pancaran medan mikroskopi elektron pengimbasan menunjukkan bahawa SiCNTs telah terbentuk dan tiada sisa SiO₂ dan MWCNTs diperhatikan untuk SiCNTs terbentuk dengan tempoh pemanasan 40 dan 60 min. Imej mikroskopi elektron pancaran menunjukkan jarak antara satah untuk SiCNTs adalah 0.263 nm dan struktur tiub nano dikekalkan. Puncak β-SiC diperhatikan pada jarak gelombang 465 nm dari spektroskopi fotoluminesen dan dikaitkan dengan jurang jalur tenaga sebanyak 2.67 eV. Belang penyerapan ikatan Si-C telah dikesan pada 806.23 cm⁻¹ dari spektroskopi transformasi Fourier inframerah. Ketulenan SiCNTs yang tinggi telah diperolehi dengan tempoh pemanasan 40 dan 60 min seperti yang ditunjukkan oleh kesusutan berat yang rendah dengan analisis termogravimetrik. 40 min adalah tempoh pemanasan yang paling sesuai untuk sintesis β-SiCNTs fasa tunggal.

Kata kunci: Pensinteran; proses gelombang mikro; silikon dioksida; tiub nano karbon berbilang dinding; tiub nano silikon karbida

INTRODUCTION

Silicon carbide (SiC) especially silicon carbide nanotubes (SiCNTs) is known to possess outstanding properties such as high mechanical strength, high stability at high temperature and high thermal conductivity which rendered SiC a suitable material for fabrication of electronic devices for high temperature, high power and high frequency applications (Wu et al. 2015). SiCNT is structurally similar to carbon nanotubes (CNTs), but SiCNT is predicted to be more superior to CNTs for the inability of CNTs to survive in high-temperature and harsh-environment applications.

SiCNT is commonly synthesized by using chemical vapor deposition (CVD) (Xie et al. 2007) and carbothermal reduction of silica by using conventional heating method (Yang et al. 2004). However, these methods possess some drawbacks. Only small quantities of SiCNTs were synthesized by using the CVD method and this method involved the use of expensive precursors. Meanwhile, carbothermal reduction of silica by carbon using the conventional heating showed some of the disadvantages such as slow heating rate, long heating duration at high temperature and non-uniform heating of raw materials

and thus incurred high cost for the production of SiCNTs. The use of microwave heating for the synthesis of SiC nano-materials such as SiC nanowires (Oh et al. 2011) and SiC nano-powder (Moshtaghioun et al. 2012) has been reported previously. Microwave heating can volumetrically heat materials with favourable dielectric properties and resulted in high heating rate, reduced reaction time and low consumption of energy. Meanwhile, conventional heating relies on the external heat source through the mechanism of conduction, convection and radiation and thus shown disadvantages such as heat loss to surrounding and irregular heat transfer between the heat source and material which in turn results in low heating rate and high energy consumptions (Oghbaei & Mirzaee 2010). Hashimoto et al. (2011) has reported the advantages of the use of microwave heating in synthesis of SiC nano-powder compared to conventional heating where pure SiC nano-powder was successfully synthesized from mixture of SiO₂ particles and carbon black in the ratio 1:3 by using microwave heating at 1300°C and maintained for 30 min. In this paper, we demonstrated the synthesis of silicon carbide nanotube (SiCNTs) from blend of SiO₂ particles and MWCNTs in the ratio of 1:3 through the microwave heating at 1400°C and maintained at heating duration of 20, 40 and 60 min. Previous studies suggested the significant effect of heating duration on the synthesis of SiC nanomaterials (Ding et al. 2014; Moshtaghioun et al. 2012). For example, Satapathy et al. (2005) studied the effect of heating duration of microwave heating (1 to 30 min) on the synthesis of SiC powder and they found that 30 min at temperature of 1300°C was sufficient to synthesize pure phase SiC powder. Ebadzadeh and Marzban-Rad (2009) also suggested that heating time at 40 min at 1450°C was sufficient to synthesize single-phase SiC nano-powder by using microwave hybrid heating. To the best of our knowledge, no study on the effect of heating duration on the synthesis of SiCNTs by microwaves heating was reported. Thus, in this paper, the effect of the heating duration on the morphology, composition, optical and purity of the SiCNTs were thoroughly studied and reported.

EXPERIMENTAL DETAILS

In this article, MWCNTs with average diameter of 18 nm and SiO₂ particles with particle size of 44 μm purchased from Sigma-Aldrich (St Louis, Missouri, USA) were used as raw materials. Ultrasonic mixing bath model D200H was used for mixing of SiO₂ particles and MWCNTs in the weight ratio of 1:3 homogeneously for 2 h and ethanol was used as liquid medium. The mixture of SiO₂ and MWCNTs then was dried by using hot plate to evaporate ethanol and the blend was then manually cold pressed into pellet form with thickness of 3 mm at 5 tons for 5 min by using Woodward Fab Shop Press 20 Ton.

Multi-mode cavity microwave model HamiLab V3 with 2.45 GHz power was used for microwave heating. Alumina crucible was filled with silica sand, graphite and SiC susceptors and then pellet of the blend of SiO₂

and MWCNTs was placed into the crucible as illustrated in Figure 1. The temperature was set to 1400°C with heating rate of 30°C/min and then maintained for 20 min. The specimen was then left to cool in microwave cavity. Similar steps were repeated for heating duration of 40 and 60 min.

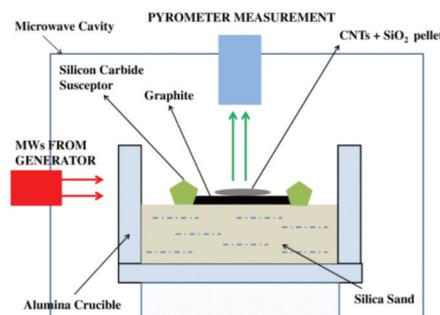


FIGURE 1. Schematic diagram of sample preparation inside microwave cavity

Characterization of SiCNTs was done by using x-ray diffraction (XRD), field emission scanning electron microscope (FESEM), transmission electron microscopy (TEM), photoluminescence spectroscopy (PL), fourier transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA). Composition of SiCNTs was investigated by using the XRD Siemens Diffractometer model D-5000 with Cu-K α radiation source in $\theta/2\theta$ mode. Fast duration scan (1s) and small step size (0.02°) was used for the measurement during XRD analysis. Meanwhile, the morphology of the SiCNTs was investigated by using FESEM model Nova Nano 450 at magnification of 200K and accelerating voltage of 5 kV. TEM model Philips Tecnai F20 TEM was used to confirm the tubular structure of SiCNTs. Optical properties of SiCNTs such as energy band gap was identified by using the PL model PL FL3-11 J81040 with xenon lamp at 400 watt and excitation wavelength at 265 nm. FTIR model FTIR MAGNA550 kBr was used to characterize the SiCNTs in the wavelength range of 450 to 4000 cm⁻¹ with spectrum resolution of 4 cm⁻¹. Quantities of SiCNTs were investigated by using Perkin-Elmer Pyris 6 TGA analyzer. Samples about 10 mg weight were heated from 30°C to 1300°C with heating rate of 10°C/min by using atmospheric air to investigate the quantity of synthesized SiCNTs.

RESULTS AND DISCUSSION

X-RAY DIFFRACTION (XRD)

Figure 2 shows the XRD patterns of SiCNTs synthesized at 20, 40 and 60 min, respectively. The XRD peaks corresponded to β -SiC at $2\theta = 36.2^\circ$ and 60° which represented the (111) and (220) planes of β -SiC were observed from XRD pattern of SiCNTs synthesized at 20 min as in Figure 2(a). Meanwhile, XRD peaks corresponded to

β -SiC at $2\theta = 36.2^\circ, 43^\circ, 60^\circ$ and 73.4° and represented the (111), (200), (220) and (311) planes of β -SiC (JCPDS Card No: 29-1129), respectively, were observed in XRD patterns of SiCNTs synthesized at 40 and 60 min as in Figure 2(b) and 2(c). This indicates that SiCNTs were successfully synthesized from heating duration of 20, 40 and 60 min. However, it can be seen from XRD pattern of SiCNTs synthesized at 20 min in Figure 2(a) that, there were peaks corresponded to unreacted SiO_2 particles and MWCNTs at $2\theta = 23^\circ$ representing (100) planes of SiO_2 and at $2\theta = 27^\circ$ representing (002) planes of carbon (JCPDS Card No: 29-1129). It can be postulated that heating duration of 20 min was insufficient for the SiO_2 particle to react completely with MWCNTs to form SiC gas and subsequently only small amounts of SiCNTs were synthesized. Furthermore, broad peak of XRD corresponded to MWCNTs showed that the MWCNTs are amorphous.

Meanwhile, XRD patterns of SiCNTs synthesized at 40 and 60 min in Figure 2(b) and 2(c) shows that only XRD peak corresponded to β -SiC was observed. This indicated that heating duration of at least 40 min were sufficient to synthesize single phase β -SiCNTs. This was due to the complete reaction of SiO_2 particles and MWCNTs to form SiCNTs. No residual of unreacted SiO_2 particles and MWCNTs was observed. Moshtaghioun et al. (2012) also has reported similar result in which they synthesized SiC nano-powder by using the microwave heating and found that complete reaction of nano silica and graphite to form SiC nano-powder with no residual of silica and graphite was achieved at heating temperature of 1450°C and heating duration of 45 min.

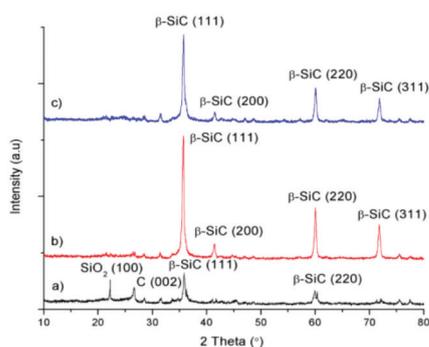


FIGURE 2. XRD patterns of SiCNTs synthesized at heating duration of a) 20 min, b) 40 min and c) 60 min

FIELD EMISSION SCANNING ELECTRON MICROSCOPY (FESEM)

Figure 3 shows the FESEM images of SiCNTs synthesized at heating duration of 20, 40 and 60 min, respectively. It can be observed from the Figure 3(a), there are unreacted MWCNTs as indicated by red circle and unreacted SiO_2 particles as indicated by blue circle in FESEM images of SiCNTs synthesized at 20 min. This shows that heating duration of 20 min was not sufficient for SiO_2 particles

and MWCNTs to react completely to form SiCNTs. This result showed good agreement with XRD pattern of SiCNTs synthesized at 20 min in Figure 2(a).

Meanwhile, Figure 3(b) and 3(c) shows the FESEM images of SiCNTs synthesized at heating duration of 40 and 60 min and it can be observed that only SiCNTs was synthesized and no residual of unreacted SiO_2 particles and MWCNTs were detected in FESEM images. These results were also in good agreement with the XRD patterns of SiCNTs synthesized at 40 and 60 min as shown in Figure 2(b) and 2(c). Ebadzadeh and Marzban-Rad (2009) reported similar result in their study of synthesis of SiC nano-powder using microwave hybrid heating where they showed that heating times at 60 min were sufficient to synthesized crystalline phase of SiC nano-powder.

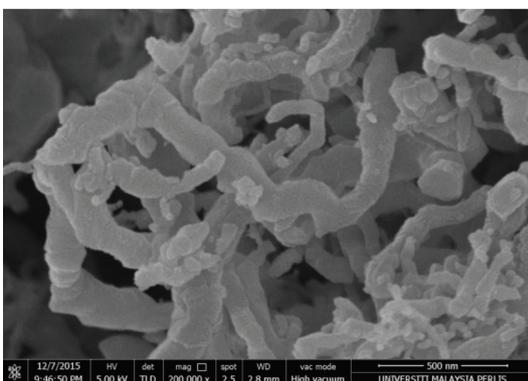
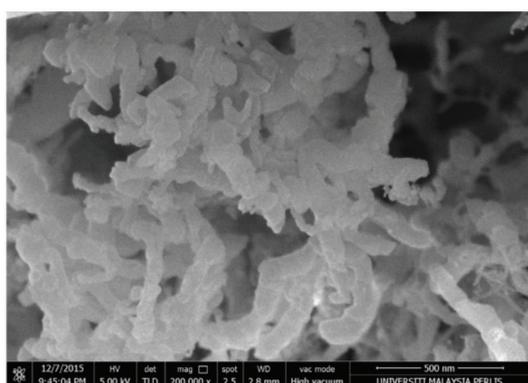
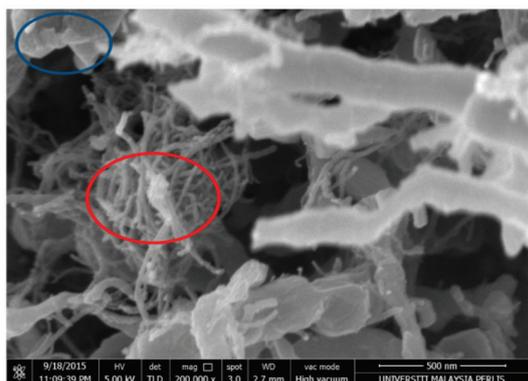
Diameters of SiCNTs were measured by using ImageJ version 1.48 and it was found out that the SiCNTs has an average diameter of 88 nm while the average diameter of MWCNTs was 18 nm. The increment of diameter of SiCNTs compared to MWCNTs may due to the difference in bond length of the C-C bond in MWCNTs and Si-C bond in SiCNTs together with the curvature of the two different types of nanotube. MWCNTs have high curvature effect of nanotube due to C-C bond of MWCNTs was tightened into the interior of the nanotube and resulted reduced diameter of MWCNTs. When SiCNTs were formed, the C-C bond of MWCNTs was changed to Si-C bond of SiCNTs. The bond length of Si-C bond are longer and spread wider inside the nanotube structure of SiCNTs and curvature effect of nanotube were reduced thus resulted the increased diameter of SiCNTs. In the study of the structure and stability of SiCNTs, Menon et al. (2004) reported that C-C bond length for MWCNTs are shorter than Si-C bond length of SiCNTs and the strain on Si-C bond was reduced which caused the diameter of SiCNTs to increase.

TRANSMISSION ELECTRON MICROSCOPY (TEM)

Figure 4 shows the TEM images of the SiCNTs synthesized at heating duration of 40 min. Figure 4 clearly shows that the tubular structure of MWCNTs was retained after the synthesis of SiCNTs from MWCNTs. The inter-planar spacing of SiCNTs was observed to be 0.263 nm while inter-planar spacing of MWCNTs was reported to be 0.35 nm (Kharissova & Kharisov 2014). This showed that SiCNTs was successfully formed. The inter-planar spacing of SiCNTs is in good agreement with the value reported by Chen et al. (2013) in their study of synthesis of SiC nanowires by thermal evaporation method in which they reported the inter-planar spacing of SiC nanowire to be 0.25 nm.

PHOTOLUMINESCENCE SPECTROSCOPY (PL)

Figure 5 shows the PL spectra of SiCNTs synthesized at 20, 40 and 60 min. Excitation wavelength of 265 nm ultraviolet fluorescent light from a Xe lamp at room temperature was used and scanned from the range of 100 to 700 nm. PL peaks corresponded to β -SiC at wavelength of 465



MWCNTs (Red Circle); SiO₂ Particles (Blue Circle)

FIGURE 3. FESEM images of SiCNTs synthesized at heating duration of a) 20 min, b) 40 min and c) 60 min

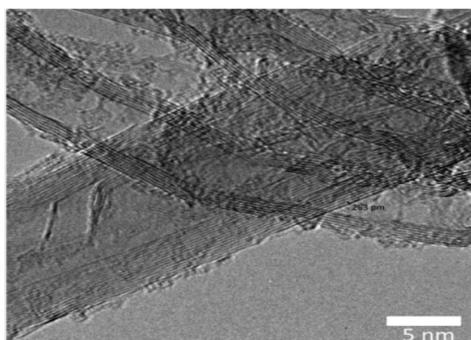


FIGURE 4. TEM image of SiCNTs synthesized at heating duration of 40 min

nm and associated with energy band gap of 2.67 eV was observed from all PL spectra of SiCNTs synthesized at 20, 40 and 60 min, respectively. This shows that SiCNTs were successfully synthesized from the heating duration of 20, 40 and 60 min. The energy band gap of the SiCNTs was obviously blue-shifted compared to energy band gap of the bulk SiC which is 2.39 eV due to the quantum confinement effect (Fan et al. 2006). This result is also in good agreement with the value of energy band gap reported by Chen et al. (2010) in which they reported that strong and sharp PL emission peak corresponding to SiC nanowire was present at 470 nm and associated with energy band gap of 2.64 eV.

The PL peaks corresponded to carbon and SiO₂ particle were detected at wavelength of 618 and 389 nm, respectively and associated with the energy band gap of 2.0 eV for carbon and 3.20 eV for SiO₂ particles in the PL spectrum of SiCNTs synthesized at 20 min as shown in Figure 5(a). This indicated that there are residual of unreacted MWCNTs and SiO₂ particle in synthesized SiCNTs for 20 min. This result shows good consistency with the XRD pattern in Figure 2(a). Chiu and Li (2009) also reported the similar result in which the PL peak corresponded to SiO₂ particles was observed centred at 390 nm in PL spectrum and indicated the oxygen discrepancy of the SiO₂ particles and showed that some SiO₂ particles not reacted with carbon to form SiC nanowires. Meanwhile, it can be observed that only PL peak corresponding to single-phase β -SiC was detected at wavelength of 465 nm in the PL spectra of SiCNTs synthesized at 40 and 60 min as shown in Figure 5(b) and 5(c), indicating SiO₂ and MWCNTs were converted completely to SiCNTs.

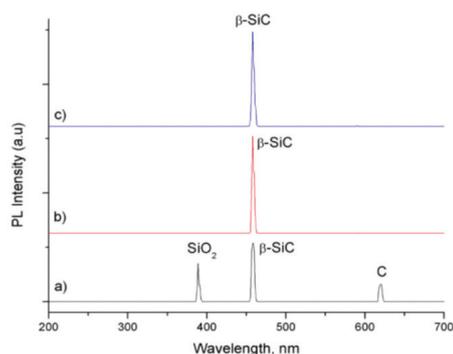


FIGURE 5. PL spectra of the SiCNTs synthesized at heating duration of a) 20 min, b) 40 min and c) 60 min

FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

Figure 6 shows the FTIR spectra of SiCNTs synthesized at 20, 40 and 60 min. FTIR absorption band corresponded to the Si-C stretching bond was detected at wavelength of 803 cm⁻¹ for all FTIR spectra of SiCNTs synthesized at 20, 40 and 60 min as shown in Figure 6. This showed that SiCNTs were successfully synthesized for blends of MWCNTs and SiO₂

heated at 20, 40 and 60 min. Chen et al. (2011) reported the similar FTIR absorption band of the Si-C stretching bond at wavelength of 800 cm^{-1} for SiC nanowires.

The FTIR absorption band of C=C bond of MWCNTs and Si-O bonding of SiO_2 particles were observed at wavelength of 1676 and 1175 cm^{-1} as observed in the FTIR spectrum of SiCNTs synthesized at 20 min in Figure 6(a), indicating the heating duration of 20 min was insufficient for the complete formation of SiCNTs. Najafi et al. (2012) also reported similar absorption band of the C=C bond of carbon that was observed at wavelength of 1623 cm^{-1} and these confirmed the presence of unreacted carbon after the synthesis of SiC nano-powder.

Figure 6(b) and 6(c) shows the FTIR spectra of SiCNTs synthesized at 40 and 60 min and it can be observed that, only FTIR absorption band corresponded to single-phase β -SiC at wavelength of 803 cm^{-1} was detected. This showed that at least 40 min was required for the complete synthesis of SiCNTs. There was no FTIR peak corresponded to C=C bond of MWCNTs and Si-O bond of SiO_2 particle was detected. This result also shows good consistency with the XRD pattern of SiCNTs synthesized at 40 and 60 min in Figure 2(b) and 2(c). Similar absorbance bands corresponded to Si-C stretching bond of SiC nanowires also were reported by Zhang et al. (2015), in which the strong absorption band of the Si-C stretching band was observed at 795 cm^{-1} in the FTIR spectra.

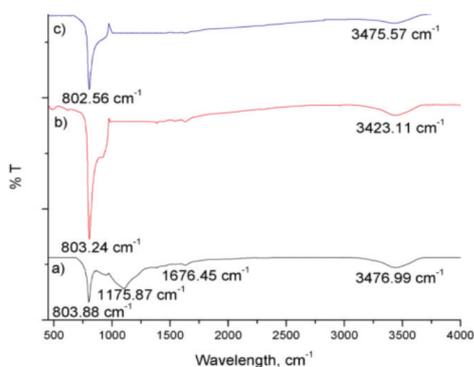


FIGURE 6. FTIR spectrums of SiCNTs synthesized at heating duration of a) 20 min b) 40 min and c) 60 min

THERMO-GRAVIMETRIC ANALYSIS (TGA)

Thermo-gravimetric analysis (TGA) was used to evaluate the quantity of the SiCNTs synthesized from blend of SiO_2 particle and MWCNTs at 20, 40 and 60 min and the TGA curves were showed in Figure 7 and the weight losses were summarized as showed from inset Figure 7. It can be observed that the weight loss of the SiCNTs commenced at about 550°C to 600°C until 1000°C . This was attributed to the decomposition of the residual of MWCNTs in the SiCNTs. In atmospheric air, MWCNTs was stable up to 400°C and decomposition of MWCNTs started at temperature higher than 400°C as reported by Mahajan et al. (2013).

High weight loss up to 44% was observed for the SiCNTs synthesized at 20 min. This weight loss was attributed to the decomposition of unreacted MWCNTs. This showed that heating duration of 20 min was insufficient for the complete formation of SiCNTs. This result also is in good consistency with the XRD pattern of SiCNTs synthesized at 20 min in Figure 2(a) in which showed that there were residual of unreacted MWCNTs.

Meanwhile, SiCNTs synthesized at 40 and 60 min in Figure 7(b) and 7(c) shows only small value of weight loss which is 3% and 4%, respectively. This may be attributed to the oxidation and decomposition of the small amounts of residual of unreacted MWCNTs which was not detected in the XRD pattern and FESEM images. It can be postulated that SiO_2 particles and MWCNTs were converted completely to SiCNTs. Similar result was reported by Najafi et al. (2011) which reported that weight loss of 9% observed from TGA analysis was due to the decomposition of the free carbon inside the SiC particles at temperature higher than 600°C .

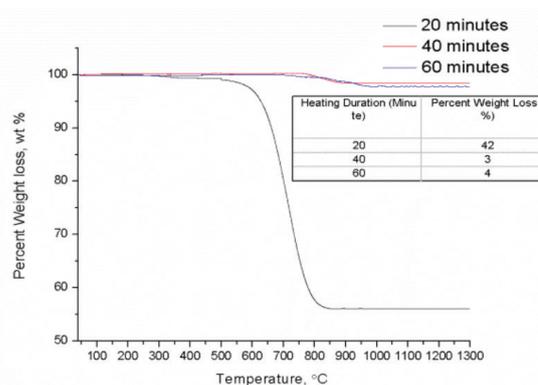


FIGURE 7. TGA curves of SiCNTs synthesized at heating duration of 20 min, 40 min and 60 min

CONCLUSION

It can be concluded that SiCNTs was successfully synthesized by microwaves heating of blended MWCNTs and SiO_2 at different heating duration of 20, 40 and 60 min. Heating duration of at least 40 min was proven as suitable heating duration of microwave heating which successfully synthesized single-phase SiCNTs with no SiO_2 particles and MWCNTs was left as residual. Meanwhile, SiCNTs formed at heating duration of 20 min consisted of the residual of unreacted SiO_2 particles and MWCNTs. Tubular structure of the nanotube was retained after the synthesis of SiCNTs and the energy band gap of the SiCNTs was 2.67 eV.

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Tony Voo Chung Sung, Voon Chun Hong*, Subash C.B. Gopinath, Mohd Khairuddin Mohd Arshad, Foo Kai Loong, Uda Hashim, Ruslinda A. Rahim & Nor Azizah Parmin
Institute of Nano Electronic Engineering (INEE)
Universiti Malaysia Perlis (UniMAP), Seriab
01000 Kangar, Perlis Indera Kayangan
Malaysia

Lee Chang Chuan
School of Manufacturing Engineering
Universiti Malaysia Perlis (UniMAP)
Kampus Alam Pauh Putra
02600 Arau, Perlis Indera Kayangan
Malaysia

Lim Bee Ying
School of Materials Engineering
Universiti Malaysia Perlis (UniMAP), Jejawi
02600 Arau, Perlis Indera Kayangan
Malaysia

Subash C.B. Gopinath
School of Bioprocess Engineering
Universiti Malaysia Perlis (UniMAP)
02600 Arau, Perlis Indera Kayangan
Malaysia

Mohd Nasha Ain Bin Nordin
Advanced Materials Research Centre
Kulim Hi-Tech Park
09000 Kulim, Kedah Darul Aman
Malaysia

*Corresponding author; email: chvoon@unimap.edu.my

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