APPENDICES

APPENDIX A

JC-PDF File

1. JCPDF of SiC

Name and formula

Reference code:	73-1665
Mineral name:	Moissanite 3*C*, syn
ICSD name:	Silicon Carbide
Empirical formula:	CSi
Chemical formula:	SiC

Crystallographic parameters

Crystal system:	Cubic
Space group:	F-43m
Space group number:	216
a (Å):	4.3490
b (Å):	4.3490
c (Å):	4.3490
Alpha (°):	90.0000
Beta (°):	90.0000
Gamma (°):	90.0000
Calculated density:	3.24
Volume of cell:	82.26
Z:	4.00
RIR:	3.53

Subfiles and Quality

Subfiles:	Inorganic Mineral Alloy, metal or intermetalic Modelled additional pattern
Quality:	Calculated (C)

Comments

Additional pattern:	See PDF 29-1129.
ICSD collection code:	024171
Test from ICSD:	No R value given. At least one TF missing. Calc. density unusual but tolerable.

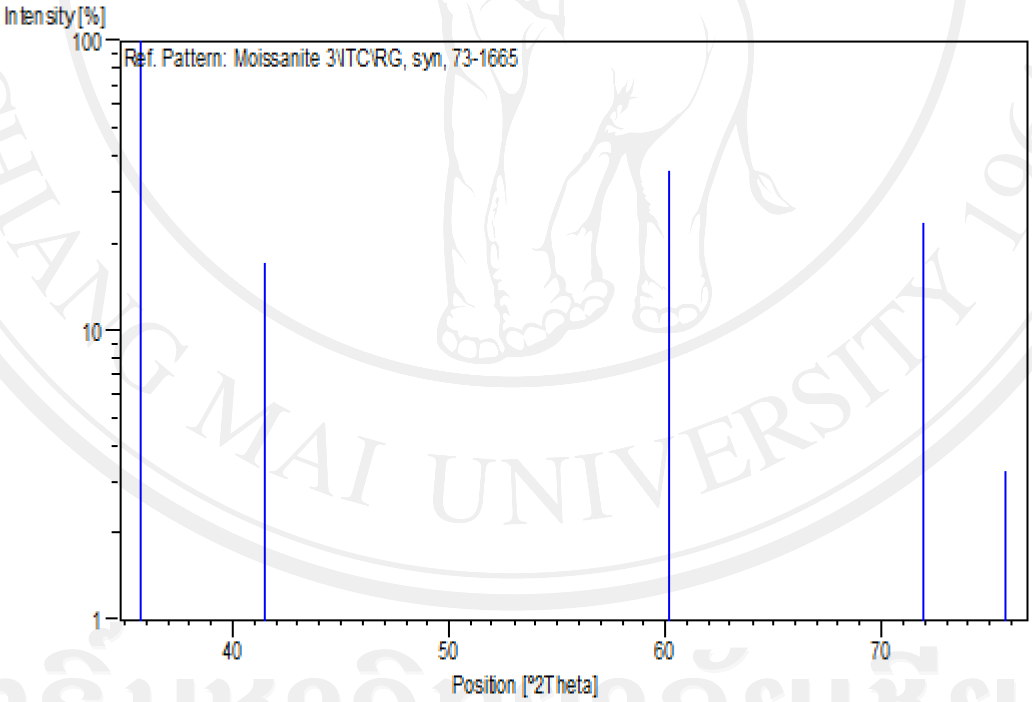
References

Primary reference:	*Calculated from ICSD using POWD-12++*, (1997)
Structure:	Thibault, N.W., *Am. Mineral.*, **29**, 327, (1944)

Peak list

No.	h	k	l	d [A]	I [%]
1	1	1	1	2.51090	100.0
2	2	0	0	2.17450	17.2
3	2	2	0	1.53760	35.6
4	3	1	1	1.31127	23.5
5	2	2	2	1.25545	3.3

Stick Pattern



2. JCPDF of FeSi

Name and formula

Reference code: 38-1397

Mineral name: Fersilicite, syn [NR]
PDF index name: Iron Silicon

Empirical formula: FeSi
Chemical formula: FeSi

Crystallographic parameters

Crystal system: Cubic
Space group: P213
Space group number: 198

a (Å): 4.4880
b (Å): 4.4880
c (Å): 4.4880
Alpha (°): 90.0000
Beta (°): 90.0000
Gamma (°): 90.0000
Volume of cell: 90.40
Z: 4.00
RIR: -

Subfiles and Quality

Subfiles: Inorganic
Mineral
Alloy, metal or intermetalic
Common Phase
Educational pattern
NBS pattern
Quality: Star (S)

Comments

Color: Dark gray
Sample source: The sample was obtained from Alfa Products, Thiokol Ventron Division, Danvers, Massachusetts, USA.
Structure: The unit cell and space group were redetermined by Wever and Moller (1).

Analysis:	Analysis by using SEM with Energy Dispersive Spectrometer (EDS) indicated no impurity.
Additional pattern:	To replace 22-632.
Temperature:	The mean temperature of data collection was 25.5 C

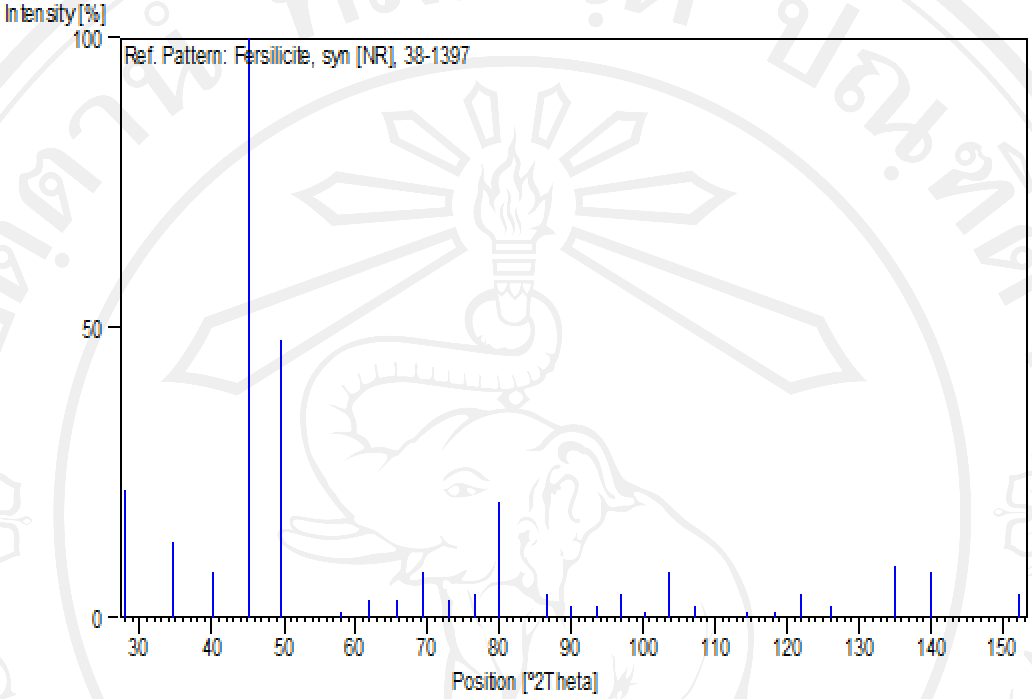
References

Primary reference:	Wong-Ng, W., McMurdie, H., Paretzkin, B., Hubbard, C., Dragoo, A., NBS, Gaithersburg, MD, USA., *ICDD Grant-in-Aid*, (1987)
Structure:	1. Wever, F., Moller, H., *Z. Kristallogr., Kristallgeom., Kristallphys., Kristallchem.*, **75**, 362, (1930)
Unit cell:	Wong-Ng, W., McMurdie, H., Paretzkin, B., Hubbard, C., Dragoo, A., *Powder Diffraction*, **2**, 261, (1987)

Peak list

No.	h	k	l	d [A]	I [%]
1	1	1	0	3.17375	22.0
2	1	1	1	2.59069	13.0
3	2	0	0	2.24328	8.0
4	2	1	0	2.00760	100.0
5	2	1	1	1.83226	48.0
6	2	2	0	1.58742	1.0
7	2	2	1	1.49562	3.0
8	3	1	0	1.41904	3.0
9	3	1	1	1.35291	8.0
10	2	2	2	1.29577	3.0
11	3	2	0	1.24472	4.0
12	3	2	1	1.19942	20.0
13	4	0	0	1.12188	4.0
14	4	1	0	1.08851	2.0
15	3	3	0	1.05792	2.0
16	3	3	1	1.02944	4.0
17	4	2	0	1.00344	1.0
18	4	2	1	0.97937	8.0
19	3	3	2	0.95671	2.0
20	4	2	2	0.91613	1.0
21	4	3	0	0.89758	1.0
22	5	1	0	0.88018	4.0
23	5	1	1	0.86372	2.0
24	5	2	0	0.83341	9.0
25	5	2	1	0.81938	8.0
26	4	4	0	0.79339	4.0

Stick Pattern



3. JCPDF of Si

Name and formula

Reference code: 27-1402

Mineral name: Silicon, syn
PDF index name: Silicon

Empirical formula: Si
Chemical formula: Si

Crystallographic parameters

Crystal system: Cubic
Space group: Fd-3m
Space group number: 227

a (Å): 5.4309
b (Å): 5.4309
c (Å): 5.4309
Alpha (°): 90.0000
Beta (°): 90.0000
Gamma (°): 90.0000

Calculated density: 2.33
Volume of cell: 160.18
Z: 8.00

RIR: 4.70

Subfiles and Quality

Subfiles: Inorganic
Mineral
Alloy, metal or intermetalic
Common Phase
Educational pattern
Forensic
Quality: Star (S)

Comments

Color: Gray

General comments:	Reflections calculated from precision measurement of a_0. a_0 uncorrected for refraction.
Sample source:	This sample is NBS Standard Reference Material No. 640.
Additional pattern:	To replace 5-565 and 26-1481.
Temperature:	Pattern taken at 25(1) C.

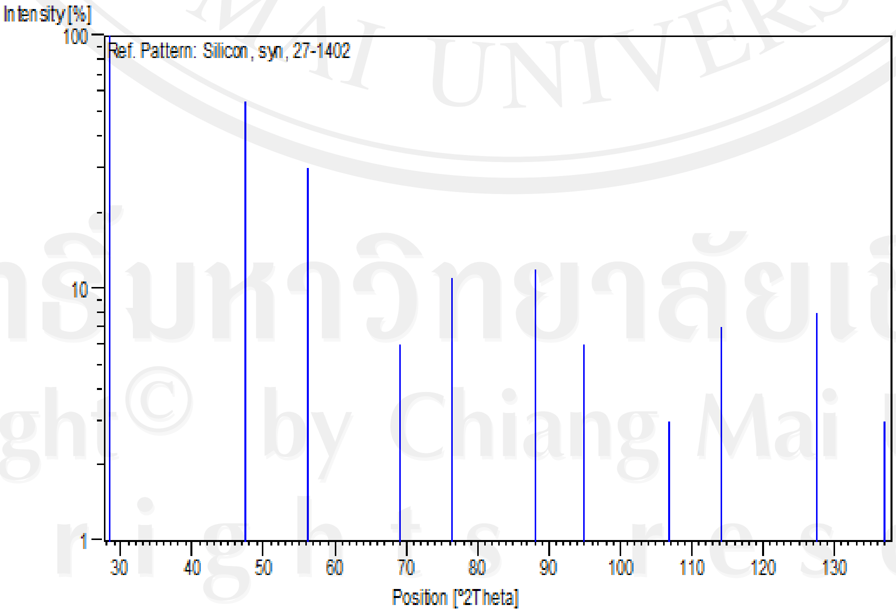
References

Primary reference: *Natl. Bur. Stand. (U.S.) Monogr. 25*, **13**, 35, (1976)

Peak list

No.	h	k	l	d [A]	I [%]
1	1	1	1	3.13550	100.0
2	2	2	0	1.92010	55.0
3	3	1	1	1.63750	30.0
4	4	0	0	1.35770	6.0
5	3	3	1	1.24590	11.0
6	4	2	2	1.10860	12.0
7	5	1	1	1.04520	6.0
8	4	4	0	0.96000	3.0
9	5	3	1	0.91800	7.0
10	6	2	0	0.85870	8.0
11	5	3	3	0.82820	3.0

Stick Pattern



4. JCPDF of C

Name and formula

Reference code: 25-0284

Mineral name: Graphite, syn
PDF index name: Carbon

Empirical formula: C
Chemical formula: C

Crystallographic parameters

Crystal system: Hexagonal
Space group: P63/mmc
Space group number: 194

a (Å): 2.4560
b (Å): 2.4560
c (Å): 6.6960
Alpha (°): 90.0000
Beta (°): 90.0000
Gamma (°): 120.0000

Calculated density: 2.28
Volume of cell: 34.98
Z: 4.00

RIR: -

Status, subfiles and quality

Status: Marked as deleted by ICDD
Subfiles: Inorganic
Mineral
Alloy, metal or intermetalic
Forensic
Quality: Calculated (C)

Comments

Deleted by: Deleted by 41-1487; good experiment pattern; Bayliss 6/90.

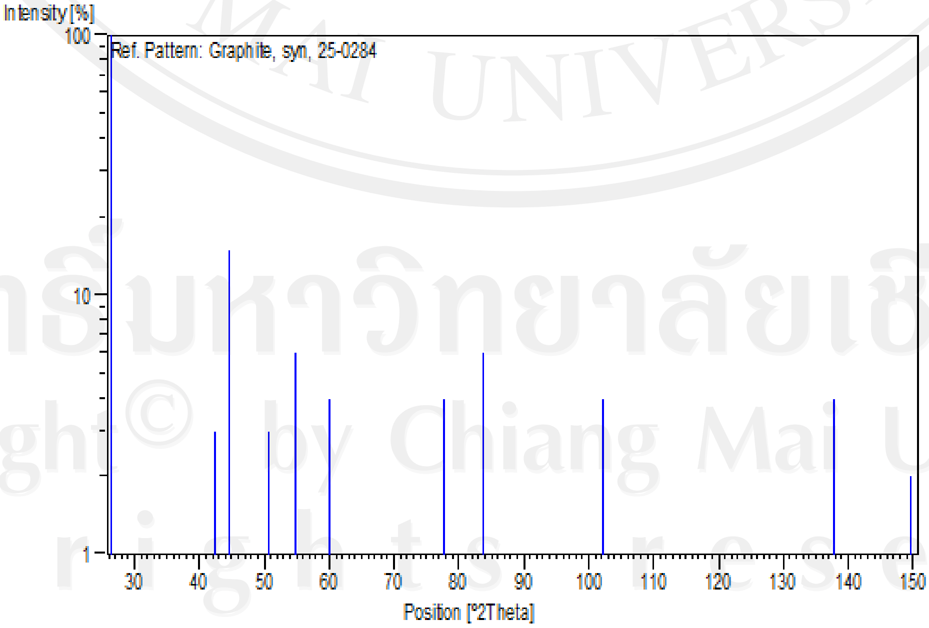
References

Primary reference:	Holcombe, USAEC Oak Ridge Y-12 Plant, Report Y1887 (1973)., *Private Communication*, (1974)

Peak list

No.	h	k	l	d [A]	I [%]
1	0	0	2	3.34800	100.0
2	1	0	0	2.12700	3.0
3	1	0	1	2.02700	15.0
4	1	0	2	1.79500	3.0
5	0	0	4	1.67400	6.0
6	1	0	3	1.53980	4.0
7	1	0	4	1.31540	1.0
8	1	1	0	1.22800	4.0
9	1	1	2	1.15290	6.0
10	1	0	5	1.13330	1.0
11	0	0	6	1.11600	1.0
12	2	0	1	1.05030	1.0
13	1	1	4	0.99020	4.0
14	2	0	3	0.96010	1.0
15	1	0	7	0.87240	1.0
16	0	0	8	0.83700	1.0
17	2	0	5	0.83280	1.0
18	1	1	6	0.82590	4.0
19	2	1	1	0.79820	2.0

Stick Pattern



5. JCPDF of SiO_2

Name and formula

Reference code: 76-0941

Mineral name: Cristobalite low
ICSD name: Silicon Oxide

Empirical formula: O_2Si
Chemical formula: SiO_2

Crystallographic parameters

Crystal system: Tetragonal
Space group: P41212
Space group number: 92

a (Å): 4.9984
b (Å): 4.9984
c (Å): 7.0242
Alpha (°): 90.0000
Beta (°): 90.0000
Gamma (°): 90.0000

Calculated density: 2.27
Volume of cell: 175.49
Z: 4.00

RIR: 5.13

Subfiles and Quality

Subfiles: Inorganic
Mineral
Alloy, metal or intermetalic
Modelled additional pattern
Pharmaceutical
Quality: Calculated (C)

Comments

Sample source: Specimen from Ellora Caves, India.
ICSD collection code: 034933

References

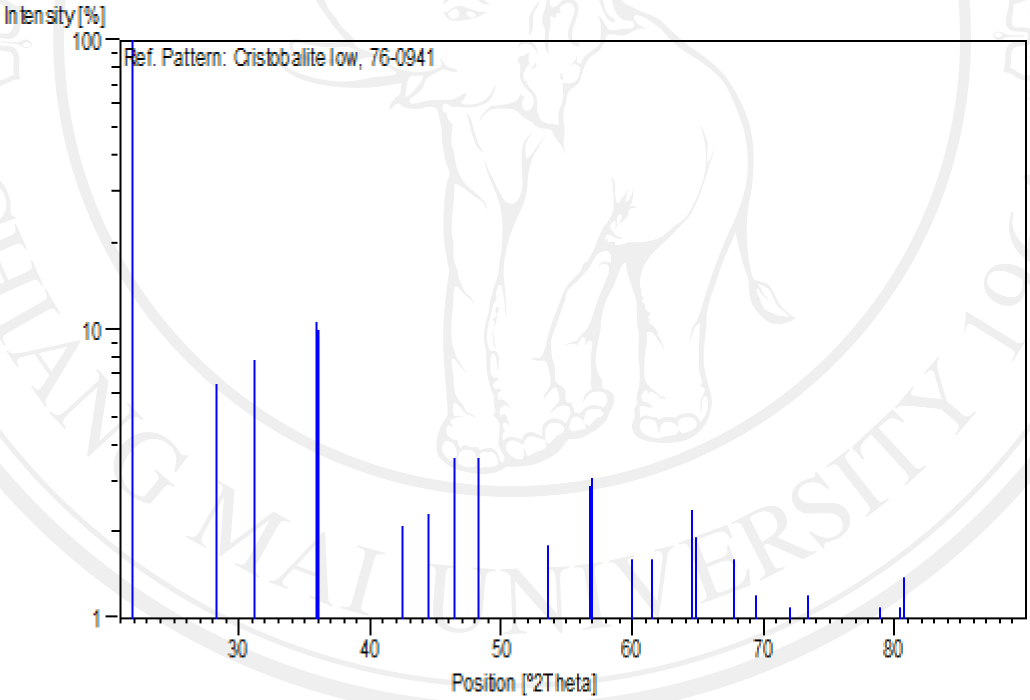
Primary reference:	*Calculated from ICSD using POWD-12++*, (1997)
Structure:	Peacor, D.R., *Z. Kristallogr., Kristallgeom., Kristallphys., Kristallchem.*, **138**, 274, (1973)

Peak list

No.	h	k	l	d [A]	I [%]
1	1	0	1	4.07254	100.0
2	1	1	0	3.53440	0.4
3	1	1	1	3.15725	6.5
4	1	0	2	2.87365	7.9
5	2	0	0	2.49920	10.6
6	1	1	2	2.49128	10.0
7	2	0	1	2.35460	0.2
8	2	1	0	2.23535	0.1
9	2	1	1	2.13009	2.1
10	2	0	2	2.03627	2.3
11	1	1	3	1.95194	3.6
12	2	1	2	1.88579	3.6
13	2	2	0	1.76720	0.3
14	0	0	4	1.75605	0.5
15	2	0	3	1.70868	1.8
16	1	0	4	1.65678	0.4
17	3	0	1	1.62115	2.9
18	2	1	3	1.61682	3.1
19	3	1	0	1.58063	0.2
20	2	2	2	1.57862	0.2
21	3	1	1	1.54207	1.6
22	3	0	2	1.50533	1.6
23	3	1	2	1.44138	2.4
24	2	0	4	1.43682	1.9
25	2	2	3	1.41053	1.0
26	3	2	0	1.38631	0.1
27	2	1	4	1.38090	1.6
28	3	2	1	1.36007	0.8
29	3	0	3	1.35751	0.8
30	1	0	5	1.35244	1.2
31	3	1	3	1.31006	1.1
32	3	2	2	1.28949	1.2
33	4	0	0	1.24960	0.1
34	2	2	4	1.24564	0.7
35	4	0	1	1.23028	0.8
36	2	0	5	1.22463	0.1
37	4	1	0	1.21229	1.1
38	3	0	4	1.20867	0.6

39	3	2	3	1.19289	1.1
40	2	1	5	1.18945	1.4
41	3	3	0	1.17813	0.2
42	3	1	4	1.17481	0.5
43	3	3	1	1.16190	0.3
44	4	1	2	1.14594	0.2
45	4	2	0	1.11696	0.4
46	1	1	6	1.11132	1.0
47	4	2	1	1.10379	0.7
48	4	0	3	1.10242	0.2
49	2	2	5	1.09970	0.2

Stick Pattern



APPENDIX B

Publications

Advanced Materials Research Vols. 93-94 (2010) pp 497-500
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Improvement in the Tensile Strength of Epoxy Resin and Hemp/Epoxy Resin Composites using Carbon Nanotubes

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Keywords: CNTs, composite, epoxy resin

Abstract. An experimental was investigated the condition of reinforcement of epoxy resin and hemp fiber/epoxy resin composites with carbon nanotubes (CNTs). The CNTs adding nanopowder were vibrated via the vibration milling technique for 6-48 h. Different volume percentages of CNTs were dispersed for hemp/epoxy resin composites. To compare properties of composites sample, CNTs were also added into epoxy resin for reference. Tensile strength of both specimens was tested. The significantly adding of CNTs and its dispersion in polymer matrix were investigated by scanning electron microscope (SEM). The results indicate that adding the milled CNTs can improve tensile properties of composites.

Introduction

Bio-composites are the smart materials of the future. It is only though these materials that the balance of ecology and economy can be maintained [1]. To concern about the environmental, the new materials such as bio-composites has improved and developed. Therefore, natural resources is interested and applied in many products. Natural fiber is one of the natural resources that are focus for future materials, especially in reinforced products. Thus, there has been raised interest in using natural fibers as reinforcements in polymer composites. The advantages of natural fibers include low price, low density, unlimited and sustainable availability, and low abrasive wear of processing machinery [2, 3]. Further, natural fibers are recyclable, biodegradable and carbon dioxide neutral and their energy can be recovered in an environmentally acceptable way. Hepworth et al. [4] produce unidirectional hemp fiber reinforced epoxy composites, with a tensile strength of 60 90 MPa and Young's modulus of 8 GPa, by pinning-decortication and hand combing. Yuanjian and Isaac [5] investigated the tensile properties of the hemp mat reinforced polyester and [±45°] glass fiber reinforced polyester. The result have shown that hemp mat have the potential to be considered for some non-critical stress bearing structures to replace glass fiber composites. To improve the mechanical properties, a type of filler, carbon nanotubes (CNTs) has been attracting considerable attention due to their excellent properties such as high tensile strength and Young' modulus [6,7]. Very limited studies have reported on the properties of nanocomposites containining natural fibers. Matuana reported the effect of CNT reinforced PVC matrix and 40-wt % wood flour resulted in greater flexural strength and modulus than their counter part made with un-reinforced PVC matrix [8]. There have been many attempts to incorporate nanoparticles, nanofibers, and nanotubes into the polymeric matrix of conventional fiber-reinforced composites by impregnating dry fiber preforms with the modified nanocomposite matrix. However, it is difficult to achieve good dispersion of the nanoparticles in low viscosity solutions due to non-bonded attractive interaction among them [9]. Thus, the treatment before being put into the matrix is very important in order to decrease the agglomeration. Vibration is one of the methods use to decrease agglomerated nanopowder. The two objectives of this experiment are the investigation on effect of adding vibrating milled CNTs on epoxy resin and the effect of adding milled CNTs on tensile properties of hemp/epoxy resin composites.

Experimental

Firstly, the CNTs were produced by chemical vapor deposition (CVD) method and vibrated by vibrated milling for 0-48 h to break and decrease the agglomeration of CNTs. For the epoxy resin samples reinforced with vibrating milled CNTs, the samples were created by mixing CNTs with ratio of 6-vol% into epoxy resin and dispersed the mixtures by ultrasonication for 3 h. and hardener was then mixed by ratio of 10:2.7 by volume. The mixtures were poured into the rubber moulds. All the samples were allowed to cure at room temperature for 24 h and then removed from the mould.

To prepare the hemp/epoxy resin composite samples, the CNTs (obtained vibration milling for 48 h) were heated treatment at 430 °C for 4 h. After that, 0-4 vol % CNTs were added and mixed into epoxy resin by ultrasonication for 3 h before being mixed with hardener. Hemp fabrics were cut into dog - bone shape and it was then performs the modified reinforced nanomatrix composites by impregnated the hemp fabric with nano-mixtures. All of the specimen sheets were allowed to cure at room temperature for 24 h and then removed from the mould. It is noted that Mylar films were covered on top and bottom in the both sides of the samples for smooth surface.

All the sheets were allowed to cure at room temperature for 6 days. To investigate the mechanical properties of composite samples, the dog – bone shape with gauge length of 30 mm, 14 mm of width and thickness about 1 mm was prepared. The tensile tests were conducted on a Hounsfield mechanical testing machine using a crosshead speed of 50 mm/min and a 250 mm extensometer. Moreover, the microstructure of composite samples was determined by scanning electron microscope (SEM).

Results and discussion

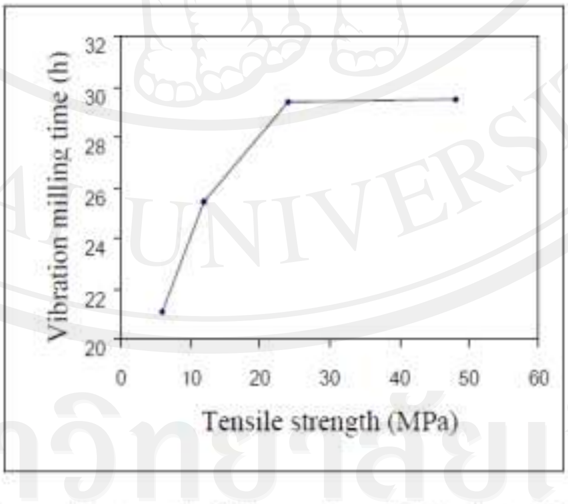

Fig. 1 shows the tensile strength of the samples, which vibrated CNTs for 6-48 h

Fig. 1, shows the tensile strength of the epoxy samples, which CNTs vibrated 6-48 h. These results show that tensile strength of the epoxy samples reinforced with vibrating milled CNTs was significantly improved. This is may be due to the vibrations of CNTs for 6-48 h can improve the dispersion of CNTs in epoxy resin (without hardener) by break through the agglomeration of nanopowder. Fig. 2, shows the SEM micrograph of vibrating milled CNTs with different vibrating time. It can be seen that dispersion of CNTs is better, which can confirm that the vibration has affect on the agglomeration of CNTs

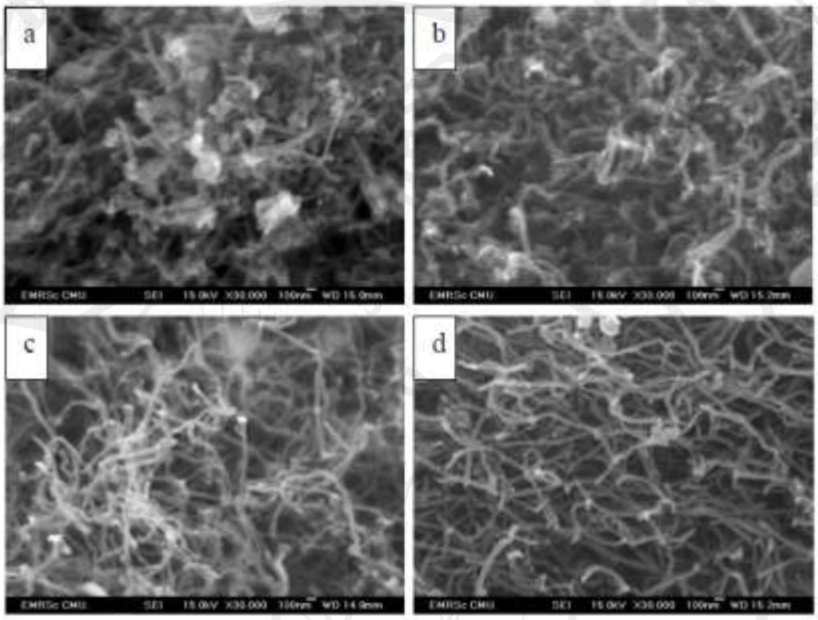

Fig. 2 SEM images of dispersion of vibrating milled CNTs: (a) 6 h, (b) 12 h, (c) 24 h, (d) 48 h

Table 1. The tensile properties of the hemp/epoxy resin composite samples filled CNTs 0-4 vol%

Samples [vol%]	tensile strength [MPa]	Max force [N]	Elongation [%]
0	21.15	281.88	9.40
0.5	22.76	305.93	7.65
1	23.70	397.18	11.51
2	24.05	344.92	9.40
4	30.02	400.58	10.24

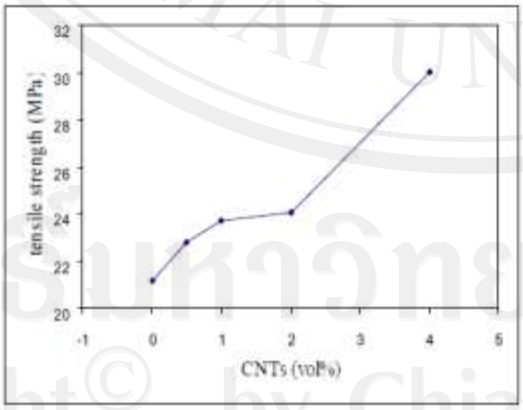

Fig. 3 Effect of quantity of CNTs fillers on the tensile strength of the hemp/epoxy resin composites

Table 1 shows the tensile properties of the hemp/epoxy resin composite samples filled with 0-4 vol% CNTs. From the results, it can be seen that using CNTs as enforcement fillers can promote the mechanical properties of composites samples. Furthermore, the highest tensile strength of these composites increased up to 41.93% when compared with non-filler CNTs samples. Fig. 3, shows the effect of quantity of CNTs fillers on the tensile strength of the hemp/epoxy resin composites. It can see that, tensile strength of specimen is significantly increased when the CNTs increased from 2 vol. % to 4 vol. %.

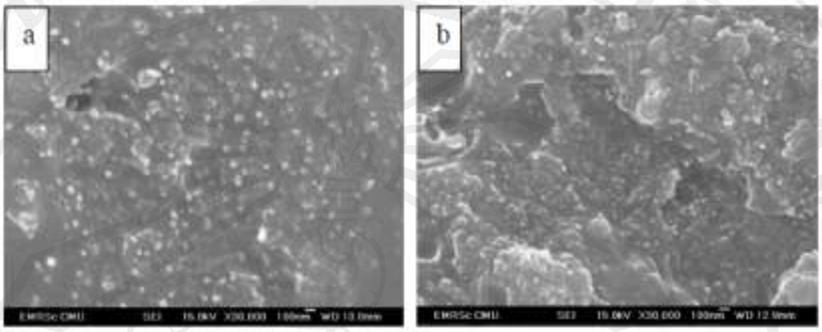

Fig. 4 SEM micrographs of CNTs dispersed in hemp/epoxy resin composites: (a) 2 vol. % (b) 4 vol. %

Fig. 4, shows SEM micrographs of CNTs dispersed in hemp/epoxy resin composites. It has been seen that the CNTs were well dispersed in the matrix without any CNTs agglomeration regardless of the increasing of CNTs content. Thus, it exhibits that CNTs has good interface adhesion with hemp/epoxy resin. It is possible that adding the reinforced CNTs fillers can improve the interface adhesion by increasing of the link ratio between nanoparticles and matrix phase and also block the molecular motion of polymer matrix.

Therefore, it can be concluded that the tensile properties of hemp/epoxy composites were improved significantly with adding of CNTs. However, the significantly change could not found in 0.5 vol. % and 1 vol. % CNTs samples which is may be due to too low content of CNTs. Moreover, vibrated milling technique can use for decreasing the agglomeration of CNTs nanoparticles. And adding the vibrated milling CNTs in composites exhibits the good mechanical property.

Conclusion

Epoxy resin reinforced with CNTs could be improved its strength by adding vibrated milling CNTs, because of the agglomeration decreasing. The optimum strength obtained from 4 Vol% of CNTs composites samples. Furthermore, adding the vibrated milling CNTs in composites exhibits the good mechanical property.

Acknowledgments

The authors would like to thank the Thailand Research Fund (TRF) for supporting this research. And the authors also would like to the Nanomaterials Research Unit, Department of Physics and Materials Science, Faculty of Science and the graduate school, Chiang Mai University.

References

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Simple Chemical Vapor Deposition Method to Synthesized SiC Nanowires

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Keywords SiC nanowires, Nanowires, Chemical Vapor Deposition

Introduction

Recently, SiC nanowires have attracted to research due to their excellent properties, such as very high melting temperature, lower thermal expansion coefficient, good thermal conductivity [1], high hardness, and high fracture toughness [2] that enables them to serve as a reinforcing phase in ceramic, metal, and polymer material composites [3]. SiC nanowires have been synthesized numerous techniques, such as arc discharge [4], laser ablation [5] and chemical vapor deposition (CVD) via the vapor-liquid-solid mechanism [6-8]

Many SiC nanowires that produced from Si wafers are expensive. This work reported that SiC nanowires were prepared by using simple CVD method and low cost route. The obtained SiC nanostructures were characterized by a field-emission scanning electron microscopy, equipped with energy dispersive spectroscopy and transmission electron microscopy.

Experimental Details

SiC nanowires were synthesized in a horizontal alumina tube furnace. 0.375 g of purity Si powder (99%, Aldrich) and activated carbon was mixed with 0.20 g of NiO and first put into an alumina boat. The mixture was placed in the center of the tube furnace and activated carbon boat was put in 10 cm spacing. The tube was heated to 1200 -1400°C by rating of 10°C/min and kept for 1 hr under argon flowing 2 L/min. SiC nanowires were formed. Finally, the furnace was cooled down to temperature by rating of 10°C/min. To purification SiC nanowires, the products were further heat-treated at 700°C for 4 hr in air to remove residual carbon. The obtained SiC nanostructures were characterized by a field-emission scanning electron microscopy, transmission electron microscopy. The crystallographic data was collected by X-ray diffraction.

Results and Discussion

After the synthesis by different heating and purification, the grayish products were obtained. The fig. 1 shows SEM images of all samples, the nanowires were randomly oriented with smooth and straight at temperature of 1300°C, 1400°C, but included straight and curve morphologies at 1200°C. These nanowires generally showed the diameter of 20-70 nm. Fig. 1(b) shows the spherical droplets of nickel particles acted as catalysts at the tip of each SiC nanowire, it indicated that the SiC nanowires were grown via the vapor–liquid–solid (VLS) growth mechanism.

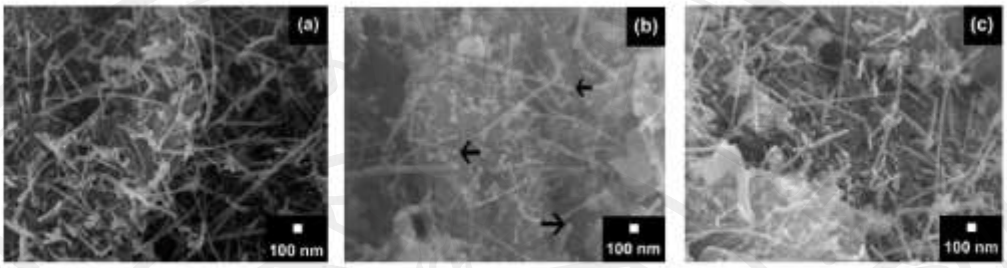

Fig. 1 SEM images of SiC nanowires at (a) 1200°C, (b) 1300°C, (c) 1400°C

In order to characterize the structure in further details, the products were investigated by TEM, at a low temperature, the nanowires tended to form as showed in Fig. 2a. TEM observations indicated that these nanowires were a combination of the amorphous and crystalline structures, whereas, the temperature was increased to 1300°C and 1400°C more crystalline structure was found. The SAED pattern in Fig. 2(b) showed a typical nanowire, and the clear spots indicated that the nanowire is a single crystal. The SAED pattern can be indexed to 3C-SiC, which grown along the [111] direction. Phase identifications of the products were characterized by XRD, as showed in Fig. 3. The peak intensity of 3C-SiC increased with process temperature, a amount of silicon and carbon acted as source remained at low temperature and decreased at higher temperature.

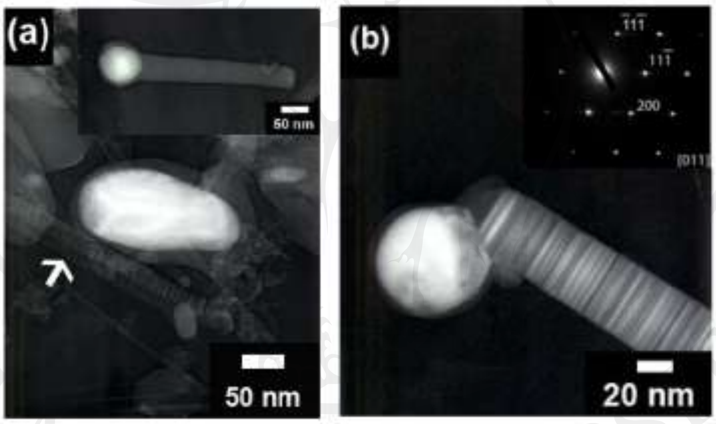

Fig. 2 TEM images of SiC nanowires at a) 1200°C, b) 1400°C and its SAED pattern (inset)

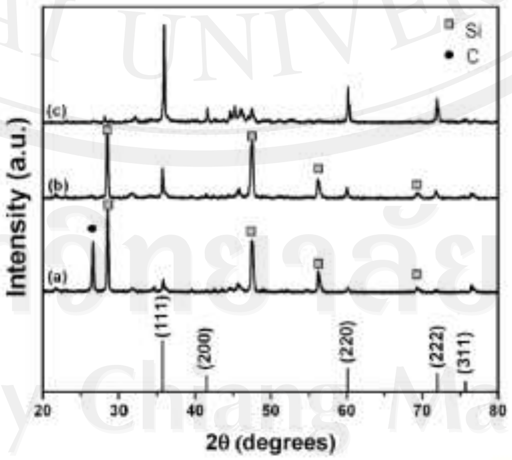

Fig. 3 X-ray diffraction spectra of products: (a) at 1200°C (b) 1300°C, and (c) 1400°C after heat-treated. It can be indexed to be β-SiC (JCPDS Card No. 73-1665).

Conclusion

The SiC nanowires were synthesized by CVD at normal atmosphere pressure have the amorphous and crystalline structures at 1200°C, whereas single crystalline feature was found at 1300°C and 1400°C. Main growth direction was [111]. This nanowires has about diameter of 20-70 nm. The droplets catalyst was found on the tips of each nanowire. It is propose that the growth mechanism is a VLS process.

Acknowledgments

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Large-scale: Synthesis, microstructure, and FT-IR property of SiC nanowires

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Large-scale SiC nanowires have been synthesized via reacting silicon powder with carbon powder using Ni_2O_3 as catalyst by simple chemical vapor deposition method. The mixed powders were heated in an alumina horizontal tube furnace up to 1350 °C for 1 h in an argon atmosphere. Characterization by scanning electron microscope, transmission electron microscope, and X-ray diffractometer indicated that the nanowires covered with a thin layer of amorphous SiO_x. The 3C–SiC nanowires were formed by vapor–liquid–solid growth mechanism and they have diameters of about 20–90 nm. The result of FT-IR spectrum shows the nanowires were composed of SiC and amorphous of silicon oxide.

1. Introduction

Recently, SiC nanowires have attracted to research due to their excellent physical and chemical properties, such as high thermal stability, lower thermal expansion coefficient, good thermal conductivity, chemical inertness [1], high hardness, and high fracture toughness [2] that enable them to serve as a reinforcing phase in ceramic, metal, and polymer material composites [3,4]. SiC nanowires have been showed to exhibit properties superior to bulk SiC such as both the elasticity and strength of SiC nanowires are considerably greater than bulk SiC [5]. SiC nanowires have been synthesized numerous techniques, such as arc discharge [6], laser ablation [7] and chemical vapor deposition (CVD) via the vapor–liquid–solid mechanism [8–10] which CVD is a widely used technique to fabricate SiC nanowires. For examples, synthesis SiC nanowires from solid phase sources on silicon wafer substrates and iron were chosen as the catalyst [11,12]. The nanorods prepared through a one-step reaction that was carried out in an autoclave by using $SiCl_4$ and CCl_4 as reactants and metal Na as-reductant at low temperature [13], the growth of SiC nanowires by carbo-thermal reactions without catalyst that Si wafers was used as substrates [14], silicon substrates act as reactant [15–17].

However, most of these synthetic approaches involved complex processes and manipulation, or requiring ultra high temperature. This work reported that SiC nanowires were prepared by using simple CVD method, low cost route, and high quantities. The obtained SiC nanostructures were characterized by a field-emission scanning electron microscopy, equipped with energy dispersive spectroscopy and transmission electron microscopy.

2. Experimental

SiC nanowires were synthesized in a horizontal alumina tube furnace. 0.03 mol of purity Si powder (99%, Aldrich) and 0.06 mol of carbon powder (ultra high pure graphite 99.9%) were mixed with 0.001 mol of Ni_2O_3. The raw materials were mixed and pulverized in a mortar for 20 min. The mixed powders were filled into an alumina boat and placed in the center of the tube furnace. Then carbon boat was put in 5 cm spacing. The tube was pumped out initially by vacuum pump and high purity argon was released into the furnace for three times to estimate the residual air. After that, the furnace was heated to 1350 °C by rating of 10 °C/min and kept for 1 h. SiC nanowires were formed. Finally, the furnace was cooled down to 300 °C by rating of 10 °C/min and then cooled down to room temperature naturally. To purify the SiC nanowires, the products were further heat-treated at 700 °C for 4 h in air to remove residual carbon. The obtained SiC nanostructures were characterized by a field-emission scanning electron microscopy, transmission electron microscopy. The crystallographic data was collected by X-ray diffraction and FT-IR spectrum shows the absorption bands.

3. Results and discussion

After the synthesis by CVD method and purification, the as-obtained product posses remaining of 60% by weight with white wool-like features of about 40 μm thick was formed on as-grown powder surface as illustrated in Fig. 1a. Fig. 1b reveals the

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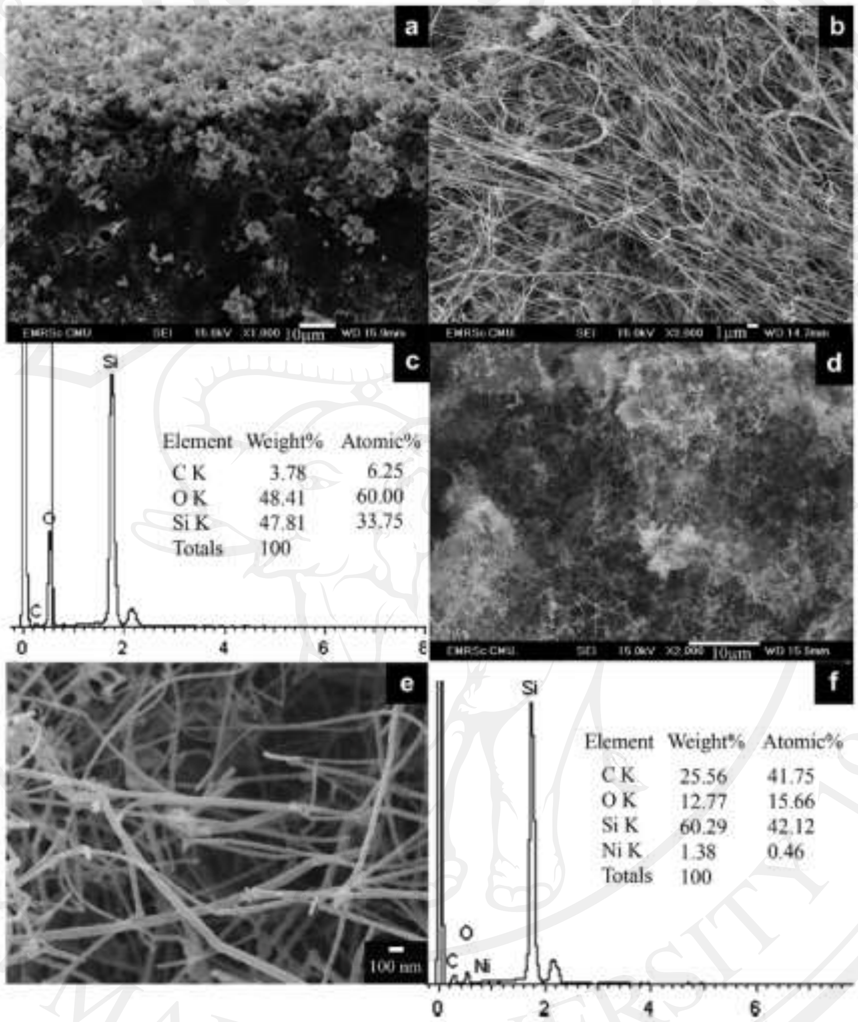

Fig. 1. SEM images of (a) cross section of white-wool covered as-grown powder surface, (b) FE-SEM image of white-wool product, (c) shows EDS spectra of the white wool-like features, (d) and (e) FE-SEM images of as-grown product and (f) is the corresponding EDS spectra.

magnification of the white wool-like features that is SiO_2 nanofibers, confirmed with EDS spectra is given in Fig. 1c. The EDS mass ratio reveals that these nanofibers consist of Si, O and C. The mass ratio of Si to O (Si:O) is 1:1.01 and the atomic ratio is 1:1.78 and this composition is very close to stoichiometric SiO2 composition. whereas, the dark gray powders were under the white-wool cover became to the light green-gray product.

Fig. 1(d) shows the FE-SEM image of the light green-gray product illustrating that the formed straight wire-like structures. Fig. 1(e) shows detail structures of the obtained nanowires which is suggested that these nanowires have a smooth and clear surface with the range of about 20–90 nm diameter and length up to several micrometer. EDS spectra of these nanowires was given in Fig. 1(f) which indicated that these of nanowires were composed of Si, C, O and Ni elements. The atomic ratio of Si:C:O is 42.12:41.75 and this composition is very close to stoichiometric SiC. The weak Ni peak came from Ni_2O_3 compound that was acted as catalyst. In order to characterize the structure in further details, the as-grown product was investigated by TEM and the SAED pattern are showed in Fig. 2 which presents a typical nanowire. The nanowire was characterized with core–shell structure which has the diameter of core of nanowire was 30 nm and it was wrapped with a uniform layer of amorphous with a thickness of 15 nm, whereas the SAED pattern along [011] zone axis in Fig. 2a (inset) shows clear spots indicated that the core of nanowire is a single crystalline structure, it can be indexed to 3C–SiC structure. Also, it can be seen that the SiC nanowire possess a high density of stacking faults which are perpendicular to the wire axis as seen in Fig. 2b. The crystal lattice image clearly shows that the spacing of lattice fringes is 0.25 nm as showed in Fig. 2c which corresponds to the {111} planes spacing of cubic SiC. It further demonstrates that the angle of lattice fringes between [111] and the wire axis is 73° which is very close to the

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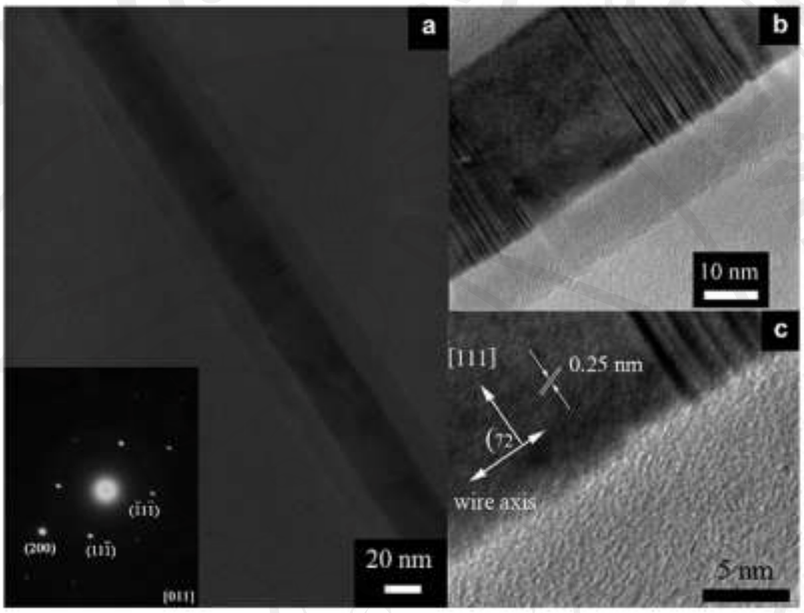

Fig. 2. (a) TEM image of SiC nanowire and its SAED pattern (inset) (b) HRTEM image of SiC nanowire shows a single crystal and shell structure (c) HRTEM image of a SiC nanowire with higher magnification.

angle between of $(\bar{1}1\bar{1})$ plane and $(11\bar{1})$ plane (70.53°) of 3C–SiC. From these results, the growth direction of the nanowires can be assumed to be [111] direction. Phase identifications of the products were characterized by XRD as showed in Fig. 4. There are five peaks in the spectrum agreeing well with the known values of (111), (200), (220), (311) and (222) diffraction peaks of cubic β-SiC as showed in Fig. 3. Besides, the lattice constants $a = 4.345$ Å, in good agreement with $a = 4.349$ Å (JCPDS card no. 73-1665).

Fig. 4 shows the FT-IR spectrum of the nanowires, which was two absorption bands from Si–O stretching vibration at about 1093 and 474 cm^{-1} transversal optic (TO) mode of Si–C vibration at about 804 cm^{-1}. The results indicated that the nanowires were composed of SiC and amorphous of silicon oxide and corresponding with previous reports [18].

The proposed growth mechanism of SiC nanowires are attributed to vapor–liquid–solid (VLS) growth mechanism [19]. Although, the spherical droplets of the catalysts were not found at the tip of each SiC nanowire, which was probable that the metal catalysts dislodged during cool down [20]. Besides, both of them were wrapped with thin SiO_x layer.

According to the results above, the possible formation mechanism of SiC nanowires in this method may be as follows, firstly, SiO_2 were generated due to reaction of Si powder with O during heating.

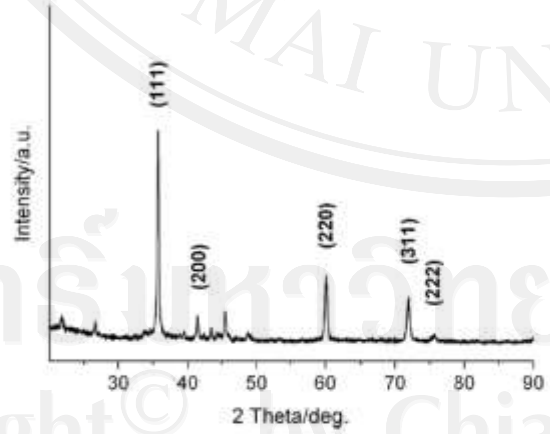

Fig. 3. X-ray diffraction spectra of product: after heat-treated. It can be indexed to be β-SiC (JCPDS Card No. 73-1665).

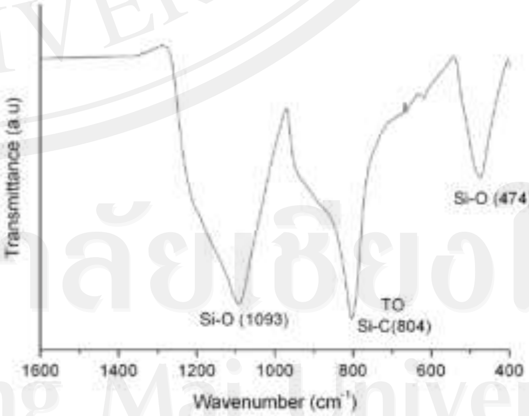

Fig. 4. FT-IR spectrum of the core–shell nanowires.

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At the same time, metal catalyst vapor and CO vapor were also generated. Secondly, a carbonthermal reduction took place and SiO vapor was formed [21]. Then, melted catalyst Si and C condensed and or SiO to formed liquid droplet alloys of the metal catalyst, Si, C and O. Since the temperature descends downstream, the vapor of Si and C condense and become supersaturated and then the coexisting Si and C precipitate as nanowires. Finally, the unreacted SiO and O took place to form the amorphous SiO_x, which was deposited on the SiC nanowires and formed on the surface of product [9].

4. Conclusions

Large-scale of SiC nanowires with 20–90 nm in diameter and length up to several microns have been synthesized by simple and low-cost CVD method at 1350 °C. These nanowires consist of a single crystalline β-SiC core and a layer of amorphous SiO_x, which correspond to the FT-IR spectrum and growth direction is [111]. This experiment, the SiC nanowires were believed that formation by VLS mechanism.

Acknowledgments

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IMPROVEMENT OF MECHANICAL PROPERTIES OF CNTs, SiCNWs AND EPOXY RESIN NANOCOMPOSITES

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Abstract: An experimental investigation was carried out to study the fabrication of carbon nanotubes (CNTs), silicon carbide nanowires (SiCNWs) and epoxy resin nanocomposites by casting technique. Different volume percentages of SiCNWs and CNTs were dispersed in epoxy resin by using the ultrasonic mixing. Physical and mechanical properties such as density, tensile strength, and compression of composite samples were examined. Furthermore, microstructures of samples were also investigated by scanning electron microscopy (SEM). The results indicated that adding the SiCNWs and CNTs could be used for improvement mechanical properties of the epoxy resin.

1. Introduction

Since the discovery of carbon nanotubes by S. Iijima in 1991 [1], an increasing number of articles were devoted to the synthesis, characterization and utilization of CNTs, as well as their applications. CNTs, as a novel crystalline carbon form, are unique nanostructured materials with remarkable physical and mechanical properties [2,3], such as high tensile strength and young's modulus [4,5], high thermal conductivity [6]. Besides, numerous nanoscale materials are being explored such as nanobelts, nanorings and nanowires because of their great potentials for understanding basic issues [7,8]. One of them is silicon carbide nanowires (SiCNWs), due to exhibits many excellent properties for high temperature, high frequency and high power applications. Especially, its excellent mechanical properties make this material a promising application in reinforcing metal, ceramic and other chemical composites [9].

To date, polymers are very important materials for a wide range of applications including aircraft, packaging, sport, electronics products, automotive and many other industrial applications [10,11]. In order to enhance the mechanical properties of original polymer materials, many researchers have attended to use CNTs as fillers in polymer composite systems, as well as another nanostructure material that is SiCNWs [12,13], due to excellent properties of them. Both hold the promise of delivering superior composite materials with high strength, light and have attracted great attention. However, using of CNTs and SiCNWs as fillers in epoxy systems, most of works focus on either CNTs or SiCNWs. Commixtures of CNTs and SiCNWs for improvement properties of materials have not yet been reported.

In this work, nanocomposites samples between CNTs, SiCNWs and epoxy resin were fabricated using ultrasonic mixture and casting techniques. The fillers are CNTs and SiCNWs, were synthesized by chemical vapor deposition (CVD) method. Physical and mechanical such as tensile strength, compressive strength and density of the nanocomposites samples were investigated. Moreover, microstructures of samples were determined by SEM technique.

2. Materials and Methods

2.1 Fillers Synthesis

CNTs were synthesized via an infusion CVD method using ethanol as the carbon source. The experiment started with a flow of infusing ethanol into a tube furnace containing an amount of nickel oxide powder. Nickel oxide was reduced by the ethanol vapor at 450 °C for 30 min before the MWCNTs were synthesized at 700 °C for 9 h [14].

SiCNWs were synthesized in a horizontal alumina tube furnace by CVD method. The raw materials, Si powder (99%, Aldrich) and C powder (ultra high pure graphite 99.9%) were mixed with a few amount of Ni_2O_3 powder. The mixed powders were filled into an alumina boat and placed in the furnace and then pumped out initially by vacuum pump and high purity argon was released into the furnace for three times to estimate the residual air. After that, the furnace was heated to 1350°C by rating of 10°C/min and kept for 1 h. SiCNWs were formed.

In order to remove impurities such as amorphous carbon and metal catalyst, the CNTs and SiCNWs were further heat-treated at 700°C for 4 h and mixed-acid treated by 1 vol% of diluted sulfuric and nitric acid. The products were washed by distilled water, absolute ethanol. After drying step at 150 °C during 4 h, the final products were obtained and subsequently mixed.

2.2 Nanocomposites Fabrication

A matrix was an epoxy resin (Thai Epoxy Resin Ltd., Thailand) mixed with its hardener; the proportion was 100 parts of resin to 27 parts of hardener by volume. To fabricate composites, the modified CNTs and SiCNWs were manually suspended in epoxy resin with the different ratios varying from 0.05% to 0.2%

by volume of each filler. Firstly, the mixtures of epoxy resin and the fillers were dispersed for 3 h in an ultrasonic bath. Secondly, hardener was added into the mixture and mixed in ultrasonic bath for 10 min. Thirdly, the mixture was poured into the rubber mold for tensile test. The shape and size of the mold is shown in figure 1. It is noted that mylar films were put at the upper and the lower of the samples in order to obtain a smooth surface. Finally, the dog-bone shaped flat composite specimens were left to settle at the room temperature for one day and then removed from the mold. Besides, the specimens for compression test were prepared in rectangular shape mold, then cut and mechanically polished surfaces and edges of samples until sizes of about 8x8x12 mm. Density of samples were measured by using Archimedes method. Tensile and compression tests of the samples were carried out by the universal testing machine (Instron Instruments) with a constant cross-head rate at 1 and 3 mm/min, respectively. The choice of this quite low loading rate is to consider the brittle character of composites. Moreover, the microstructure of the nanocomposites samples were investigated by SEM.

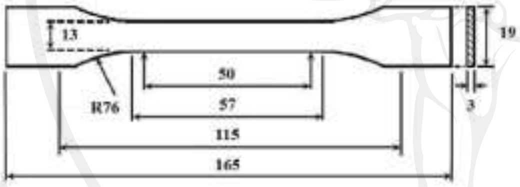

Figure 1. The tensile test sample geometry.

3. Results and Discussion

3.1 The morphologies of CNTs and SiCNWs

Field emission scanning electric microscopy (FESEM) of the random CNTs and SiCNWs are shown in figure 2a and b, respectively. As can be seen from figure 2a, CNTs with diameters in ranges of 50-100 nm were highly entangled and randomly organized. The microstructures of SiCNWs are illustrated in figure 2b shows with diameters in ranges of 40-110 nm. The density of CNTs and SiCNWs were taken as 2.0 g cm^{-3}[14] and 3.217 g cm^{-3}, respectively.

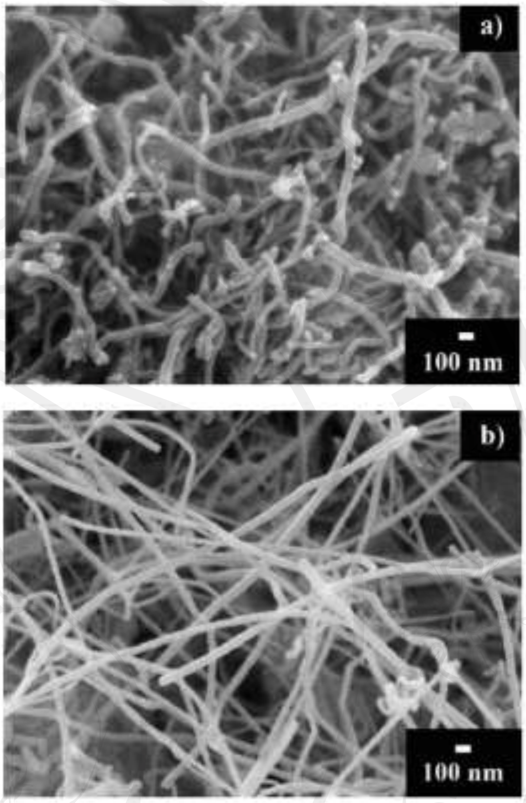

Figure 2. FESEM images of: a) CNTs and b) SiCNWs.

3.2 Effect of fillers on the mechanical properties of epoxy composites

Table 1 shows the density of epoxy resin nanocomposites were reinforced with CNTs and SiCNWs. The results show the density of nanocomposite samples that increased consistent with increasing of the fillers content, which is clearly more than neat epoxy resin (1.1586 g cm^{-3}). Besides, the nanocomposites were varied the ratio of CNTs:SiCNWs of each volume percent, the density slightly increased as the SiCNWs content increased.

Table 1 Density data for CNTs, SiCNWs and epoxy resin nanocomposites

Ratio of CNTs: SiCNWs	Content of nano-fillers by vol%		
	0.05	0.10	0.20
Epoxy		1.1586	
100:0	1.1605	1.1616	1.1626
75:25	1.1604	1.1622	1.1631
50:50	1.1598	1.1629	1.1642
25:75	1.1603	1.1625	1.1643
0:100	1.1626	1.1653	1.1696

Tensile and compression responses of nanocomposites are shown in figure 3. it is can be seen that epoxy with nano-fillers have the higher strength than neat epoxy and increased simultaneously with the addition of CNTs and SiCNWs which indicated that the addition of these nano-fillers could promote the strength of the composites. Moreover, the results exhibited that the ratio of CNTs: SiCNWs is 25:75 at 0.2 vol%, the tensile and compressive strength have the maximum values of 48.59 MPa and 122.30 MPa, respectively.

The samples were added either SiCNWs or CNTs, it as found that both nano-fillers added samples show nearly strength. Except in the case of 0.2 vol%, the SiCNWs added samples showed clearly higher strength than CNTs added samples. Possible reasons for such behavior could be that there are impurities contents in CNTs powder more than SiCNWs powder, due to treatment by acid-treated with 1 vol% of mixed acids could not remove all impurities and can be seen clearly when added more contents of CNTs powder in the matrix.

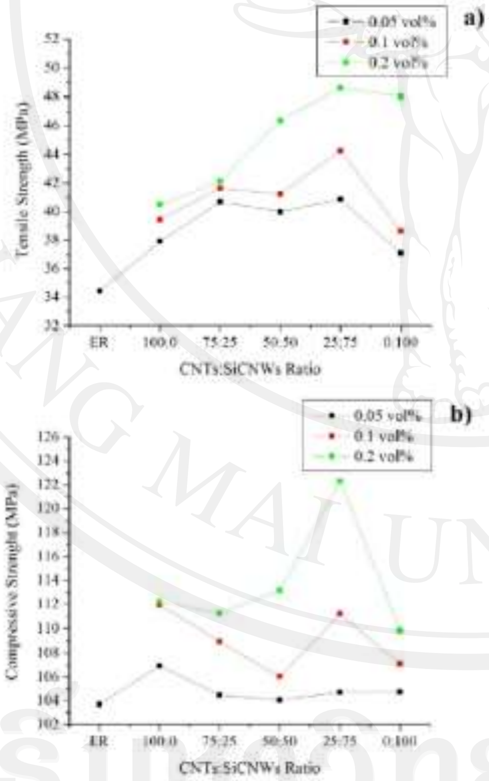

Figure 3Tensile response of CNTs and SiCNWs loading content (vol %) on the mechanical properties of epoxy nanocomposites: a) tensile strength, b) compression strength.

Gain in tensile and compressive strength of the ratio of 25:75 (CNTs:SiCNWs; 0.2 vol%) is about 41.13% and 17.65 % respectively over the neat as shown in Table 2 and 3. Adding SiCNWs content was 75 parts mixed with 25 parts of CNTs content on condition 0.2% by volume provided the highest percentage of tensile test. However, there are no significantly gains in compression tests in the same condition.

Table 2 the gain of tensile strength in percentage

CNTs:SiCNWs	Gain tensile strength in (%)		
100:0	10.09	14.52	17.63
75:25	18.15	20.96	22.34
50:50	16.11	19.75	34.68
25:75	18.63	28.54	41.13
0:100	7.77	12.20	39.47

Table 3 the gain of compression strength in percentage

CNTs:SiCNWs	Gain compression strength in (%)		
100:0	3.14	7.96	8.22
75:25	0.77	5.06	7.28
50:50	0.36	2.27	9.16
25:75	0.96	7.23	17.94
0:100	1.01	3.31	5.95

3.3 Morphology of the nanocomposite interface and dispersion of nanno-fillers in epoxy matrix

In order to have a better view of effect of CNTs and SiCNWs on the nanocomposites, SEM micrographs were taken and investigated for the nanophased systems. the samples were observed that the CNTs were not very well dispersed in the matrix. There are agglomerations of CNTs in some areas of matrix is shown in Figure 4a. Besides, the SEM image (inset) shows particles-to-particles interaction which became almost like impurities [15]. The possible reasons might be a weak van der waals attractive force between tube which effect to homogenization process in the viscous matrix was difficult. Increases of mechanical properties in this case are hence less than adding SiCNWs. From Figure 4b, the SEM image exhibits well dispersion of SiCNWs in the matrix and shows particles-polymer-particles interaction. However, the EDS analysis shows the elements of nanocomposites sample, it is found that there are dispersion of all CNTs and SiCNWs in the matrix and can promote the mechanical properties of epoxy resin especially, tensile and compressive strength.

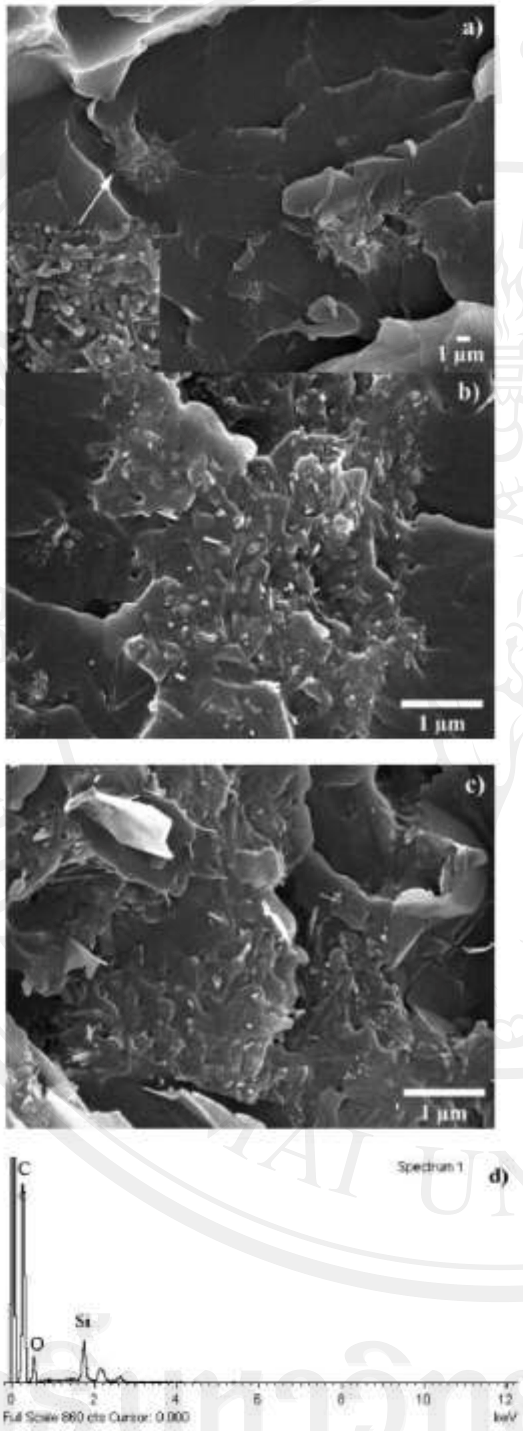

Figure 4. The SEM images of the surfaces of CNTs, SiCNWs and epoxy resin nanocomposites containing 0.2 vol% of the nano-fillers: a) CNTs 100% b) SiCNWs 100% c) CNTs:SiCNWs is 25:75 and d) EDS spectrum of the ratio 25:75 (CNTs:SiCNWs)

4. Conclusions

In this work, CNTs, SiCNWs and epoxy resin nanocomposites were successfully fabricated by using the ultrasonic mixing and casting technique. CNTs and SiCNWs were used as the nano-fillers to promote the mechanical properties of the epoxy resin. The results revealed that combination of CNTs and SiCNWs is a useful approach to improve strength of epoxy matrix. This experiment, the optimum of percentage of the nano-fillers added in the matrix is 0.2 vol% and proportion of CNTs:SiCNWs is 25:75 which shows the highest strength are 48.59 MPa of tensile and 122.30 MPa of compression.

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