เอกสารอ้างอิง

1. P.K. Mallick, *"Fiber-reinforced composites: materials, manufacturing and design"*, Marcel Dekker, Inc., New York 10016, 1993.
2. เสนีย์ พันโยธา. <u>การผลิตรถยนต์</u>. สำนักพิมพ์ศูนย์ส่งเสริมอาชีวะ: กรุงเทพฯ. 2545.
3. Jones RM. "Mechanics of Composite Materials", Hemisphere Publishing Company, New York, 1987.
4. Friedriech K, Walters R, Carlsson LA, Smiley AJ, Gillespie JW, Mechanisms of rate effects on interlamina fracture toughness of carbon/epoxy and carbon/PEEK composites. *J.Mater.Sci.* 1989; **24**:3387-98.
5. Truss RW, Hine PJ, Duckett RA. Interlamina and intralaminar fracture toughness of uniaxial continuous and discontinuous carbon fibre/epoxy composites. *Composites Part A* 1977; **28**: 627-36.
6. Albertsen H, Ivens J. Interlaminar fracture toughness of CFRP influenced by fibre surface treatment:: Part 1. Experimental results. *Compos Sci. Technol.* 1995; **54**: 133-45.
7. Kotaki M, Hamada H. Effect of interfacial properties and weave structure on mode I interlaminar fracture behaviour of glass satin woven fabric. *Composite Part A* 1997; **28**: 257-66.
8. Wagner HD, Wiesel E, Green AK, Evidence of dynamic effect in the fragmentation of optical fiber in epoxy. *Composites Part A* 1998; **29**: 989-91.
9. Day RJ, Rodriges JVC. Investigation of the micromechanics of the microbond test. *Compos Sci Technol* 1998; **58**: 907-14.
10. Varelidis PC, Papakostopoulos DG, Pandazis CI, Papaspyrides CD. Polyamide coated Kevlar fabric in epoxy resin: mechanical properties and moisture absorption studies. *Composites Part A*. 2000; **31**: 549-58.
11. Kllantar J, Drzal LT. The bonding mechanism of aramid fiber to epoxy matrices. *J.Mater.Sci.* 1990; **25**: 4186-93.
12. Mai YW, Castino F. Fracture toughness of Kevlar-epoxy composites with controlled interfacial bonding. *J.Mater.Sci.* 1984; **19**: 1638-55.
13. Li R, Ye L, Mai YW. Application of plasma technologies in fibre-reinforced polymer composites: a review of recent developments. *Composites Part A*, 1997; **28**: 73-86.
14. Brown JR, Chappell PJC, Mathys Z. Plasma surface modification of advanced organic fibers. *J.Mater.Sci.* 1990; **26**: 4172-8.

15. Andrews MC, Bannister DJ, Young RJ. Review: The interfacial properties of aramid/epoxy model composites. *J.Mater.Sci.* 1996; **31**: 3893-913.

16. Aronhime MT, Neumann S, Marom G. The anisotropic diffusion of water in Kevlar-epoxy composites. *J.Mater.Sci.* 1984; **22**: 2435-46.

17. Krishan K. Chawla, "*Composite Materials: Science and Engineering*", Springer-Verlag, New York, Inc., 1998.

18. E. Dujardin, T.W. Ebbessen, A. Krishnan, P.N. Yiailos, M.M. Treacy, *Phys. Rev. B*, **58**, 14013-14019 (1998).

19. M.F. Yu, B.S. Files, S. Arepalli, R.S. Ruoff, *Phys. Rev. Lett.*, **84**, 5552 (2000).

20. S. Iijima, *Nature*, **345**, 56 (1991).

21. J.W. Mintmire, B.I. Dunlap, and C.T. White, *Phys. Rev. Lett.*, **68**, 631 (1992).

22. N. Hamada, S. Sawada and A. Oshiyama, *Phys. Rev. B*, **68**, 1579 (1992).

23. R. Saito, M. Fujita, G. Dresselhaus and M.S. Dresselhaus, *Phys. Rev. B*, **46**, 1804 (1992).

24. Y. Ye, C.C. Ahn, C. Witham, B. Fultz, J. Liu, A.G. Rinzler, D. Colbert, K.A. Smith, R.E. Smalley, *Appl. Phys. Lett.*, **74**, 2307-2309 (1999).

25. C. Liu, Y.Y. Fan, M. Liu, H.T. Cong, H.M. Cheng, M.S. Dresselhaus, *Science*, **286**, 1127-1129 (1999).

26. H.J. Dai, J.H. Hafner, A.G. Rinzler, D.T. Collbert, R.E. Smalley, *Nature*, **384**, 147-150 (1996).

27. S.S. Wong, E. Joselevich, A.T. Woolley, C.L. Cheung, C.M. Lieber, *Nature*, **394**, 52-55 (1998).

28. R.S. Ruoff, D.C. Lorents, *Carbon*, **33**, 925-930 (1995).

29. William, F.S. *Principle of Material Science and Engineering*, 3rd ed., New York: McGrawHill, 1996.

30. *Institute for Research in Construction. (no date). "Properties and Behavior of Plastics" [Online]. Available http://www.irc.nrc-cnrc.gc.ca (20 February 2008).*

Output จากโครงการวิจัยที่ได้รับทุนจาก สกว.

1. **Nhuapeng W.,** Thamjaree W., Kumfu S., Singjai P. and Tunkasiri T., Fabrication and Mechanical Properties of Silicon Carbide Nanowire / Epoxy Resin Composites, *Current Applied Physics*, 2008, 8: 295-299.
2. **Nhuapeng W.**, Kumfu S., Thamjaree W.and Tunkasiri T., Fabrication of Double Layers of Aramid Fiber/Alumina/Epoxy Resin Laminate Composite Using Low-Pressure Technique, *Advanced Materials Research Vols. 55-57 (2008) pp 457*
3. W. Thamjaree, **W. Nhuapeng** and T. Tunkasiri, Fabrication of Barium Zirconium Titanate Ceramics Using Ultrasonic Ball Milling Technique, *Advanced Materials Research Vols. 55-57 (2008) pp 213*
4. H. Longkullabutra, **W. Nhuapeng**, W. Thamjaree and T. Tunkasiri, Mechanical Properties of Hemp Fiber Composites with Carbon Nanotubes Reinforcement, *Advanced Materials Research Vols. 55-57 (2008) pp 553*

ภาคผนวก

Available online at www.sciencedirect.com
ScienceDirect
Current Applied Physics 8 (2008) 295–299
ELSEVIER
Current Applied Physics
An official journal of the K&S
www.elsevier.com/locate/cap
www.kps.or.kr

Fabrication and mechanical properties of silicon carbide nanowires/epoxy resin composites

W. Nhuapeng, W. Thamjaree *, S. Kumfu, P. Singjai, T. Tunkasiri

Department of Physics, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand

Abstract

An experimental investigation was carried out to study the fabrication of silicon carbide nanowires (SiC NWs)/epoxy resin composites. This material could exhibit excellence mechanical properties for example, hardness, wear resistant and especially lightweight which may be used to replace metal parts in vehicles. The SiC NWs/epoxy resin composites were prepared by using the ultrasonic mixing and casting techniques. The physical and mechanical properties such as density, tensile strength, hardness and wear test of composite samples were examined. Furthermore, microstructures of samples were also investigated by scanning electron microscopy (SEM). It was found that the ultrasonic mixing could be used for fabricating high wear strength samples of SiC NWs/epoxy resin composites.

PACS: 81.05.Mh; 81.05.Ni; 81.05.Pj; 81.05.Ok

Keywords: Composites; Silicon carbide nanowires; Epoxy resin

1. Introduction

In the recent years, there is a great need for composites because the combination of two or more materials can lead to enhance performance and outstanding properties compared to their constituents [1,2]. Especially, the polymer-based composites reinforced with a small percentages of strong fillers can significantly improve the mechanical, thermal and barrier properties of the pure polymer. Moreover, these improvements are achieved through conventional processing techniques without any detrimental effects on processability, appearance, density and aging performance of the matrix [3]. The realization of their unique properties, it has been considering for a wide range of applications including packaging, coating, sport, electronics, aerospace industries, aircraft and military, automotive, and marine engineering [3–6]. The conventional fibers, for examples, glass-fiber, carbon-fiber and aramid-fiber are widely used in nowadays. However, these materials are still limited in some applications, especially, the aircraft engine and aerospace industries which need high temperature materials, high mechanical properties that are lightweight [7]. Therefore, there are many works focus on the reinforcing polymer-based materials have incorporated various particle/whisker-type fillers especially, the functionally graded materials (FGMs) to fabricate the high performance materials for space-planes [8,9]. The new type filler, silicon carbide nanowires (SiC NWs) have been attracting considerable attention due to their excellent properties such as high thermal stability, high thermal conductivity, good mechanical properties and chemical inertness [10,11]. Besides, it has been suggested as good reinforcement materials and suitable to be used as the reinforcing material for composites due to their much larger strength over their bulk counterparts and strong interfacial bonding [12]. It is reported that the elastic modulus and ultimate bending strengths of SiC nanorods with several tens of nanometer thick were 610–660 and 53.4 GPa, respectively. The strength of these nanorods is a factor of two or more times higher than earlier observations for

---

* Corresponding author. Fax: +66 5335 7512.
E-mail address: msrlwt@yahoo.com (W. Thamjaree).

1567-1739/$ - see front matter © 2007 Elsevier B.V. All rights reserved.
doi:10.1016/j.cap.2007.10.074

SiC whiskers of micrometer diameter [13]. Therefore, they can be used in nanoelectronics, field emission device, biomedical engineering, nanocomposites and applications in high temperature nanoscale devices [10,14,15]. Although there are many research works on SiC NWs, most of the works focus on the fabrication of SiC NWs and properties of SiC/ceramic composites [12,16–18]. However, improvement properties of materials by using SiC NWs as fillers in polymer matrix composites have not yet been reported.

In this work, composites samples between SiC NWs and epoxy resin were fabricated using ultrasonic mixing and casting techniques. The reinforced fiber, SiC NWs was synthesized by current heating technique (CHT). Physical and mechanical properties such as hardness, wear resistance, tensile strength and density of the composites samples were investigated. Moreover, microstructure of samples was determined using SEM technique.

2. Experimental procedure

The SiC NWs were synthesized via current heat technique (CHT) [19]. The raw materials, carbon powder (ultra high pure graphite, 99.9%), SiO_2 (Silica gel 60, Fluka) and Al_2O_3 (Extra pure, 98%, Reidel-de Haen) were mixed and pressed into rod shape with 12 mm in diameter and 25 mm in length. The rod was heated by DC power supply for 5 min under argon atmosphere. SiC NWs was taken out from surface of the rod were ground by using agate mortar for 10 min to get rid of hard agglomeration. A matrix was created by mixing epoxy resin (Thai Epoxy Resin Ltd., Thailand) with its hardener in the ratio 100:27 by volume. The density of the matrix is 1.176 g/cm^3. To fabricate composites, firstly, SiC NWs were added into epoxy resin with the different ratios varying from 5% to 15% of volume. Firstly, the mixtures of epoxy resin and SiC NWs were dispersed for 3 h in an ultrasonic bath. Secondly, hardener was added into the mixture and mixed in ultrasonic bath for 5 min. Thirdly, the mixture was poured into the rubber mould in the dog-bone shape (Fig. 1) with a sample thickness of 1 mm. 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 composite samples were left to settle at the room temperature for one day and then removed from the mould. Density of samples was measured by using Archimedes method. Tensile strength of the samples was measured by the universal testing machine (Lloyd Instruments, LRX) with a cross-head rate at 50 mm/min. The hardness was tested by Knoop's hardness testing machine (HK, Brooks Inspection Equipment Ltd., England). The wear resistance was examined by the Pin-on-Disk Tribometer with ball radius of 4 mm. Moreover, the microstructure of the composites samples was investigated by SEM.

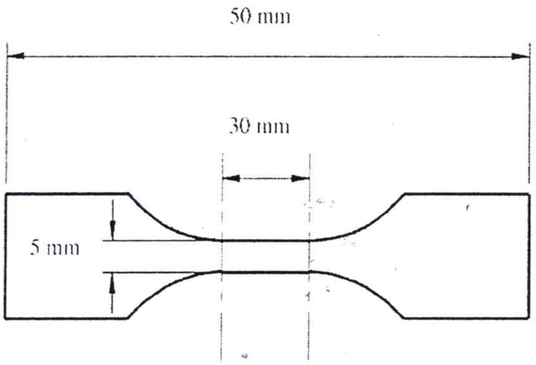


Fig. 1. The tensile test of composites samples.

3. Results and discussion

Table 1 shows density, hardness, width of wear track and tensile strength values of SiC NWs/epoxy resin composite samples. The results showed that density of composites samples increased with increasing of SiC NWs content which is in the range of 1.1455–1.1567 g/cm^3. However, it is noted that the density of the composites samples is higher than pure SiC NWs (0.996 g/cm^3) and slightly less than that of the pure epoxy resin (1.176 g/cm^3). Moreover, the hardness (HK) and tensile strength are in the range of 1.3–6.3 HK and 5.40–23.81 MPa, respectively. Furthermore, it is observed that the widths of wear track of samples are in the range of 329–550 μm. It is observed that hardness and tensile strength of 15 vol% SiC NWs composites increased to 384% and 341%, respectively, and the wear track of samples decreased to 40% when compared with that of pure epoxy resin. Fig. 2 shows the relationship between coefficient of friction and distance, it shows that friction values of pure epoxy resin are much higher than that of composites. The coefficient of friction of pure epoxy decreased to 83% when adding the SiC NWs into polymer phase. It could be assumed that adding SiC NWs into polymer-based, epoxy resin would decrease the friction and promote the wear resistance of the pure polymer. Fig. 3 shows the photographs of the width of wear track of pure epoxy resin and composites samples. It exhibits that the width of wear track are dramatically decreased (from 550 to 329 μm) with increasing of SiC NWs (0–15 vol%) contents which confirm that wear resistance of soft and ductile materials could be improved by using the SiC NWs filler.

Table 1
The density, hardness, width of wear track and tensile strength of composites samples

vol% of SiC NWs (%)	Density (g/cm^3)	Hardness (HK)	Width of wear track (μm)	Tensile strength (MPa)
0	1.176	1.3	550.0	5.4
5	1.14549	3.7	371.0	9.40
10	1.14747	4.7	356.0	15.41
15	1.15674	6.3	329.8	23.81

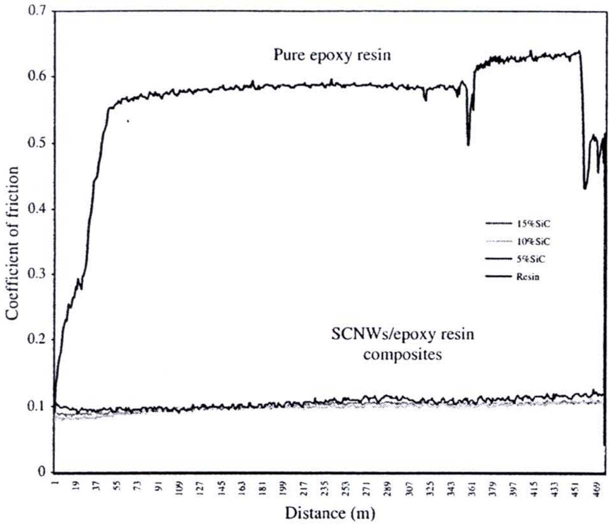


Fig. 2. Wear resistance of pure epoxy resin and SiC NWs/epoxy resin composites.

Fig. 4 shows the typical SEM micrographs at different magnifications and EDS spectrum of the obtained products. It reveals that the products consist of a large quantity of wire-like nanostructures with several micrometers in length and diameters varying from 50 to 70 nm. It is also revealed that SiC NWs were randomly oriented with straight or curved morphologies. These nanowires generally display smooth surface and very uniform diameter. Besides, the catalyst particles are observed at the end tip of wires as shown in the higher magnification SEM micrograph (Fig. 4). The EDS analysis showed that the elements of nanowires with a wrapping layer contain silicon, carbon and oxygen. The wrapping layer is thought to be SiO_2. This may be due to the reaction between SiC NWs and the remaining oxygen in the furnace. Fig. 5 shows the SEM micrographs of composites samples with different SiC NWs contents. It can be seen that the brighter phase is the filler phase (SiC NWs) whereas, the grey phase is matrix phase (epoxy resin). The fracture surface of the nanocomposites was coarse, which may imply that the composites have good toughness because of the presence of nanowires. Mostly area covered with polymer phase when small quantity of SiC NWs (1.0 vol%) was added into the sample as shown in Fig. 5a. However, very well distribution of nanowires can be obtained when the content of SiC NWs is increased for 5 vol% and 10 vol%, respectively. Moreover, it shows that epoxy resin evenly impregnated throughout the nanowires, which indicates good resin impregnation (Fig. 5b and c). Fig. 5d shows the micrograph of 15 vol% composites, it is found that the SiC NWs was covered by polymer matrix and some parts of SiC NWs embedded into epoxy resin which may cause to the much higher of mechanical properties. Therefore, it can be reported that adding the nanowires into the polymer-based phase can promote the mechanical of single phase polymer-based especially, hardness and tensile strength.

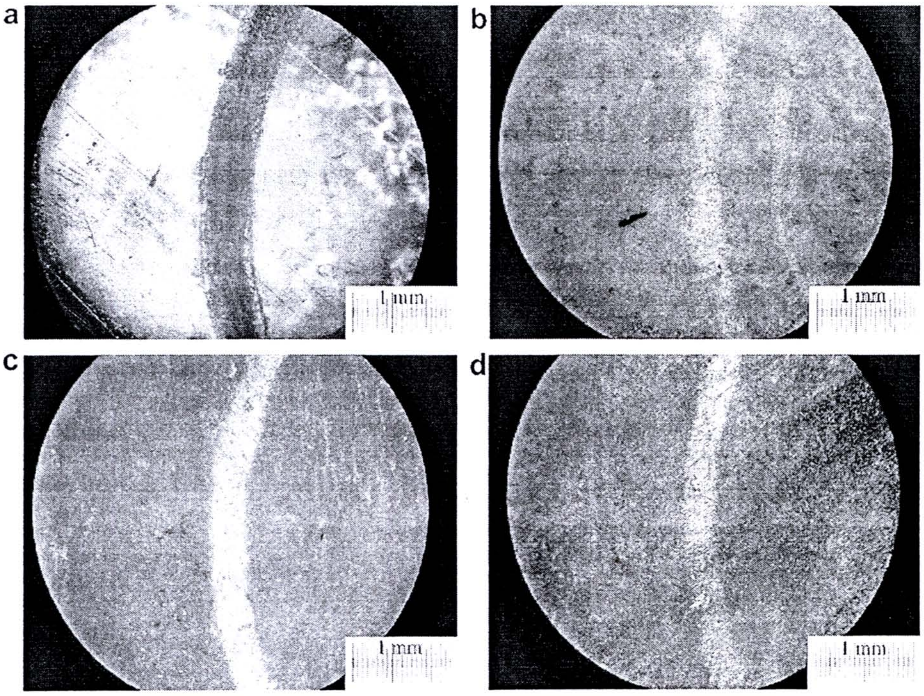


Fig. 3. Wear track of composites samples: (a) pure epoxy resin, (b) 5 vol%, (c) 10 vol% and (d) 15 vol% of SiC NWs.

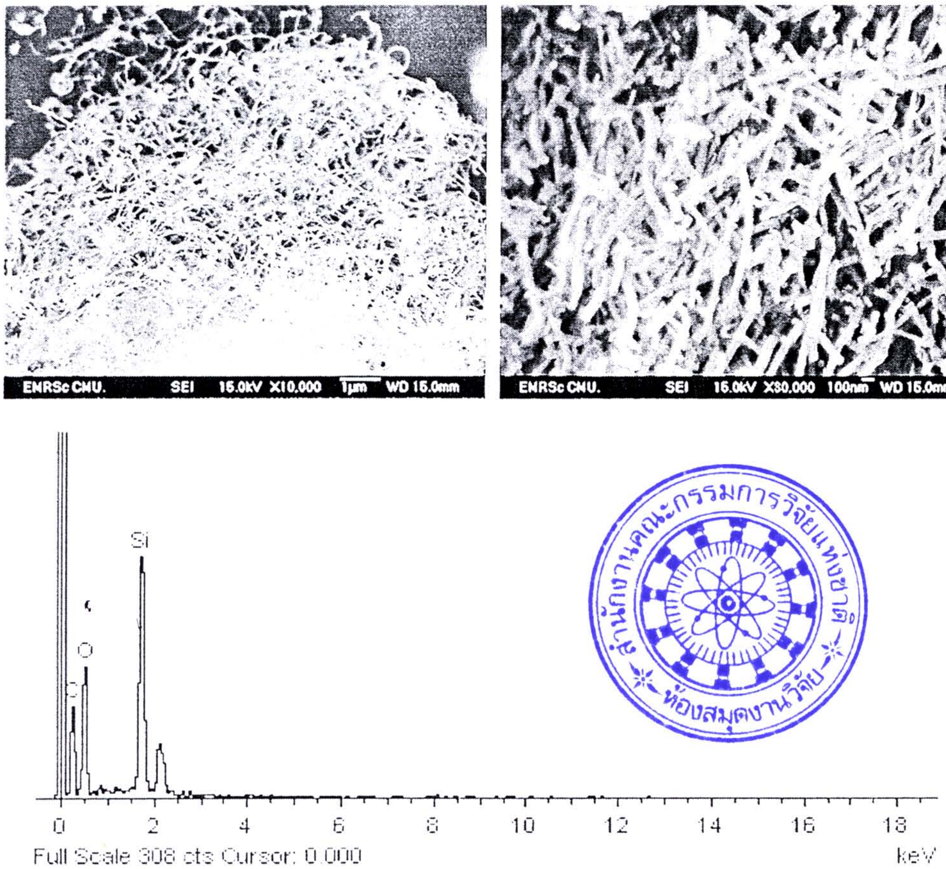


Fig. 4. SEM micrographs and EDS spectrum of SiC NWs.

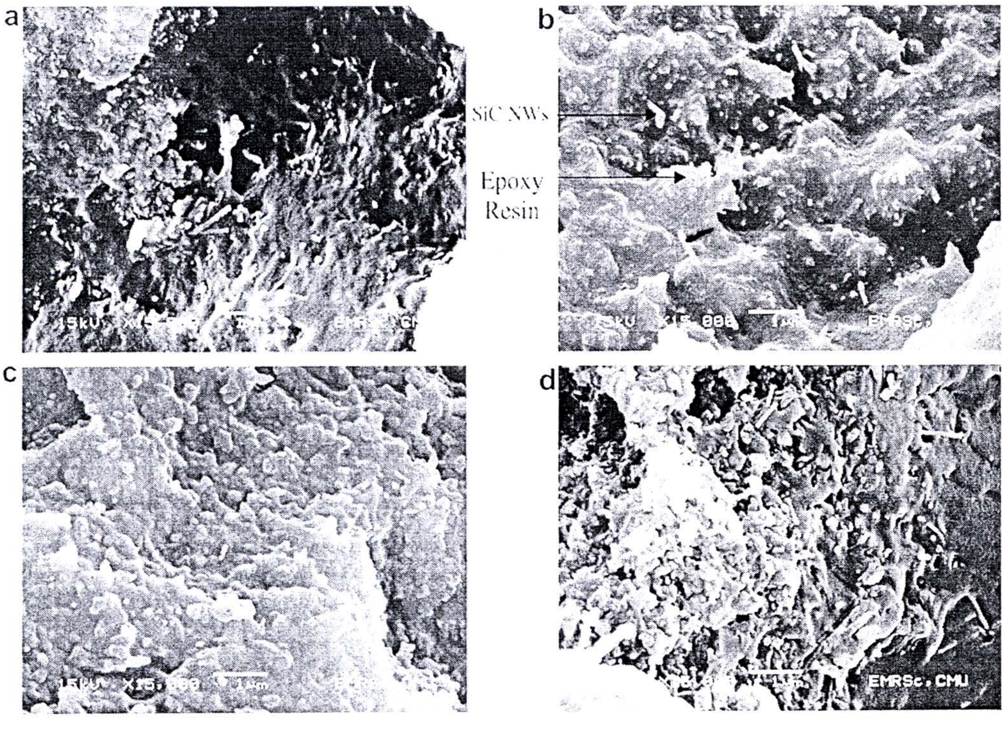


Fig. 5. SEM micrographs of SiC NWs/epoxy resin composites (a) 1 vol%, (b) 5 vol%, (c) 10 vol% and (d) 15 vol%.

4. Conclusion

SiC NWs/epoxy resin composites were fabricated by using the ultrasonic mixing and casting techniques. The SiC NWs were used as the reinforced fiber to promote the mechanical properties of the composites. The results revealed that the SiC NWs were well-dispersed within the epoxy resin matrix. The wear resistance of polymer-based materials could be improved by adding SiC NWs. The composites with 15 vol% of SiC NWs show the best wear resistance with the width of wear track of 329.8 μm which is much less than that of pure epoxy resin (550 μm) while there is no significant change in weight. Moreover, the hardness and tensile strength of composites was improved to 384% and 341%, much higher than that of pure epoxy resin.

Acknowledgements

The authors would like to thank the Thailand Research Fund (TRF) and National Nanotechnology Center (Thailand) for supporting this research. And the authors also would like to express their sincere thanks to the Department of Physics, Faculty of Science, Chiang Mai University for all the facilities.

References

[1] X.F. Liu, C.X. Xiong, H.J. Sun, L.J. Dong, R. Li, Y. Liu, Mater. Sci. Eng. B 127 (2006) 261–266.
[2] R.J. Day, J.V. Cauich Rodrigez, Comp. Sci. Techol. 58 (1998) 907–914.
[3] N. Chisholm, H. Mahfuz, V.K. Rangari, A. Ashfaq, S. Jeelani, Comp. Struct. 67 (1) (2005) 115–127.
[4] P.K. Mallick, Fiber-reinforced Composites: Materials, Manufacturing and Design, Revised and Expanded, second ed., Marcel Dekker, Inc., NY, 1993.
[5] M. Walker, R. Smith, Comp. Struct. 80 (2002) 1457–1460.
[6] M.S. Mel, Composite Materials, Properties, Nondestructive, Testing and Repair, vol. 1, Prentice-Hall, NJ, 1994.
[7] K.L. Luthra, H.D. Park, J. Am. Ceram. Soc. 75 (7) (1992) 1889–1898.
[8] S. Chand, J. Mater. Sci. 35 (2000) 1303–1313.
[9] M. Krumova, C. Klingshirm, F. Haupert, K. Friedrick, Compos. Sci. Technol. 61 (2001) 557–5563.
[10] Q.G. Fu, H.J. Li, X.H. Shi, K.Z. Li, J. Wei, Z.B. Hua, Mater. Chem. Phys. 100 (2006) 108–111.
[11] Y. Ying, Y. Gu, Z. Li, H. Gu, L. Cheng, Y. Qian, J. Solid State Chem. 177 (2004) 4163–4166.
[12] W. Yang, H. Araki, A. Kohyama, S. Thaveethavorn, H. Suzuki, T. Noda, Mater. Lett. 58 (2004) 3145–3148.
[13] E.W. Wong, P.E. Sheehan, C.M. Lieber, Science 277 (1997) 1971.
[14] X.W. Du, X. Zhao, S.L. Jia, Y.W. Lu, J.J. Li, N.Q. Zhao, Mater. Sci. Eng. B 136 (2007) 72–77.
[15] J.Y. Fan, X.L. Wu, P.K. Chu, Prog. Mater. Sci. 51 (2006) 983–1031.
[16] Y. Baek, Y. Ryu, K. Yong, Mater. Sci. Eng. C. 26 (2006) 805–808.
[17] S. Tang, J. Deng, H. Du, W. Liu, K. Yang, J. Am. Ceram. Soc. 88 (1) (2005) 3253–3255.
[18] T. Ito, K. Sano, T. Akiyama, K. Nakamura, Thin Solid Films 508 (2006) 243–246.
[19] T. Jintakosol, P. Singjai, Key Eng. Mater 353–358 (2007) 2171–2174.

Advanced Materials Research Vols. 55-57 (2008) pp 457-460
online at http://www.scientific.net
© (2008) Trans Tech Publications, Switzerland

Fabrication of Double Layers of Aramid Fiber/Alumina/Epoxy Resin Laminate Composite Using Low-Pressure Technique

W. Nhuapeng[a], S. Kumfu[b], W. Thamjaree*[c] and T. Tunkasiri[d]
Department of Physics, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand
[a]nhuapeng@yahoo.com, [b]supreyak@yahoo.com, [c]msrlwt@yahoo.com, [d]tawee@chiangmai.ac.th

Keywords: Composites, Aramid, Al_2O_3, Epoxy resin

Abstract. An experimental investigation was carried out to study the fabrication of double layers of aramid fiber/alumina/epoxy resin laminate composites using low-pressure technique. To obtain the sample with high wear resistance property, the micron size of alumina particles were added into epoxy resin using the ultrasonic processor. The physical and mechanical properties such as density, hardness, impact test, wear resistance and tensile strength of the composites samples were investigated. Furthermore, microstructures of specimens were also examined by scanning electron microscope (SEM). From the results, it was found that the mechanical properties of double layers laminate composites of aramid fiber/alumina/epoxy resin could improve by using the low-pressure technique.

Introduction

Epoxy resin systems are increasingly used as matrices in composite materials for a wide range of automotive and aerospace applications, and for shipbuilding or electronic devices because of its excellent bonding, thermal and mechanical characteristics [1-2]. Aramid fiber is one of the most interesting fibers, because of their good mechanical properties for example, light–weight, high specific modulus and strength, high thermal resistance and chemical inertness. Furthermore, it exhibits low electrical conductivity when compared to metal alloy and carbon fiber [3]. However, it has erosion resistance poorer than metallic materials [4]. The scientist added ceramic phase into the composites to improve the property of the fiber–reinforced composite materials [5]. Al_2O_3 is known to be an important ceramic material because of its outstanding combination of chemical and physical properties such as excellent resistance to heat and wear, high specific strength, and good oxidation resistance. However, the major problem with the use of Al_2O_3 as a structural material is its inherent brittleness, which is the common characteristic of ceramic materials [6]. In order to enhance the bending force and interaction between Al_2O_3 and epoxy resin phase, the ultrasonic mixing was used for fabricating these laminate composites due to its high energy sonic wave utilization that can force an intrinsic mixing of particles with the matrix via sonic cavitations [7].

In the present work, the double layers laminated composites of aramid /Al_2O_3/epoxy resin were fabricated by using ultrasonic mixing and low-pressure technique. The physical and mechanical properties such as density, hardness, impact test, wear resistance and tensile strength were investigated. Moreover, microstructure of the samples was examined by using the scanning electron microscopy (SEM).

Experimental

In this work, the epoxy resin (Thai Epoxy Resin Ltd., Thailand) was used as matrix phase. The density of the matrix is 1.176 g/cm^3. The plain weave aramid fiber and Al_2O_3 powder (Aldrich, purity 99%, 10 microns) were used as the reinforcement phase. The Al_2O_3 powder was added into epoxy resin with the different ratios varying from 2, 5, 10, 12, 15 and 20 percents by volume. To fabricate the double layers laminated composites, firstly, aramid fiber was cleaned by dipping in the ethanol (Merck, purity 99%) and dried. Secondly, the mixtures of epoxy resin and Al_2O_3 powder were dispersed for 3 h in an ultrasonic bath. Thirdly, hardener was added into the mixture and mixed in ultrasonic bath for 5 min. Fourthly, aramid fiber was laid down and the mixture of epoxy resin and hardener filled with Al_2O_3 powder was then poured into the plastic mould with thickness of 1 mm. Thereafter, the other aramid fiber was laid down upon the 1st layer. In this step, the mylar films were put at the upper and the lower of the samples in order to obtain smooth surface. Finally, the composite samples were left to settle for 1 day at the room temperature and then removed from the mould. Density of the samples was measured by Archimedes method. To investigate the mechanical properties, hardness by Vicker's hardness testing machine (HV30, Brook Inspection Equipment Ltd., England). The impact was tested using the impact testing machine (6705CE, Dension Mayes Group, DMG). The wear resistance was measured by the Pin-on-Disk Tribometer with the ball radius of 4 mm. The tensile strength was examined by using the universal testing machine with a cross-head rate at 50 mm/min, the composites samples were cut into the dog-bone shapes. Moreover, the microstructure of the samples, which cut by ceramic cutting tool, was also determined by scanning electron microscopy (SEM).

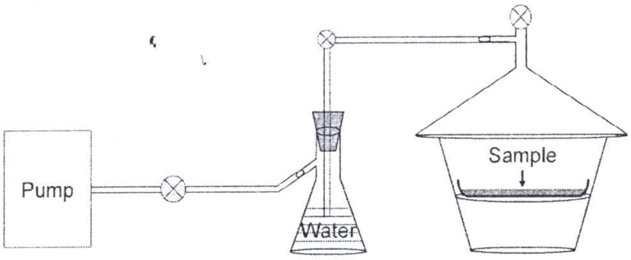


Fig. 1. The schematic diagram of low - pressure technique.

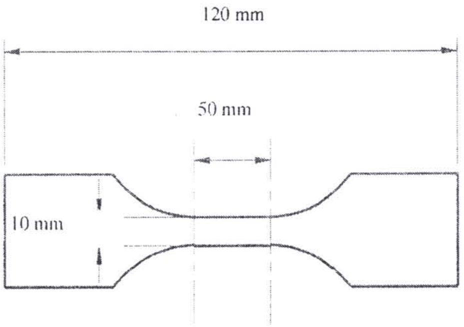


Fig. 2. The tensile test of composites samples.

Results and Discussion

Table 1 shows density, hardness, impact, wear rate and tensile strength values of aramid/Al_2O_3/epoxy resin laminated composite samples. The results showed that the density of composite samples which is in the range of 1.265-1.476 g/cm^3 was increased with increasing Al_2O_3 powder content. However, it is noted that the density of the composites samples is less than the density of Al_2O_3 powders (3.61 g/cm^3) and slightly higher than the pure epoxy resin (1.176 g/cm^3). The hardness and tensile strength of composites sample are in the range of 84.7 - 102.3 HV and 129.8 - 290.3 MPa, respectively. Furthermore, it is observed that the impact and wear rate values are in the range of 3.7 - 4.3 J. and 0.2074 - 0.3924 μm^2, respectively. It is observed that at 10 vol% Al_2O_3 powder content is minimum hardness (85.0 HV) whereas tensile strength is maximum and much higher than the pure resin (887% increased). Besides, at 20 vol% Al_2O_3 powder content are highest hardness, which increased to 174% and lowest tensile strength (102.3 HV and 129.8 MPa, respectively). Fig. 3 shows the photographs of the width of wear track of composites samples. It exhibits that the wear rate are dramatically decreased with increasing of Al_2O_3 powder content when compared to the pure epoxy resin (from 2.9100 to 0.2074 μm^2). It might be said that the wear resistance of the composites samples could be improved by using the Al_2O_3 powder filler. The SEM micrographs of composites samples with the different Al_2O_3 powder contents are shown in Fig. 4. It was found that the small amount of porous appears at the contact area. It may assume that composites show good adhesion between reinforced and epoxy resin.

Table 1. The properties of reinforced composites.

Vol% of Al_2O_3	Density (g/cm^3)	Hardness (HV30)	Impact (J)	Wear rate (μm^2)	Tensile strength (MPa)
0	1.176	37.3	3.2	2.9100	29.4
2	1.265	84.7	3.7	0.3924	255.8
5	1.312	87.7	3.9	0.2970	282.4
0	1.339	85.0	4.1	0.2573	290.3
12	1.363	90.6	4.0	0.2271	282.0
15	1.436	92.6	4.1	0.2074	212.1
20	1.476	102.3	4.3	0.1575	129.8

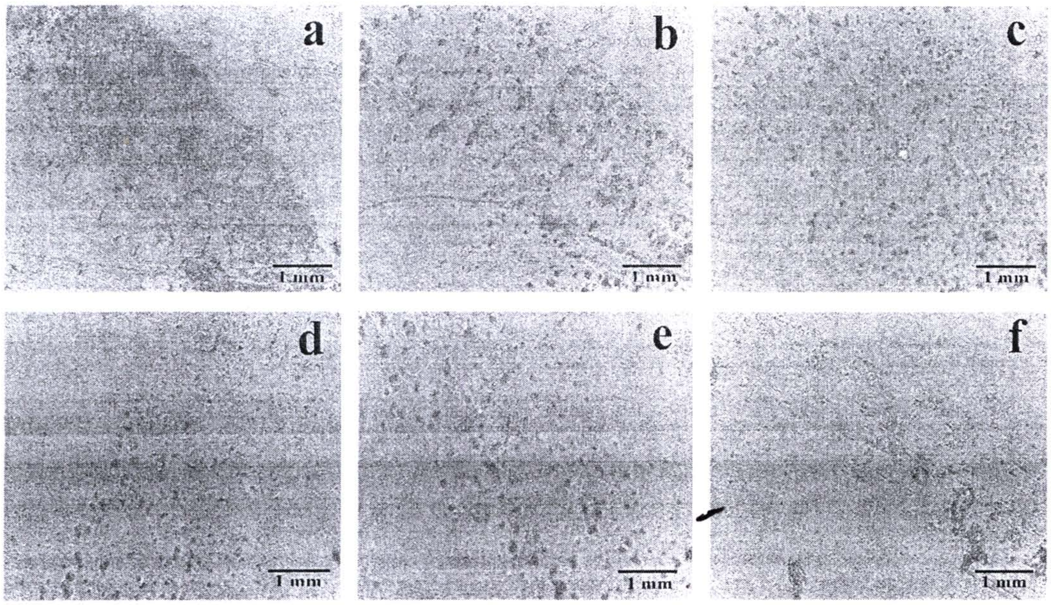


Fig. 3. Wear track of composites samples (a) 2 vol%, (b) 5 vol%, (c) 10 vol%, (d) 12 vol%, (e) 15 vol%, (f) 20 vol%

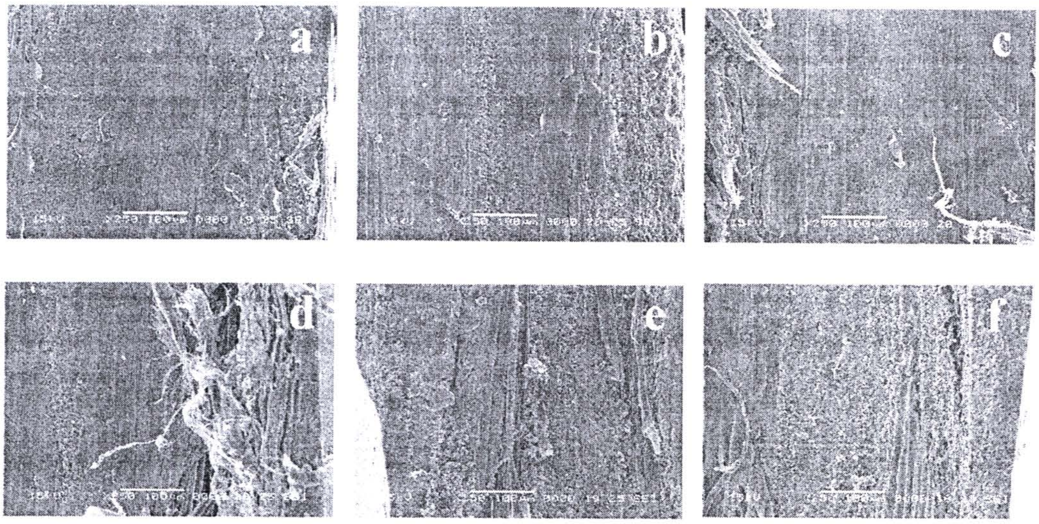


Fig. 4. SEM micrographs of aramid/Al_2O_3/epoxy resin composites (a) 2 vol%, (b) 5 vol%, (c) 10 vol%, (d) 12 vol%, (e) 15 vol%, (f) 20 vol%.

Summary

Aramid/Al_2O_3/epoxy resin composites were fabricated by using the ultrasonic mixing and casting technique. The plain weave aramid fiber and Al_2O_3 powder were used as the reinforcement to promote the mechanical properties of the composites. The results revealed that the aramid and Al_2O_3 powder were well-dispersed within the epoxy resin matrix. The tensile strength of the 10 vol% Al_2O_3/epoxy resin composites compared with the pure epoxy resin increased 887%. The hardness and impact were maximum at 20 vol% Al_2O_3. The composites samples with 20 vol% of Al_2O_3 show the best wear resistance with the wear rate 0.1575 μm^2, much less than the pure epoxy resin (2.9100 μm^2).

Acknowledgement

The authors would like to sincere thanks the Thailand Research Fund and The Graduate School, Chiang Mai University for their financial supporting. And the authors also would like to express their sincere thanks to the Department of Physics, Faculty of Science, Chiang Mai University for all the facilities.

References

[1] B. Wetzel , P. Rosso, F. Haupert and K. Friedrich: Eng. Fract. Mech. Vol. 73 (2006), p. 2375.
[2] M. Alagar, A.A. Kumar, K.P.O. Mahesh and K. Dinakaran: Eur. Polym. J. Vol. 36 (2000), p. 2449.
[3] P.J. De Lange, E. Mäder, K. Mai, R.J. Young and I. Ahmad: Composites Part A. Vol. 32 (2001), p. 331.
[4] U.S. Tewari, A.P. Harsha, A.M. Häger and K. Friedrich: Compos Sci. and Technol. Vol. 63 (2003), p. 549.
[5] M. Hussian, A. Nakahira and K. Niihara: Mater. Lett. Vol. 26 (1996), p. 185.
[6] Z. Su, Z. Jizhong, Z. Shiqi, L. Wenzhi and L. Hengde: Compos. Sci. and Technol. Vol. 63 (2003), p. 1009.
[7] C. Nathaniel, M. Hassan, K.R. Vijaya, A. Adnan and J. Shaik: Compos. Struct. Vol. 67 (2005), p. 115.

Advanced Materials Research Vols. 55-57 (2008) pp 213-216
online at http://www.scientific.net

Fabrication of Barium Zirconium Titanate Ceramics Using Ultrasonic Ball Milling Technique

W. Thamjaree, W. Nhuapeng* and T. Tunkasiri

Department of Physics, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand

*nhuapeng@yahoo.com

Keywords: BZT, milling time, ultrasonic ball milling

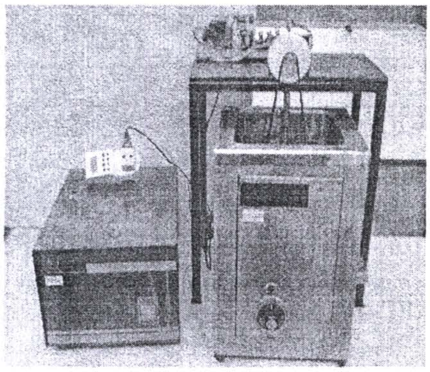
Abstract. In this work, barium zirconium titanate (BZT) ceramics were fabricated by using a ultrasonic ball milling technique. BZT with the ratio of $BaCO_3$:ZrO_2:TiO_2 was 1:0.05:0.95 was mixed in ultrasonic ball milling for 1, 3 and 4.5 hr and calcined at temperature of 1000 °C. Phase formation of BZT powder was investigated using XRD technique. Moreover, physical and electrical properties were examined. It was found that the tetragonal structure of BZT can be observed. Particle size of sample powder obtained from new milling technique is smaller than that of powder obtained from conventional mixed oxide method. Moreover, this new technique also requires the less time fabrication. Furthermore, the dielectric properties are increased with milling time and sintering temperature.

Introduction

In the past, innovations in many applications for example, actuators, sensors, hydrophones and ultrasonic transducers have been the driving force for new developments in piezoelectric ceramics [1]. Lead–containing materials including mainly lead zirconate titanate (PZT) and lead-based perovskite compositions have been the mainstay for high performance applications due to their excellent dielectric, piezoelectric and electromechanical coupling coefficients [1,2]. However, from the sustainable development point of view, the toxicity of lead oxide and its high vapor pressure during materials processing may bring serious problems and the growing of concerning on health and safety worldwide have drawn the attention owing to the need for environmental protection [2,3]. In recent years, lead-free ferroelectric and piezoelectric materials have received much attention to produce the electronic products to protect the environment and human health. Therefore, it is necessary and urgent to develop lead-free materials with excellent properties for replacing the lead-based materials. One of the most important and promising lead-free materials is barium titanate (BT) and its solid solutions for example, barium zirconium titanate (BZT) [4]. Normally, the fine, high purity and uniform BT and its solid solution powder which is advantages in many engineering applications can be synthesized by such chemical methods for example, sol-gel, co-precipitation, hydrothermal, the poly vinyl alcohol evaporation route, spray dry technique and spray pyrolysis technique,..etc [5]. However, the chemical synthesis is not suitable for mass production due to low yield and high cost of powder. Therefore, the solid-state reaction process using oxides as the starting materials are more favorite to prepare BT and its solid solution powder because this technique is very simple, low cost and can be prepared for mass product [6]. Unfortunately, the conventional method requires a high calcinations temperature to synthesize the designed BT compound, making the fabrication of BT ceramics very complicated and time-consuming [7]. Therefore, in this work the authors attempt to prepare the BZT powder by using ultra-ball milling technique. The effect of time on formation and morphology of powder was studied. Furthermore, the physical and electrical properties of samples were also examined.

Experimental

BZT with the mole ratio of $BaCO_3$:ZrO_2:TiO_2 was 1:0.05:0.95 was prepared via the ultrasonic ball-milling technique (Figure 1). $BaCO_3$ (99%, Fluka), ZrO_2 (99%, Fluka) and TiO_2, (99%, Reidel) were employed as precursors. The starting powders were weighed according to appropriate molar fractions, mixed and wet milled (in ethanol) for different time (1, 3 and 4.5 hr) in the zirconia grinding media. The mixtures were dried, calcined in closed alumina crucible at temperature of 1000 -1250 °C for 2 hr with heating and cooling rate of 3 °C/min. Moreover, the calcined powder was sieved and sintered in closed alumina crucible at temperature of 1300 °C and 1350 °C for 2 hr with heating and cooling rate of 3 °C/min., respectively. An X-ray diffractometer (XRD, Philips) and a scanning electron microscope (SEM, Philips XL 30 ESEM) were employed to study phase evolution and microstructure of the samples, respectively.



Figture 1. Fabrication of BZT powder using the ultrasonic ball-milling technique.

Results and Discussion

The phase identification of calcined and sintered BZT ceramics were conducted by X-ray diffraction (XRD) technique. Figure 2 show the XRD pattern of BZT powder which milled for 4.5 hr and calcined as different temperature. From Fig. 2, the series of peak can be matched with the JCPDS file no. 83-1880. It can be seen that the uncompleted perovskite-like BZT phase was found in powder with a milling time of 1 hr and the single BZT phase was obtained from a milling time of 3 hr and 4.5 hr (Figure 3). It can be seen that the ultrasonic ball-milling technique take the less time consuming when compare with the conventional method which normally take 24 hr for milling time. This may be due to that the ultrasonic processing may promote the ability of grinding, breaking down the agglomeration of powder. Next, effect of milling on particle size of BZT powder was examined by particle size analyzer (Mastersizer S ver 2.19, Malven Instrument, Ltd.) as show in Table 1. It can be seen that average particle size of uncalcine powder and calcined powder are in the range of 0.83-0.16 μm and 0.72-0.22 μm, respectively. Besides, it is also found that there is a significantly change in size of particle with the further milling time of ultrasonic ball mill. Table 2 exhibits the density, dielectric properties and Curie temperature of BZT ceramics. It can be found that density and dielectric constant of BZT ceramics is increasing with the milling time which effect from the decreasing of particle size when the milling time is increased. The optimum values of density and dielectric constant (77.67% and 1555) obtain from ceramics sintered at 1350 °C with the milling time of 4.5 hr. It has been observed that density of samples is not extremely high which may be due to its sintering behavior of the oxide mixture which shows the volumetric expansion [7]. Moreover, there is a significantly change in Curie temperature of BZT ceramics sintered at 1350 °C which may be due to the density of sample and the Zr concentration from the media. The relationship between dielectric constant of BZT ceramics and temperature is shown in Figure 4.

The normal curve of dielectric shows that higher dielectric constant values obtain from ceramics which sintered higher temperature.

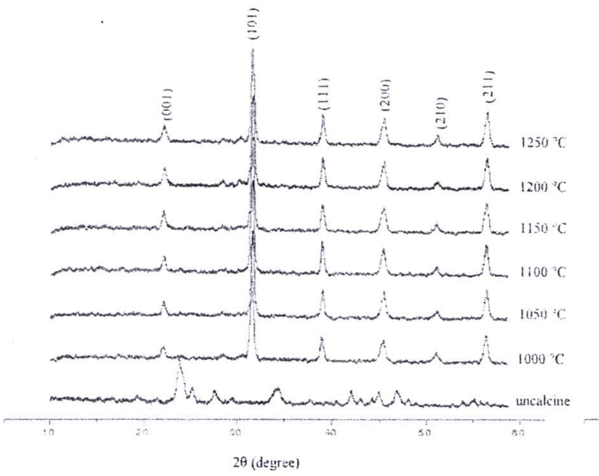


Figure 2. XRD pattern of BZT powder Milled for of 4.5 hr and calcined different temperatures.

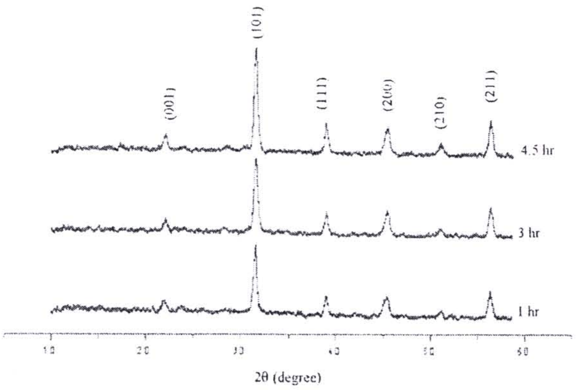


Figure 3. XRD pattern of BZT powder calcined at 1000 °C which mixed for different of milling times.

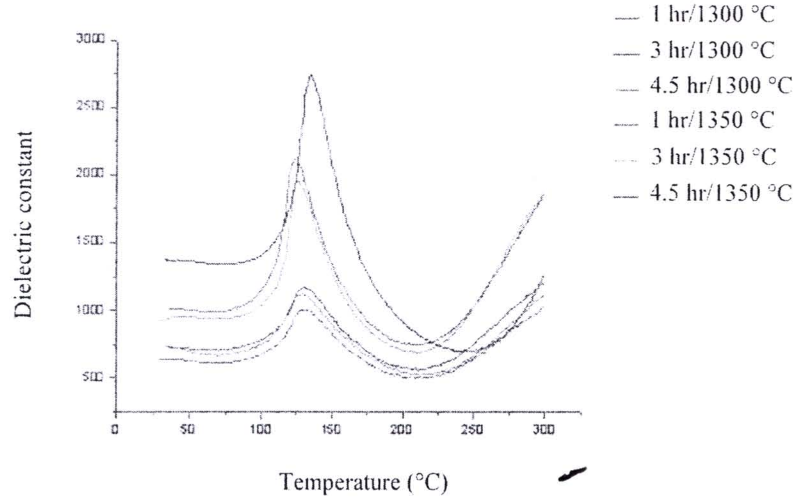
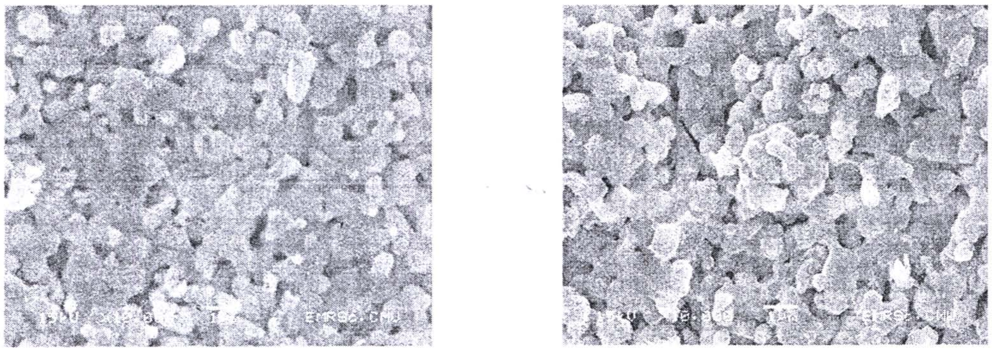


Figure 4. Relationship between dielectric constant of BZT ceramics and temperature.



(a) (b)

Figure 5. SEM micrographs of BZT samples sintered at 1350 °C (a) as received surface and (b) fracture surface.

Table 1. Average particle size of uncalcine and calcined BZT powders

Milling Time (hr)	Mean diameter (μm)	
	Uncalcine powder	Calcined at 1000 °C
1	0.83	0.72
3	0.18	0.23
4.5	0.16	0.22

Table 2. Density, dielectric properties and Curie temperature of BZT ceramics

Sintering Temperature (°C)	Milling Time (hr)	Density (%)	Frequency (1 kHz)		Curie Temperature (°C)
			ε_r	tanδ	
1300	1	67.62	866	0.38	130
	3	71.69	970	0.35	130
	4.5	73.08	1308	0.60	129
1350	1	74.72	1128	0.19	123
	3	75.16	1193	0.13	126
	4.5	77.67	1555	0.25	134

The SEM micrographs of as receive surface and fracture surface of BZT ceramics sintered at 1350 °C are shown in Figure 5. There is many pores appear in the bulk which may be due to the effect of low density of the samples. However, this problem can be improved by using higher sintering temperature. The time employed in the ball milled method has the effect on the porosity in the bulk (not show here). The grains agglomerate in irregular shape. Furthermore, the average grain size of sintered ceramics is about 1.2 μm. Therefore, it could be concluded that the ultrasonic ball milling technique could produce fine powder but consuming less milling time when compared to the conventional technique.

Summary

It can be concluded that homogeneous and fine powder can be obtained by using the ultrasonic ball-milling technique which could be fabricated in mass product and low cost. The most significant change occurs in the size of the particles. Moreover, the ultrasonic ball-milling technique results in smaller particle size and less milling time than the powder processed via the conventional ball milling method.

Acknowledgement

The authors would like to express their sincere thanks to the Thailand Research Fund (TRF) and Department of Physics, Faculty of Science, Chiang Mai University for supporting this research.

References

[1] T.R. Shrout and S.J. Zhang: J. Electroceram Vol. 19 (2007), p. 111.
[2] J. Wu, D. Xiao, Y. Wang, J. Zu and P. Yu: J. Appl. Phys. Vol. 103 (2008), p. 1.
[3] H. Yan, D. Xiao, P. Yu, J. Zhu, D. Lin and G. Li: Mats Design Vol. 26 (2005), p. 474.
[4] Z. Yu, R. Guo and A.S. Bhalla: Mats. Lett. Vol. 57 (2002), p. 349.
[5] J. Lappalainen and V.Lantto: App. Surf. Sci. Vol. 154-155 (2000), p. 118.
[6] Shirane, K. Suziki and A. Takeda: J. Phys. Soc. Jpn. Vol. 7 (1952), p. 12.
[7] L.B. Kong, J. Ma, H.T. Huang, W. Zhu and O.K. Tan: Mater. Lett. Vol. 50 (2001), p. 129.

Advanced Materials Research Vols. 55-57 (2008) pp 553-555
online at http://www.scientific.net
© (2008) Trans Tech Publications, Switzerland

Mechanical Properties of Hemp Fiber Composites with Carbon Nanotubes Reinforcement

H. Longkullabutra, W. Nhuapeng[a], W. Thamjaree and T. Tunkasiri
Department of Physics, Faculty of Science, Chiang Mai University, Chiang Mai 50200, Thailand
[a]nhuapeng@yahoo.com

Keywords: composites, carbon nanotubes, reinforcement

Abstract. An experimental was investigated the condition of reinforcement of hemp fiber and hemp fiber/epoxy resin composites with carbon nanotubes (CNTs).The CNTs were mixed with several liquid such as ethanol, latex, water glue and epoxy resin. These mixtures were added to hemp fiber and prepared the hemp fiber/epoxy resin composites. The mechanical properties of both fiber and composite samples were measured. It was found that the mixture of epoxy resin and CNTs with CNTs ratio of 20 vol% in hemp fiber showed the highest tensile strength of 25.43 N. and the same mixture in hemp fiber/epoxy resin composites showed the highest tensile strength of 31.82 MPa and elongation of 7.40 %.

Introduction

Bio-composites are the smart materials of the future. It is only through these materials that the balance of ecology and economy can be maintained. In the past decade the interest and research in this area has grown exponentially. Natural/bio-fibers offer many advantages such as energy efficiency, low cost, low density, high toughness, acceptable specific strength, and bio-degradability [1]. CNTs have a number of properties (mechanical and physical) that make them attractive for use in a broad spectrum of applications, especially as reinforcement in nanocomposites [2-6]. There are two kinds of carbon nanotubes: single-walled carbon nanotubes (SWNTs) (one graphene layer) and multi-walled carbon nanotubes (MWNTs) (many graphene layers) wrapped onto themselves.They can be produced in various ways such as arc discharge, laser ablation, solar energy, molten salt electrolysis and chemical vapour deposition (CVD). The nanotubes are often with more or less impurity (metal catalytic particles, amorphous carbon, etc.) depending on the synthesis method used [7].

There have been many attempts to incorporate nanoparticles, such as metal–oxide nanoparticles, nanoclays, carbon nanofibers, graphite nanoplatelets and carbon nanotubes into the polymeric matrix of conventional fiber-reinforced composites by impregnating dry fiber preforms with the modified nanocomposite matrix or introducing nanoparticle/epoxy films between composite prepreg plies [8]. The aim of the experimental was investigated the condition of reinforcement of hemp fiber and hemp fiber/epoxy resin composites with CNTs and mechanical properties of them were measured.

Experimental

The CNTs were produced by CVD and burned at 430 °C for 4 h for treatment. After that CNTs were mixed with several liquid such as ethanol, latex, water glue and epoxy resin by CNTs ratio of 20 vol %. Hemp fibers were impregnated with these mixtures and tensile strength was measured with Hounsfield H10KS mechanical testing machine in order to choose the best of liquid for prepared the hemp fiber/epoxy resin composites. Then, the epoxy was chosen for prepared hemp fiber/epoxy resin composites by two conditions, untreated and treated CNTs. The CNTs were dispersed in the epoxy by ultrasonication before being mixed with the hardener by ratio 10:2.7 by volume, impregnating the hemp fabric performs with the nanocomposite solution and the matrix (epoxy resin) was mixed to hemp fabric and CNTs by hand lay-up technique, the flat sheet samples were produced.

All the sheets were allowed to cure at room temperature for 6 days. The dog – bone shape composite samples for mechanical testing had the gauge length of 30 mm, width 14 mm and thickness about 1 mm. The tensile tests were conducted on a Hounsfield mechanical testing machine using a crosshead speed of 2.5 mm/min and a 250 mm extensometer.

Results and Discussion

The Table 1 shows tensile properties of the hemp fibers, hemp fibers with several liquid were un-reinforced/reinforced CNTs.

Table 1. The tensile properties of hemp fibers

Reinforcing hemp fiber	Force (N)	Elongation (%)
Hemp	15.01	6.67
Hemp + ethanol + CNTs	17.47	7.07
Hemp + ethanol	17.16	7.77
Hemp + latex +CNTs	21.56	14.40
Hemp + latex	25.57	14.08
Hemp + water glue + CNTs	23.61	12.37
Hemp + water glue	24.67	15.93
Hemp + resin + CNTs	25.43	10.80
Hemp + resin	26.53	14.03

It was found that they are slightly different because of the strength of hemp fiber have more influence than CNTs. However, they have better strength than hemp fiber, hemp fiber with ethanol + CNTs and hemp fiber with ethanol. It is possible that the reinforcement was effect from several liquid and CNTs.

The resin liquid was chosen for hemp fiber/epoxy resin composites because of its high strength

Table 2. The mechanical properties of hemp fiber, CNTs untreated/treated reinforced hemp fiber/epoxy resin composites

Reinforcing hemp fiber/epoxy resin composites	Strength (MPa)	Modulus (GPa)	Strain to break (%)
Hemp	25.34	0.18	13.74
Hemp + resin + CNTs (untreated)	23.58	0.18	18.61
Hemp + resin + CNTs (treated)	28.63	0.20	21.29

The average values of the strength, the Young's modulus, and the fracture strain of these composites were shown in Table 2. These results show that the addition of the CNTs (treated) increased the strength and the modulus of the composites. The tensile strength of CNTs treated/untreated reinforced hemp fiber/epoxy resin composites are values of 28.63 and 23.58 MPa respectively. The decrement of the tensile strength of the composite may be due to the impurity (metal catalytic particles, amorphous carbon, etc.) of CNTs (untreated) that disperses into the matrix [7]. The CNTs were burned to improve the purity of nanofiber.

Scanning electron microscope (SEM) images were obtained to study the dispersion and mechanical role of CNTs in the epoxy matrix of the composites. There were some zones have very high local CNTs concentrations, as shown in Fig.1. The distribution of CNTs on CNTs (treated) reinforced hemp fiber/epoxy resin composite was more homogeneous than CNTs (untreated) reinforced hemp fiber/epoxy resin composite. It is possible that the particles of impurity of CNTs (untreated) disperse into composite instead of CNTs. Another parameter is the CNTs added into resin with CNTs ratio of 20 vol% in hemp fiber was very viscous and the homogenization process was difficult.

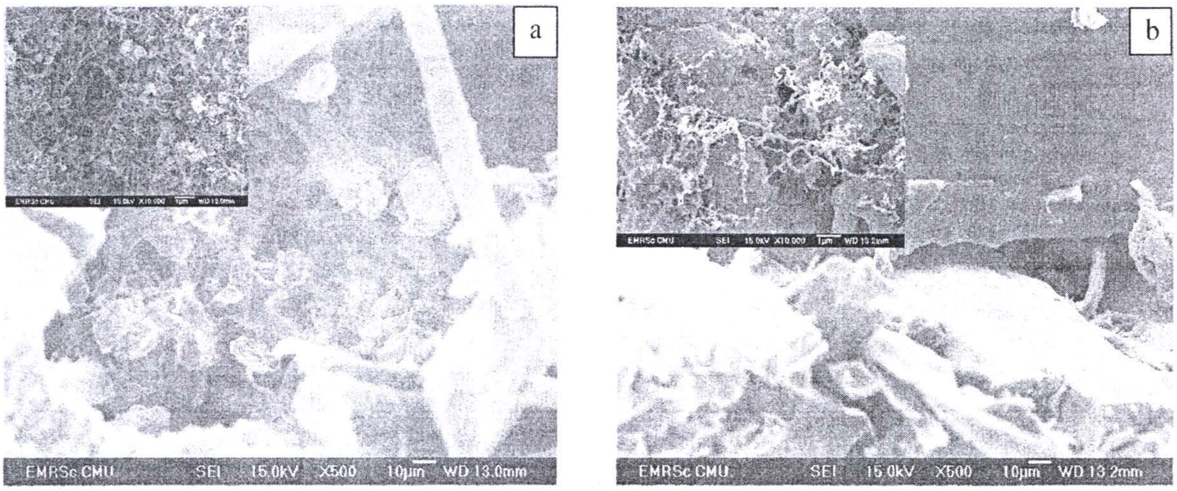


Fig. 1 SEM images of the fracture surface of (a) CNTs (untreated) reinforced hemp fiber/resin epoxy composite (b) CNTs (treated) reinforced hemp fiber/resin composite.

Summary

CNTs were dispersed into epoxy resin better than another liquid. The mechanical properties of hemp fiber composites with CNTs reinforcement made by hand lay-up technique and CNTs (treated) reinforced hemp fiber/resin epoxy composite was higher tensile strength than CNTs (untreated) reinforced hemp fiber/resin epoxy composite. In order to make significant improvements to the mechanical properties of the composites it was found necessary to CNTs treatment.

Acknowledgments

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

References

[1] M G. Mehta, A. K. Mohant, M. Misra, L. T. Drzal: J. Mater. Sci. Vol. 39 (2004), p. 2961.
[2] D.H. Robertson, D.W. Brenner, J.W. Mintmire: Phys Rev B Vol. 45 (1992), p. 2592.
[3] B.I. Yakobson, C.J. Brabec, J. Bernholc: Phys Rev Lett Vol. 76 (1996), p. 2511.
[4] J.P. Lu: Phys Rev Lett Vol. 79 (1997) p. 1297.
[5] E.W. Wong, P.E. Sheehan, C.M. Lieber: Science Vol. 277 (1997), p. 1971.
[6] C.F. Cornwell, L.T. Wille: Solid State Commun Vol.101 (1997), p.555.
[7] J.B. Bai, A. Allaoui : Composites: Part A Vol. 34 (2003), p. 689.
[8] J. Cho, I.M. Daniel: Scripta Mater. (2007), in press.