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OUTPUT

1. W. Thamjaree, S. Kumfu, W. Nhuapeng* and T. Tunkasiri, Effect of Nd_2O_3 adding on electrical properties of bismuth sodium titanate ceramic, NU Science Journal 2009;6(S1): 23-27.
2. Wim Nhuapeng1, Supreya Kumfu2, Wandee Thamjaree1*, Haruthai Longkullabutra1 and Tawee Tunkasiri1Fabrication and Mechanical Properties of Carbon Nanotubes and Rubber Composites, NU Science Journal 2009;6(S1): 51-55..
3. Supreya Kumfu1*, Pisith Singjai2, Wandee Thamjaree2 Haruthai Longkullabutra2 and Wim Nhuapeng2 Mechanical Properties of Silicon Carbide Nanowires /Carbon Nanotubes / Rubber Composites, NU Science Journal 2009;6(S1): 80-85.
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Effect of Nd_2O_3 adding on electrical properties of bismuth sodium titanate ceramics

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ABSTRACT

This research, fabrication and effect of neodymium oxide (Nd_2O_3) adding on electrical properties of bismuth sodium titanate (BNT) ceramics were studied. The sample powder, BNT/x Nd_2O_3 (x = 4, 8 and 12 wt %) was prepared via mixed oxide method from BNT and Nd_2O_3 starting powders. The starting powder were mixed by using zirconia grinding media in ethanol for 24 h and dried by an oven. The mixture powder was calcined at temperature of 800 °C and was then pressed and sintered at the temperature of 1075 °C. Phase formation of samples was examined by XRD technique. Moreover, the physical and electrical properties and microstructure of samples were determined. From the results, it was found that electrical properties of BNT ceramics can be improved by adding Nd_2O_3. Furthermore, grain size of BNT ceramics are decreasing with increasing of Nd_2O_3 quantity.

Keywords: BNT, Nd_2O_3, Electrical Properties.

INTRODUCTION

In the present day, bismuth sodium titanate, $Bi_{0.5}Na_{0.5}TiO_3$ (BNT) is widely used in many applications. Since BNT ceramics are strongly ferroelectrics and exhibit the outstanding advantages in a free control atmosphere and produce no lead pollution (Lam *et al.*, 2005). Because health and environmental concerns with lead-containing materials, BNT is considered to be excellent candidate for use as lead-free piezoelectric ceramics to take place lead-containing piezoelectric ceramics (Lam *et al.*, 2005; Nakamura and Nomura, 1966; Takanaka, 2001). Their crystal structures are the perovskite type with rhombohedral symmetry at room temperature. They show a strong ferroelectric property with a relative high Curie temperature of 320 °C. However, the application of pure BNT ceramics are limited by some its shortcomimgs in electric properties, such as low relative dielectric permittivity (ε_r), narrow sintering temperature range and high conductivity at room temperature (Takanaka, 2001). Therefore, it is necessary to improve BNT ceramics on dielectric properties for applications. From the previous works found that the rare-earth additives have effect to the piezoelectric and dielectric properties of PZT ceramics (Garg and Argrawal, 2001; Shannigrahi *et al.*, 1999; Garg and Goel, 1999). Thereafter, it is very interesting to study the effect of adding Nd_2O_3 into non-lead based materials. In this work, the authors have studied the effects of rare earth additive (Nd_2O_3) on physical and electric properties of bismuth sodium titanate ceramics. Various weight percent of Nd_2O_3 were used for doping materials and adding to BNT ceramics *(like our previous work which adding Nd_2O_3 into PZT ceramics (Thamjaree, et al., 2007)*. The phase formation, physical properties, dielectric and piezoelectric properties were also studied. Moreover, the SEM micrograph was employ for microstructure determination.

METHODOLOGY

$(Bi_{0.5}Na_{0.5})TiO_3$ powder is prepared by a conventional one-step mixed oxide method. Commercially metal oxide and carbonate powder of high purity, for example Bi_2O_3 (98%), Na_2CO_3 (99.9%), TiO_2 (99%) and Nd_2O_3 (99%) were used as raw materials. The Nd_2O_3 was added to the mixture in various quantities (4, 8 and 12 percent by weight). Firstly, the precursors and dopants were weighed, mixed and milled by wet-milling in ethanol (Merck) using zirconia grinding media for 24 h. The dried powder was crushed and calcined in a closed alumina (Al_2O_3) crucible at temperatures of 800 °C for 2 h with a heating and cooling rate of 3 °C/min. The calcined powder was grinded in an agate mortar, pestle and sieved using a 100-mesh nylon sieve. The sieved powder was pressed to a cylindrical shape with diameter of 15 mm and thickness about 2 mm Polyvinyl alcohol (PVA, Fluka) 2 wt% was used as binder and added to the sieved powder. The green bodies were then sintered in a closed alumina crucible at temperature of 1075 °C for 2 h with heating/cooling rate of 5 °C/min. The sample was firstly heated at 5 °C /min to 500 °C for 1 h to burn out the binder. Then, the pellets were polished and electroded using silver paste (Acheson Electrodag 1415M) for dielectric and piezoelectric property measurements. Moreover, microstructures of the ceramic samples were examined by SEM technique.

RESULTS AND DISCUSSION

Figure 1 shows the typical of X-ray diffractogram of BNT powder which was calcined at temperature of 800 °C for 2 h. It can be seen that the perovskite-like BNT materials with orthorhombic structure were formed with the extra peak which mostly matched with neodymium oxide (Nd_2O_3). The series of peaks are matched with JCPDS no. 36-0340 (Powder Diffraction File, 2000). A weak reflection peak of unreact Nd_2O_3 phase increased with higher dopant quantities.

Table 1 shows physical and electrical properties of BNT samples. From the results, it could be found that the quantity of Nd_2O_3 have effects to physical and electrical properties of BNT ceramics. High density (93-97%) and shrinkage (~45-48%) of samples are increased with quantity of dopant. Furthermore, the particle and grain size of BNT powder and ceramics decrease with increasing of Nd_2O_3 quantity which is in the range of 0.28-0.24 μm and 1.25-0.43 μm, respectively. Morever, dielectric constant (670-830) and piezoelectric property (36-75 pC/N) of samples are improved by adding Nd_2O_3. It may be the effect of inhibiting grain growth of Nd_2O_3 on grain growth processing which effect to high density and high piezoelectric property. Besides, it can be observed that tendency of d_{33} value of 4 wt% and 8 wt% is not consistent with density. It may be due to the inhomogeneous mixing of Nd_2O_3 in BNT ceramics. However, adding Nd_2O_3 can promote the piezoelectric and dielectric properties of BNT ceramics which can develop to capacitor like PZT materials.

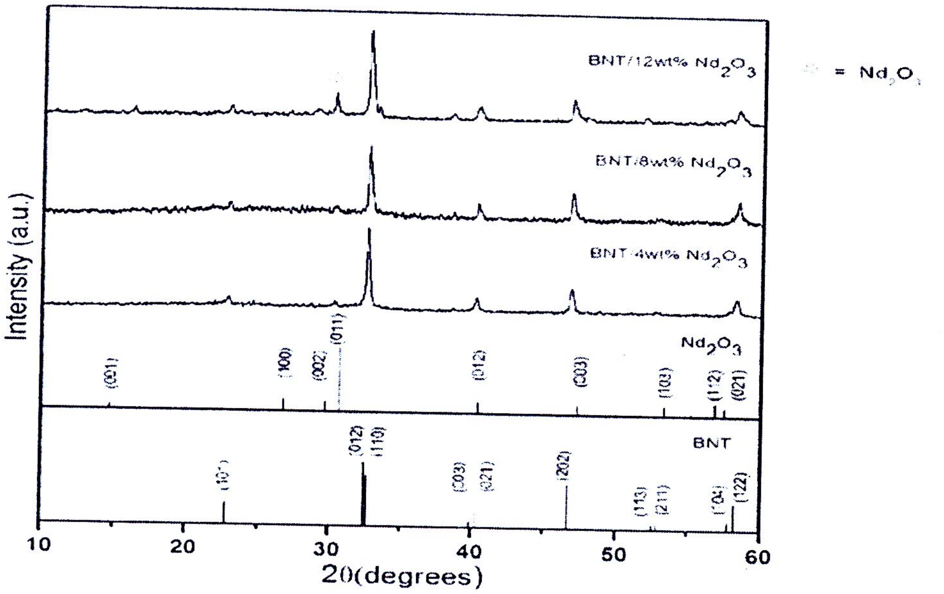

Figure 1 X-ray diffractogram of BNT powder doped with different quantities of Nd_2O_3 and calcined at temperature of 800 °C for 2 h.

Table 1 shows the physical and electrical properties of BNT samples.

Quantity of Nd_2O_3 (wt%)	Density (%)	Shrinkage (%)	Particle size (µm)	Grain size (µm)	ε_r	d_{33} (pC/N)
0 wt%	93.18	45.13	0.28	1.25	670	36
4 wt%	94.95	46.23	0.26	0.85	742	65
8 wt%	96.52	47.14	0.25	0.63	800	48
12 wt%	97.05	47.89	0.24	0.43	830	75

Figure 2 and Figure 3 show the SEM micrographs of BNT powder and ceramics, respectively. Figure 2 shows the morphology of BNT powder doped 4-12wt% Nd_2O_3. The agglomeration of irregular shape of BNT powder can be observed. Figure 3 shows SEM micrographs of BNT ceramics sintered at temperature of 1075 °C for 2 h. It can be seen that grain size of BNT ceramics decrease with increasing of Nd_2O_3 which corresponded with density of samples. The cube shape could be observed in all samples. The EDS was employed to determine the ratio of element in the sample which as shown in Figure 4. From the spectrum, it can be seen that all elements observed could be matched with the use starting powders.

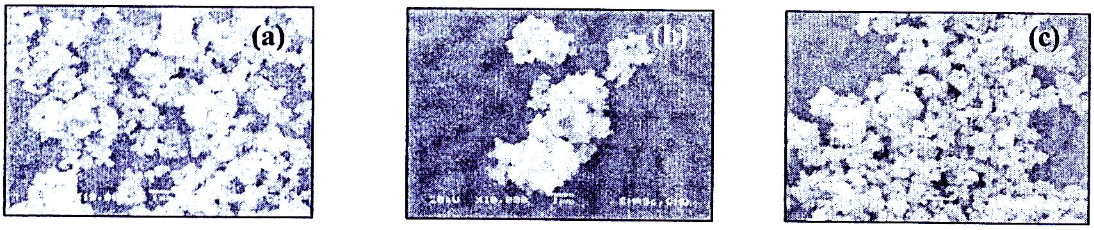

Figure 2 SEM micrographs of BNT powders (a) 4 wt% (b) 8 wt% and (c) 12 wt% of Nd_2O_3.

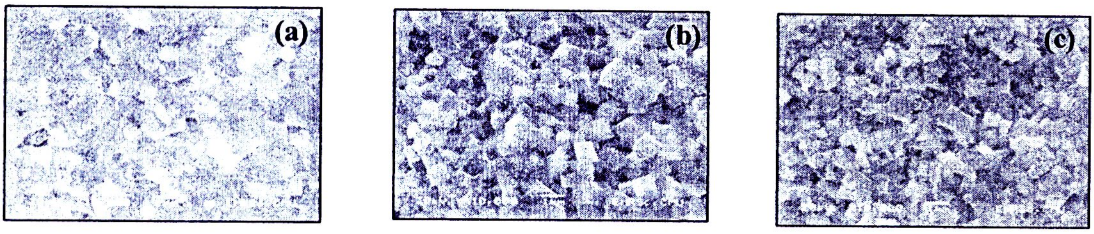

Figure 3 SEM micrographs of BNT ceramics (a) 4 wt% (b) 8 wt% and (c) 12 wt% of Nd_2O_3

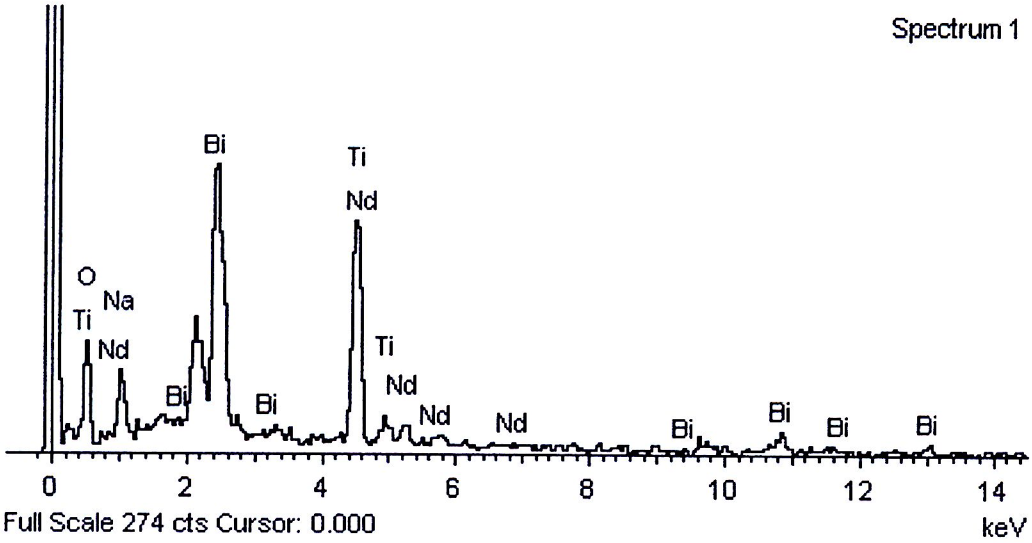

Figure 4 EDS spectrum of BNT ceramics.

CONCLUSION

The effect of adding various quantities of Nd_2O_3 on physical and electric properties of BNT ceramics were studied. From the result, it can be concluded that the density increased with the increasing of wt% of Nd_2O_3. The dielectric and piezoelectric properties of BNT ceramics are also improved with increasing of wt% of Nd_2O_3. The SEM micrographs show the grain growth with grain size in the range of 1.25-0.43 μm.

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Fabrication and Mechanical Properties of Carbon Nanotubes and Rubber Composites

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ABSTRACT

In this research, the composite materials between carbon nanotubes (CNTs) and natural rubber was studied. CNTs which used as dispersed phase were synthesized from NiO via chemical vapour deposition (CVD) method. The composites sample was fabricated by casting technique. CNTs was added into rubber for 0 – 1.0 % by volume. The physical and mechanical properties such as density, tensile strength, toughness, hardness and wear test of composite samples were examined, respectively. Furthermore, microstructures of samples were also investigated by scanning electron microscopy (SEM). From the results, it can be seen that the mechanical properties such as hardness of rubber was improved with the adding of CNTs.

Keywords: CNTs, Rubber, Composites

INTRODUCTION

In the present day, Polymer Matrix Composites (PMCs) with a small percentage of strong disperse are designed for many applications because of the significantly improve the mechanical, thermal and barrier properties of the pure polymer matrix (Chisholm *et al.*, 2005). With their excellent stiffness and weight characteristic, PMCs especially, fiber reinforced composites are widely used in many structural materials and industries which take place of metal, for example, aerospace, automobile, sporting goods, marine and other industries (Walker and Smith, 2002; Mel, 1994). There are many type of reinforcing fibers using in nowadays such as, glass fiber, carbon fiber, alumina fiber and aramid fiber (Kevlar), etc. which exhibits different properties. However, fiber with the extremely high mechanical properties which much more than that of metal is the challenging of technology improvement. Carbon nanotube (CNTs) is one of the novel material which can be the candidate of fillers in PMCs (Krumova *et al.*, 2001; Allaoui *et al.*, 2002; Kim *et al.*, 2006). Because of their unique properties, interesting mechanical (axial Young modulus 1-5 TPa) (Allaoui *et al.*, 2002), high flexibility, bending fully reversible up to a 110° critical angle for SWNT (Kim *et al.*, 2006). The CNTs-based composites, the new type of materials which can be one of the most promising applications have been intensively researched. In this work, the composites samples between multi-wall carbon nanotubes (MWNTs) and rubber were fabricated using ultrasonic mixing and casting techniques. The reinforced fiber, MWNTs were synthersized by a novel method, infusion chemical vapor deposition (Singjai *et al.*, 2007). Physical and mechanical properties such as hardness, tensile strength and density of composites samples were investigated. Besides, microstructure of samples was determined using SEM technique.

METHODOLOGY

The carbon nanotubes were synthesized from NiO via chemical vapour deposition (CVD) method (Singjai *et al.*, 2007). CNTs were milled for 3 hr to get rid of the agglomeration. To fabricate composites samples, firstly, the nanotubes were then weighed (vary from 0.1-1.0 vol%) and dispersed in water using ultrasonic technique for 2 hr. Secondly, the dispersed nanotubes were mixed with rubber in ultrasonic bath. Thirdly, the mixture was poured into plastic mould 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 on the both sides. Finally, the composite sample was left to settle at the room temperature for 2 days and then removed from the mould. Density of samples was measured by using Archimedes method. Composites samples were then cut into standard shape (Figure 1) to investigate mechanical properties. Tensile strength and maximum force of the samples were measured by the universal testing machine (Lloyd Instruments, LRX) with a cross-head rate at 50 mm/min. The hardness was tested by Durometer technique (Shore A). Furthermore, the microstructure of CNTs and CNTs composites was examined using SEM technique.

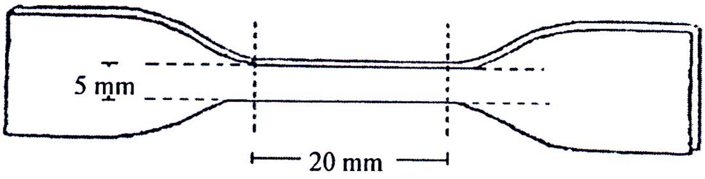


Figure 1 Schematic of standard sample for tensile testing.

RESULTS AND DISCUSSION

Figure 2 shows SEM micrograph of CNTs with the mean diameter of 68.21 nm. Figure 3 shows the photographs of casting rubber and casting CNTs/rubber composites. It can be seen that after mixing CNTs and rubber together, the color of rubber changed from grey to black which is the color of CNTs. Table 1 shows the physical and mechanical properties of CNTs/rubber composites. It is shown that quantity of CNTs is not significantly changed in density of composites (1.07-1.11

g/cm^3). It is may be due to adding quantity is too small. In the contrary, CNTs adding shown the significantly changed in mechanical properties, for example, tensile strength (19.34-10.90 MPa), toughness (135-85 N/cm^2) and hardness (54.1-70.0 type A). It can be noted that tensile strength and toughness of elastic rubber are decreased with increasing of CNTs. This is the effect of ceramics filler characteristics. However, the ceramics filler promote the hardness of rubber which is meaning the rubber is encourage its compressive property. Furthermore, SEM micrographs of the samples were shown in Figure 4. It has been seen that the dark phase and the bright are belong to rubber and CNTs phase, respectively. Besides, it is also found that rubber phase covered entirely of CNTs. It indicated that very good distribution and adhesion between CNTs reinforcement fiber and rubber matrix phase obtained from composites samples.

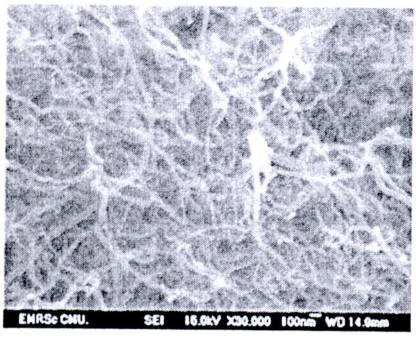
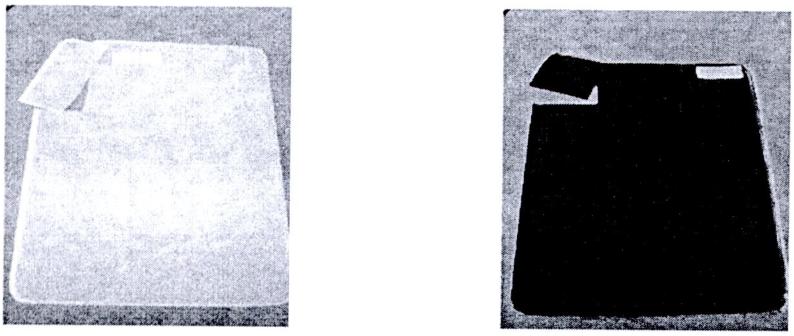


Figure 2 SEM micrograph of CNTs

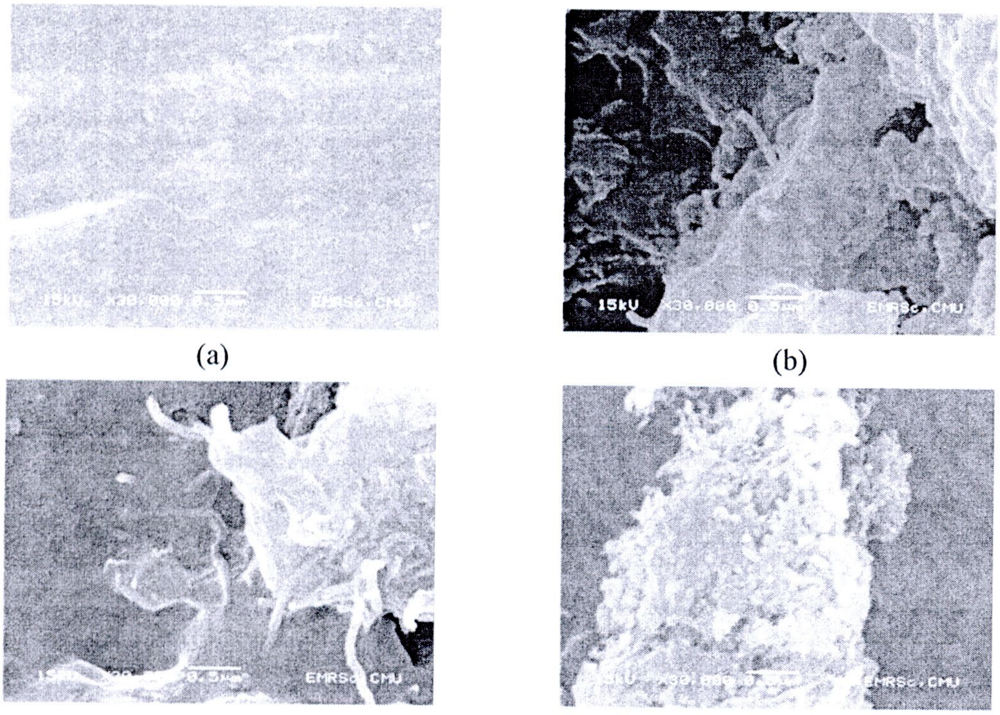


(a) (b)

Figure 3 Photographs of (a) rubber and (b) CNTs/rubber composites

Table 1 The physical and mechanical properties of CNTs/rubber composites

CNTs (vol%)	Density (g/cm^3)	Tensile strength (MPa)	Toughness (N/cm^2)	Hardness (Type A)
0.0	1.10	19.34	135.00	54.1
0.1	1.08	16.26	116.25	55.3
0.2	1.11	17.60	117.50	55.5
0.3	1.10	17.80	115.00	58.8
0.4	1.08	16.20	105.00	59.5
0.5	1.11	16.60	100.00	61.7
0.6	1.08	17.66	108.75	63.5
0.7	1.10	18.20	123.75	63.8
0.8	1.07	16.60	105.00	66.8
0.9	1.07	16.00	108.75	66.9
1.0	1.08	10.90	85.00	70.0



(a) (b)

(c) (d)

Figure 4 SEM micrographs of CNTs/rubber composites (a) 0 (b) 0.3 (c) 0.7 and (d) 1.0 vol%

CONCLUSION

CNTs/rubber composites were fabricated by using ultrasonic mixing and casting technique. The CNTs were used as reinforcement fiber to promote the mechanical property (hardness) of composites where as there is no significant change in weigh. The SEM results also revealed the well-dispersion of CNTs within rubber matrix.

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Mechanical Properties of Silicon Carbide Nanowires / Carbon Nanotubes / Rubber Composites

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ABSTRACT

In this work, 0-0-3 composites materials of silicon carbide nanowire (SiC NWs)/ carbon nanotube (CNTs)/ rubber were fabricated. The SiC NWs and CNTs reinforcement were prepared using the current heating technique (CHT) and infusion chemical vapor deposition (infusion CVD), respectively. The 0-10% by volume of CNTs + SiC NWs were mixed into rubber matrix phase to obtain 0-0-3 composites. Mechanical and physical properties of composite samples such as density, tensile strength and hardness were examined and compared with that of single phase of rubber. The results shown that tensile strength, hardness and density of composites were increased with adding reinforcement phase. Moreover, microstructure of sample was investigated by using scanning electron microscopy (SEM).

Keywords:silicon carbide nanowires (SiC NWs), carbon nanotubes (CNTs), rubber, composites

INTRODUCTION

In the recent years, polymer based composites reinforced with a small percentage of strong fillers can significantly improve the mechanical, thermal and barrier properties of the pure polymer matrix (Chisholm *et al.*, 2005). Therefore, fiber reinforced composites (FRCs) are increasingly used as structural materials, due primarily to their excellent stiffness and weight characteristics. Additionally, the effectiveness can be increased in many applications including packaging, coating, sport, electronics, aerospace industries, aircraft and military, automotive, and marine engineering (Chisholm *et al.*, 2005), (Walker and Smith, 2002), (Mel, 1994) and (Mallick, 1993).

Many types of fiber employed nowadays are glass-fiber, carbon-fiber and aramid-fiber or Kevlar. The traditional carbon fiber and glass fiber reinforced composites, both fiber and porous structures are at microscale and only a very small portion of the resin molecules are able to directly interact with reinforcement (Wang *et al.*, 2004). Therefore, these fibers are still limited in some applications, especially, the aircraft engine and aerospace industries which need high temperature materials, high mechanical properties (Luthra and Park, 1992). So that, 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 (Chand, 2000) and (Krumova *et al.*, 2001).

The new type filler phase, 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 (Fu *et al.*, 2006) and (Ying *et al.*, 2004). Besides, it has been suggested as good reinforcement materials and suitable to be used as the reinforcing material for composites due to their much higher strength over their bulk counterparts and strong interfacial bonding (Yang *et al.*, 2004).

Carbon nanotubes (CNTs), one graphene layer (SWNT) or many graphene layers wrapped onto themselves (MWNTs) are a novel crystalline carbon form which is another one of the novel materials which have been utilized as fillers in polymer matrix composites (Allaoui *et al.*, 2002) because of their unique structure and remarkable mechanical properties such as high elastic properties, large elastic strain and fracture strain sustaining capability, exceeding those of any previously existing reinforcement materials of composites (Xie *et al.*, 2005) and (Zou *et al.*, 2004). Moreover, the CNTs also shows great flexibility compared to conventional fiber. Due to their exceptional mechanical, physical, thermal, optical and electrical properties, CNTs are being dispersed in polymer using variety of approaches.

In this work, composites samples between SiC NWs, CNTs and rubber were fabricated using casting techniques. The reinforced fiber, SiC NWs and CNTs were synthesized by current heating technique (CHT) and infusion chemical vapor deposition (infusion CVD). Physical and mechanical properties such as hardness, tensile strength and density of the composites samples were investigated. Moreover, microstructure of samples was determined using SEM technique.

METHODOLOGY

The SiC NWs were synthesized via current heat technique (CHT) (Jintakosol and Singjai, 2007) and (Nhuapeng *et al.*, 2008). SiC NWs were taken out from surface were ground by using agate mortar for 10 min to get rid of hard agglomeration. The CNTs were synthesized by the infusion chemical vapor deposition (infusion CVD) (Singjai *et al.*, 2007). CNTs were milled for 3 h to get rid of the agglomeration. A matrix phase of the composites was natural rubber. The density of the matrix is 0.928 g/cm^3. The SiC NWs and CNTs were used as the reinforcement phase (Figure 1). The SiC NWs and CNTs were added into rubber with the different ratios varying from 1 to 10 percents by volume.

To fabricate the composites, firstly, the mixture of SiC NWs and CNTs was ground for 10 min. Second, the mixture of reinforcements was added into the water and mixed in ultrasonic bath for 5 min. Thereafter, mixture of rubber filled with SiC NWs and CNTs was then poured into the plastic mould with thickness of 1 mm. Finally, the composite samples were left to settle for 3 day at room temperature and then removed from the mould.

Density of the samples was measured by Archimedes method. The mechanical properties, tensile strength were investigated 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 (Figure 2). The hardness was examined by Shore durometer hardness testing technique (SHORE A). Furthermore, the microstructure of the samples was determined by scanning electron microscopy (SEM).

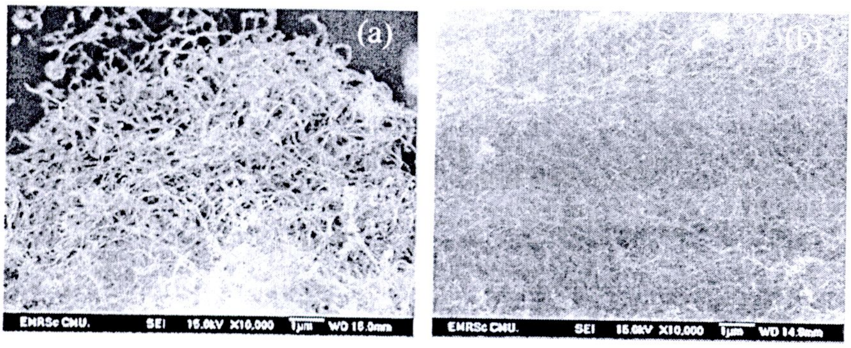

Figure 1 SEM micrographs of (a) SiC NWs and (b) CNTs

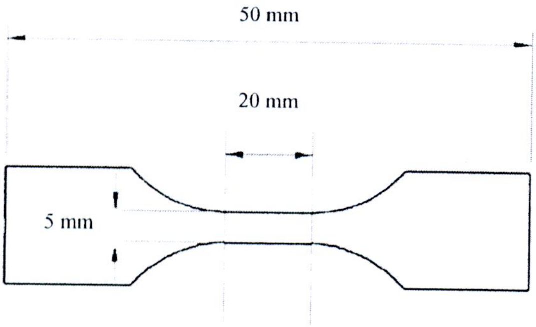

Figure 2 The standard sample for tensile testing.

RESULTS AND DISCUSSION

The results of the physical and mechanical properties testing of of SiC NWs/ CNTs/rubber composites sample were shown in Table 1. The density of composite samples is in the range of 0.938-1.006 g/cm^3. It was also found that the density of the composites is close to that of the rubber (0.928 g/cm^3). It may be due to adding small quantity.

The tensile strength and hardness of the composite samples are in the ranges of 6.31-17.63 MPa and 42.9-50.6 (type A), respectively. It can be noted that tensile strength of composite samples is decreased, where as the hardness of elastic rubber composite samples is increased with increasing the volume percentage of SiC NWs and CNTs. This result shows the effect of ceramics filler characteristics. However, the SiC NWs and CNTs filler can promote the hardness property of rubber composites.

Moreover, SEM micrographs of the samples are shown in Figure 3. It has been seen that the dark phase is rubber phase and the bright phase is belong to SiC NWs and CNTs phase. Besides, it is also interesting to note that SiC NWs and CNTs are entirely wrapped by rubber. This result indicated that very well distribution and adhesion between SiC NWs and CNTs reinforcement phase and rubber matrix phase obtained from composites samples.

Table 1 The physical and mechanical properties of SiC NWs/CNTs/rubber Composites

SiC NWs + CNTs (Vol%)	Density (g/cm^3)	Tensile strength (MPa)	Hardness (Type A)
0.0	0.928	13.32	42.9
1.0	0.956	11.88	44.8
2.0	0.938	16.36	46.8
3.0	0.972	17.46	47.7
4.0	0.954	17.63	47.8
5.0	0.962	16.13	48.2
6.0	0.988	11.99	49.1
7.0	0.995	11.88	49.4
8.0	0.987	13.38	49.5
9.0	0.986	14.48	49.7
10.0	1.006	6.31	50.6

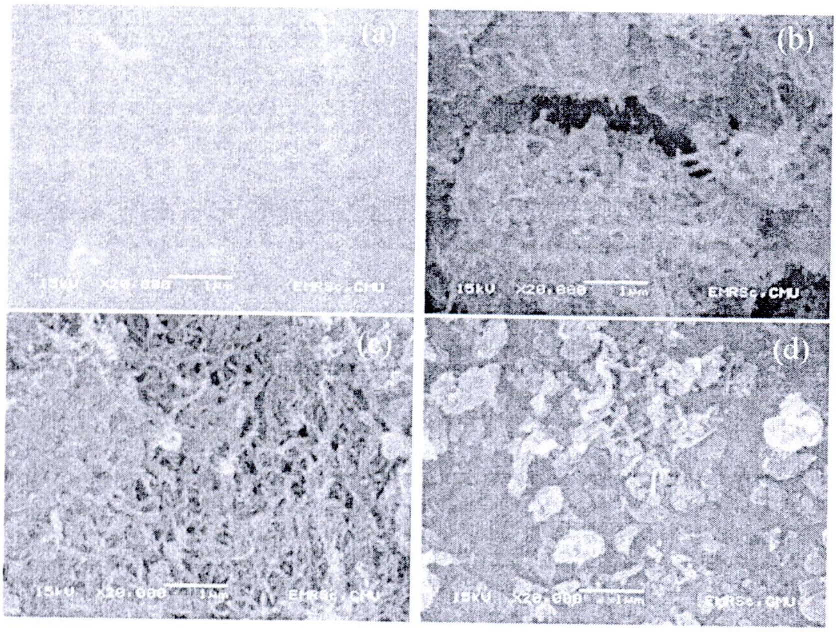

Figure 3 SEM micrographs of (a) 0.0, (b) 2.0, (c) 6.0 and (d) 10.0 Vol%

CONCLUSION

SiC NWs/CNTs/rubber composites were fabricated using the ultrasonic mixing and casting technique. The SiC NWs and CNTs were used as the reinforcement to promote the mechanical properties (hardness) of the composites. The results revealed that the well distribution and adhesion between SiC NWs and CNTs reinforcement phase and rubber matrix phase were obtained.

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Improvement in the Tensile Strength of Epoxy Resin and Hemp/Epoxy Resin Composites using Carbon Nanotubes

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eywords: CNTs, composite, epoxy resin

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

troduction

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

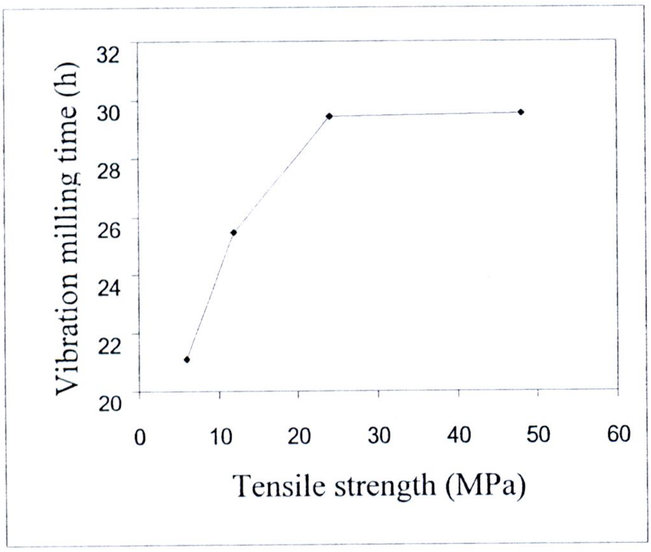

Fig. 1 shows the tensile strength of the samples, which vibrated CNTs for 6-48 h

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

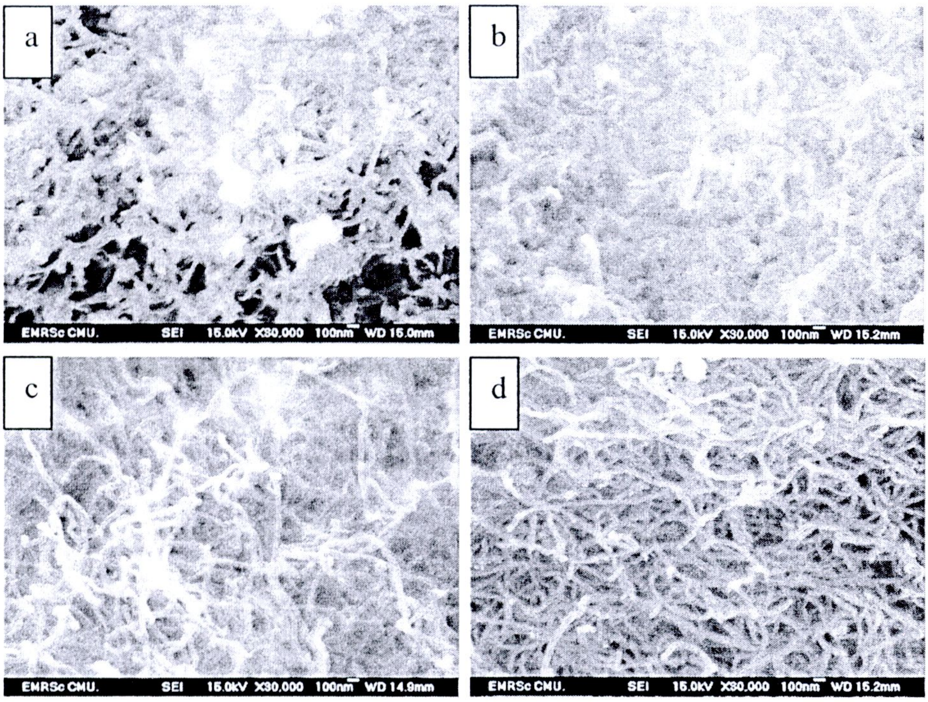

Fig. 2 SEM images of dispersion of vibrating milled CNTs: (a) 6 h, (b) 12 h, (c) 24 h, (d) 48 h

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

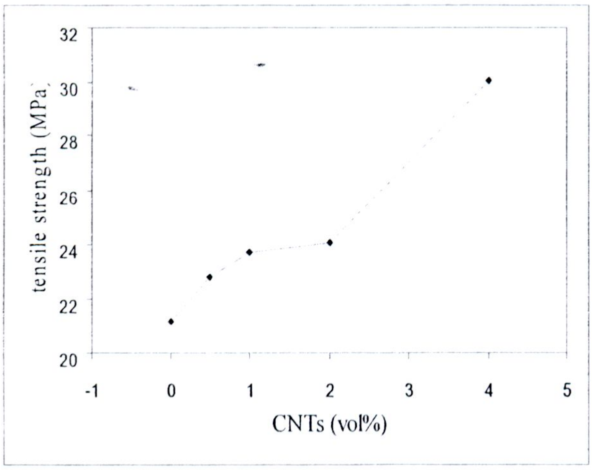

Fig. 3 Effect of quantity of CNTs fillers on the tensile strength of the hemp/epoxy resin composites

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. %.

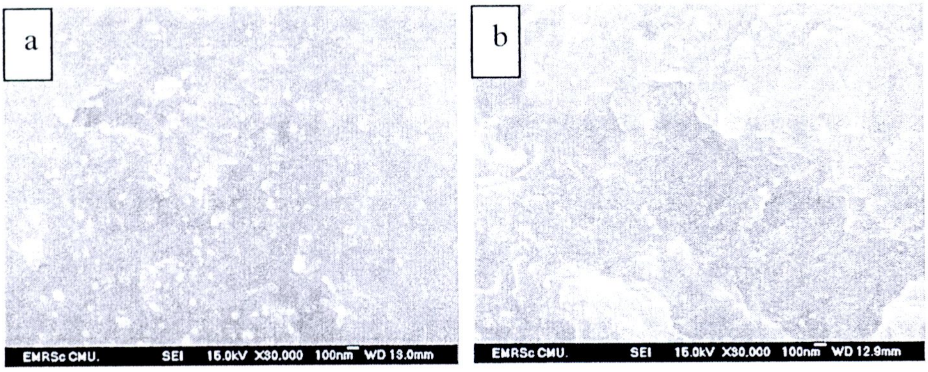

Fig. 4 SEM micrographs of CNTs dispersed in hemp/epoxy resin composites: (a) 2 vol. % (b) 4 vol. %

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

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EFFECT OF SiC NANOWIRES ADDITION ON MICROSTRUCTURE AND DIELECTRIC PROPERTIES OF BARIUM ZIRCONATE TITANATE CERAMICS

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***Keyword**: SiC NWs, nanocomposite ceramics*

Abstract

The perovskite ferroelectric BZT ceramics adding SiC NWs has successfully fabricated from BZT powder and SiC NWs by a simple solid-state mixed oxide and conventional sintering method. Phase transformation was showed the formation of only single phase perovskite structure in all cases. SEM showed that the SiC NWs randomly located in BZT matrix and only at the grain boundary, obviously indicated in 1.0 and 5.0 wt% SiC NWs. The densification was decreased with addition of SiC NWs has been observed. The dielectric properties was improved, the best result of dielectric properties have been obtain at 1.0 wt% SiC NWs and the dielectric values of all nanocomposites ceramics more than monolithic BZT ceramics.

INTRODUCTION

In the present, $BaTiO_3$ – based composition is one of the most popular and attractive materials. But, the monolithic $BaTiO_3$ ceramics have not enough properties and can be used for applications in narrow range. Therefore, BZT ($Ba(Zr_xTi_{1-x})O_3$) has been chosen for ceramic capacitors because this material has high dielectric constant and has potential for various device applications including multilayer ceramic capacitors (MLCs), sensor and actuator, etc. Almostly, the dielectric properties deteriorate with enhancement in mechanical properties. Thus, the enhancement of dielectric properties of BZT is very important for these applications. No previously report that investigated BZT/SiC NWs. In this present work, the small amount SiC NWs (0.1–5.0 wt%) into BZT ceramics which has fabricated by mixed oxide and conventional sintering method. We attempted to investigate the effect of SiC NWs on microstructure and dielectric properties of BZT ceramic.

EXPERIMENTAL

The BZT ($Ba(Zr_xTi_{1-x})O_3$) powder were prepared by ball milling method in alcohol and zirconia grinding media for 24 h. The starting raw materials including barium carbonate ($BaCO_3$, 99.9%), titanium dioxide (TiO_2, 99.9%) and zirconium oxide (ZrO_2, 99.5%) which the investigated system was $Ba(Zr_{0.05}Ti_{0.95})O_3$. The SiC NWs were synthesized by using a simple chemical vapor deposition. Monolithic BZT ceramics and BZT/SiC nanocomposite ceramics were fabricated by using a conventional ceramic method. The different amounts of SiC NWs (0.1–5.0 wt%) were dispersed by ultrasonic technique in ethanol for 20 min before mechanically vibro-mixing with the BZT powders and 1 wt% of PVA binder for 30 min . Slurry was dried at 120 °C and sieved into a fine powder. The mixed powders of each composition were pressed into disc-shape pellets with a diameter 15 mm, placed into an alumina crucible. The PVA binder was burn out at 500 °C for 1 h and green samples were sintered at 1450 °C for 2 h with slow heating/cooling rates of 3 °C/min, respectively. BZT ceramics were electrode by silver paste. Phase formation, physical and electrical properties were examined. Furthermore, microstructure of nanocomposites was investigated by SEM technique.

RESULT AND DISCUSSION

Phase characteristics of BZT/SiC NWs powder

XRD pattern of monolithic BZT and BZT/SiC NWs nanocomposites ceramics are displayed in Fig. 1, indicating the formation of only single phase perovskite structure in all cases. All pattern were identified main Ba(Zr$_x$Ti$_{1-x}$)O$_3$ peak, which could be matched with JCPDS file no. 36-0019. No trace of the SiC phase was detected in all nanocomposites. It may be due to good mixing between the BZT matrix and SiC NWS during vibro-milling. Moreover, the content of SiC NWs containing in the nanocomposites may have been too small to be detected by XRD.

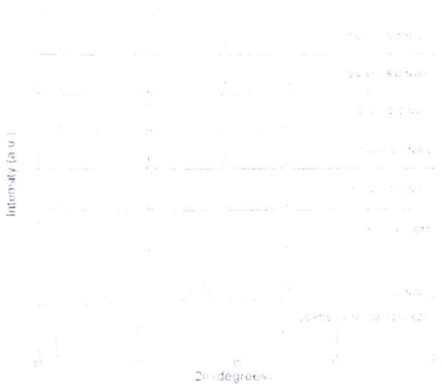

Fig 1. XRD patterns of BZT/SiC NWs powders.

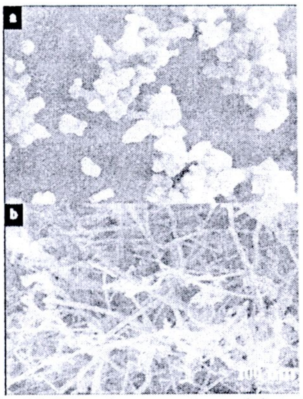

Fig 2. SEM micrograhs of fabricated (a) BZT powders and (b) SiC nanowires

Physical properties and microstructure of BZT/SiC NWs nanocomposites ceramics

The influence of SiC NWs additions on relative density and shrinkage of all BZT/SiC NWs nanocomposites ceramics are shown in Fig 3. The relative density was decreased markedly with increasing the content of SiC NWs but the values of BZT/SiC NWs with 0.1 wt% additives was almostly the same of monolithic BZT ceramics. It shows that the small amount of SiC NWs has small effective on sinterability of BZT ceramics, whereas increasing of SiC NWs additives exhibits more effect on the densification, shrinkage and sinterability of the ceramic matrix. Furthermore, adding a small amount of SIC NWs also has affect to grain growth mechanism of BZT ceramic matrixIt believed that the grain boundary movement is depressed by the SiC NWs. This may be caused by the pinning effect of SiC NWs at grain boundary, which depressed the migration of BZT grain boundary during the sintering process, similar with previous works [1-3]. In addition, the inhibition of densification was evidenced from the large fraction of open pores in 3.0 and 5.0 wt% SiC NWs/BZT samples (Fig 4 (d, e)), indicating that SiC NWs inhibited grain growth of ceramic matrix phase. Furthermore, shrinkage of all samples was increased with the increasing of SiC NWs. It can be ~~said~~ concluded that the SiC NWs is very good sintering aid for BZT ceramics ~~[5]~~ due to it has high thermal conductivity (3.6 W/cm.k) which agree with results of Hwang et al [5].

Microstructure of monolithic BZT and BZT/SiC NWs nanocomposites ceramics sintered at 1450°C for 2 h are observed by SEM technique and shown in Fig 4 It can be seen that there are many close pores in monolithic BZT ceramic matrix phase (Fig 4a.) which are gradually decreased with the increasing of SiC NWs. Moreover, it has been also seen that SiC NWs randomly located at grain boundary of BZT matrix phase, which obviously indicated in 3.0 and 5.0 wt% SiC NWs samples.

Table 1 Physical properties of BZT/SiC NWs nanocomposites ceramics after sintered at 1450 for 2 h.

SiC NWs content (wt%)	Bulk density ($g.cm^{-3}$)	Relative density (%)	Shrinkage (%)
0	5.42 ± 0.023	88.8	10.8 ± 0.768
1.0	5.37 ± 0.012	87.7	17.8 ± 0.145
3.0	4.55 ± 0.013	87.6	20.6 ± 0.291
5.0	4.37 ± 0.004	74.7	23.7 ± 0.145

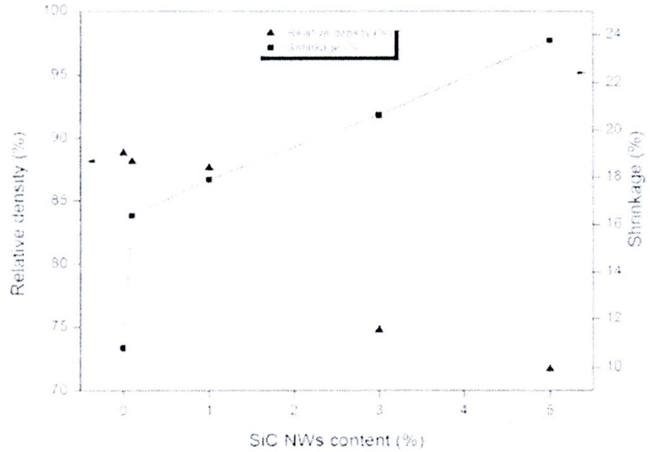

Fig 3. Plots of relative density as a function of content of BZT/SiC NWs nanocomposites ceramics sintered at 1450°C.

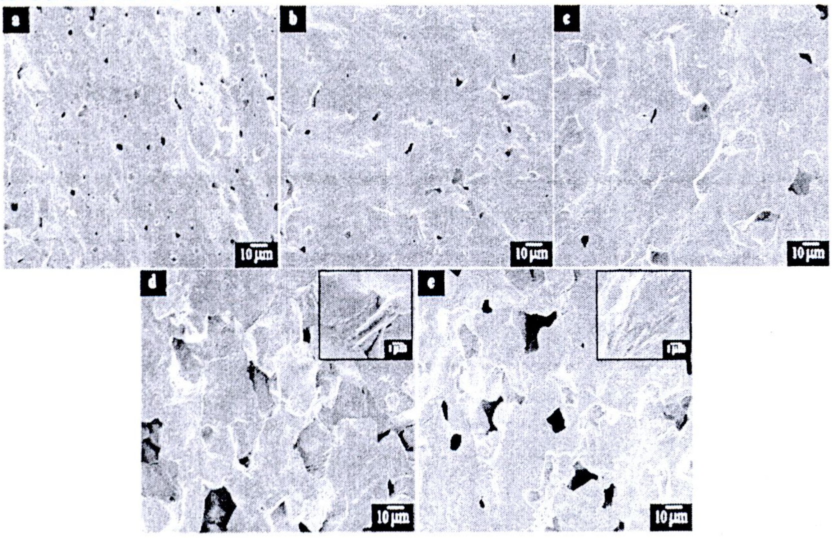

Fig 4. SEM micrographs of fracture surfaces of BZT/SiC NWs nanocomposites ceramics, where (a) monolithic BZT, (b) – (e) represent x = 0.1, 1.0, 3.0, 5.0 wt%, respectively.

Dielectric properties of BZT/SiC NWs nanocomposites ceramics

Dielectric constant (ε_r) and dielectric loss (tan δ) measured at room temperature were plotted as a function of frequency as shown in Fig 5. The results exhibited that the addition of SiC NWs can improve the dielectric properties of BZT ceramic Dielectric constant of BZT ceramics are increased with the increasing of amount of SiC NWs. It may be due to the smaller grain size which affect from grain growth inhibiting mechanism of nanoparticles on ceramic matrix phase.

However, it is also have the effect on higher dielectric loss of ceramic matrix which It may be the affect of a large fraction of porosity in matrix phase of monolithic BZT ceramicsTherefore, it can be concluded that adding SiC NWs can promote dielectric property of BZT ceramics which agree with the previous work [4].

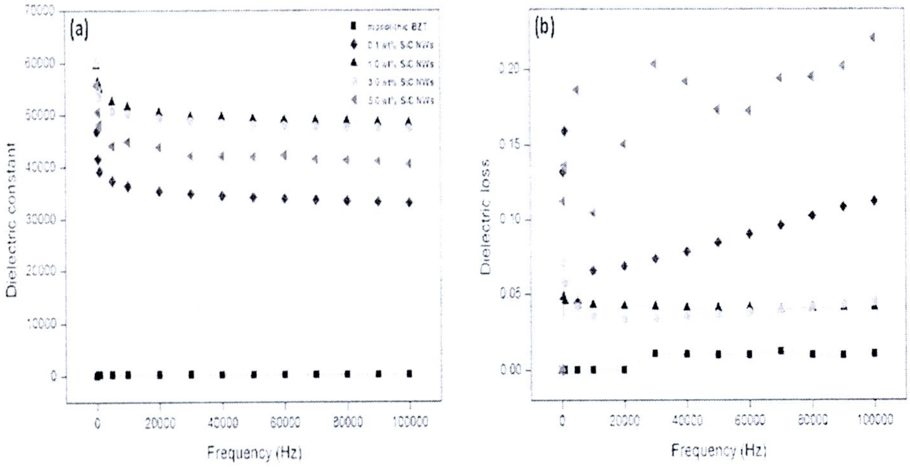

Fig 4. Variation with frequency of (a) dielectric constant and (b) dielectric loss of BZT/SiC NWs nanocomposites ceramics as a function of SiC NWs content.

CONCLUSIONS

In the fabrication of BZT/SiC NWs nanocomposites ceramics, adding the small amount of SiC NWs were improved dielectric properties could be successfully produced by a simple solid-state mixed oxide conventional sintering method. Addition of SiC NWs was found to depress densification, caused inhibited grain growth mechanism. The dielectric constant measured at room temperature was observed in all compositions. However, the dielectric constant at room temperature of BZT ceramic could be improved in sample contained 1.0 wt% SiC NWs. It was suggested that the optimum composition of BZT/SiCs nanocomposites ceramics.

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