2012 4th International Conference on Chemical, Biological and Environmental Engineering
IPCBEE vol.43 (2012) © (2012) IACSIT Press, Singapore
DOI: 10.7763/IPCBEE. 2012. V43. 7

NiTiO3/polyimide Nanocomposite: Synthesis and Magnetic Properties

S. Khanahmadzadeh[1+], S. Simani[2], M. Enhessari[3] and B.Khezri[4]

[1, 2, 4] Department of Chemistry, Mahabad Branch, Islamic Azad University, Mahabad, Iran
[3]Department of Chemistry, Islamic Azad University, Naragh Branch, Naragh, Iran

Abstract. Nickel titanate (NiTiO3)/ polyimide composite was successfully prepard in wet-chemistry synthesis method, using nickel titanate nanopowder, pyromellitic dianhydriede (PMDA) and 4, 4-oxydianiline. In this method. Fourier taransform infrared spectrometry (FT-IR), X-ray diffraction (XRD), scanning electron microscopy (SEM) and vibrating sample magnetometer (VSM) were used to characterize morphology, particle size and the magnetic properties. The results indicated that nickel titanate polyimide nanocomposite with particle size between 20 and 40 nm could be obtained after calcinations of the dried gel at 300 °C.

Keywords: Nanocomposite, Magnetic properties, Nickeltitanate, Polyimide

1. Introduction

Composite consisting of a polymer matrix and dispersed ceramic particles is a kind of materials with great potential properties and applications [1]. Titanium based oxides containing metals such as MTiO3 (M: Ni, Pb, Fe, Co, Cu, and Zn) are universally known as inorganic functional materials with wide applica -tions [2]. Nickel titanate has been investigated as a tribological coating to reduce friction and wear at high temperature applications without using liquid lubricants[3].The wet-chemistry synthesis technique used in this study, including sol-gel, sol-precipitation, combustion synthesis, chemical coprecipition, and hydrothermal synthesis, offers many distinctive advan -tages over solid-state method in the production of powders such as a controlled morphology, a narrow size distribution and high purity[4-8]. Aromatic poly - imides exhibit many useful properties such as high transition temperatures, excellent dimensional sta-bility, low dielectric constants, and outstanding thermal and thermo – oxidative stability. Therefore some of these materials are being used in such application as high per formance structural materials and packaging in printed electronic circuity. Polyimides are primarily used in the aerospace and microelectronics industries in the forms of films, moldings, and foams [9].

2. Experimental

A solution containing 1.0 gr coupling agent of APTS in ethanol (5 ml), and 1.0 gr nickel titanate particles were added into a flask .Under vigorous stirring, this suspension was ultrasonicted at room temperature for 10 min, and heated at 80 Ċ for 1 h. The condensation between PMDA and ODA in DMAC at room was synthesized (synthesis method see Ref. [6]). After adding the modified NiTiO3 particles into DMAC, temperature yielded a poly (amic acid) (PAA, the precursor of polyimide) solution with certain solid content the mixture was vigorously stirred under ultrasonication for 4 h at room temperature giving a suspension.Then, the above PAA solution was added into this suspension and stirred for 24-48 h at temperature of 80 Ċ and rate of 400-500 rpm and finally heating the dried gel at temperatures to obtain (at each temperature of 100, 200, 250, and 300 Ċ for 1 h, respectively), the polyimide/NiTiO3 composite was

+ Corresponding author. Tel.:+984422336000 fax: +984422330601
E-mail address: khanahmad_s@yahoo.com

obtained. In this process, the content of NiTiO3 in the composite was controlled by the portion ratio of NiTiO3 and PAA.

3. Results and Discussion

3.1. X-ray Diffraction Patterns and IR Spectra

Fig. 1. Is the FT-IR spectra of NiTiO3 particles,pure polyimide, and polyimide/NiTiO3 composite.is this spectrum the NiTiO3 powder calcined at 750 Ċ for 2h showed peaks below 800 cm-1 which are assigned to the Ti-O stretching vibration(see Ref.[2]).In the spectrum of pure polyimide, the absorptions of imide carbonyl band at 1780 and 1720 cm-1 , and the absorption of imide C-N band at 1380 cm-1 were the characteristics of PMDA/ODA polyimide[9]

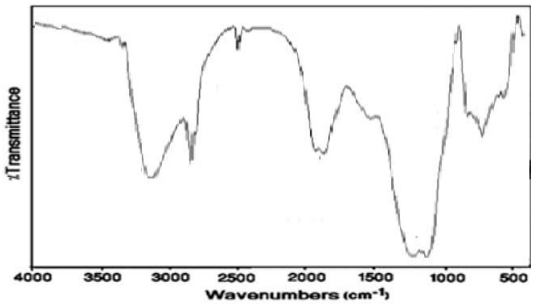

Fig. 1: FTIR spectra of the nanocomposites calcined at 300Ċ.

In the spectrum of NiTiO3/ polyimide comoosites, the absorption of NiTiO3 below 800 cm-1 ,as well as that of polyimide was found inunchanged state.These results the success in synthesi -zing the polyimide/NiTiO3composites.To investigate the stability of crystal structure of NiTiO3 in the process of composite preparation, XRD measurements were carried out. Fig. 3 shows the XRD patterns of the original NiTiO3 particle, the pure polyimide and the obtained PI/NiTiO3 composite. It was found that the used NiTiO3 particles possessing a crystal rhombohedral structure [2].In comparison with the pure NiTiO3 in the composite had no obvious differences. It was indicated that the crystal structure of NiTiO3 was still stable when it was doped into polyimide matrix. However, the XRD pattern of polyimide matrix in the composite was different to that in pure polyimide.In the XRD of pure polyimide, the broad peak with 2 centered at 17.5 revealed that the polyimide molecules have amorphous structure.As well, this peak becomes narrow in comparison with the correspondence at 17.5 in pure polyimide.

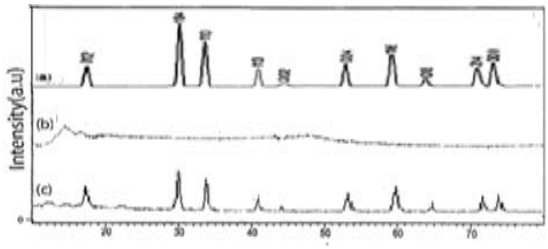

Fig2: X-ray patterns of (a) NiTiO3 particles, (b) pure polyimide and (c) polyimide/NiTiO3 (NiTiO3 content is 10 %)

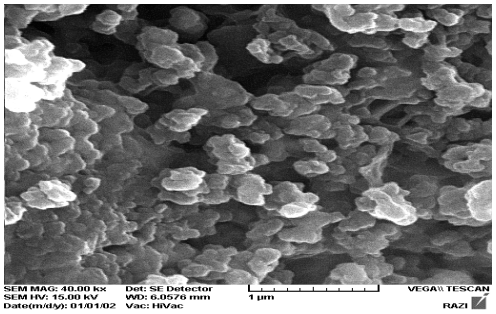

Fig. 3: SEM images of polyimide-NiTiO3 nanocomposites calcined at 300 Ċ.

3.2. Morphology of Samples

The morphology of the composites measured by SEM is shown in Fig. 3. It could be seen that $NiTiO_3$ particles were uniformly distributed throughout the polyimide matrix. There was no obvious aggregation of $NiTiO_3$ particles in the composites.The size of discrete phase was still remained in near sphere shape with diameter of 21-40 nm.

3.3. Magnetic Properties

There are few reports regarding magnetic properties of NiTiO3. The VSM magnetic measurements for the NiTiO3 Fig. 4a. Show the magnetic properties of nanoparticles calcined at 750Ċ for 1 h.In the face-centered cubic crystallographic structure, the magnetic interactions of neighboring Ni+2 spins are ferro - magnetic within the a-b planes and antiferromagnetic between adjacent a-b planes.The sample exhibits a fully reversible transition, representive of a genuine antiferromagnet. This fact reflects the good sample quality, since magnetic irreversibilities are often present in magnetic ceramic samples. The synthesized NiTiO3 indicate a superparamagnetic behavior, as evidenced by zero coercivity and remanance on the magnetization loop. In a system of superparamagnetic particles does not show hysteresis in the M-H curves, hence HC and MR are near zero. A saturation magneti -zation of ~ 0.2 emu/g . The VSM magnetic measure-ments for the polyimide/NiTiO3 Fig. 4b. Show the magnetic properties of nanocomposites calcined at 300ċ for 1 h.

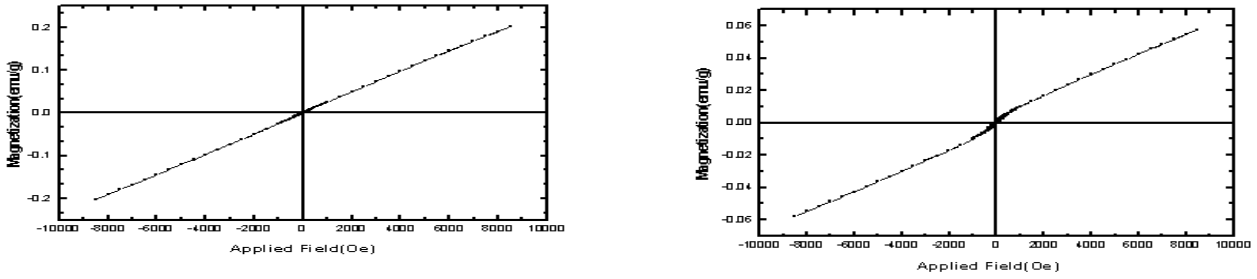

Fig. 4: VSM curves of a) NiTiO3 nanoparticles calcined at 750 Ċ, .b) polyimide-NiTiO3 nanocomposites calcined at 300 Ċ.

4. Conclusions

This study has demonstrated the feasibility of synthesis of polyimide\ NiTiO3 nanocomposites using wet chemistry synthesis route, stearic acid gel. Well crystallized polyimide-NiTiO3 nanocomposites could be synthesized at 300Ċ for 1 h.Morever, the synthesized polyimide \ NiTiO3 has been indicated a superpara magnetic behavior, as evidenced by using VSM at room temperature

5. Acknowledgement

The authors express thanks to Islamic Azad University and the Iranian Nanotechnology Initiative for supporting this work.

6. References

[1]. Shu-Hui, X., Bao-Ku, Zh., Xiu-Zhen, W., Zhi-Kang, X., and You-Yi, X., Polyimide/$BaTiO_3$ composites with controllable dielectric properties**,** *J. Composites:Part A*. 2005, **36**:1152-1157.

[2]. Enhessari, M., Parvizi A., Ozaee, K., Karamali, E., Magnetic properties and heat capacity of $CoTiO_3$ nanopowders prepared by stearic acid gelmethod, *J Experimental Nanoscience.*2011, **5** : 61-6.

[3]. Sadjadi, M-S., Zare, K., Khanahmad zadeh, S., Enhesari, M. Structural characterization of $NiTiO_3$ nanopowders prepared by stearic acid gel method , *J Materials letters*. 2008, **62** , 3679-3681.

[4]. Sedaghat, A., Taheri-Assaj, E., and Naghizadeh, E.An Alumina Math with a NanoMicrostructure Prepared by Centerifugal Spinning Method, *J. Nano – Crystalline Solids*. 2006, **352**: 2817-2828.

[5]. Chao, F., Liang, G., Kong, W., and Zhang XStudy of Dielectric Property on$BaTiO_3$/BADCy Composite, *J. Mat Chem. Phys*.2008,108: 306-311.

[6]. Wang, S.F., Wang, Y.R., Cheng, K.C., and Hasio, Y.P, Charactristics ofPolyimide/Barium Titanate Composite

Films,*J. Ceram. Int.*2008, 35**:** 265-268.

[7]. Mortezaei, M., Navid Famili, M.H., and Kokabi, M., 2009, Effect of Surface Modification of nanosilica on the Viscoelastic Properties of Its polystyrene nanocomposite, Iran. *J. Polym. Sci. Technol*. **21**: 523-530.

[8]. Chao, F., Liang, G., Kong, W., Zhang, Z., and Wang, J. dielectric properties of Polymer/Ceramic Composite Based on thermosetting Polymers, *J. Polym. Bull*. 2008. **60**: 129-136.

[9]. Hsiao, Sh-H., Chen, Yu-Jen. Structure-property study of polyimides derived from PMDA and BPDA dianhyarides with structurally different diamines , *J European polymer*. 2002, 38**:** 815-828.