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Abstract

Raman shift, FTIR spectra and SEM were used to detect the percolation threshold of nano titania filler ceramic $nTiO_2$ dispersed in a silicon rubber matrix SR8100 by hot vibrate dispersion. It has been found that an additional frequencies, and stock lines appears in Raman shift when $nTiO_2$ reach (5)wt% which mean a change in polarity of the SR8100/$nTiO_2$ PMNC polarity.

Keywords: PMNC, nano ceramic oxide fillers, $nTiO_2$, Raman shift

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1. Introduction

It is increasingly being recognized that new applications for materials require functions and properties that are not achievable with single materials. In this sense, composites formed by a polymer matrix and a conductive nano second phase are very interesting materials [1], these conducting polymer matrix nano composites PMNCs are capable of dissipating electrostatic charges and shielding devices from electromagnetic radiation [2]. Dielectric property of PMNCs with different conductivity fillers, such as carbon nano tube (CNT), cupper (Cu) and nickel (Ni) powders was investigated by several researchers [2-5]. Dependence of the dielectric property of the PMNC composites on frequency and volume fraction of fillers was also studied [6,7]. With gradually increasing the conducting filler content, composites undergo a percolation transition where the electrical conductivity of the composite jumps up several orders of magnitudes and its nature changes from an insulator to a conductor. This behavior is attributed to the formation of conducting network through the insulating matrix material when the filler content is at or above the percolation threshold [8,9]. The critical content of any filler that characterizes a drastic increase in conductivity is commonly termed as the electrical percolation threshold (EPT) [10]. On the other hand the use of nano ceramic oxides as a filler in several polymer matrixes can be viewed as one of the most promising area driven towards electronic applications [11]. Among all the ceramic oxide nano fillers $nTiO_2$ is documented to have the highest effect on the PMNCs, especially on the electrical and thermal properties. The improvement in these properties observed for $nTiO_2$ filled polymers could be due to one and/or more of the following factors (i) the large surface area of nano particles which creates a large interaction zone or region of altered polymer behavior, (ii) change in the polymer morphology due to the surfaces of nano particles, (iii) a reduction in the internal field caused by the decrease in the size of the particles, (iv) change in the space charge distribution, (v) scattering mechanism [12-17]. Aim of this research was to determine the percolation threshold of a new PMNC composed of SR8100 and $nTiO_2$ ceramic filler fabricated by hot vibrate dispersion (HVD).

2. Experiments

2.1 Materials

SR8100 polymer containing resin and hardener from (SICOMEN, USA), and (99.9%), and (10 nm) TiO_2 from (HORIBA, Germany).

2.2 Method

Polymer matrix discs were prepared using mould with 1cm diameter and 1cm height, the ratio of resin and hardener was 1:3. They were stirred by a magnetic stirrer for 5 min, then it was purred into the mould leaving for 24 hrs. After solidification the polymer discs were put in 1×1 cm cylindrical mould with a moving base with TiO_2 nano powder. The mould was then heated up to 80 °C and vibrate ultrasonically for 1 hr to ensure a uniform disperse for TiO_2 nano particles in SR8100 polymer matrix. Then it was left to cool down at room temperature.

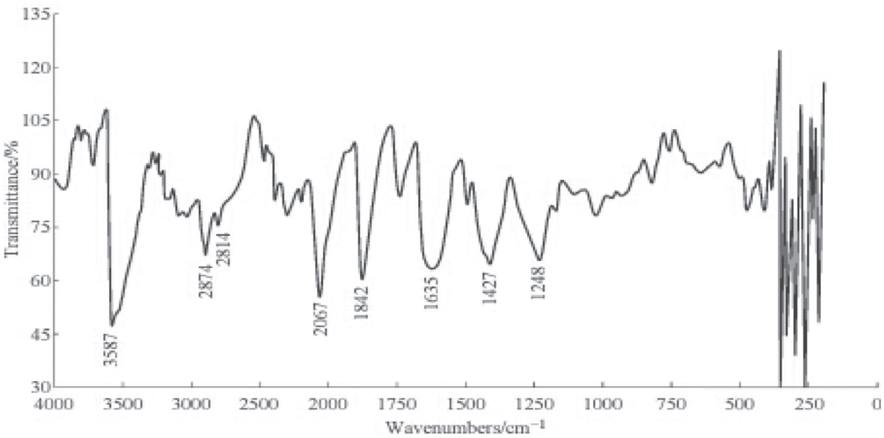

Fig. 1 *FTIR of SR8100/nTiO₂ PMNCs*
1. ábra *SR8100/nTiO₂ PMNC FTIR spektruma*

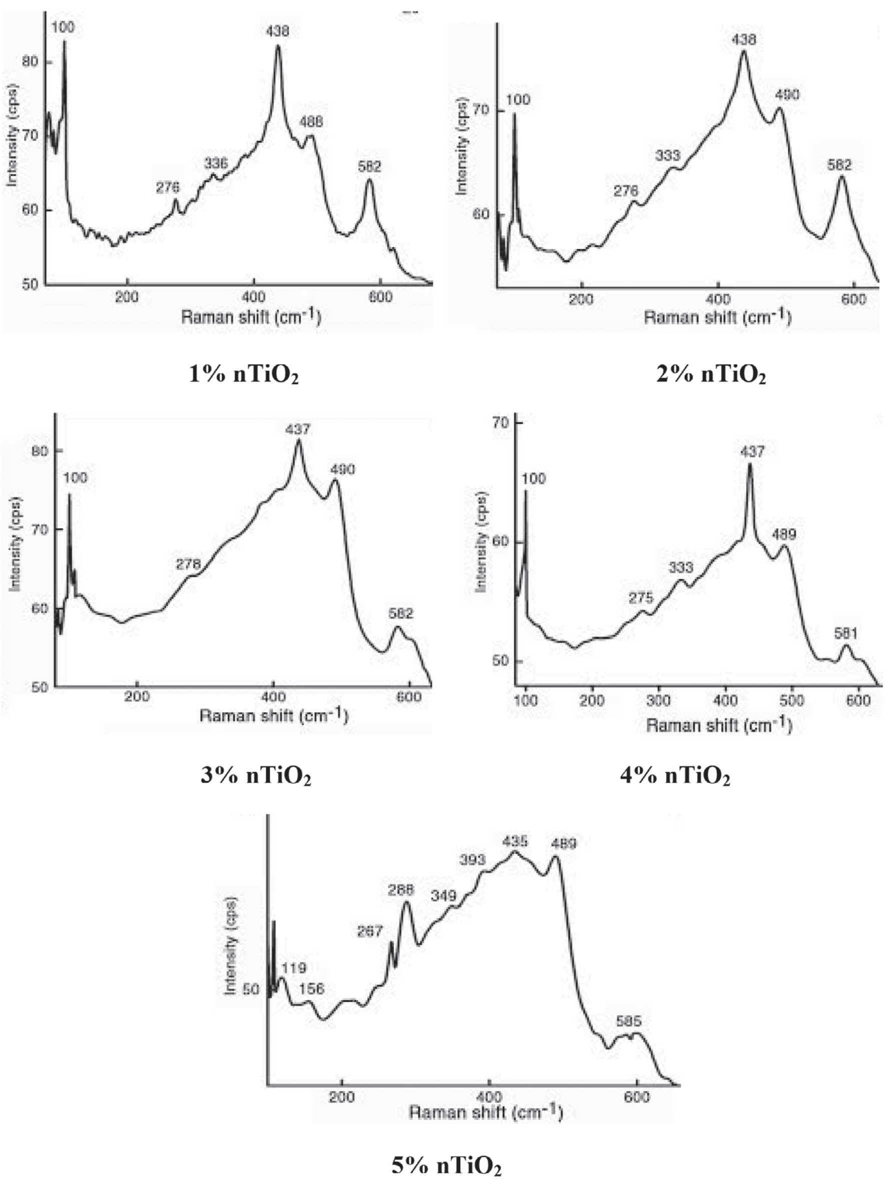

Fig. 2 *Raman Shift of SR8100/nTiO₂ PMNCs*
2. ábra *SR8100/nTiO₂ PMNC Raman eltolódása*

Specimens were tested by Raman shift, FTIR, and SEM to detect their properties.

3. Results and discussion

In the present work we succeeded to change polarity of silicon rubber (SR8100) by the addition of nano titania filler with 10 nm particle size using hot vibrate dispersion technique to prepare a polymer matrix nano composite material (PMNC) with high electrical conductivity. Both heating and vibrating used in PMNC preparation stimulate molecules and give it the energy it need to vibrate and spin creating a large change in the polarity of the polymer matrix as we shall see from Raman shift of the SR8100/nTiO$_2$ PMNC specimen. *Fig. 1* illustrates FTIR of SR8100/TiO$_2$ PMNC. PMNC specimen peaks at (1472.32,1635.64) refers to phases where SR8100 epoxy is the dominant, while peaks at (3587.60, 3525.88, 3464.15) are places where nTiO$_2$ concentrate [20].

Fig. 2 illustrates the Raman shift of SR8100 1-5 wt% nTiO$_2$. It is easy to notice the additional frequencies in Raman shift related to the internal energy of the SR8100/nTiO$_2$ PMNC especially when nTiO$_2$ weight percent is 5% as can be seen from *Fig. 1*, where stock lines has also appeared which is a very interesting result that we shall relay for our future work. From *Fig. 1* we may see the large similarity between Raman shift for the 1-4 wt% nTiO$_2$ PMNC specimen, while the 5 wt% nTiO$_2$ has much different peaks which may refer to a percolation threshold phenomena. According to this fact we may distinguish two types of Raman shift peaks; (i) the repeated peaks that appear in SR8100/nTiO$_2$ PMNC specimen with 1-4 wt% nTiO$_2$, which are as follow, peak at (100) which is related to symmetric stretching of C=O, (275,267 and 278) peaks indicate the stretching mode of C-H bond, (333,336) refer to a combination of C-O stretching and O-H deformation, peaks at 437,438 refer to nTiO$_2$, and/or stretching mode of saturated C-O, it may also refer to a slight entrance between SR8100 polymer matrix and nTiO$_2$ filler. Peaks (488,489,490) all refer to the presence of nTiO$_2$ phase, while (552,581,582,585) peaks indicate the deformation of C-H and C-O-H, ring deformation of polymer matrix and nTiO$_2$ presence [18,19]. Raman shift of 5 wt% nTiO$_2$ shows completely different peaks than the other SR8100/nTiO_2 PMNC specimens. In this specimen nTiO_2 is more obvious in the peaks (256,267,288), peak (349) refer to combination of C-O stretching and C=H deformation, peak (393) refers to nTiO_2 [20,21], and/or combination of C-O stretching and O-H deformation. Peak (435) indicates stock line that is not defined as polymer or nTiO_2; it may indicate percolation threshold and tunneling effect [18] that may take place within SR8100/nTiO_2 PMNC. Finally (585) peak refers to nTiO_2 and/or deformation of C-C and C-O [19,22,23]. The Raman shift results of this work are in agreement with FTIR results shown in *Fig. 1*.

Fig. 3 illustrates SEM of SR8100/nTiO_2 (PMNCs). We can see the good dispersion and high interaction between polymer matrix and nano reinforcing particles; this may lead us to conclude that there is, we may notice that at ultra low nTiO_2 concentration 1-3 wt% nTiO_2 there is no obvious pattern for dispersion of nTiO_2 in SR8100 matrix, hence no percolation is expected. But this regular pattern starts to appear at 4% nTiO_2 and continue with the same regular dispersion especially at 5% nTiO_2 which gives a very regular dispersion of nTiO_2 phase through the SR8100 matrix.

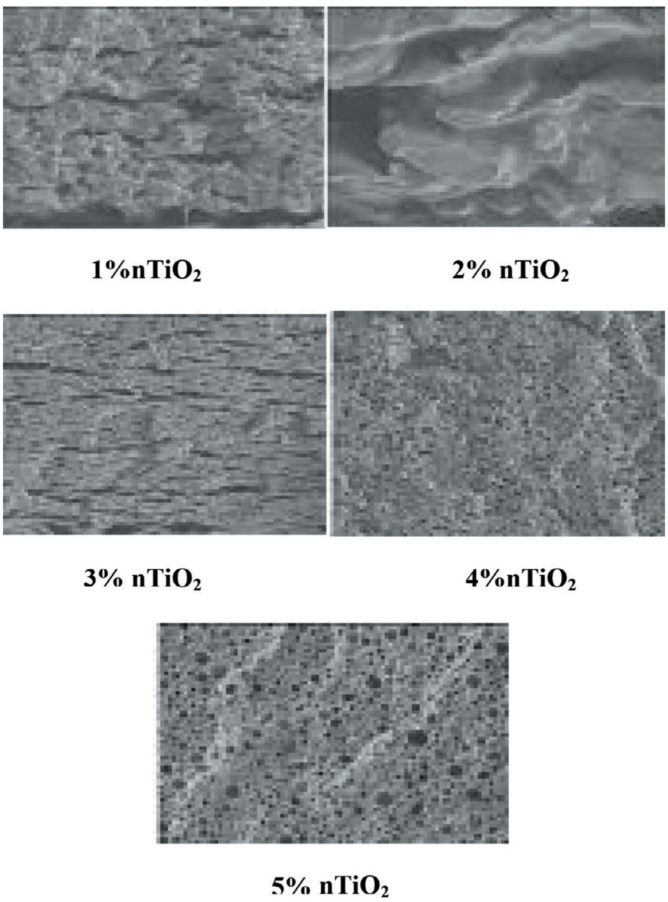

Fig. 3 SEM of SR8100/nTiO_2 PMNCs 1-5 wt% nTiO_2
3. ábra SR8100/nTiO_2 PMNC (1-5 wt% nTiO_2) pásztázó elektronmikroszkópos felvételei

Values of electrical resistivity, and thermal conductivity of the SR8100/nTiO_2 are listed in Table 1. From the table we may notice that addition of nTiO_2 effects on both electrical resistivity, and thermal conductivity when concentration of nano titania filler exceed 3%.

4. Conclusions

Raman shift, FTIR spectra and SEM were used to detect the percolation threshold of nano titania filler ceramic nTiO_2 dispersed in a silicon rubber matrix SR8100 by hot vibrate dispersion. It has been found that an additional frequencies, and stock lines appears in Raman shift when nTiO_2 reach (5) wt% which mean a change in polarity of the SR8100/nTiO_2 PMNC polarity.

Specimen Composition	Electrical Resistivity (Ω.cm)	Thermal Conductivity (W/m.k)
SR8100	1.0×10^{13}	0.120
SR8100+1%nTiO_2	0.821×10^{13}	0.416
SR8100+2%nTiO_2	3.07×10^{12}	1.002
SR8100+3%nTiO_2	5.48×10^{10}	2.714
SR8100+4%nTiO_2	2.36×10^{10}	5.227
SR8100+5%nTiO_2	4.65×10^{8}	7.186

Table 1..Electrical Resistivity and Thermal Conductivity of SR8100/nTiO_2
1. táblázat SR8100/nTiO_2 elektromos ellenállása és hővezetési tényezője

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Szilikon gumi-nano titándioxid PMNC Raman eltolódása

A szerzők nanoszemcsés TiO_2 töltőanyagot alkalmaztak SR8100 szilikon gumi ágyazóanyagban. A kialakuló struktúrát és az anyagjellemzőket pásztázó elektronmikroszkóppal, Raman spektroszkópiával és Fourier-transzformációs infravörös spektroszkópiával vizsgálták, és meghatározták az FTIR spektrumokat és a Raman eltolódást a perkolációs határ azonosítása érdekében. Az eredmények rámutattak, hogy 5 m% TiO_2 töltőanyag tartalom felett megváltozik a kompozit polaritása és Raman eltolódása.

Kulcsszavak: PMNC, nano kerámia oxid töltőanyag, $nTiO_2$, Raman eltolódás