The Effect of Titania Content on the Physical Properties of Polyimide/Titania Nanohybrid Films

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Received 12 July 2010; accepted 16 July 2011
DOI 10.1002/app.35292
Published online in Wiley Online Library (wileyonlinelibrary.com).

ABSTRACT: In this study, a series of PI/TiO_2 nanohybrid materials were prepared from polyamic acid of 3,3′,4,4′-benzophenonetetracarboxylic dianhydride/3,3′-diaminodiphenyl sulfone, and titania precursor by the sol-gel method. The titania content in the hybrid system was varied from 0 to 5 wt %. The physical and mechanical properties of the hybrids such as refractive index, optical transmission, and tensile strength were investigated. It was determined that incorporation of titania precursor into the PI matrix improved the refractive indices and tensile modulus of the hybrid films. It was observed that the optical transmittance and tensile strength of the nanohybrids were slightly decreased with the increasing titania content. It was determined that the hybrid films might have enhanced the UV shielding properties compare to the PI films. Furhermore, the hybrid materials showed better thermal stability than the PI. SEM studies demonstrated that titania particles (1 and 3 wt %) were distributed homogeneously through the PI matrix. The effect of the titania content in the PI on DC conductivity and dielectric constant were also analyzed. For the PI film containing 5 wt % titania, activation energy value increased to 1.0 eV from the value of 0.65 eV. DC conductivity value of the films depending on titania content varied between 3.0×10^{-11} and 1.4×10^{-10} S/cm at room temperature. Relative dielectric constants of the films were calculated from capacitance measurements depending on frequency (40–100 kHz) at different temperatures (303–360 K). The values increased with the increasing titania content. © 2012 Wiley Periodicals, Inc. J Appl Polym Sci 000: 000–000, 2012

Key words: nanocomposites; polyimides; thin films; dielectric properties; activation energy

INTRODUCTION

Last three decades, organic/inorganic hybrids have gained considerable importance as new-generation advanced materials because of the fact that they usually exhibit better properties than traditional composites and conventional materials. They combine some advantages of organic polymers (toughness, ductility, and easy processing) and those of inorganic materials (heat resistance, retention of mechanical properties at high temperatures, low thermal expansion, and transparency).[1,2] The enhanced properties of these nanocomposite materials mainly emerged from the fact that the discrete inorganic phases, which can be nanoscale level, provide a very large interfacial area between the organic and inorganic phases.

The sol-gel process based on hydrolysis and condensation reaction of alkoxy derivates of materials such as Zr, Al, Si, and Ti is most widely used method for the preparation of the organic–inorganic hybrid materials since sol-gel chemistry offers a versatile approach to design new nanocomposites.

Polyimides (PIs), a well-known class of high-performance polymers have found various applications in many high-tech fields. Among them, the cyclic aromatic imides are the most intriguing materials for optical waveguide applications due to the fact that they have excellent thermal stability, high mechanical strength, low dielectric constant, low thermal expansion coefficient, high glass transition temperature, and good resistance to solvents.[3–6] PIs are particularly suitable for the sol-gel process since they can be produced from polyamic acid (PAA) precursors. As the condensation reaction for the conversion of PAA to the corresponding PI is an intramolecular process, there will be no steric hindrance by the surrounding inorganic network after gelation.

Polyimide/titania hybrid materials (PI/TiO_2) have attracted lots of interests due to their high refractive index. For this reason, these hybrids were used as interference filters, antireflective coatings, protective layers, and optical waveguides.[7–12] Nevertheless, there are some problems for the preparation of

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PI/TiO_2 hybrids: First one is the decrease of thermal stabilities of the hybrids caused by the decomposition of TiO_2 and incomplete imidization of PAA. Second one is the difficulty to control of hydrolysis reaction of TiO_2 precursors owing to their high reactivity, which often results in the agglomeration of inorganic particles. Therefore, acetyl acetone (acac) act as a chelating agent which is employed to stabilize the titanium alkoxside.[13,14]

Until now, numerous titania-based nanocomposites have been reported in the literature, but the studies on polyimide/TiO_2 nanohybrid materials are limited. Chiang et al.,[15] reported the preparation of polyimide/TiO_2 hybrid materials via sol-gel process by employing titania-acac complex. The results indicated that the polyimide/titania hybrids exhibited lower thermal expansion and resistivity compared to neat PI. It is observed that the incorporation of small amount titania into the PI matrix improved the mechanical properties of the hybrid films at both low and elevated temperatures. Yoshida et al.[8] reported a new approach for the preparation of polyimide/TiO_2 nanohybrid materials. This approach is a unique solid-state impregnation using prepared nano-sized particles in reverse miscellar micro-reactors. The resultant composites possess optical clarity due to the fact that the particle size is very small. Chiang and Whang[14] prepared new PI/TiO_2 hybrid materials from 2,5-bis (4-amino phenyl)-1,3,4-oxadiazole (BAO), 4,4′-oxydiphtalic anhydride(ODPA) and Ti : acac via *in situ* sol-gel process. It was found that these nanohybris exhibited fairly good optical transparency up to 40 wt % of TiO_2 content. Transmission electron microscope measurements of the hybrids also showed that the TiO_2 particles (10–40 nm) are well dispersed in the polyimide matrix. Naganuma and Kagawa[12] prepared polyimide/titania hybrid films from a PAA of pyromellitic dianhydride (PMDA)/4,4′-oxydianiline (ODA) and titanium *n*-butoxide by the sol-gel process. The effects of titania content on the refractive index of the polyimide/titania hybrid materials were examined. It was determined that the refractive index at a wavelength of 633 nm increased from 1.67 for pure polyimide to 1.77 for the polyimide/titania hybrid film.

The development of PIs to investigate the electrical properties has also been the focus of several investigators. Because of this fact, measurement of the electrical properties of polyimide thin films is necessary.[16] Therefore, determination of dielectric properties of PIs with different dopants such as PI/$BaTiO_3$,[17,18] Boron[19] and fluorine diffused PI,[19,20] polyimide/silica/titania nanohybrids[10] has been investigated. According to best of our knowledge, DC electrical properties and dielectric properties depending on titania content in a wide range of temperature were rarely studied.

In the present work, we prepared the polyimide/TiO_2 (PI/TiO_2) nanohybrid materials by the sol-gel technique. The effects of TiO_2 content of the hybrids on the various properties (thermal, mechanical, and morphological properties) were investigated. Additionally, a detailed study on the effects of the titania content of the nanohybrids on DC conductivity and dielectric constant (between the temperatures of 303 and 363 K) were done and the optical properties were investigated.

EXPERIMENTAL PROCEDURE

Materials

3,3′-Diaminodiphenyl sulfone (DADPS) was obtained from Merck (Istanbul, Turkey) and recrystalized from ethanol prior to use. 3,3′,4,4′-Benzophenonetetracarboxylic dianhydride (BTDA) was purchased from Merck (Istanbul, Turkey). *N*-Methyl-2-pyrrolidone (NMP) was supplied by Merck (Istanbul, Turkey) and it was dried and freshly distilled over phosphorous pentoxide (P_2O_5) before use. Titanium (IV) isopropoxide [(Ti $(O^iPr)_4$)] and acetylacetone (acac) was provided by Merck (Istanbul, Turkey). Glass panels ($50 \times 100 \times 2$ mm^3) were used as a substrate to prepare free thin films. Au (99.98%) was purchased by commercially.

Measurements

FTIR spectras were recorded on Shimadzu 8303 FTIR Spectrometer.

Thermo gravimetric analyzes (TGA) were performed using a TA Instruments Q50 model TGA. Samples were run from 30 to 800°C with a heating rate of 10°C/min under nitrogen atmosphere.

The morphology of the nanohybrid materials were examined using scanning electron microscopy (SEM JEOL JSM-5910 LV). The fractured surfaces were coated with a gold layer by vacuum sputtering. Elemental concentrations for titania and oxygen were determined with an energy dispersive spectrum (EDS) Oxford Instruments-INCA, Model No.7274.

The stress–strain tests were performed at room temperature on a Universal Test Machine (Zwick Rolle, 500*N*) with a crosshead speed of 2 mm/min. Reported data represents the average of at least four measurements.

UV–Visible transmission spectra of the hybrid films were obtained in the wave length range of 200–800 nm using a Shimadzu UV 6010 spectrometer.

Refractive indices of the hybrid films were measured on a Microphotonics Ellipsometer EL X-01 R.

Both DC conductivity and dielectric measurements of the nanohybrid films were carried out by preparing gold electrode system in a sandwich structure (gold/polyimide-Ti/gold). Gold (Au) electrodes were

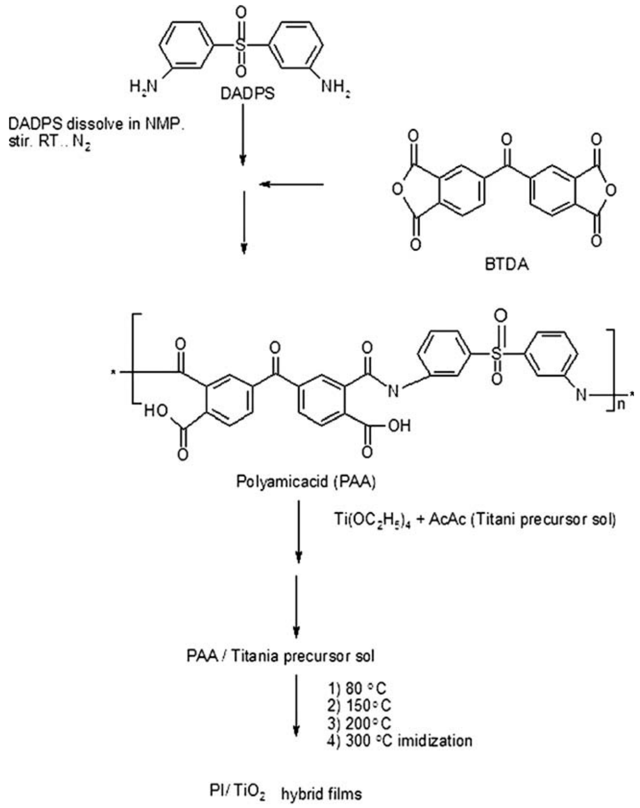

Figure 1 Flow chart showing the procedure for preparing of the PI/TiO_2 hybrid films.

formed by thermal evaporation of gold onto both sides of the films in a vacuum level at 2×10^{-6} mbar (Edwards Auto 500 Deposition System). Before the evaporation, the films were dried at 363 K under high vacuum ($\sim 10^{-6}$ mbar) for 1 h to avoid the effect of the adsorbed gas molecules on the electrical properties. The film thickness values were between 50 and 120 μm and radius of the Au electrodes was 2 mm. After the Au electrodes were performed, the films were placed in a chamber for electrical measurements. Electrical contacts between sample and chamber were taken using silver paste. During the measurements vacuum level of the chamber was 3×10^{-3} mbar. Current–voltage (I–V) measurements were carried out with a Keithley 617 model programmable electrometer. Electrical measurements were performed in the temperature range of 303–363 K. The frequency and temperature dependencies of the dielectric constant were investigated for the films. Capacitance values of the films were measured with a Keithley 3330 model LCZ meter between the frequencies 40 and 100 kHz at different temperatures (303–363 K). All the measurement system was computerized.

Preparation of the titania precursor

Titanium(IV) isopropoxide [(Ti ($O^iPr)_4$)] is very reactive toward moisture due to the presence of electronegative isopropoxide groups that make possible a nucleophilic attack. Therefore, titanium isopropoxide needs to be complexed with a chelating agent such as acetylacetone (acac), methacrylic acid (MA), and 2-(methacryloyloxy) ethyl acetoacetone (MAEA) to control the rate of hydrolysis. To meet this condition, acac was used for titanium isopropoxide stabilization. For the preparation of titania precursor, 0.05 mol (14.2 g) of titanium tetraisopropoxide $Ti(O^iPr)_4$ and 0.1 mol (10 g) of acac were stirred under a nitrogen atmosphere. The clear yellow Ti : acac solution was obtained.

Preparation of the polyimide/titania hybrid films

PAA used as a PI precursor was prepared in NMP as follows:

DADPS (0.02 mol, 4.97 g) was dissolved in 57 mL of dried NMP. Then, BTDA (0.02 mol, 6.44 g) was added in small portions into the above solution. The resulting solution was stirred for 24 h at room temperature under N_2 atmosphere. The solid content of PAA solution was kept at about 20% (w/v). The solution with various amount of titania precursor was added drop by drop into the PAA solution with vigorous stirring to minimize the occurrence of local precipitation. After stirring for 4 h, the reaction went to completion. After mixing, the obtained clear and viscous hybrid solution was cast on to the glass plates with the aid of a 30 μm applicator. Then the wet coating was cured at 80, 100, 150, 200, and 300°C for 1 h at each temperature. The cured films were retrieved from the glass surface by immersing in distilled water at 80°C.

The preparation route of PI/TiO_2 hybrids was schemed in Figure 1. The recipe for the hybrid formulations was also listed in Table I.

RESULTS AND DISCUSSION

A series of nanohybrid materials were prepared from PAA solution and titania precursor which is composed of [(Ti ($O^iPr)_4$)] and acetylacetone, via the

TABLE I
The Recipe Preparation of PI and PI/TiO_2 Hybrid Materials

Samples	PAA solution (g)	Titania precursor (Ti : acac) (g)	TiO_2 (wt %)	PI (wt %)	Appearance
PI	5	–	0	100	Clear
PI/TiO_2-1	5	0.0575	1	99	Clear
PI/TiO_2-2	5	0.1164	2	98	Clear
PI/TiO_2-3	5	0.1767	3	97	Clear
PI/TiO_2-4	5	0.2383	4	96	Clear
PI/TiO_2-5	5	0.3007	5	95	Clear

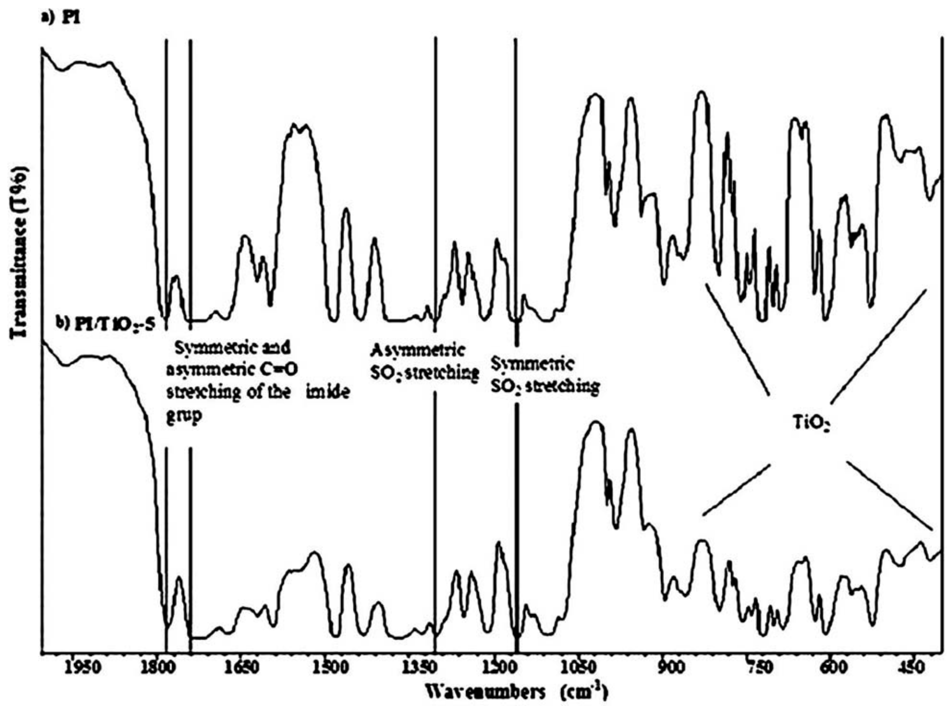

Figure 2 FTIR spectra of (a) PI and (b) PI/TiO_2-5 hybrid films.

sol-gel method. The preparation route of the hybrids and their compositions were depicted in Figure 1 and Table I, respectively.

Characterization of the nanohybrid materials by FTIR

The FTIR spectra of PI and PI/TiO_2-5 hybrid films were shown in Figure 2(a,b). Both samples exhibited the characteristic peaks of symmetric and asymmetric C=O stretching of the imide group at 1720 and 1780 cm^{-1}, respectively. Additionally, C—N stretching of the imide ring was observed at 1380 cm^{-1}. It can be seen in Figure 2(b), with the addition of inorganic components into the PI matrix, a broad and strong absorption band was observed, with slightly upward shift of the base lines in the range of 400–850 cm^{-1}, which corresponds to Ti-O-Ti network.[14,21] Both spectra consist of other peaks located at 1310 cm^{-1} for asymmetric SO_2 stretching and at 1155 cm^{-1} for symmetric SO_2 stretching. The mentioned peaks emerge from DADPS monomer. However, one can be seen from the spectra given in Figure 2(a,b); the absorption bands related to PI and PI/TiO_2-5 confirm the formation of the expected structures.

Morphology of the nanohybrid materials

The morphologies of the fractured surfaces of the hybrid films with different titania content (1, 3, and 5 wt %) were investigated by SEM. From Figure 3(a,b), it can be observed that titania particles are homogeneously dispersed through the polyimide matrix. This result can be attributed to the fact that the hybrid films showed an intense interaction interface between the titania particles and the PI matrix. When the titania content of the hybrids was increased, the chelation between titania and PI main chains (PAA) enhanced due to the adhesion between organic and inorganic phases.[22] So more homogeneity of disperse of the nanoparticles in the polymer matrix can be attained. However, from Figure 3(c) very small agglomeration was observed in PI/TiO_2-5 hybrid film. It was explained that concentration of the titania : acac solution near the critical limit for agglomeration. The diameter of the particles for PI/TiO_2 hybrids were in the range of 50–60 nm. Besides that, SEM-EDS were used to identify the chemical composition or the distribution of elements within the hybrid system [Fig. 3(d–f)]. It was determined that the nanohybrids were composed of titania and oxygen elements. The other peaks at the spectra seem to be related to gold coating needed for sample preparation.

TGA Analysis of the nanohybrid films

The thermal stability of PI and PI/TiO_2 nanohybrid materials was evaluated employing the TGA technique and curves of all materials taken at nitrogen atmosphere shown in Figure 4. The analyzing data was also collected in Table II. As seen from the Figure 4, all samples exhibited a 5% weight loss at

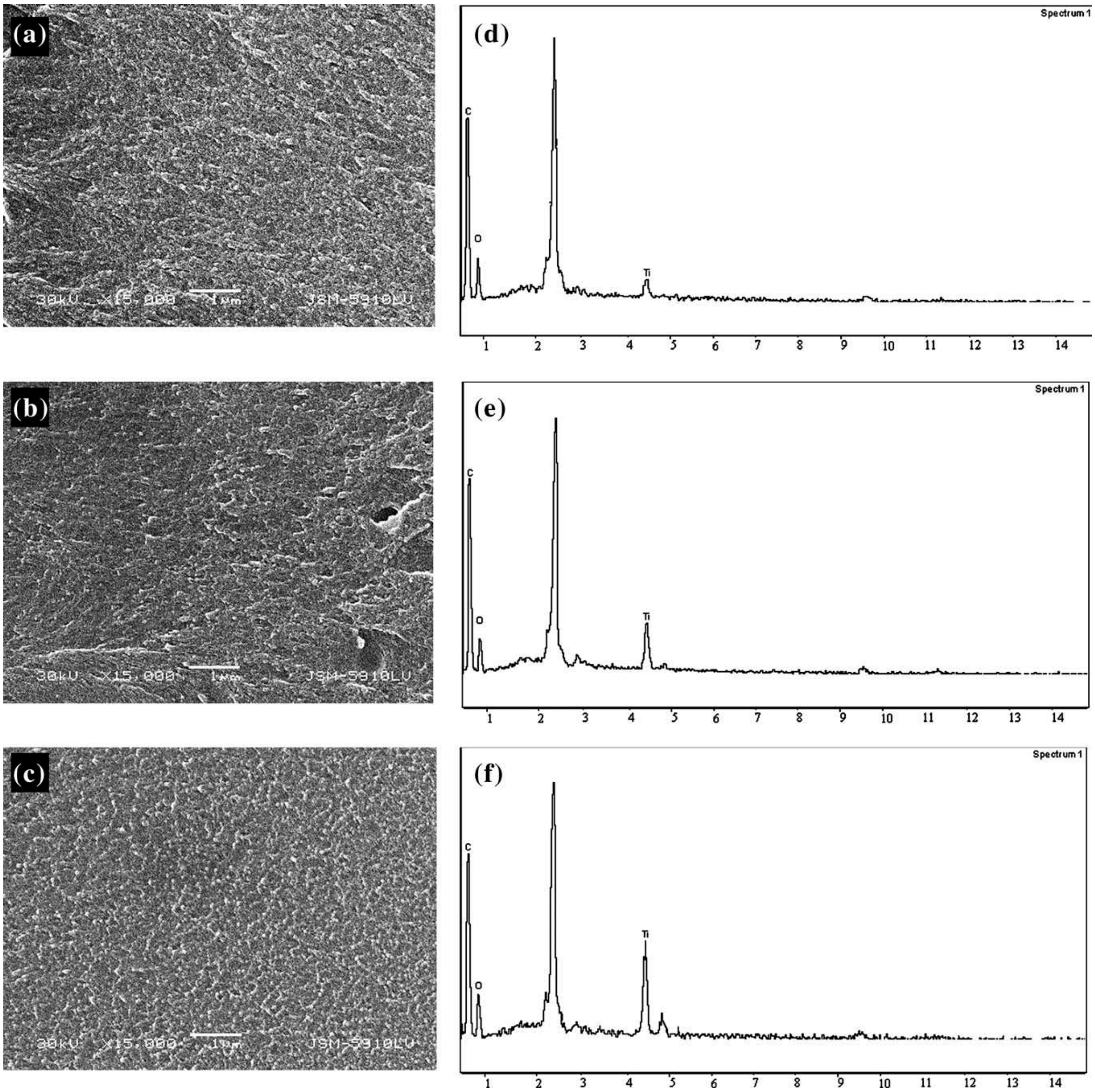

Figure 3 SEM micrographs of (a) PI/TiO_2-1, (b) PI/TiO_2-3, (c) PI/TiO_2-5. SEM-EDS spectrum of (d) PI/TiO_2-1, (e) PI/TiO_2-3, (f) PI/TiO_2-5.

280–350°C. This is probably due to the fact that incomplete imidization occurs, which is consistent with our FTIR results. As seen from the Figure 1(a,b) the small absorption peak at around 1660 cm^{-1} attributed to incomplete imidization. The thermal decomposition temperature (T_d) (at 10% weight loss) of PI was 500°C. When an increase in the titania content of the polyimide based hybrids, T_d decreased from 500 to 400°C. The decrease in thermal stability of the hybrid films can probably be attributed to metal-catalyzed oxidative decomposition pathways in the composite.[14,22,23] However, one can see from the Figure 4 that the maximum decomposition temperatures of the hybrid materials were between at 585 and 600°C. The char yields at 800°C are also increased with the increase of titania content due to the fact that metal oxides (TiO_2) cannot decompose at high temperature. Therefore, all the hybrid films still posses good thermal stability.[7]

Mechanical properties

The ultimate tensile strength (σ), tensile modulus (*E*), and elongation at break (ε) values of PI/TiO_2 hybrid films were compared with the pure PI and the results were summarized in Table III. Due to the

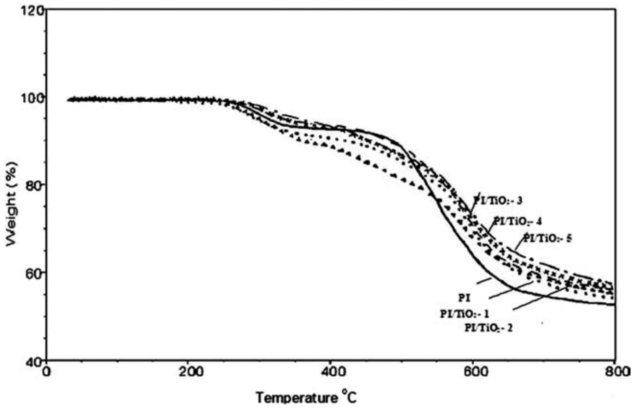

Figure 4 TGA curves of PI and PI/TiO_2 hybrid materials with different silica content.

effect that the presence of TiO_2 tends to make the films brittle, there is an upper limit for the introduction of TiO_2 used, 4 wt % in our case, for the material to be able to from a film.[24] Furthermore, the influence of titania content on the tensile modulus, tensile strength, and elongation at break was depicted in Table III, respectively. The pure PI film had a tensile modulus of 1.72 GPa. One can see from Table III, incorporation of TiO_2 greatly enhanced the tensile modulus, which increases linearly from 1.72 to 2.05 GPa (19%).[15] At the same time it can be seen from Table III, the values of tensile strength and elongation at break of the hybrids were decreased with the increase of titania content. Compared to neat PI, at 10 wt % of titania content, the tensile strength decreased by 76%, while the elongation at break decreased by 67%. This may be attributed to the fact that TiO_2 particles acts as stress concentrators and affects the ultimate fair or point during extension.[25]

Optical properties

Optical devices such as photonic integrated circuits generally require the control of the refractive index as well as the dimension of the waveguide structure and other optical properties of the material. Due to the high refractive index of TiO_2, its introduction may lead to change of the refractive index of the hybrid material.[21,23] As can be seen from Table IV the refractive index of the neat polyimide and the hybrid film, at 633 nm, increase from 1.596 for pure PI to 1.688 for PI-TiO_2-4. These results can be attributed to the titania content of the hybrid films. The more titania in the hybrid system, the higher the refractive index.

Figure 5 shows the UV–VIS transmission spectra of the pure polyimide and PI/TiO_2 hybrid thin films with different TiO_2 content. The absorption band moved toward longer wavelength and the transmittance was slightly decreased with the increasing titania content in the hybrid films. The results may be attributed to the increase of the degree of TiO_2 aggregation and formation of titania : acac complex during preparation of the precursor.[14,24] Consequently, titania content in the hybrid system (π-Ti-1 to 5) might be improved the UV shielding property of the pure PI due to slightly decreasing the transmittance like sunglasses.

DC Conductivity

DC electrical properties of the thin film of the compounds were investigated by means of current–voltage (I–V), measurements. The measurements were carried out at different temperatures between 303 and 363 K. I–V measurements for each film were carried out by cycling voltage, quassi statically in 50 mV steps with a delay time of 100 ms. I–V curves of the films showed ohmic behavior.

Figure 6 shows the dependence of the calculated DC conductivity on inverse of temperature for the pure and hybrid PI films in the temperature range of 303–363 K.

As seen from Figure 6, the exponentially dependence of the measured conductivity on temperature indicating the applicability of the well-known expression for conductivity.

$$\sigma_{dc} = \sigma_0 \exp(-E_a/kT) \qquad (1)$$

where E_a is an activation energy, T is temperature, k is Boltzmann's constant, and σ_0 is a constant of proportionality. The other five nanohybrid films containing different titania content show similar

TABLE II
Thermal Decomposition Temperatures of PI and PI/TiO2 Hybrid Films

Samples	5% Weight loss temperature (°C)	10% Weight loss temperature (°C)	Maximum weight loss temperature (°C)	Char yield (wt %) (800°C)
PI	300	500	560	52.4
PI/TiO_2-1	350	443	585	54.1
PI/TiO_2-2	280	397	590	55.1
PI/TiO_2-3	310	498	600	56.2
PI/TiO_2-4	290	469	600	56.8
PI/TiO_2-5	325	469	600	57.6

TABLE III
The Mechanical Properties of PI and PI/TiO_2 Hybrid Nanocomposite Films

Samples	Tensile modulus (Mpa)	Tensile strength (Mpa)	Elognation at break (%)
PI	1718.00	776.88	4.60
PI/TiO_2-1	1826.00	660.00	3.49
PI/TiO_2-2	1903.79	588.37	3.03
PI/TiO_2-3	1950.00	487.14	2.42
PI/TiO_2-4	2049.23	260.04	1.09

behavior. The values of the activation energies which are determined from straight portions of the slope of the ln conductivity versus $1/T$ graphs, are $E_A = 0.65$ eV for the pure PI film. Figure 7 shows the variation of activation energy values of the films versus titania content. As seen from Figure 7 activation energy values increases with increasing titania content. The values take places in the range of 0.65–1.00 eV.

The effects of the titania content on DC conductivity of the polyimide film were also investigated. Figure 8 shows the variation of DC conductivity of the films depending on titania content at room temperature. As seen from the Figure 8 we have three distinct region (0–2 wt %), (2–4 wt %), and a small greater than 4 wt % in our content of titania range.

The behavior can be explained as follows; In the first region, conductivity of the film increases with increasing titanium content in the PI film. In this region titanium atoms create new energy levels near to the conduction band in band gap. Since, the energy between the new levels and conduction band is smaller than the pure PI film, the conductivity increases with increasing titania content. The behavior is in agreement with other works.[10,15] However, further addition of titanium (the second region) decreases the conductivity of the film. The behavior may originate from the deep traps arising from titania content greater than 0–2 wt %. In the third region as seen from the figure, increase in conductivity is small. We may say that the change in titania content from 4 to 5 wt % does not mainly affect the conductivity. This low effect can also be seen from Figure 6. As seen from Figure 6 conductivity of the

TABLE IV
The Refractive Index of PI and PI/TiO_2 Hybrid Nanocomposite Films

Samples	Refractive index (632 nm)
PI	1.596
PI/TiO_2-1	1.637
PI/TiO_2-2	1.646
PI/TiO_2-3	1.675
PI/TiO_2-4	1.678
PI/TiO_2-5	1.688

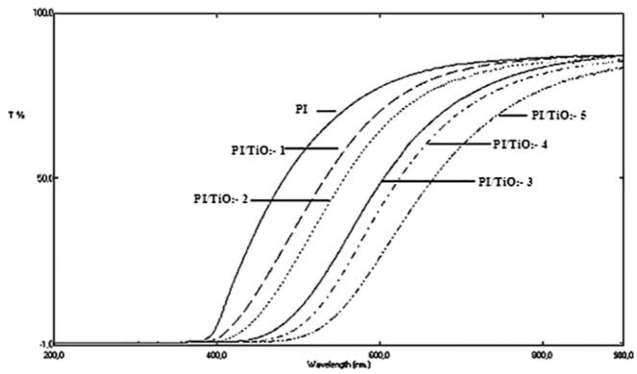

Figure 5 UV–visible transmission spectra of the PI/TiO_2 hybrid films.

hybrid films with titania content 4 and 5 wt % oscillates with each other in the measurement temperature range. The change in conductivity can be interpreted as small variations in energy levels.

As a result, conductivity of the films depending on titania content, we can conclude that main parameter in conductivity is energy levels created by doped titanium atoms. However, the conductivity of the films needs to analyze in a wide range of titania content. The issue is out of scope of this study and its underway.

Dielectric constant of the films

Capacitance values of the films were measured between the frequencies of 40 and 100 kHz at different temperatures (303–363 K). The dielectric constants of the films were calculated from their capacitance measurements by using the eq. (2).

$$\varepsilon = \frac{C}{\varepsilon_0}\frac{d}{A} \qquad (2)$$

The dielectric constant of polymers is generally known to decrease gradually with increasing frequency because the response of the electronic,

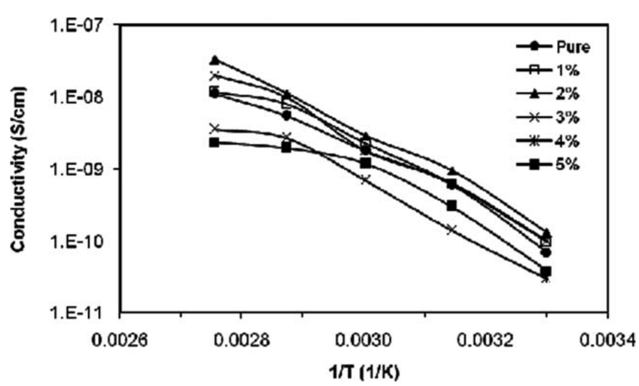

Figure 6 The variation of DC conductivity of pure and hybrid polyimide films as a function of inverse of temperature.

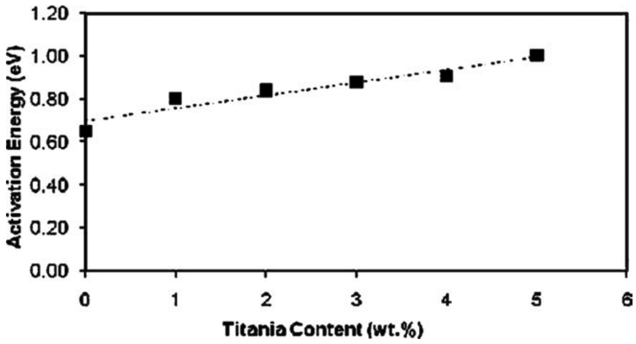

Figure 7 Variation of activation energy values of the hybrid films as a function of titania content (wt %).

atomic, and dipolar polarizable units varies with frequency. In a polymer the magnitude of the dielectric constant, ε, is therefore determined by the ability of the polarizable units to orient fast enough to keep up with an applied AC electric field of varying frequency.[19] These behaviors can be attributed to the frequency dependence of polarization mechanisms. We have observed small decrease (~ 2%) in dielectric constant of the films with increasing frequency. This is in good agreement with other works.[19,26] For polar polymers the dielectric constant increases with increasing temperature. We have observed small increase in dielectric constant (~ 2%) with increasing temperature. Our results are also in good agreement with other works.[19,26]

Figure 9 shows dielectric constant values of the films depending on titania content at 10^5 Hz at room temperature. It is clear that dielectric constant of the films increases with increasing titania content. The value of dielectric constant for the pure PI film is 6.35 and those of the PI containing 5 wt % titania are increased to the value of 10.35 with increasing titania content. The behavior we obtained here is in consistent with that reported by Chiang et al.[15] The increase in dielectric constant of the films could be attributed to the increase in polarizability of the PI/TiO_2 nanohybrid due to adding of titania.

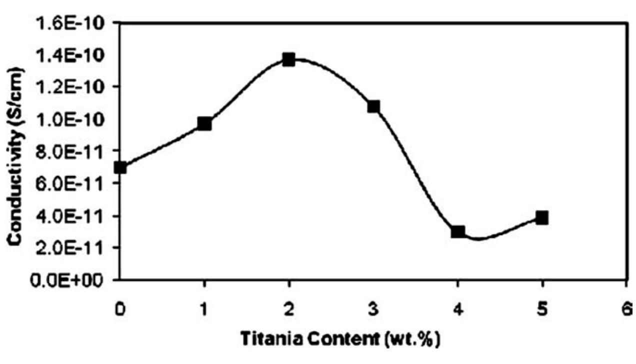

Figure 8 Conductivity of the hybrid films versus titania content (wt %) at room temperature.

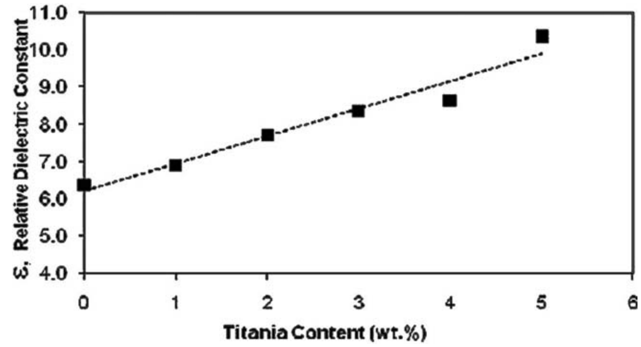

Figure 9 The effect of titania content (wt %) on dielectric constant of the polyimide films at 100 KHz at room temperature.

CONCLUSIONS

In this study, a series of polyimide/titania hybrid materials (PI/TiO_2) were prepared by the sol-gel process. The FTIR spectrum confirmed the Ti-O-Ti bonding in the PI matrix. The morphological study showed that the nanometer-scaled inorganic particles (1 and 3%wt.) disperse homogeneously within the PI matrix. The PI/TiO_2 hybrid films exhibited good mechanical properties. The incorpoaration of titania might be improved the UV-shielding property of the pure PI. Although, the thermal stability of the hybrid films was slightly lower than the pure PI, these films may be favored for various applications.

The increase in titania content causes increase in conductivity in the titania content range of 0–2 wt % and decrease in conductivity in the titania content range of 2–4 wt %. The results of dielectric measurements indicate that the increase in titania content causes increase in dielectric constant. The variation is linear in the titania content range of 0–5 wt %.

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