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# Preparation and characterization of highly transparent epoxy/inorganic nanoparticle hybrid thin films



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#### ARTICLE INFO

Available online 17 May 2013

Keywords: Epoxy-titania hybrid materials High transparent films Thermal polymerization Refractive index Sol-gel method

#### ABSTRACT

This paper presents the preparation of epoxy/inorganic-nanoparticle hybrid materials synthesized from diglycidyl ether of bisphenol A and colloidal titania (TiO<sub>2</sub>) with coupling agent, 3-isocyanatopropyltriethoxysilane, and curing agent, hexahydro-4-methylphthalic anhydride, by using a thermal polymerization. The precursor was spin-coated and thermal-cured to form hybrid films. The experimental results showed that the refractive index of hybrid films can be tuned by adding various solid contents of TiO<sub>2</sub> to hybrid films. The refractive index at 633 nm increased from 1.450 to 1.639 as the TiO<sub>2</sub> content increased from 0 to 50 wt.%. UV-vis analysis showed that the transparency of hybrid films was over 90%. La.b. color analysis indicated that the luminance of films was above 95%, and no yellowing was observed. In addition, the hybrid materials exhibited a low hydroscopic property under a high-humidity environment.

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### 1. Introduction

Organic/inorganic hybrid materials have recently been studied extensively. Organic materials have been widely used because of their flexibility, toughness, and processability. Although inorganic materials have high heat resistance [1], they have excellent mechanical and optical properties [2-5]. Organic/inorganic hybrid materials have a combination of the superior properties of both organic polymers and inorganic materials. The properties of hybrid materials can be tuned through the functionality or segment size of each component. Mixing hybrid materials in the molecular level using Vander Waals forces, hydrogen bonding, and ionic or covalent bonding [6] can overcome the traditional composite macroscopic phase separation to ensure excellent characteristics of organic and inorganic substances [7–11]. The properties (optical, mechanical, and thermal) of hybrid materials are relative to those of each component, the composite phase morphology, and interfacial properties. The mild characteristics offered using the sol-gel process allow inorganic and organic components to be mixed at the nanometric scale [12,13].

Epoxy resin systems are commonly used as matrices in composite materials for a wide range of automotive and aerospace applications, shipbuilding, and electronic devices. Highly cross-linked epoxy matrices

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can provide high stiffness and strength; however, they are often brittle because plastic deformation is constrained [14,15]. To develop technical nanocomposites, several researchers applied inorganic nanoparticles to improve various properties. Bauer et al. [16] showed that a high content of nanosized silica, alumina, and titania was embedded in epoxy adhesives. The reinforced nanocomposites exhibited shifts of glass transition temperatures of approximately 20 K, indicating improved thermal stability. Chau et al. [17] prepared epoxy/titania (TiO<sub>2</sub>) nanocomposite coatings with a high refractive index and optical transparency by using the sol-gel method. Nanocomposite coating with a refractive index of 1.668 can be obtained by adding 30 wt.% TiO<sub>2</sub> nanoparticles into the polymer matrix. Coatings with various amounts of TiO2 exhibited excellent optical transparency of more than 90%. Although a nanocomposite with a higher refractive index can be obtained by increasing the TiO<sub>2</sub> content, cracks appear on the surface of the hybrid coating. They also showed that the refractive index of hybrid films can be tuned using various forms of titania nanoparticles and by changing the solid content of titania. The solid content of titania in the epoxy matrix can be more than 70 wt.% without affecting the optical transparency of the hybrid film [18]. Sowntharya et al. [19] synthesized hybrid nanocomposite coatings from titanium tetraisopropoxide, and epoxy or acrylic modified silanes were deposited on polycarbonate (PC). The results showed that the coatings from a freshly prepared sol of acrylic modified silane and titania exhibited a maximal nanoindentation hardness of 0.52 GPa compared to 0.23 GPa for bare PC.

We prepared epoxy/TiO<sub>2</sub> hybrid films using the colloidal titania and diglycidyl ether of bisphenol A (DGEBA) with a 3-isocyanatopropyltriethoxysilane (IPTES) coupling agent and hexahydro-

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$$\begin{array}{c} \text{CH}_2\text{C} \\ \text{CHCH}_2\text{O} \\ \text{CH}_2\text{C} \\ \text{CHCH}_2\text{C} \\ \text{CH}_2\text{C} \\ \text{CHCH}_2\text{C} \\ \text{CH}_2\text{C} \\ \text{CHCH}_2\text{C} \\ \text{CH}_2\text{C} \\ \text{CHCH}_2\text{C} \\ \text{CH}_2\text{C} \\ \text{C} \\ \text{CH}_2\text{C} \\ \text{C} \\ \text{C}$$

$$+ O = C = N - CH_2CH_2CH_2 - Si - OEt$$

$$(IPTES) OCH_2CHCH_2 - CHCH_2 - CH$$

$$\xrightarrow[\text{curing}]{\text{e-MHHPA+BDMA}} \xrightarrow[\text{backing}]{\text{Spin coating}} \\ = \text{epoxy} - \text{TiO}_2 \text{ hybrid film}$$

**Fig. 1.** Reaction scheme for preparing epoxy/ ${\rm TiO_2}$  hybrid films.

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