

# Barium titanate dispersion obtained by a high pressure methods and light resistant composites containing the nanoparticles

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To improve the light resistance of the organic-inorganic composite for optical use, barium titanate (BTO) dispersion was prepared instead of titanate ( $\text{TiO}_2$ ) dispersion by a high pressure reaction between titanium oxide ( $\text{TiO}_2$ ) dispersions treated with silane-coupling reagent and barium hydroxide [ $\text{Ba}(\text{OH})_2$ ]. The obtained BTO was observed by transmission electron microscope (TEM) and its diameter was less than 10 nm. This seemed the result from the treatment of the  $\text{TiO}_2$  before the reaction. In addition, X-ray diffraction analysis (XRD) indicated  $\text{TiO}_2$  crystals didn't remain in the BTO. Transparent composites composed of 2-hydroxy-butylacrylate as the organic matrix and the BTO or the  $\text{TiO}_2$  as the inorganic filler were prepared. The composite including the BTO showed much longer decomposition time than that of  $\text{TiO}_2$  in the light (405 nm) resistance test. This indicates that the BTO has lower photocatalytic activity than the  $\text{TiO}_2$ .

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## 1. Introduction

Transparent inorganic-organic composite materials are very attractive for optoelectronics devices.<sup>1)</sup> Especially the composites possessing a high refractive index are applied for optical lens, light extraction layers and anti-refraction layers.<sup>1),2)</sup> Titanium oxide ( $\text{TiO}_2$ ; refractive index = 2.5) is generally used as a part of high refractive composite materials.<sup>3)-6)</sup> However,  $\text{TiO}_2$  should not be applied for inorganic-organic composites since  $\text{TiO}_2$  has photocatalytic activity and destroy the organic components in the composites.<sup>7)</sup> Instead of  $\text{TiO}_2$  materials, barium titanate oxides (hereafter referred to as BTO, refractive index = 2.4) can be used as a component of high index composites because they have a quite low photocatalytic activity.<sup>8)</sup>

BTO nanoparticles are prepared using sol-gel methods<sup>9),10)</sup> and hydrothermal methods.<sup>11)-14)</sup> Kuwabara et al. prepared BTO nanoparticles using a Ba metal and Ti alkoxides from the high-content sol-gel method.<sup>9)</sup> However, transition metal alkoxides are very difficult to use since they are higher moisture sensitive compounds and very expensive. On the other hand, relatively large BTO particles can be prepared easily by the high pressure hydrothermal reaction between  $\text{Ba}(\text{OH})_2$  and  $\text{TiO}_2$ . The prepared BTO was too agglomerated (more than 20 nm) to apply for transparent devices.

In this study, we prepared  $\text{TiO}_2$  covered with a silane-coupling reagent as a covering material for affinity to the organic matrix of the composite before the high pressure reaction. And the reaction was carried in a heterogeneous environment (so we call this method improved high pressure method hereafter referred to as

“IHM” in distinction from a traditional aqueous hydrothermal method hereafter referred to as “AHM”). As a result, the transparent dispersion of BTO (hereafter referred to as “IHM-BTO”) was obtained. The diameter of the IHM-BTO particle observed by transmission electron microscope (TEM) was less than 10 nm. At this time, we could prepare the sample for the initial objective; transparent BTO-organic composite as an optical material sample that should have high refractive index and a light resistance against 405 nm laser. Due to the process of IHM, the IHM-BTO particle already had a dispersibility in the organic matrix. So we just mixed IHM-BTO and the matrix. The obtained composite had been improved on light resistance compared with  $\text{TiO}_2$ -organic composite.

## 2. Experimental procedure

### 2.1 Materials

Titania sol; SAD01-W (diameter: 5 nm, 15 wt %, specific gravity = 1.1) was obtained from Sakai Chemical Industry Co., Ltd. Barium Hydroxide Octahydrate  $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$  was obtained from Wako Pure Chemical Industries, Ltd. Silane-coupling reagent; 3,3,3-Trifluoropropyltrimethoxysilane (hereafter referred to as “FAS-3”) was obtained from Tokyo Chemical Industry Co., Ltd. Distilled water, aqueous ammonia (28%), methanol, ethanol, acetic acid and methyl isobutyl ketone (hereafter referred to as MIBK) were obtained from Junsei Chemical Co., Ltd. Aqueous ammonia (28%) was diluted by the same volume of ethanol. This solution is hereafter referred to as “ $\text{NH}_4/\text{EtOH}$ ”. The acrylic resin; 2-hydroxybutylacrylate for the organic matrix of the composite and the photoinitiator; 1-hydroxycyclohexylphenylketone were obtained from Tokyo Chemical Industry Co., Ltd.

### 2.2 Aqueous hydrothermal method (AHM)

SAD01-W was measured by a micro pipette to involve 200 mg as  $\text{TiO}_2$  particles and diluted by the distilled water up to 20 ml in

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