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The novel optical diffusers based on the fillers of boehmite hollow microspheres

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ABSTRACT

The novel optical diffusers based on boehmite (γ -AlOOH) hollow microspheres were successfully prepared by solvent-free UV curing process. The optical properties of the novel diffusers were also measured for the first time. Boehmite hollow microspheres had been synthesized via high efficient and facile hydrothermal synthesis method. The microscope analysis revealed that the boehmite hollow microspheres were about 5 μ m in diameter and with a shell thickness of approximately 1.6 μ m. The novel diffuser based on the fillers of boehmite hollow microspheres possessed suitable light transmittance, good diffusion capacity, and low incident angle dependence, which were critical and necessary for excellent optical diffusers. Thus, boehmite hollow microspheres were quite suited to being as optical fillers and such microspheres can be widely used for preparing different multifunctional diffusion materials such as touch-panel functions, monitors, military projectors, night vision LCD, etc.

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1. Introduction

The optical diffuser is one of the most important materials in the liquid crystal display (LCD) industry. Recently, many researchers have made a great deal of effort to obtain a good back-lighting unit (BLU) in LCD screens. The optimal BLU has a good luminance uniformity, wide scattering angle, well light transmittance, high luminance, and great thermal stability, which requires that the optical diffuser should have wide scattering angle, low incident angle dependence, high transmittance and haze, and good heat durability [1–3]. As well known, there are currently two types of optical diffusers that achieve sufficient optical diffusion for LCD screens [4]. One is called volumetric diffusers which mainly are made by adding the fillers to films and the fillers cause the uniformly inside the plates to scatter light [5–7]. The other is called surface-relief diffusers, which depend on the surface structure to scatter light, such as rough surfaces, pyramids, microlenses, and other microstructures [8–10].

Optical diffusing particles can be divided into two types, which are organic particles and inorganic particles. Nevertheless, the thermal stability is very low when using organic fillers, and the agglomeration problem is very serious when using inorganic fillers. Hence, well-controlled methods for fabricating good optical diffusers are still very much expectable. Among the many target

http://dx.doi.org/10.1016/j.matlet.2014.08.018 0167-577X/© 2014 Elsevier B.V. All rights reserved. materials, hollow spheres with nanometer or micrometer size have been especially interesting in the recent years due to their special physical and chemical properties different from solid particles, owing to their low density, larger specific surface area, hollow structure and nanostructured wall, as well as their potential applications of these materials for example in catalysis, controlled delivery, artificial cells and light-weight fillers [11–13].

Herein, we first reported that boehmite hollow microspheres can be used as optical diffusing fillers of diffusers, and the optical properties of the novel diffusers were also measured. Compared with conventional optical diffuser, the novel diffusers based on the fillers of boehmite hollow microspheres combined the advantages of volumetric diffusers and surface-relief diffusers for the first time. Boehmite hollow microspheres were quite suited to being as optical fillers because of good light transmittance and diffusion capacity. Thus, we all believed that boehmite hollow microspheres also have a potential to fabricate diffusion materials, especially optical diffusers.

2. Experimental procedures

In a typical synthesis, $Na_2C_4H_4O_6 \cdot 2H_2O$ (0.0003 mol), $CO(NH_2)_2$ (0.007 mol), and $Al(NO_3)_3 \cdot 9H_2O$ (0.0035 mol) powders were dissolved in 70 ml of distilled water and stirred for 15 min. The solution was placed in a 100 ml autoclave with a Teflon liner. The autoclave was maintained at 150 °C for 3 h and then air cooled





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to room temperature. After reaction, the precipitate was filtered, washed three times with distilled water and one time with anhydrous alcohol, and finally dried in a vacuum oven at 80 °C for 6 h. Took down a few of the sample, and then calcined at 400 °C for 2 h.

Subsequently, the complex films were prepared by solvent-free UV curing process. 5 g Tri(propylene glycol) diacrylate, 0.4 g photoinitiator 184, and 15 g polyurethane acrylate were mixed in dark space. The concentration of relative particles with 2–10 wt% were added to the above. The film samples were obtained by double-sided coating in 50 μ m PET. Then, the film was placed in 100 w UV lamp (365 nm) for 10 min each side. Finally, it was placed in an oven at 60 °C for 24 h. The optical diffuser for comparison derived from Dong Xucheng Chemical Co., Ltd. (DXC Diffusion 50-BDN).

The structure and morphology of the multifunctional microspheres were studied by X-ray diffractometer(XRD) equipped with CuKa radiation, powder transmission electron microscopy (TEM) and Scanning Electron Microscopy (SEM). The transmittance haze meter (WGT-S) was used for measuring the properties of optical diffusers. Optical diffusing properties were measured by light intensity distribution measuring instrument (HD-4960-LED).

3. Results and discussion

The X-ray diffraction (XRD) patterns of hydrothermal product and the dispersion property were shown in Fig. 1. Diffraction peaks corresponding to boehmite (JCPDS 21-1307) had been found for the product. No obvious XRD peaks arising from other phases of alumina were found indicating pure γ -AlOOH phase in the hydrothermal product. In addition, the image of 5 wt% boehmite dispersed in ethanol was presented in Fig. 1 (right). It displayed that the product possessed good dispersion and there was no aggregation problem.

Fig. 2(a), (b) and (d) presented the SEM and TEM images of the γ -AlOOH sample obtained after hydrothermal synthesis. As shown in Fig. 2(a), (b) and (d), template-free hydrothermal fabrication of organized γ -AlOOH microspheres can be observed. Approximately 5–8 μ m microspheres were displayed, and the scales of them were suitable for optical diffusing materials when the thickness of coating film was in the range from 8 to 24 μ m. Those can assured optimal diffusing effect according to the design rule reported

[14,15]. To investigate the internal structure of the microspheres, the calcined sample was measured by SEM (shown in Fig. 2(c)). As can be seen in the red circle of Fig. 2(c), the broken hollow shells obviously reveal the hollow structure of the microspheres. The microscope analysis revealed that the boehmite hollow microspheres were about 5 μ m in diameter and with a shell thickness of approximately 1.6 μ m. The hollow structure caused the difference of refractive index difference between shell and hole. It meant that the optical diffuser containing γ -AlOOH microspheres existed three kinds of media with different refractive index. Thus, the novel diffusers based on the fillers of boehmite hollow microspheres combined the effects of volumetric diffusers and surface-relief diffusers.

The novel optical diffusers were examined to understand the effect of diffusing ability for the concentration of diffusing fillers. The quantity of boehmite microspheres was modulated, including 2, 5, 8 and 10 wt%. A He–Ne laser with 630 nm wavelength was used to illuminate directly on diffusers and the optical pattern was recorded by digital camera (shown in Fig. 3). It was clear that diffusion range gradually increased with the concentration of boehmite microspheres increasing. Moreover, the prepared diffusers had good visible light transmittance. When the concentration of boehmite microspheres was up to 10 wt%, the diffuser possessed super light diffusing effect, similar to optical diffusers on market.

The transmittance and haze of novel diffuser with different concentration of boehmite hollow microspheres were measured by the transmittance haze meter. Fig. 4(a) showed the transmittance and haze of the diffusers. The transmittance of the diffusers slightly decreased with the concentration of boehmite microsphere increasing while the haze increased. When the concentration of boehmite hollow microspheres was up to 10 wt%, the haze of novel diffusers can exceed the optical diffusers on market. Fig. 4(b) showed the relationship between incident angle and scattering angle based on diffusing films with different concentration fillers. It can be observed that the scattering angle, also named as scattering range, increased with the concentration of boehmite hollow microspheres increasing. Due to multi-scattering derived from boehmite hollow microspheres structure, the novel diffuser presented low incident angle dependence. In other words, all different angles of incident light can be scattering through the novel diffuser when the concentration of novel diffusing fillers was certain value. When the concentration of boehmite hollow



Fig. 1. XRD patterns of boehmite sample (left) and the image of 5 wt% boehmite dispersed in ethanol (right).

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