Evaluation of curing process of UV resin in PFP gas ambient by photo-differential scanning calorimetry

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UV-nanoimprinting in 1,1,1,3,3-pentafluoropropane (PFP) gas ambient is proposed to eliminate the bubble defects¹⁾. In this study, we evaluated the curing processes of synthesized radical- and cationic-UV-curable resins that the monomers were disclosed in PFP gas ambient by photo-differential scanning calorimetry (photo-DSC).

We used C-TGC-02 (radical polymerization type; Toyo Gosei CO., LTD.) and NICT103 (cationic polymerization type; Daicel Chemical Industries, LTD.). Figures 1(a) and 1(b) show the monomer structures of C-TGC-02 and NICT103, respectively. 365 nm-UV intensity and irradiation time were 2 mW/cm² and 10 min. The sample weight was 10 mg. We carried out photo-DSC in air and PFP gas ambient and compared the measurement results in air with those in PFP gas ambient.

Figures 2(a) and 2(b) show the photo-DSC results of C-TGC-02 and NICT103 in air and PFP gas ambient, respectively. In this experiment, we defined the time from UV irradiation start to exothermic peak as the curing time and height of exothermic peak as the calorific value. As the results, although the curing times in PFP gas ambient were almost the same values as those in air, the calorific values in PFP gas ambient were higher than those in air. These results indicate that the UV-curable resins were efficiently polymerized in PFP gas ambient. However, we found that the photo-DSC results depended on UV intensity. We therefore examined the UV intensity dependence of the curing time and calorific value in air and PFP gas ambient.

Figures 3(a) and 3(b) plot the UV intensity dependence of curing times of C-TGC-02 and NICT103 in air and PFP gas ambient, respectively. When the UV intensity was 0.2 mW/cm^2 , the curing time in PFP gas was very faster than that in air. However, in the case of other intensity, the curing times in PFP gas ambient were almost the same values as those in air. On the other hand, we confirmed the UV intensity dependence of calorific value as shown in Fig. 4. In the case of the UV intensity under 20 mW/cm², the calorific values of C-TGC-02 in PFP gas ambient were higher than those in air as shown in Fig. 4(a). However, when the UV intensities were over about 20 mW/cm², the calorific values in PFP gas ambient were lower than those in air. The measurement results of NICT103 also showed the same tendency as shown in Fig. 4(b). These results indicate that the UV-curable resins were efficiently polymerized in PFP gas ambient compared to air when the UV intensity was low. However, in the case of the high UV intensity, the calorific value in air was higher than that in PFP gas ambient.

In the presentation, we will present the relationship between the calorific value and the polymerization degree measured by Fourier transform infrared spectroscopy.

1) H. Hiroshima and M. Komuro: Jpn. J. Appl. Phys. 46 (2007) 6391.







Fig.2 Photo-DSC results of (a) C-TGC-02 and (b) NICT103.



Fig.3 Relationship between UV intensity and curing time of (a) C-TGC-02 and (b) NICT103.



Fig.4 Relationship between UV intensity and calorific value of (a) C-TGC-02 and (b) NICT103.