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# Effect of substrate reflecting conditions on the curing of UV curable resin layers on aluminum and the formation of surface wavy structures

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## ABSTRACT

We have investigated the formation of surface wavy structures (wrinkles) depending on the diverse surface conditions of a substrate, and now report the effect of various levels of reflection of incident ultraviolet (UV)-light. UV-curable resin layers with a thickness of 0.15 mm were coated on three different surface conditions; transparent glass, fine and rough surface aluminum plate, to compare the formation of wrinkles on each surface. Short irradiations of UV-light for 10, 15, 20, 25 and 30 s were exposed, resulting in the weak to full polymerization of the skin of a resin layer, respectively. The wavy structures were formed during thermal curing under room temperature after the short exposure of UV-light. The difference in reflection conditions resulting from the various surface roughnesses of the substrates led to changes in the amounts of polymerization, and the distribution of polymerization intensity through the layer thickness. Due to these different mechanisms, wrinkling shapes were quite distinguished. Through this work, we observed that controllable producing an approximately 33% maximum difference in the line width of wrinkles by using different substrate surface conditions.

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## 1. Introduction

Surface structures with a wavy morphology are easily found in nature. Wrinkled structures have also been applied in various technological fields; for instance, to enhance wettability [1,2], for surface patterning [3], tissue engineering [4], diffraction gratings [5], photonic applications [6], flexible electronics [7,8] and surface modification [9]. Until now, many research works have focused on methods which fabricate wrinkles on a soft substrate by controlling pre-stress and pre-strain. For example, Watanabe formed wrinkles on a thin gold film deposited onto various elastic polymer substrates by using compression and stretching [10]. Chen et al. fabricated herringbone buckling patterns on compliant substrates by exploiting the large thermal expansion mismatch between the substrates and deposited film [11]. Ni et al. indicated the origin of such kinds of branched wrinkles and provided a guideline for controllable hierarchical wrinkles by patterning the stiffness gradient [12]. Huck et al. described the spontaneous formation of wrinkle patterns comprised of aligned buckles in a thin film of gold deposited on the elastomer polydimethylsiloxane (PDMS) [13]. In addition, Ohzono et al. proposed that wrinkle patterns on PDMS were controllably ordered by compressive strain [14]. Kim

et al. proposed a simple method for generating hierarchical wrinkles via weak photopolymerization followed by thermal curing [15]. Zhao et al. investigated an effective way of generating various wrinkle shapes by varying the layer thickness of a photocurable resin [16]. Park et al. proposed a step-and-repeat method to form large-area wrinkle patterns by a repetitive volume dividing (RVD) process [17]. Yu et al. revealed the thickness gradient-guided spontaneous formation of ordered wrinkling patterns in metal films deposited on soft elastic substrates [18]. Recently, Li et al. created microarchitecture for a three-dimensional wrinkled surface platform to finish cell culture [19]. Kim et al. generated the wrinkles and deep folds that form on polymer surfaces when subjected to mechanical stress, to guide and retain light within the photo-active regions of photovoltaics [20]. Chung et al. examined the wettability of rough surfaces through a measurement approach that harnessed a wrinkling instability to produce model substrate topographies [21]. And recently, some other reports related to wrinkle generations and applications have been introduced [22–29].

However, in terms of manufacturing technology, finding a simple and low cost process to fabricate wrinkles remains an important issue, and thus we have proposed continuative processes such as weak-polymerization and thermal curing in order to realize microscale wrinkles in a large area. In this letter, as further extension of the previous works, we investigated the correlation between the surface conditions of a substrate and wrinkle

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formation to determine the most effective range. For this, three different UV-reflection conditions were utilized to compare the generated wrinkle shapes on each of three types of plate: a transparent glass plate, finely uneven and rough opaque aluminum plates. We believe that the effects produced by the surface conditions of a substrate can be utilized as a simple and easy way of generating diverse microscale wrinkles.

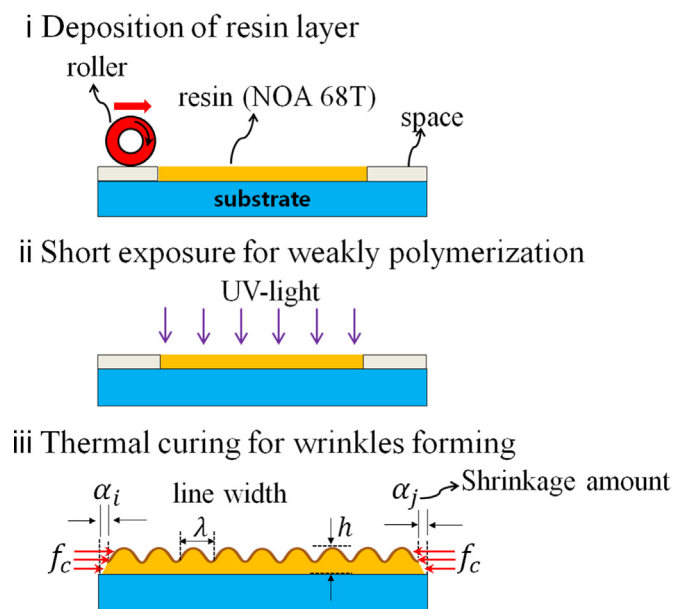
## 2. Experiments

### 2.1. Materials

Different substrate surface conditions were prepared to evaluate how the surfaces affected wrinkle formation. Transparent glass, finely uneven and rough aluminum plates having different surface roughnesses (Ra) of 0.139, 0.154, and 0.234  $\mu\text{m}$ , respectively, were used. An UV curable resin, NOA-68T (Norland Co., Germany), was used to generate microscale wrinkles. The resin's viscosity is between 20,000 and 25,000 cps at room temperature (25–28  $^{\circ}\text{C}$ ), and its UV absorption is highest at a wavelength between 350 and 380 nm. We determined theoretically that a UV lighter with a power of 400  $\text{mW}/\text{cm}^2$  made the resin polymerize during irradiations of 10–30 s.

### 2.2. Methodology

A film of the resin (NOA-68T) with a thickness of 0.15 mm was uniformly coated on the surface of each substrate by a roller as shown Fig. 1-i. The film was exposed to a short irradiation of UV-light to produce somewhat strong polymerization on the top surface of the resin (see Fig. 1-ii). After that, as shown in Fig. 1-iii, a thermal curing process was conducted in a chamber at temperature of about 30  $^{\circ}\text{C}$ . During thermal curing, the resin layer exhibited a partial gradient shrinkage depending on the intensity of polymerization of the film's interior, which was done by the UV-exposure. This state resulted in shrinkage of the volume of the resin layer as shown Fig. 1-iii ( $\alpha_i$  and  $\alpha_j$  are the shrinkage amount at both sides). Simultaneously, compressive force ( $f_c$ ) caused by the volume shrinkage generated a pattern of wrinkles on the



**Fig. 1.** Fabrication process of wrinkle patterns (i) uniform deposition of resin layer by a roller; (ii) short exposure for weak polymerization; (iii) thermal curing of wrinkles formed at room temperature.

surface of the resin layer (see Fig. 1-iii). Here,  $\lambda$  is the line width of the wave structure;  $h$  is the height of the resin layer.

The mechanism of Wrinkling was affected by the surface roughness of a substrate, and the mechanisms for different surface roughnesses are shown in Fig. 2a and b. The surface roughness of each substrate was measured using atomic force microscopy (AFM) are shown in Fig. 2a.

Generally, a surface under irradiation by light can reflect light rays, and the amount of reflected light can be modified by the nature of the surface. The surface feature of transparent glass used in this work, as shown in Fig. 2a-①, allowed the irradiated dose of UV-light to pass through the glass, leading to a reduced absorption of light. In contrast, the fine uneven surface of the opaque aluminum plate could absorb more energy under UV-light irradiation due to specular and partially scattered reflection (see Fig. 2a-②). Similarly, the rough surface of the opaque aluminum plate produced fully scattered reflection (see Fig. 2a-③). As a consequence, when the UV-light exposure time was 10–15 s, only weak polymerization appears on the skin resin layer on the transparent glass due to the relatively weak absorption of light, and the intensity of polymerization gradually weakens with thickness of resin (Fig. 2b-①). With increasing irradiation time, the intensity of polymerization was increased, but the amount ( $\delta$ ) of difference of polymerization along thickness is almost equivalent  $d$  (Fig. 2b-①,  $\delta_1 \cong \delta_2 \cong \delta_3$ ). As a result, the line width ( $\lambda$ ) of the wrinkle pattern generated on the glass substrate looks almost same (see Fig. 3a-d). When the resin layer on the fine aluminum substrate was irradiated for 10–15 s, weakly polymerized region was similar to that of the transparent glass substrate, but there was somewhat difference in the intensity of polymerization. Thus, the line widths of wrinkle patterns are almost identical, due to the similar gradient of polymerization. However, we believe, when the UV-exposure exceeded 20 s, strongly harden appeared on the skin of the resin layer, while the interior was still in a weakly polymerized soft state (sol-gel state, it means that the strength difference between skin and inner side becomes larger). Therefore, the line widths of the wrinkles became decreased with the greater the amount of gradient (see Fig. 2b-②  $\delta_1 < \delta_2 < \delta_3$ ). In addition, the mechanism for the rough aluminum substrate shows a similar phenomenon depicted in Fig. 2b-③; one thing difference is the degree of absorption of light. So, the intensity of polymerization on the resin layer can appear different change due to much higher light absorption ( $I_p < I'_p < I''_p$ ).

## 3. Results and discussion

As described in the previous section, the shape and dimensions of generated wrinkles can vary depending on the different surface roughness of the substrate because of the effect it has on the volume shrinkage of the resin layer during the thermal curing process. In this paper, we chose three substrates with distinctive characteristics to study; a transparent glass plate, fine uneven aluminum plate and rough aluminum plate, respectively. In order to precisely investigate the effect of different surface roughness on the difference of wrinkles shape on the surface of the resin, we applied UV-light exposures for 10, 15, 20, 25 and 30 s to a resin layer having a thickness of 0.15 mm, and observed the wrinkles' shapes (see Fig. 3a-l) using an optical microscope (ECLIPSE MA200, Nikon, Japan). Following UV-exposures of 10–25 s, wrinkles were generated after about 30 min of thermal curing, as shown Fig. 3a-l (see 10 times magnified images). However, after UV-exposure for 30 s, no wrinkle patterns were generated due to the full polymerization of resin layer. The average widths of wrinkles on the transparent glass substrates were 80.5  $\mu\text{m}$ , 78.1  $\mu\text{m}$ , 77.9  $\mu\text{m}$  and 76.9  $\mu\text{m}$  following UV-exposure for 10–25 s,

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