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Morphology and surface characteristic control of dimpled wrinkles using microscale water-soluble powders [†]

Sung-Woo Choi[1] and Sang-Hu Park[2,*]

[1]*Graduate School of Mechanical Engineering, Pusan National University, Busandaehak-ro 63beon-gil, Geumjeong-gu, Busan 46241, Korea*
[2]*School of Mechanical Engineering and ERC/NSDM, Pusan National University, Busandaehak-ro 63beon-gil, Geumjeong-gu, Busan 46241, Korea*

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Abstract

We proposed a novel method for the morphological shape change of dimpled wrinkles using water-soluble powders to modify surface characteristics. Ultraviolet (UV)-curable resin was coated thinly on an aluminum plate and three water powders with 70, 100 and 200 μm in average diameters were dredged on each resin-layered plate. After a short exposure in UV light for less than 15 s on the plate and subsequent thermal curing, we observed microscale wrinkles with diverse morphological structures. Next, dimpled wavy structures were observed by dissolving water-soluble powders on the skin using water rinsing. Through experimental results, wrinkle morphology was affected by the powder size and resin layer thickness. Using this simple approach, various types of dimpled wavy structures can be realized and may be utilized in engineering applications, such as hydrophobicity control.

Keywords: Microscale wrinkle; Weak polymerization; Thermal curing; Surface modification; Dimpled wrinkle; Water-soluble powder

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1. Introduction

Microscale surface structures or wrinkles have various forms and have specific functions in animals and plants. For example, the microscale skin structure of a shark generates a vortex to reduce fluid resistance, and the surface of the lotus leaf with fine protrusions is hydrophobic to avoid being wet from water drops [1-3]. Furthermore, wrinkles are being used for surface treatments in industrial fields, such as aerospace and shipbuilding [4]. These naturally inspired mimetic structures have been artificially fabricated to be used in engineering fields, such as heat transfer enhancement, micro-flow control, superhydrophobic reactions, and reactions with contact material [5-11]. Moisture control in the forest is essential for the reproductive cycle of mosses, which absorb water droplets like sponges using numerous dimples on their stems; moreover, the absorbed water is expelled from the stems to maintain ambient humidity [12].

So far, studies have studied the fabrication of microscale wrinkles and their applications by using different strains on surface-induced in-plane tension [13], contraction and expansion by water absorption in polymer films [14], repetitive contact and separation processes [15], weak polymerization and thermal curing [16], out-of-stretching method for directional wrinkles [17], irradiation of ion beams on poly-dimethylsiloxane [18], wrinkle shape changes by resin layer thickness control [19], solvent-assisted swelling and subsequent drying process [20, 21], hierarchical wavy structuring by post-processing [22], and many other methods [23-25]. Moreover, theoretical studies have been reported to predict wrinkle shapes depending on the process parameters [26-28]. Despite these numerous works, advances on fabrication still face issues. For example, a simple and effective way to fabricate wrinkles with diverse and controllable shapes is still considered as an important research topic. Most current studies have been limited to making a single wrinkle shape. For improved functionality, the shape of hierarchically dimpled wrinkles is required to mimic the surface of moss stems.

This work focused on the fabrication of a surface wrinkle with microscale dimples on its surface to allow large area fabrication and analyze the surface properties. To this end, we proposed a method of producing wrinkles with dimples by sprinkling 70, 100 and 200 μm water-soluble microscale powders on UV-curable resin layers and adjusting the shape and size of wrinkles through weak polymerization and thermal curing.

2. Experiments

2.1 Materials

The material used for fabricating the dimpled wrinkles is

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[*] Corresponding author. Tel.: +82 51 510 1011, Fax.: +82 51 514 0685
E-mail address: sanghu@pusan.ac.kr

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NOA68T (Norland Co. Germany) resin, which has a high affinity with other materials and is widely used as a photo-curable adhesive. This resin has a viscosity of 20000-25000 cps at 25 °C and is cured completely in UV conditions of about 4.5 J/cm^2. In addition, it absorbs the UV in the wavelength range of 350-380 nm. Full curing reaction occurs when UV light with output of 400 W/cm^2 is irradiated for 30 s. Moreover, in this study, we used grinded sugar powders as water-soluble microscale powders.

2.2 Fabrication process

To understand the formation of wrinkle structure characteristics depending on the size of the water-soluble microscale powder, we fabricated four types of water-soluble powders after classifying their sizes into 70, 100 and 200 μm in diameter using several plain meshes. To produce a surface wrinkle structure, we uniformly coated NOA68T resin on the aluminum sheet, as shown in Fig. 1, with an area of 50 × 50 mm^2 and resin layer thickness of 0.05 and 0.1 mm. Next, we sprinkled the water-soluble microscale powders that were classified in size uniformly on the resin layer at a proportion of 0.004 g per in^2 using a specially designed container. After the weak photo-curing on the resin layer by UV light irradiation for 15 s, we fabricated the wavy structures through thermal curing for 10 h at 30 °C.

Through short UV light exposure, the local material density of the resin layer changed along the layer thickness, that is, the skin of the resin layer became weakly solidified by UV exposure, but the inside of the layer remained as a sol-gel or in the liquid state due to insufficient UV exposure for full polymerization. Thus, wrinkles were created on the skin during thermal curing. Finally, to create dimples on the fabricated wrinkling surface, we dissolved the water-soluble microscale powder embedded in the surface of wrinkle structure in hot water at 50 °C. By using an optical microscope at contact angle of a water droplet and 3D shape-measuring apparatus, we analyzed the role of the water-soluble powder size in shaping wrinkles and observed the properties of the fabricated dimpled surface.

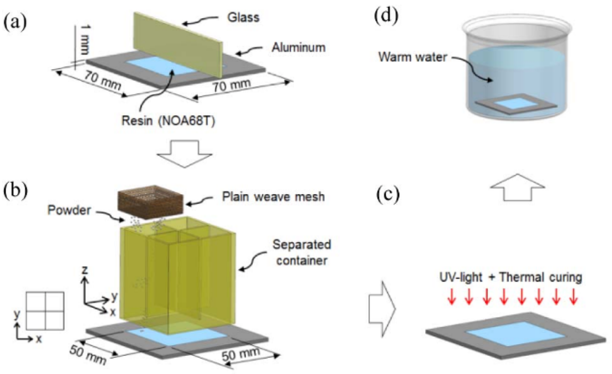


Fig. 1. Schematic diagram of fabrication process for dimpled wrinkles.

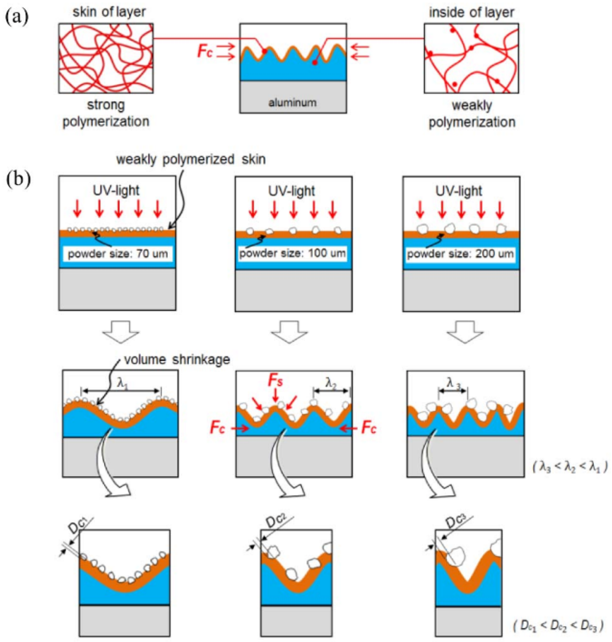


Fig. 2. Schematic diagram of wrinkling mechanism: (a) Wrinkling mechanism on a single layer; (b) line width of dimpled surface wrinkling mechanism. Compressive force (*Fc*) and surficial compressive force (*Fs*) are generated in volume and on surface during thermal curing.

3. Results and discussion

The mechanism that created wrinkles by polymerization and thermal curing is as follows: As shown in Fig. 2(a), the resin layer surface is directly exposed to UV light through weak polymerization and caused a relatively more active polymerization than the inside. Thus, we can find that a weak polymerization causes the difference in material properties in the direction of the thickness of the resin layer. The resin layer was cured fully under the thermal curing process, but it contracted as a whole volume and caused *Fc* and *Fs* in the skin zone to form wrinkles.

We analyzed the effect of the water-soluble powder sizes in the formation of morphological wrinkle structures. Fig. 2(b) shows the characteristic shape change for the wrinkle periodic interval (λ) in the powder-embedded resin layer depending on the size of the powder. When we indicated the wrinkle interval as λ_1, λ_2 and λ_3, according to the powder sizes of 70, 100 and 200 μm, respectively, λ was larger when the size of the water-soluble microscale powder was smaller: $\lambda_3 < \lambda_2 < \lambda_1$. When the powder increased, the contact area and contact depth (*Dc*) between resin layer and single powder increased. However, if the total weight of the powder sprinkled on the surface is the same amount, then the total contact area between them increases because of the total number of powder sprinkled. Assuming that the powder is spherical and the total weight is the same, the relation formula of $n_1 D_1^3 = n_2 D_2^3$ can be obtained, where *n* and *D* indicate the number of the total powder and powder diameter, respectively. In this case, the number of the 70 μm powder was approximately 23.3 times higher than the

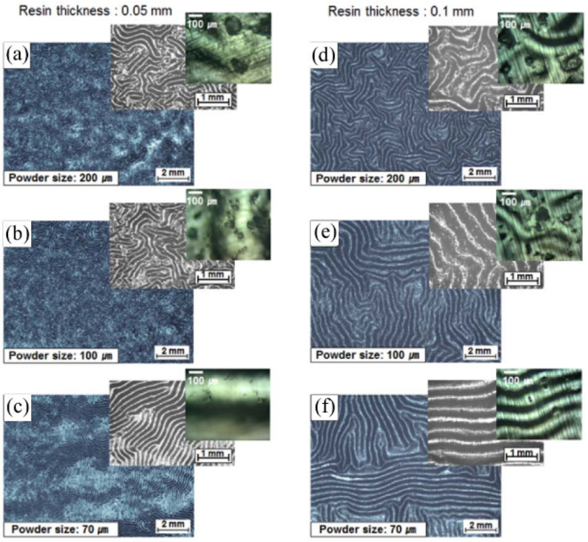


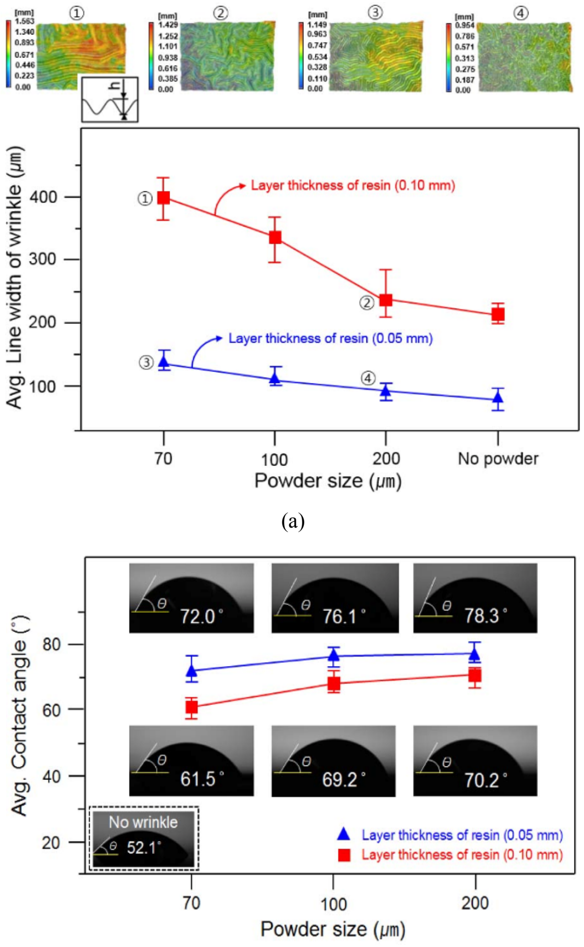
Fig. 3. Wrinkle image on dimpled surface depending on the powder sizes.

that of the 200 µm powder. Therefore, the small-sized powders were distributed evenly and densely on the skin, thereby increasing the total contact area with the resin layer, which caused the high rigidity of the skin by tightly combining resin and powders during UV exposure. However, the wrinkle interval was known to be proportional to the rigidity difference between the skin and the foundation (inside layer), as shown in the Eq. (1) [21]:

$$\lambda \propto \left(E_S / E_f\right)^{1/3} . \tag{1}$$

E_S and E_f indicate the elastic modulus of the skin and the foundation, respectively. Thus, the morphological shape of wrinkles can be controlled by applying different powder sizes.

Dimpled wrinkling shapes, which were generated with different resin layer thicknesses (0.05 and 0.1 mm) and water-soluble powder with various sizes (70, 100 and 200 µm), are shown in Figs. 3(a)-(f). From the experimental results, the smaller the microscale water-soluble powder, the larger the wrinkle interval becomes; moreover, the resin layer with thickness of 0.1 mm allowed λ to be larger than the resin layer thickness of 0.05 mm. As mentioned, the wrinkle shape can be changed by scattering different sizes of powder on the resin surface. In addition, the overall wrinkle interval was narrow when powders with large sizes were embedded and when the resin thickness was 0.05 mm. The dependence of the resin layer thickness on wrinkle formation has already been reported [22]. However, the average λ changed considerably with the scattered powder size and resin thickness of 0.1 mm, compared with that of 0.05 mm. Figs. 3(a)-(c) show the dependence of the dimple size on the wrinkling surface, scattered powder size, and variation of dimple diameters of 30, 50 and 100 µm, with powder sizes of 70, 100 and 200 µm.



(a)

(b)

Fig. 4. (a) Line width of wrinkles according to layer thickness of resin (NOA68T) and 3D view; (b) contact angles according to layer thickness of resin and powder sizes.

Another interesting point is the position of dimples. By comparing the positions of the dimple, we found that the dimples mainly existed in the valley instead of the ridge of the wrinkle when the powder size was 200 µm (see Figs. 3(a) and (d)) and randomly distributed dimples in the other two cases of powder sizes of 70 and 100 µm. A certain rule exists for the formation of wrinkles affected by the scattered powder size. Therefore, the wrinkling shape can be changed by regularly positioned powders with critical sizes on a resin layer; however, further studies are required to understand clearly the regulation of wrinkle formation related with initially scattered particles.

Figs. 4(a) and (b) show the variation of wrinkle formation depending on powder sizes and the changes of contact angle that appear on the dimpled wrinkle surface. As depicted in Fig. 4(a), λ varied according to the resin thickness and the sprinkled powder size. The wrinkle shapes, as shown in the top images of the figure, were measured by 3D shape-measuring apparatus (VHX500, Keyence Co. Japan). When the resin thickness was 0.05 mm, the average λ of each wrinkling structure was 132, 124 and 110 μm with powder size of 70, 100 and 200 μm, respectively. In addition, if the resin thickness was 0.1 mm when the powder size was 70, 100 and 200 μm, then the average value of λ changed to 399, 338 and 235 μm, respectively. Without the powder, the average λ of each wrinkle was measured as 90 and 207 μm according to the layer thickness of 0.05 and 0.1 mm, respectively. The sample sizes of wrinkling plate were equal: 24 × 24 mm. This result shows that the dimpled wrinkle interval tends to become narrower with the increase of the powder size, and the height of the wrinkle ("h" in Fig. 4(a)) was in the range of 0.95-1.15 mm with a resin thickness of 0.05 mm and 1.43-1.56 mm with a resin thickness of 0.1 mm. The height of the wrinkle with a resin thickness of 0.1 mm was larger by about 36 %-49 % than that of the wrinkle with a resin thickness of 0.05 mm.

Fig. 4(b) shows the change in contact angle of the dimpled wrinkle. The contact angle varied from 61.5°-78.3° according to the powder size and layer thickness. Compared with the contact angle of the flat surface with no wrinkle, the maximal contact angle increased to 50.3 %. However, the contact angle has a relationship with the wrinkle interval, and the smallest λ, 110 μm, made the highest contact angle. We believe that the dimples contributed in increasing the contact angle due to the change in surface roughness. Therefore, we can make a conclusion that the contact angle increases as the interval of dimpled wrinkle becomes narrower, and the porosity generated on the surface increases as the water-soluble powder increases.

4. Conclusions

In this study, we proposed a process of producing a dimpled wrinkle by varying the size of the water-soluble microscale powder and the thickness of the resin and used sugar as a solid yet water-soluble microscale powder to produce various sizes of porosity on the surface wrinkle structure. By reducing the powder size on the surface wrinkles to observe the change in the surface properties, we found that the wrinkle intervals tended to increase. This result showed that the wrinkle interval (λ) is proportional to the rigidity difference between the skin and the foundation, as shown in Eq. (1) as smaller powders can be distributed more evenly than larger ones on the skin of the resin to increase surface rigidity. In addition, this tendency appeared more significantly in the resin with thickness of 0.1 mm rather than that of 0.05 mm due to the increased rigidity of the skin.

Based on the resin thickness and the powder size during the contact angle-measuring test, the contact angle was larger when the average λ on the wrinkle surface was decreased and the porosity was increased. The contact angle increased due to the surface tension between water droplets and surface on the surface wrinkle having a double structure. The maximum height of the wrinkle in the 3D shape measurement was affected more by the resin thickness than by the powder size, and the height of surface wrinkle tended to increase in proportion to the resin thickness. We considered that a low-cost dimpled wrinkle applicable to large areas can be fabricated by using water-soluble microscale powder. We will continue to utilize and study it in the surface treatment field requiring heat transfer enhancement and surface modification.

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Sung-Woo Choi is a Ph.D. candidate student in Department of Mechanical Engineering of Pusan National University. Present research field is development of wrinkling processes and their applications. He is also presently a Researcher in Human Resources Development Service of Korea.

Sang-Hu Park is a Professor in the School of Mechanical Engineering (ME), Pusan National University. His research fields are nano/microfabrication, sheet metal forming, and optimal structure design. And he is a member of KSME, KSPE, and KPEA. Also, he is a Director in Division of Precision Manufacturing Systems of ME.