Drying-Induced Variation in Pressure inside Packed Colloidal Films and Subsequent Formation of Periodic Cracks.

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Drying is indispensable for coating. All wet film has to be dried in production process. However, many undesired defects in films, such as formation of cracks, uneven thickness and roughened surface, are frequently observed in drying step. Especially in the formation of colloidal films from suspension, crack formation due to drying is one of the crucial problems. No crack is allowed in these films because even one crack severely reduces the quality of films. Cracks are caused by large stresses which originate from drying of solvent. In general, colloidal particles are condensed and packed due to solvent evaporation. When pores among packed particles are still filled with solvent, formation of cracks does not occur. On the other hand, when solvent liquid is extracted from these pores due to further evaporation of solvent, cracks form. Capillary force, which originates from small meniscus between particles, is proposed as an important factor for crack formation[1,2]. In practical processes, binders are frequently used to avoid cracks, however, fundamental mechanism of crack formation is not well-understood yet. We consider that study on drying-induced cracks in colloidal films is still necessary for further understanding of crack formation phenomena.

2. Experimental[3]

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Colloidal silica particles dispersed in water was used for film formation. The average diameter of silica colloids is ca. 10 nm (Snowtex OS, Nissan Chemical, Japan). For particulate film formation, we used a handmade small glass chip as shown in Figure 1. Thin silicone rubber was used as a spacer. The thickness of the spacer was 100 μm. A set of rubber spacers, which were ca. 1 mm in width and ca. 35 mm in length, were put on a cut slide glass (ca. 40 × 26 mm^2). The distance between two spacers was set to be 5 mm. A cut cover glass was put on the spacers in order to create a narrow space with two open ends. Because of good adhesion between the silicone rubber and glass surface, we do not have to use any glue to make glass chip.

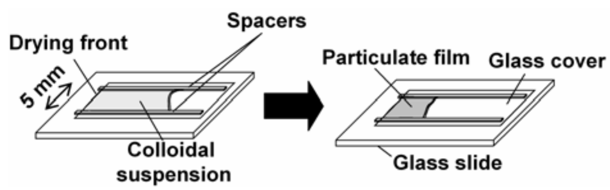

Figure 1. Schematic illustration of handmade glass chip used in our study.

Glass chip was put on a temperature control stage. After setting the glass chip, a slight amount of colloidal suspension was poured into the chip from one open end so that we can keep the air-liquid meniscus ca. 10 mm away from the other open side[1]. Because the latter open side is filled with solvent vapor, evaporation of solvent mainly occurs from the open side filled with suspension, resulting in a unidirectional drying of colloidal suspension. We put a glass chip with colloidal suspension on a temperature controlled transparent stage made of glass and observed drying dynamics using an optical microscope (SMZ800, Nikon) for observation of suspension drying. Observed images were recorded at 10 s time intervals by a digital camera (D1x, Nikon). For observation of reflected image, we used a magnetron sputtering equipment (MSP-1S, Vacuum Device, Japan) to make a thin metallic film of gold-palladium mixture on the surface of the glass cover of handmade glass chip. Reflected images were recorded with the same optical microscope.

3. Results and Discussion

Typical example of cracks in formed colloidal film is shown in Figure 2. The drying temperature was 50°C. While many cracks parallel to the drying directions formed, periodic cracks perpendicular to the drying direction also formed.

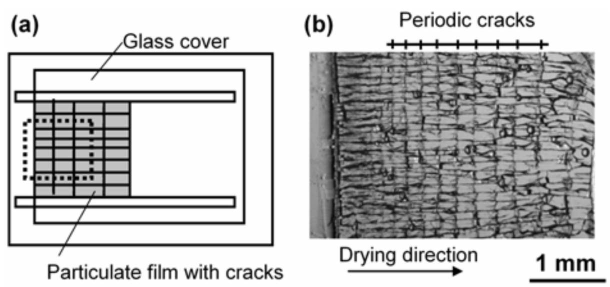

Figure 2. Typical example of formed cracks. (a) an overview of formed colloidal film with cracks. (b) is optical microscope image of the dotted square in (a).

These perpendicular periodic cracks had wider spacing when drying rate was small. We also found that the top glass cover became distorted during drying and the distorted glass cover suddenly got back to flat surface after a period of time. Each periodic perpendicular crack form when this relaxation occurs. To visualize this, we conducted reflection image observation. Figure 3a shows schematic illustration of this experiment. Thin metal film was deposited by sputtering on a glass chip to make the top glass cover into a half mirror. Then we were able to observe distortion by recording images reflected by the glass cover. When the glass cover distorts, the reflected image also distorts. Figures 3b-3d show reflected images. As clearly seen in Figure 3c, reflected lines near the drying edge got distorted compared to the image taken right after injection of colloidal suspension, Figure 3b. The distortion on the glass cover was released and we observed parallel lines as seen in Figure 3d, 10 s after Figure 3c. We found that the top glass cover formed a concave surface and this distortion occurs at which colloidal particles were packed but still wet. This indicates that the pressure in the packed particles becomes low compared to the atmospheric pressure during drying. Furthermore, we also found that this variation in pressure occurs periodically as drying proceeds. In our presentation, we report the details for this drying-induced self-excited system of pressure variation inside packed colloidal particles.

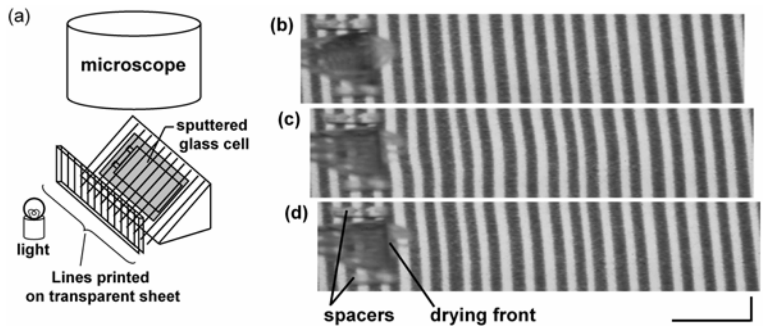

Figure 3. (a) Schematic illustration for reflection image observation. Reflected images (b) right after injection of colloidal suspension, (c) just before perpendicular crack formation and (d) 10 s after (c). The aspect ratio of each image of (b) – (d) was intentionally changed from the original ratio of 1 : 1.6 to 1 : 5 to highlight the distorted reflection image of parallel lines. The scale bar is 2 mm in each direction.

Acknowledgements

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