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Dong Kee Yi [a] & Dong-Yu Kim [a]

[a] Department of Materials Science and Engineering, Kwangju Institute of Science and Technology, Kwangju, 500-712, Korea

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Thin Film Formation Through the Array of Colloidal Meso Beads

DONG KEE YI and DONG-YU KIM

Department of Materials Science and Engineering, Kwangju Institute of Science and Technology, Kwangju 500-712, Korea

A convective two-dimensional self-assembly array was used to fabricate dense hexagonal colloidal monolayer on a glass substrate. The dense monolayer sintered at various time and temperature conditions. When the temperature was 160°C, the fully sintered film was achieved within 10 minutes. The resulting film thickness was about 260 nm. The sintering mechanism was explained following Frenkel's viscous model.

Keywords self-assembly; dense monolayer; sintered; Frenkel's model

INTRODUCTION

Fabrication of polymer thin films is important requirement for use of optical devices, molecular electronics, and corrosion protection applications. Some of such fabrication methods are spin coating, immersion in reaction mixture, and self-assembly array.[1-3] Denkov *et al.* used the vertical self-assembly method to fabricate the monolayer of colloids on substrates. The monolayer retained dense hexagonal array of colloids. However, there are many pores between the colloids and the pore contained air, therefore no continuous film was made. We report on a novel thin film fabrication method by using colloidal self-assembly and sintering.

EXPERIMENTAL

Glass slides were cleaned by ultrasonication in a hot mixture of H_2SO_4/H_2O_2 (8:2) for 1h. And they were treated with a mixture of H_2O/NH_3 (9:1) for 2h at 60 ^oC, and then immersed in a mixture of acetone/ethanol (5:5) at 30 ^oC for 2h. After this procedure, the substrate became hydrophilic. The glass slides were placed vertically into a vial containing aqueous polystyrene colloidal suspensions (diameter of 500 nm, *Duke Scientific*) The vial was placed in an oven for drying at 50 ^oC for 3h. The dried samples were heated for 10 minutes at 120 ^oC, 160 ^oC, 200 ^oC, and 240 ^oC. And they were also placed under the various time conditions (30 to 300 seconds) at 150 ^oC. The heat-treated samples were gold coated and the surfaces were observed by scanning electron microscopy (SEM) JEOL-5800 and the topologies were measured by atomic force microscopy (AFM, Auto Probe CP) in non-contact mode.

RESULTS AND DISCUSSION

When the water of suspensions evaporated at 50 ^oC, some colloids were nucleated and others were under the convective forces forming dense hexagonal arrays (fig. 1(a)).[3] As the densification proceeds, the lateral capillary force greatly affects between the beads drastically (fig. 1(b)).[4] At a temperature higher than 160 ^oC, the colloidal sphere structure can not be clearly identified with SEM images (fig.1(c),(d)) due to the fully developed sintering. Figure 2(a) shows that the root mean square roughness (RMS) of surface decreases as the heating temperature increases. However, the average film thickness lowered as the temperature increases because the leveling process worked.[5] Frenkel proposed a model to explain the earlier stage of viscous sintering of amorphous polymers.[6]

$$(X/R)^2 = 1.5\,(\sigma/\eta)\,(t/R)$$

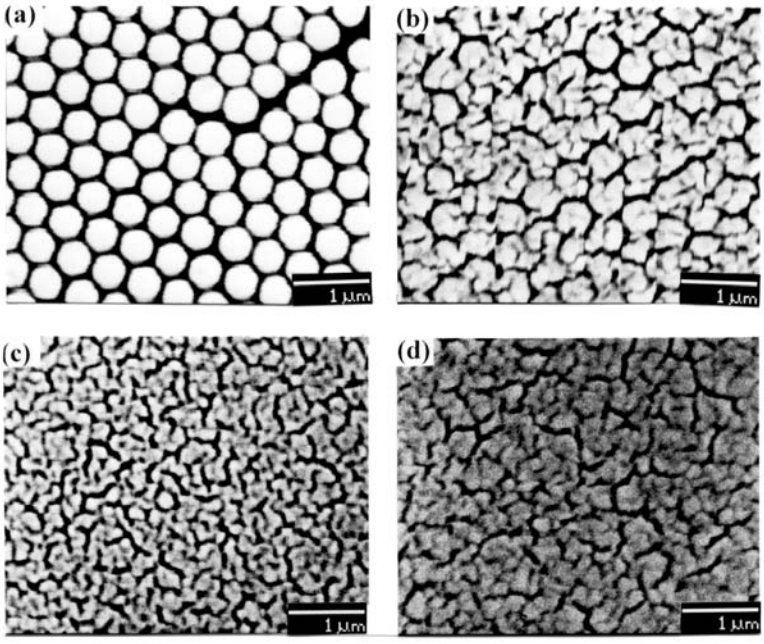

FIGURE 1. The SEM images of the colloidal film at various temperatures (a) green, (b) 120°C, (c) 160 °C, and (d) 200 °C

where, 2X is a neck diameter of the sintered beads, R is a bead diameter, σ is the surface tension of the bead, η is the Newtonian viscosity of the bead, and t is the time. Figure 2(b) shows the plot of $(X/R)^2$ vs t/R at 150°C. $(X/R)^2$ and t/R have a linear relationship with a slope of 5.79e-3. This means that the sintering was a viscous phenomenon.

In summary, we have demonstrated a new thin film fabrication method using colloidal self-assembly and sintering. When the dense colloidal assembly sintered, the surface roughness decreased and the film thickness decreased to the half of the neat bead diameter. The sintering mechanism was explained by the viscous model.

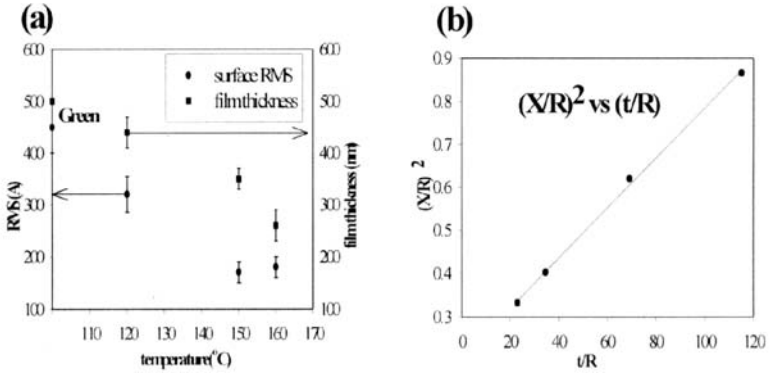

FIGURE 2. RMS values determined on 400 μm^2 spot and the film thickness are shown in (a) and (b) shows the the plot of $(X/R)^2$ vs t/R at 150°C.

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