Assembly of Colloidal Nanoparticles into Anodic Aluminum Oxide Templates by Dip-Coating Process

Il Seo, Chang-Woo Kwon, Hyun Ho Lee, Yong-Sang Kim, Ki-Bum Kim, and Tae-Sik Yoon

***Abstract*—In this paper, the assembly behavior of colloidal nanoparticles into anodic aluminum oxide (AAO) templates is investigated. Approximately 20-nm-diameter iron oxide (Fe_2O_3) particles stabilized by oleic acid and 5-nm-diameter CdSe coated by thin ZnS and stabilized by trioctylphosphine oxide and dispersed in octane solvent are integrated into AAO pores with an average pore diameter of ∼30–100 nm by dip-coating process. The particles assemble selectively at the bottom of pores. Also, the multiple stacks of particles are obtained selectively inside the pores by sequentially repeating dip coating and removing the surfactants (oleic acid) from the particle layer. The nanoparticles integrated into nanometer-scale AAO templates produce the nanostructures for potential applications such as high-density patterned magnetic media, patterned nanoparticle layers for memory device, seeds for nanowire growth, and so on.**

***Index Terms*—Anodic aluminum oxide, colloid nanoparticle, dip coating, self-assembly.**

I. Introduction

CHEMICALLY synthesized and surfactant-stabilized colloidal nanoparticles have attracted wide interest for their unique electrical, optical, and magnetic properties. Their peculiar properties are originated from their nanometer-scale dimension, which also offers an excellent sensing and catalytic capability, application to seed for nanowire and nanotube growth, biological diagnostic, to name a few [1], [2]. In particular, these particles can be synthesized with a narrow size distribution and form the ordered, close-packed self-assembly upon evaporation of the solvent [1]–[5]. In order to utilize these particles as part of devices, the construction of mono and multiple layers of them on the substrates has been pursued by Langmuir–Blodgett method [6], [7], spin-coating [8], [9], and dip-coating method [10]–[15]. While each of these processes demonstrates some successful results, at the same time, each of these processes has some drawbacks. For example, the Langmuir–Blodgett method, even though there is a huge benefit of forming the largest area of self-assembly, has a problem of crack formation after drying. The spin coating is simple and compatible to conventional device fabrication processes. However, it is difficult to deliver the exact amount of particles onto the substrate as well as it has an inherent problem of wasting the particles. Compared to these, the dip-coating process is a simple and a cost-effective one delivering the particles on many different types of substrates despite that it does not produce a long-range ordered structure as compared to Langmuir–Blodgett method. In addition, the mono and multiple layers can be constructed through simply repeating the dip coating after the surface treatment by annealing or depositing a glue layer on the preformed particle layer to promote the adsorption of particles.

With these advantages, the dip-coating process is popularly used for the particle layer formation. We previously reported the monolayer formation of ∼10-nm-diameter γ-Fe_2O_3 particle stabilized by oleic acid through the multiple dip-coating process, where the dip coating is repeated to increase the particle coverage without any glue layer [10]. By repeating the dip coating, we could obtain the particle coverage to ∼80% on Si substrate. In addition, the Coulombic interaction and strong intermolecular force between particles and the glue layer on the substrate surface were used for the particle attachment onto the substrate. Through the sequential adsorption of glue layer and particles, called layer-by-layer adsorption, the mono- and multilayer of particles were obtained [12], [13]. As another approach, Dimitrov and Nagayama [14] and Kim *et al.* [15] formed the particle layer driven by capillary force between particles and convective assembly of particle at the meniscus of solution during dip coating.

The patterned particle layer enables the applications of particles to be further extended to integrated nanostructures and devices. One of the popular approaches of patterned layer formation is to selectively deposit the particles on prepatterned regions. For example, Yin *et al.* [16] and Cui *et al.* [17] reported the delivery of particles into nanometer-scale hole and line patterns by capillary force at the edge of solution and substrate interface during the solvent evaporation. Maury *et al.* [18] and Kim *et al.* [19] also patterned the substrate with glue layer and adsorbed the particles selectively on the patterned glue layer driven by intermolecular force and Coulombic interaction, respectively. All of these efforts provide the routes for the patterned particle layer formation on the planar substrate.

Along the same line, the particle layer on 3-D nanostructures having high aspect ratio (height/width) is believed to widen the applications of particles, for instance the electrodes of sensor and capacitor with large surface area, 3-D photonic and electronic devices, etc. For this end, the integrations of nanomaterials such as nanoparticles or nanowires to nanoporous anodic aluminum oxide (AAO) templates, for example, are actively

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explored. AAO template has nanometer-scale ordered pores and their depth could be tuned up to about micrometers range [20]. It can be a template for the growth of nanowire, nanotube, and nanoparticles and be also integrated to the various substrates such as Si wafer, metallic, and plastic plates. Lee *et al.* [21] reported the formation of Ru nanowire and nanotube in AAO by atomic layer deposition. Also, Guo *et al.* [22] integrated the Au and Pt nanoparticles into AAO pores by preparing polyacrylamide hydrogel in the pores and subsequently introducing particles into the polymer nanowire in the pores.

In this study, we investigate the integration and assembly behavior of colloid nanoparticles into AAO templates by dip-coating process. The particle assembly can be controlled by tuning the pore size and dip-coating conditions. Here, we report the particle assembly by repeating dipping and pulling out AAO templates from the colloid solution without any chemical modification of AAO surface. This approach integrates nanoparticles in nanometer-scale templates with simple dip-coating process, which can be utilized for practical applications.

II. Experimental Details

Sterically stabilized iron oxide (Fe_2O_3) nanoparticles with ~20-nm diameter were chemically synthesized by decomposition of iron pentacarbonyl ($Fe(CO)_5$) in a mixture of octadecene and oleic acid ($C_{18}H_{34}O_2$), and then dissolved in octane (C_8H_{18}) solvent [5]. The particles were identified to be maghemite (γ-Fe_2O_3, JCPDS No. 39-1346) measured by X-ray power diffraction method. As-synthesized colloidal particles were repeatedly precipitated with ethanol and redispersed in octane. During the particle synthesis, the oleic acid molecules encapsulate the particle surface, as a stabilizing agent (surfactant), to prevent the aggregation of particles in the solution. A hydrophilic head of oleic acid is attached on particle surface and hydrophobic tail faces the solvent. Therefore, the particles have a hydrophobic character and are well dispersed in nonpolar solvent such as alkane solvent. Here, we dissolved the particles in octane solvent. In addition, CdSe nanoparticles with a diameter of approximately 5 nm, coated with thin ZnS and trioctylphosphine oxide (TOPO), and dispersed in octane with a concentration of 10 mg/mL roughly calculated to be about 7.2×10^{16}/mL when neglecting the mass of surfactants in the solution for the calculation, were used as purchased commercially from Nanosquare Inc.

AAO templates were prepared by two-step anodization of Al plate as previously reported [23]. The first anodization of Al plate was performed at a constant voltage of 50 V in 0.3 M oxalic acid at 15 °C for 4 h after degreasing Al plate in acetone and following electropolishing in a mixed solution of perchloric acid and ethanol under a constant voltage of 15 V at 3 °C for 2 min. Then the anodic oxide was removed in phosphoric acid and chromic acid at 60 °C for 2 h. The second anodization was carried out and then the pore widening step has been done in a 0.5 M phosphoric acid, which makes the surface smoother at the same time. Two sets of AAO templates were prepared with different diameters of ~30–50 nm for 5 min of pore widening procedure and ~80–100 nm for 1 h.

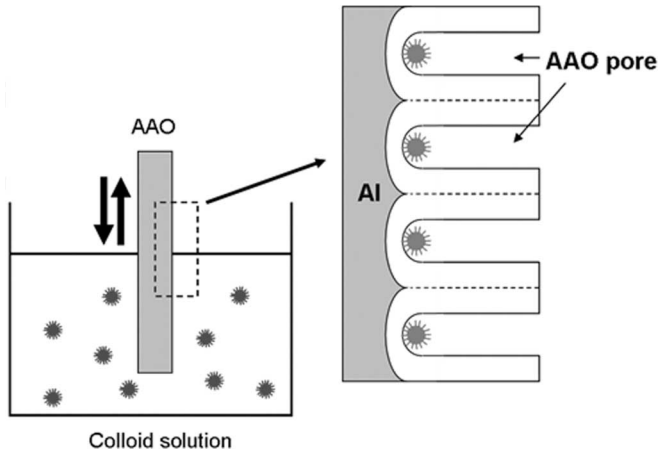

Fig. 1. Schematic illustration of dip-coating process for the particle assembly into AAO templates.

These AAO templates were vertically dipped in a colloidal solution and pulled out at a speed of 0.1 mm/s and was dried in air at room temperature. The Fe_2O_3 particle concentration in the solution is 1.2×10^{13}/mL as optimized to form uniform assembly of particle in templates, which were measured by inductive coupled plasma spectrometer (ICP, Leeman Labs Inc.) [10], [24]. The dipping and pulling out templates were repeated up to five times. The schematic illustration of dip-coating process for the particle assembly into AAO templates is shown in Fig. 1. Besides simply repeating dip coating, the dip-coated templates were also annealed at 200 °C for 1 h in air environment, during which the surfactants (oleic acid) desorb from the particle layer on template, and subsequently dipped again in the solution for additional adsorption of particles onto the particle layer. The dipping and annealing procedures were repeated thrice to make multiple stacks of particles inside the pores. The morphologies of particle layer were analyzed using scanning electron microscope (SEM). Oblique angle-view images were obtained by folding and tilting AAO templates formed on Al plate.

III. Results and Discussion

Fig. 2 is the plan-view and oblique angle-view SEM images of ~20-nm-diameter Fe_2O_3 particles in AAO templates with smaller pore diameter of ~30–50 nm after dip coating for one time (a, b) and repeating for five times (c). The particles are uniformly and selectively incorporated inside the pores and assemble at the bottom. Also, the particles are found to form the multiple stacks inside the pores even by dip coating for one time [Fig. 2(b)]. However, repeating dip coating rarely increases the number of particles inside the pores [Fig. 2(c)]. This tendency is consistently observed from larger pores with a diameter of ~80–100 nm, as shown in Fig. 3. The particles assemble selectively at the bottom of all the pores as uniformly forming close to monolayer. As similar to the results in smaller pores in Fig. 2, the number of particles in the pores is slightly increased by repeating dip coating for five times [Fig. 3(c)]. For comparison, we have previously reported that the particle coverage gradually increased close to monolayer by repeating dip

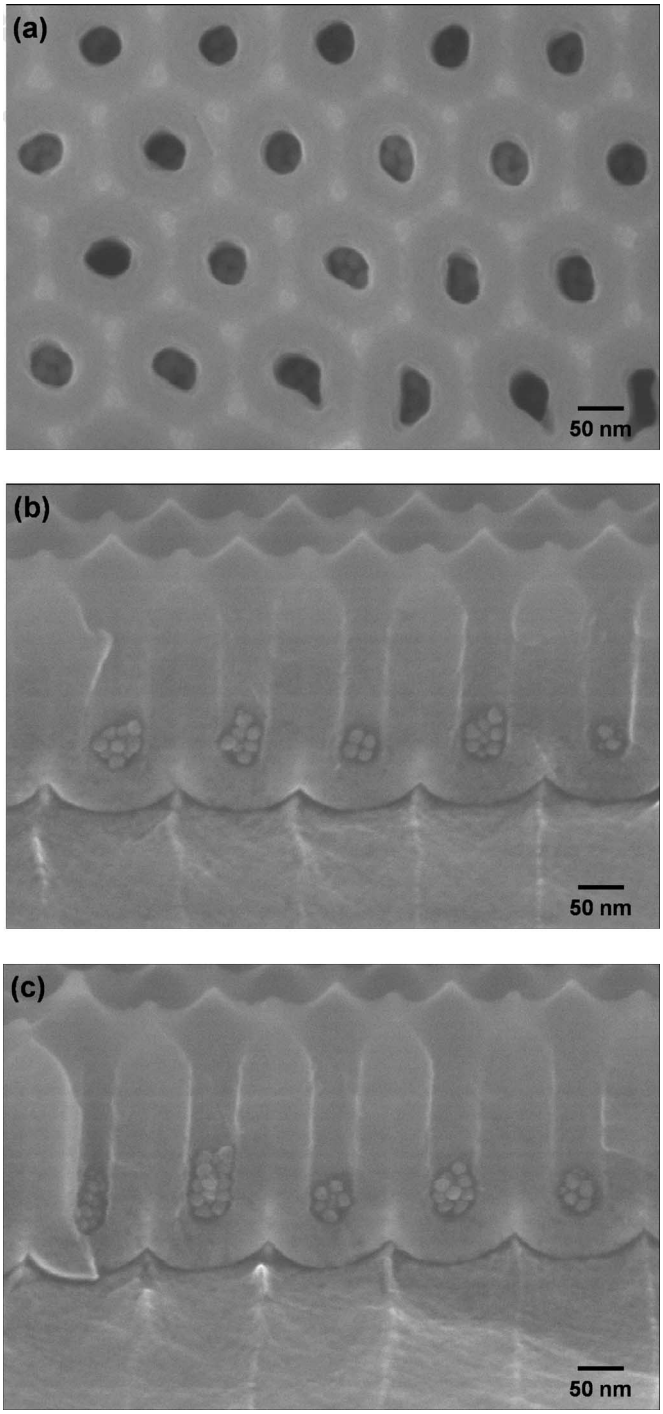

Fig. 2. Plan-view and oblique angle-view SEM images of ∼20-nm-diameter Fe_2O_3 particles in AAO templates with a pore diameter of ∼30–50 nm after dip coating for (a, b) one time and (c) five times.

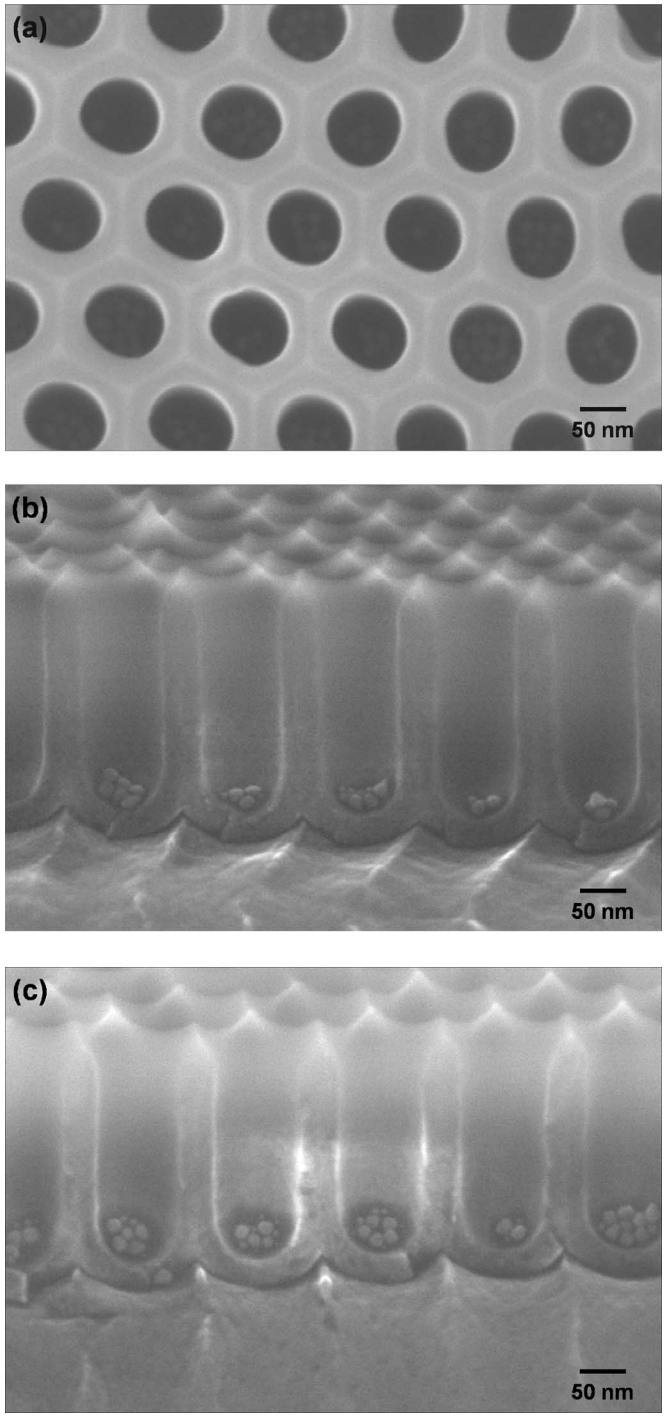

Fig. 3. Plan-view and oblique angle-view SEM images of ∼20-nm-diameter Fe_2O_3 particles in AAO templates with a pore diameter of ∼80–100 nm after dip coating for (a, b) one time and (c) five times.

coating on planar Si and SiO_2 substrates [10]. We suggested that the particles adsorb on the substrate by van der Waals interaction proportional to the Hamaker constant of particle and substrate and subsequently assemble during the evaporation of solvent [10], [25]. The particle coverage on Si, Si_3N_4, and SiO_2 substrates was revealed to be highest on Si and lowest on SiO_2, as qualitatively corresponding to the order of Hamaker constants of substrates: 18.65×10^{-20} J for Si [26], 16.71×10^{-20} J for Si_3N_4 [26], [27], and 6.5×10^{-20} J for SiO_2 [26], [27]. In addition, even on SiO_2 substrate with the lowest Hamaker constant, the particles coverage increases from ∼10 to 50% of monolayer coverage by repeating dip coating for ten times. The coverage was defined as the ratio of the number of particles on the surface to the number forming the close-packed hexagonal array on the surface. However, despite of larger Hamaker constant of Al_2O_3

of 15.2×10^{-20} J [27] than that of SiO_2, the particle coverage does not increase by repeating dip coating up to five times. In order to clarify this, the multiple dip coating was performed on planar Al_2O_3 surface prepared by thermal oxidation of Al plate at 500 °C. The particles were found to assemble as irregularly scattered with multilayers and the coverage of assembly gradually increases by repeating dip coating. This indicates that the particle assembly behaviors are different between the planar surface and patterned one.

As for the selective assembly of particles inside the patterns, Yin *et al.* [16] and Cui *et al.* [17] suggested that the capillary force at the edge of solution interface with substrate (vapor-solution-substrate contact line of solution meniscus) drives the particles into patterns upon drying solvent. A magnitude of capillary force for 20-nm-diameter particles, $F_c = 2\pi r\sigma \sin\varphi$, where σ is the surface tension of solution (roughly assumed to be 21.62 mN/m of octane solvent), r is the particle-solution contact-line radius same with particle radius when the solution meniscus moves down to a thickness of particle radius, φ is the angle between radial axis of particle and solution (assumed to be 90° of complete wetting between nonpolar octane solvent and hydrophobic tail of oleic acid covering the particle surface) [17], is roughly calculated to be ~330 *kT*/nm, which is sufficient to overcome the thermal fluctuation. Therefore, the particles can be driven inside the pores when the solution thickness becomes close to particle radius during evaporation.

It should be also noted that the particles form multiple stacks in smaller pores while form the single layer in larger ones. It is postulated that the particles adsorb first on AAO surface and all the adsorbed particles are driven into the pores during solvent evaporation. The top surface area of AAO template with smaller pores is wider than larger ones. Therefore more particles adsorb on the top surface and are driven inside smaller pores as forming multiple stacks. On the other hand, fewer particles adsorb and are driven into larger pores resulting in single layer formation. However, it is not clear why the number of particles inside the pores is rarely changed even by repeating dip coating.

One probable reason for self-limited stacking of particle layers even by repeating dip coating is that the surfactants covering the particle surface prevent the particles from adsorbing onto the particle layer, which was previously demonstrated on planar Si and SiO_2 substrates [10]. Though the particles are driven into pores by capillary force, they may escape from the pores before the solvent is completely dried because the surfactants on particle surface inhibit the adsorption on particle layer. In order to clarify the effect of surfactants, the AAO templates were dip coated and then annealed at 200 °C for 1 h in air environment after dip coating. During annealing, oleic acid molecules (surfactants) desorb from Fe_2O_3 particle layer, thereby they do not prevent the particle adsorption during subsequent dip-coating step. Pérez-Dieste *et al.* reported that the oleic acid molecules covering Co and Ni particle surface readily desorb from the surface above 200 °C [28]. Fig. 4 is the oblique angle-view SEM images of Fe_2O_3 particles in the pores with a diameter of ~80–100 nm after repeating dip coating and annealing at 200 °C for 1 h for three times. It is clearly observed that more particles are driven into pores and form the multiple stacks as different from the results after dip coating for five times without annealing [Fig. 3(c)]. It demonstrates that the surfactants covering the particle surface inhibit the formation of multiple stacks, and removing the surfactants by annealing and subsequently repeating dip coating successfully form the multiple stacks of particle layer with high density selectively inside the pores. However, further study is necessary to clarify all the observations. For example, it is still questionable why the multiple stacks were formed without removing surfactants in smaller pores [Fig. 2(b)], which implies that more parameters are complicatedly involved in determining the single and multiple stack formation.

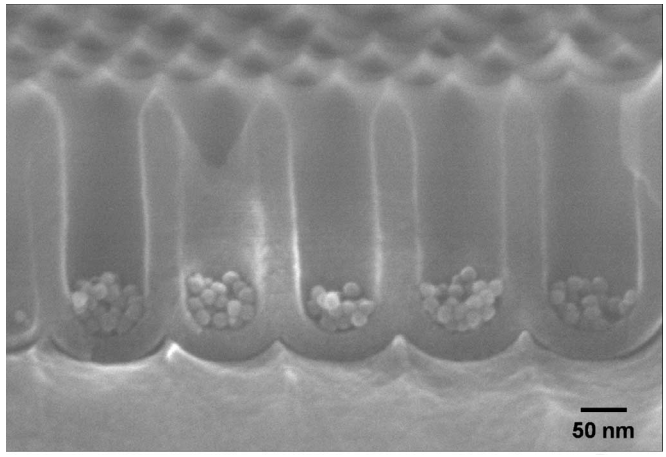

Fig. 4. Oblique angle-view SEM image of Fe_2O_3 particles in AAO templates with a pore diameter of ~80–100 nm after repeating dip coating and subsequent annealing at 200 °C for 1 h for three times.

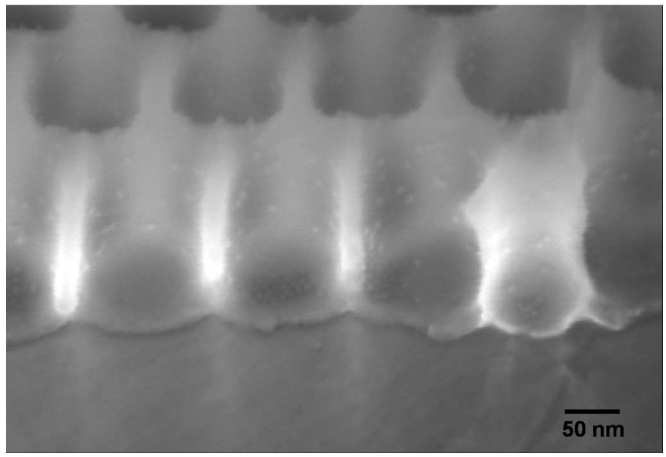

Fig. 5. Oblique angle-view SEM image of ~5-nm-diameter CdSe particles in AAO templates with a pore diameter of ~80–100 nm after repeating dip coating for five times.

Besides Fe_2O_3 particles, 5-nm-diameter CdSe particles coated with thin ZnS layer and TOPO and dispersed in octane were delivered into the pores with a diameter of ~80–100 nm. Fig. 5 is the oblique angle-view SEM image after repeating dip coating for five times. The CdSe particles assemble at the bottom of pores as similar to Fe_2O_3 particles. But, some of them adsorb on the sidewall and top of pores. A rough calculation

of the magnitude of capillary force for 5-nm-diameter CdSe is ~80 *kT*/nm. Since the capillary force is proportional to particle radius, a larger population of CdSe particles at the sidewall and top of pores is due to a lower capillary force than that of 20-nm-diameter Fe_2O_3 particles.

All of these results imply that the assembly behavior in AAO templates or other patterned structures is comprehensively determined by various parameters such as the interaction of particle with substrate, particle migration into the patterns determined by capillary force through solution meniscus, size of patterns, and the presence of surfactants on the particle layers. The selective assembly of nanoparticles inside nanotemplates such as AAO as either single layer or multiple stacks is expected to be utilized to form self-aligned nanoparticles for nonvolatile memory, electrode of sensors with large surface area, seeds for nanowire and carbon nanotube growth, surface reaction catalysts with high and uniform density, high-density patterned magnetic media, and so on.

IV. Conclusion

We have integrated colloidal nanoparticles into AAO templates. Dipping and pulling out the templates with ~30–50 nm and ~80–100 nm diameter pores from the colloidal solutions presents the selective assembly of particles inside the pores with a high density. The multiple stacks of particle layers were formed by removing surfactants from the particle layer and subsequent dip-coating procedures. The selective assembly inside the pores was interpreted with the capillary force-driven particle assembly at the bottom of pores. The approach of selective assembly of nanoparticles inside nanotemplates such as AAO as single and multiple stacks with high areal density can be a route for the nanoparticle integration into nanotemplates for various applications utilizing patterned particle layers.

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