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Colloidal nanoparticle sorting and ordering on anodic alumina patterned surfaces using templated capillary force assembly



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ABSTRACT

A new, robust technique of size-selective nanoparticle ordering on porous anodized aluminum oxide (PAAO) templates is presented. Simultaneous particle sorting and array formation is achieved for the first time using a polydisperse suspension of irregularly shaped diamond nanocrystals. The array parameters can be tuned through a balance of evaporation driven particle flux, capillary, electrostatic, and adhesion forces, which are influenced by the asperities of the surface during the capillary and convective assembly dip-coating process. The resulting structures are dense (lower limit approximately 50 nm center separation), isolated (non-touching) nanoparticle arrays with a size distribution that matches the topology of the PAAO template surface. The method is demonstrated using nanodiamonds (ND) and Ag nanoparticle colloids of various sizes, on PAAO surfaces with different pore diameters as well as a patterned Al surface whose oxide layer had been removed. The transfer of the ND array to a polydimethylsiloxane (PDMS) matrix further illustrates the versatility of the method. The procedures presented here extend the range of templated nanoparticle array production to technologically important materials, which are difficult to handle using conventional evaporation, electrodeposition or imprint techniques, yet are available as polydisperse suspensions.

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1. Introduction

Patterned assembly of colloidal nanoparticle arrays is essential for fundamental and applied research, including the design of metamaterials on the size scale of optical wavelengths [1]. Although colloid synthesis is capable of producing relatively monodisperse suspensions of certain materials [2], individual particles typically have different sizes and shapes, which hinders the production of ordered arrays with cell dimensions in the 50 nm range. Such particles usually form irregular aggregates during deposition on a flat surface. Techniques of close-packed (touching) nanoparticle superlattice self-assembly have been developed [3] for a wide range of materials. However, production of non-touching arrays remains challenging (see Schumde et al. [4] and references therein) and

requires multiple steps. In a work by Abdelsalam et al. [5] close-packed monodisperse colloid crystals were used to pattern surfaces for subsequent deposition of isolated smaller nanoparticle arrays.

A separate class of array production techniques is templated deposition, where prefabricated structures, such as PAAO are used to create patterns in different materials [6,7]. PAAO is a well-known self-organized structure [8] with numerous synthesis protocols, which allow to control the pore size and distribution [9], as well as perform various manipulations including transfer to different surfaces [10]. PAAO applications include production of nanoparticle or nanowire arrays, either using electrochemical deposition [11,12], vacuum filling [13,14], the supercritical fluid method [15], masked deposition [10], crystallization [16], imprinting [17], or other methods [6]. In a recent work we have demonstrated production of sub-20 nm metal nanoparticle arrays using PAAO as an evaporation mask [18].

The main objects in the present study are nanodiamond (ND) arrays. Diamond is an emerging multifunctional optical and semiconductor material and is expected to be of technological importance for the electronics industry [19]. Fluorescent nanodiamonds (FND)

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have been proposed in the context of quantum computing, and the ability to create ordered arrays of such structures may open new possibilities for the design of complex devices [20]. Other potential FND applications include biomolecular labeling [21], nanophotonic light sources [22], and magnetic imaging [23]. Unfortunately, due to the extreme synthesis conditions, production of ordered ND structures using masked deposition techniques is difficult. However, nanodiamonds can be obtained relatively easily in colloidal suspensions [24]. Here we study templated deposition of ND arrays from a colloid onto a PAAO patterned surface and find optimal parameters for a uniform pore filling using initially polydisperse ND suspension. Further, we show that the method can be applied to transfer an ND array to a polymer matrix and to assemble non-touching Ag nanoparticle arrays, which are of importance for synthesis of plasmonic metamaterials [25].

The proposed method is novel and differs from reported capillary force assisted (CFA) colloid assembly procedures [26–28] by making it possible to obtain non-touching arrays from initially polydisperse suspensions of irregularly shaped particles. The template in our case is an easily producible, self-ordered structure, whereas other studies have used lithographically patterned substrates [27,29]. Moreover, we achieve sorting and array ordering of particles in the 20 nm range, whereas CFA on patterned surfaces is typically demonstrated using larger diameter spheres (>50 nm).

2. Experimental

Most of the PAAO membranes were produced on bulk 0.25 mm thick high purity (99.999% GoodFellow) electropolished Al sheet using a two-stage anodization protocol [10] in a 0.3 M sulfuric acid solution (s-PAAO) at a constant voltage 20 V as described elsewhere [18]. The resulting membrane thickness was 150–200 nm, the pore diameters were approximately 25 nm, and the average center separation was 50 nm. For the purpose of comparison, PAAO substrates with larger pore diameters (≈ 60 nm) and center separation (≈ 100 nm) were produced using oxalic acid 0.3 M electrolyte (o-PAAO) and 40 V anodizing potential. It should be mentioned that for optical applications the sulfuric acid anodization protocol is advantageous, since the resulting s-PAAO has significantly lower photoluminescence in the visible spectral range [30].

A solution of ND particles in water (DiaScience 25, Quantum Particles) with a specified size $d_{50} = 28$ nm (number mean value) and a concentration of 0.29 mg/ml was used as received. The particle size distribution with d_{50} value 28.5 nm and standard deviation $\sigma = 8.7$ nm was confirmed using dynamic light scattering (Malvern Nano ZS).

Ag sol was produced following the protocol of Lee and Meisel [31]. Using this method the Ag nanoparticle size varies for different batches depending on when the Ag reduction reaction stops. Further, the nanoparticle concentration could be adjusted by centrifugation.

Dip-coating was done using a homebuilt water bath thermostat placed on a vertical translation stage with controllable withdrawal velocity (Fig. 1a).

The colloid solution was kept in a polystyrene cuvette inside a water bath for ca. 3 min to reach thermal equilibrium before inserting the sample. The small meniscus angle at the sample surface during withdrawal (Fig. 1b) confirms the hydrophilic nature of the PAAO substrate. Finally, the samples were left to dry overnight in air at room temperature. Array particle size and distribution analysis was done using a field emission scanning electron microscope (SEM, Hitachi S4800).

During a separate run, the air humidity and Al sheet temperature were measured inside the cuvette approximately 5 mm above the surface of the solution using an HIH-4010-001 (Honeywell) humidity sensor and a DS18B20 digital thermometer (Maxim Integrated).

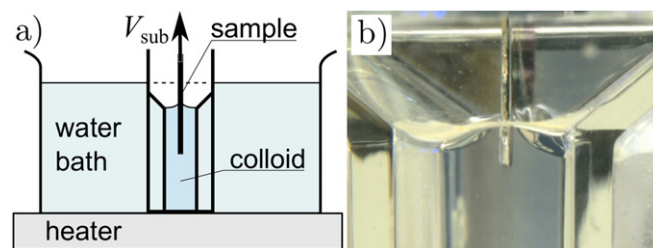


Fig. 1. a) Schematic of dip-coating setup and b) photograph of the meniscus area during sample withdrawal.

The PDMS for the array transfer was produced using Sylgard 184 elastomer kit (Dow Corning). The PDMS and the curing agent were mixed following the manufacturer's instructions, and a droplet was placed on the ND coated s-PAAO surface, which was subsequently kept in a degassing vacuum chamber for 5 min. The curing was done at 50 °C overnight. The Al substrate was dissolved in a $\text{CuCl}_2/\text{FeCl}_3$ mixture [18]. Finally, the PAAO was removed by a 5% H_3PO_4 solution, washed with distilled water and dried in air.

3. Results and discussion

The ND arrays on the s-PAAO surface were produced in a set of control parameters defined by a variation of colloid (water bath) temperature (5–40 °C) and substrate pull-out velocities $0.4 < V_{\text{sub}} < 11 \mu\text{m/s}$. A feature common to all the samples obtained using the s-PAAO substrate is the size-dependent sorting effect, which can be seen in the SEM images (Fig. 2), which show very few NDs with sizes exceeding the specified $d_{50} = 28$ nm value of the colloid. From the size distribution (Fig. 3c) one would expect a significant number (about 10%) of the particles to have a size larger than 40 nm, which is not the case. However, large particles were observed if deposition was done at similar conditions on a flat electropolished Al surface (not shown). Further, nearly all NDs are located above or close to the pore openings (Fig. 2), clearly indicating the preferred attachment sites.

Although the SEM analysis of pores, which are more than 100 nm deep, does not allow accurately to distinguish individual particles from small aggregates, the temperature dependence can be summarized by the following qualitative observations. Firstly, the number of filled pores increases with increasing temperature. Secondly, at higher temperatures, more pores tend to host multiple particles. However, even at a temperature of 40 °C, no large aggregates of connected particles or continuous nanoparticle layers are formed.

From a series of samples that were produced at different temperatures and withdrawal velocities, it was found that a good balance between sorting, ordering and filling density can be achieved at a colloid temperature of 25 °C and a withdrawal rate of $V_{\text{sub}} = 2 \mu\text{m/s}$. At higher withdrawal speeds, sparse arrays with many empty sites are produced (Fig. 2a). The particle density increases at lower pull-out rate and nearly 100% site filling or overfilling can be achieved (Fig. 2b,c). As will be discussed in more detail shortly, the balance between V_{sub} and convective particle flux j_p due to evaporation is the dominant factor that determines the array density, since the capillary number $Ca \approx 10^{-8}$ cannot be sufficiently increased at velocity range (0.4–11 $\mu\text{m/s}$) used in this study. In this regime the liquid film thickness above meniscus does not depend on V_{sub} [32]. The opposite case would correspond to the Landau-Levich regime $10^{-6} < Ca < 10^{-1}$, which is of interest for more viscous liquids or high velocity drag.

The dynamics of colloid deposition on a PAAO surface is a complex process and involves multiple mechanisms and competing forces. Apart from colloid particle-particle and particle-solid surface interactions [33] convection and water-air surface tension plays an

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