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Low-Dimensional Arrangement of SiO₂ Particles

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Novel processes to realize low-dimensional arrangement of SiO₂ particles were proposed. Particle wires and a pattern of the close-packed particle monolayer were fabricated in the solution at room temperature. SAMs (self-assembled monolayers) were formed on Si substrates and modified to be suitable for templates of precise arrangement. Particles were arranged precisely in the desired positions in the solution using well-controlled electrostatic interactions and chemical bond formation between particles and substrates.

Introduction

Particles show various interesting properties¹ that are not observed in the bulk form of a material. Recently, monodispersed particles have been synthesized by various methods. Particles may be thus treated as if they were atoms, and one-, two-, or three-dimensional arrangements of particles have been studied by many researchers. For instance, photonic crystals can be constructed by monodispersed particles. Additionally, electronic energy transfer in close-packed CdSe² or InP³ quantum dots arises from dipole–dipole interdot interactions between proximal dots. For instance, the metal–insulator transition was observed in a silver quantum dot monolayer.^{4,5} To apply these novel properties for future devices, precise particle arrangement techniques must be developed.

Assembly techniques of fine particles have been studied by several groups^{6–14} (Table 1). Assembly of Au particles was realized by a casting method^{8,9} at first. The electrostatic interaction was also used for assembly and alternate adsorption of Au particles.^{11,12} Yonezawa et al.¹⁴ demonstrated that the close-packed ammonium layer of an adsorbed bilayer provides superior binding sites for negatively charged Au particles (19 nm diameter). They are mostly adsorbed in a closely packed arrangement, due

to the regular, dense disposition of positive charges on the surfaces. This research confirmed that alternate layer-by-layer assembly is possible in this system. Furthermore, a closely packed layer of polystyrene particles (1 μm diameter) was fabricated using the capillary attractive force, and the optical properties of this particle array were studied.¹⁴ Particles are successfully assembled in an orderly arrangement in this array.

Patterning of assembled particles was realized by several methods^{15–31} (Table 1). Micropatterns of randomly deposited particles were fabricated by gas jet^{15,16} and ink jet^{17,18} printing methods or electrostatic interaction.¹⁹ Aizenberg et al.¹⁹ supported substrates micropatterned with anionic and cationic regions in a particle suspension for 2 min. The substrates were then removed and rinsed the excess suspension. The observation of drying process of this wetted substrate showed the particles within each negatively charged regions moved toward the center of the circles and formed dense clusters. They fabricated micropatterns of randomly deposited particles and dots using electrostatic interactions and a drying process. Two-dimensional patterns with regular repeatability in the particle array were also made by some techniques such as probe technique,^{20,21} electrophotography method,^{22–24} and micromold method.²⁵ In addition, patterns with high

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Table 1. Assembling and Patterning Techniques of Particles

assembling methods	particle	structure	ref
casting	Au, Pt, Ag (1–10 nm diameter)	two-dimensional hexagonal structure	8
casting	Ag	ring of Ag particles	9
alternate adsorption (macromolecule)	Au	monolayer, multilayer	11
alternate adsorption (amphiphilic molecule)	Au (20 nm diameter)	monolayer, multilayer	12
laser standing-wave field (laser trap)	polystyrene (1 μ m diameter), TiO ₂ (0.24 μ m diameter)	two-dimensional close-packed structure	13
capillary force	polystyrene (1 μ m diameter)	two-dimensional close-packed structure	14
patterning methods	particle	structure	ref
gas jet deposition	various kinds of particles (nm to μ m diameter)	micropattern of randomly deposited particles	15, 16
ink jet printing	ZrO ₂ (100–200 nm diameter)	micropattern of randomly deposited particles	17, 18
electrostatic and lateral capillary interaction	polystyrene (1 μ m diameter)	micropattern of randomly deposited particles, dot	19
probe manipulation	Au (50–100 μ m diameter)	two- and three-dimensional structure, particle tower	20, 21
electrophotography method	SiO ₂ (5 μ m diameter)	micropattern of randomly deposited particles dot	22–24
micro mold method	Al ₂ O ₃ (47 μ m diameter)	dot, particle tower, etc.	25
probe manipulation.	latex (0.6–2 μ m diameter)	micropattern of close-packed structure, fcc structure	26, 27
micro mold method	polymer (450 nm diameter)	micropattern of close-packed structure	28
photolithography and etching	Si (855 nm diameter)	micropattern of close-packed structure, etc.	29, 30
SAM patterning method	SiO ₂ (500 nm diameter)	micropattern of close-packed structure	1, 31, 32

arrangement accuracy have been realized by some techniques. Miyazaki et al.^{26,27} fabricated photonic crystals such as two-dimensional closely packed structure²⁶ and fcc (or hcp) structure²⁷ from latex particles (0.6–2 μ m diameter) by the micromanipulation technique in a scanning electron microscope. Though this can be applied to many kinds of particles and it is possible to arrange particles to an accuracy of several percent, this technique requires hours for the arrangement. Kim et al.²⁸ fabricated micropatterns of fcc (or hcp) structure from polymer particles (200 nm diameter) using a micromold. The mold was adhered to silicon substrate, and the particle suspension was then poured into clearance between the mold and the substrate. After drying, the mold was removed and microstructures were obtained. However, the position of individual particles cannot be controlled, and a highly precise mold is required for the arrangement of nanosized particles. Patterning of close-packed three-dimensional structure (fcc) constructed from silicon particles was also fabricated using photolithography and etching techniques. Planar, single-crystalline silicon photonic crystals that have low defect densities can be obtained by assembling a thin layer of colloidal particles on silicon substrate.^{29,30}

Recently, we succeeded in two-dimensional arrangement of particles using self-assembled monolayers (SAMs).^{1,31,32} Particles are arranged in silanol regions selectively to form a closely packed monolayer at room temperature. However, improvement in arrangement precision is necessary to apply this method to photonic or electronic devices. Here, we present novel modification processes of SAMs to fabricate templates for precise arrangement of particles. Additionally, interactions between particles and substrates were well controlled by surface modification of particles and substrates to improve arrangement precision. SiO₂ particles that have silanol groups were arranged on silanol regions of SAM to form particle wires. SiO₂ particles covered by carboxyl groups were arranged on silanol groups of SAM modified by AFM lithography. Furthermore, SiO₂ particles covered by

carboxyl groups were arranged selectively on amino groups of SAM, resulting in a pattern of the close-packed particle monolayer.

Experimental Section

Preparation of an OTS-SAM and a SAM Patterned with Octadecyl Groups and Amino Groups. OTS-SAM was prepared by immersing the Si substrate in an anhydrous toluene solution containing 1 vol % OTS for 5 min under a N₂ atmosphere.^{33–36} SAMs were exposed for 2 h to UV light (184.9 nm) through a photomask. The UV-irradiated regions became hydrophilic due to Si–OH group formation, while the nonirradiated part remained unchanged; i.e., it was composed of hydrophobic octadecyl groups, which gave rise to patterned OTS-SAM. To check successful film formation and functional group change, water drop contact angles were measured for irradiated and nonirradiated surfaces. Initially deposited OTS-SAM had a water contact angle of 96°, while the UV-irradiated SAM surface was saturated (contact angle <5°). The patterned SAM was further immersed in a toluene solution containing 1 vol % APTS ((aminopropyl)triethoxysilane) in air for 1 h. APTS molecules combined to silanol groups of SAM, and hence, octadecyl/amino groups patterned SAM was fabricated. OTS-SAM exhibited a water contact angle of 96°, while that of the amino surface was 28°. These observations indicated successful fabrication of octadecyl/amino groups patterned SAM.

Surface Modification of SiO₂ Particles with Carboxyl Groups. Silica particles (1 μ m diameter, HIPRESICA UF, UNK, Ltd.) were immersed in a dicyclohexyl and sonicated for 10 min under a N₂ atmosphere for good dispersion. 1 vol % of trichloroethoxyethylsilane (TCES) was added to the dicyclohexyl solution under a N₂ atmosphere, and the solution was stirred gently for 30 min in order to chemisorb TCES onto the SiO₂ particle surfaces. SiO₂ particles with TCES were centrifuged several times to remove unreacted TCES using dicyclohexyl.

The SiO₂ particles with TCES were further dispersed in a tetrahydrofuran solution containing potassium *tert*-butoxide (*t*-

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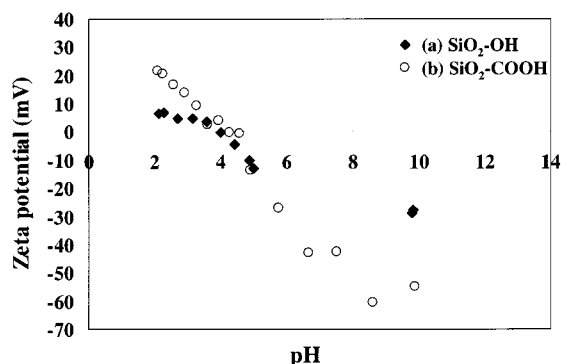


Figure 1. Zeta potential of (a) SiO₂ particles and (b) SiO₂ particles modified with carboxyl groups.

BuOK) and 18-crown-6 ether for 48 h under an ambient atmosphere to oxidize the CN groups to carboxyl groups.³⁷ The solution was centrifuged several times using distilled water to remove *t*-BuOK, 18-crown-6 ether, and a tetrahydrofuran. SiO₂ particles modified with carboxyl groups were thus obtained.

SiO₂ particles covered by silanol groups or carboxyl groups were arranged selectively in silanol regions or amino regions of SAM using interactions between particles and SAMs. Zeta potentials of SiO₂ particles that have silanol groups and SiO₂ particles modified by carboxyl groups were measured (Zetasizer 3000HSA, Malvern Instruments Ltd.) as shown in Figure 1. Zeta potentials measured in aqueous solutions (pH = 7.0) for the surface of silicon substrate covered with silanol groups, phenyl groups (PTCS), and amino groups (APTS) are -38.23, +0.63, and +22.0 mV, respectively.³⁸

Results and Discussion

Fabrication of Particle Wires Employing Selective Arrangement Process. OTS-SAM was modified by a diamond tip to form a line of silanol groups of approximately 0.5 μm width (Figure 2). The diamond tip was contacted to OTS-SAM surface lightly and traced with low contact pressure in order to modify the SAM surface. The surface modified by a diamond tip, i.e., the white area in Figure 2 corresponds to silanol groups, showed low contact angle (<5°). This modified region was shown to be white compared with the OTS-SAM region in a scanning electron micrograph (SEM; S-3000N, Hitachi, Ltd.). Octadecyl groups were broken mechanically by contact pressure with the diamond tip, and they possibly changed into silanol groups. The diamond tip was used to avoid contamination from a metal tip and the influence of a chemical reaction between the tip and the SAM.

Patterned SAMs were immersed in the aqueous solution containing SiO₂ particles and a hydrochloric acid as a condenser,³¹ rinsed in water, and observed by a SEM. SiO₂ particles were observed on lines of silanol groups selectively, indicating particles were successfully arranged well (Figure 3a).

Because particles were not easily removed by sonication, it was judged that siloxane bonds had been formed by condensation of silanol groups between particles and a SAM.

It is clearly seen that the accuracy of particle arrangement has been improved compared to our former experiments.^{31,32} A double particle wire and a triple particle wire were likewise fabricated on wide silanol groups regions with about 1.4 and 2.2 μm in width, respectively (Figure 3b,c). The double particle wire that has a triangular lattice

also demonstrates a high arrangement accuracy, though there is a defect in arrangement between seventh particle from left and eighth particle. Additionally, a curved double particle wire was fabricated on curved region of silanol groups. Curved double particle wires have not been reported previously, and they may have useful applications for an optical waveguide.

The accuracy of the particle arrangement was evaluated from Figure 3a. The center position ((*x_i*, *y_i*) μm) of each particle was plotted to estimate the standard deviation (Figure 4). The bottom left corner of Figure 3a was set to be the origin of the *x*-*y* coordinate. The approximated straight line (*f*(*x*)) and its slope (*θ*) are represented as follows:

$$f(x) = 0.0061x + 2.9609 \quad (1)$$

$$\cos \theta = 0.9940 \quad (2)$$

The standard deviation from the approximated straight line is described by the expression

$$S(\text{standard deviation}) = \frac{[\sum_i \{\cos \theta (f(x_i) - y_i)\}^2]^{1/2}}{n - 1} \quad (3)$$

where *n* is the number of particles (*n* = 6). We obtained *S* = 0.0126. The accuracy of particle arrangement in Figure 3b,c was estimated by the same manner. Standard deviations of seven particles from left in an upper particle line and a bottom particle line in Figure 3b were estimated to be *S* = 5.66 × 10⁻³ and *S* = 3.84 × 10⁻³. The standard deviations of a bottom particle line, a middle particle line, and a bottom particle line in Figure 3c were estimated to be *S* = 8.11 × 10⁻⁴, 8.27 × 10⁻³, and 2.30 × 10⁻², respectively.

Precise Arrangement of Particles on Small-Area Silanol Sites Modified by AFM Lithograph. OTS-SAM was modified to silanol groups by an AFM (atomic force microscope, Nanoscope E, Digital Instruments) to control the position of arrangement accurately (Figure 5). A source measure unit (SMU model 236, Keithley) was installed in the AFM in order to control the electric current passing through the probe and a SAM. The SAM was biased positively; the AFM probe was scanned with constant current mode (50 nA), and the scanned area was used as a template for arrangement. The scanning area (100 nm × 100 nm) was set smaller than the diameter of the particles (500 nm diameter) to facilitate the precise arrangement of particles.

SiO₂ particles (500 nm diameter, powder, Admatechs Co., Ltd., SO-E2) modified with carboxyl groups were sonicated for 10 min in tetrahydrofuran or dichloromethane, and this solution was refrigerated to -20 °C for 1 h. *N,N*-Dicyclohexylcarbodiimide was added to this solution as a condenser^{39,40} to form ester bonds between carboxyl groups of SiO₂ particles and silanol groups of a SAM.³² Modified OTS-SAM was then immersed in this solution for 2 h. The temperature of the solution was increased slowly to 25 °C and kept for 2 h. After having been rinsed in water, a SAM was observed by a SEM. Particles were arranged in silanol regions, and the line of particle was fabricated (Figure 5). A two-dimensional arrangement with required features can easily be realized with this technique, though it takes a long time to modify a SAM with an AFM probe. Particles were not removed

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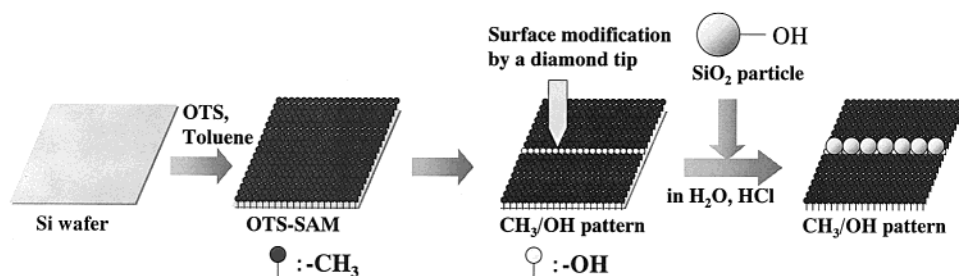


Figure 2. Conceptual process for fabrication of a particle wire on a patterned SAM modified by a diamond tip.

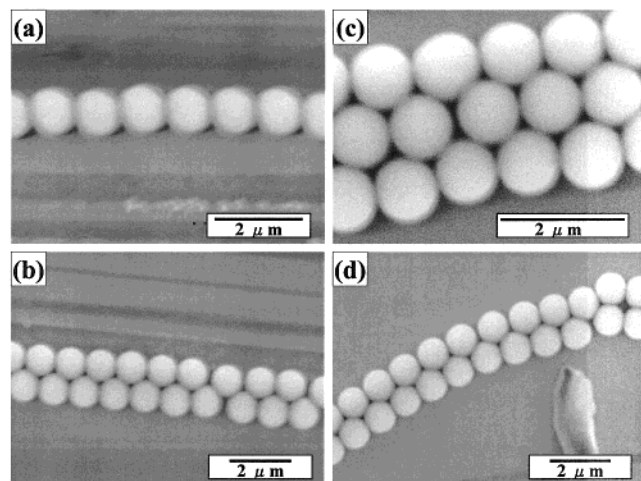


Figure 3. SEM micrographs of (a) a single particle wire, (b) a double particle wire, (c) a triple particle wire, and (d) a curved double particle wire.

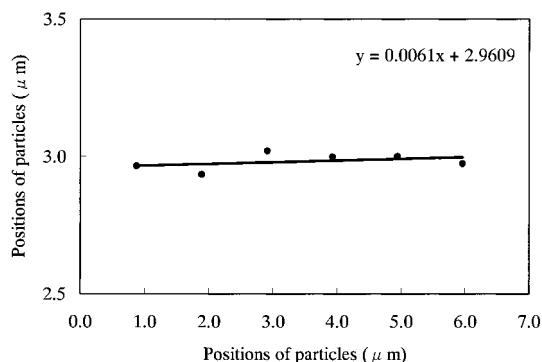


Figure 4. Positions of particles in Figure 3a showing the accuracy of particle arrangement.

easily from a SAM by sonication, indicating that ester bonds were formed by condensation. The accuracy of particle arrangement in Figure 5 was estimated to be $S = 1.17 \times 10^{-2}$. This might be decreased by decreasing the dimension of each silanol region.

To verify the formation of ester bonds between carboxyl groups and silanol groups, bromopropionic acid, whose molecule has a carboxyl group at one end and a bromo group at the other, was reacted with silanol groups of a Si substrate using the same reaction scheme as we used to attach SiO_2 particles to silanol groups. After having been sonicated in acetone for 5 min, the substrate surface was analyzed by X-ray photoelectron spectroscopy (XPS; ESCALAB 210, VG Scientific Ltd., $(1-3) \times 10^{-7}$ Pa, measurement area; $3 \text{ mm} \times 4 \text{ mm}$). The X-ray source (Mg K α , 1253.6 eV) was operated at 15 kV and 18 mA. The spectrum corresponding to Br 3d binding energy centering at 74.35 eV was observed.³² Although the observed binding

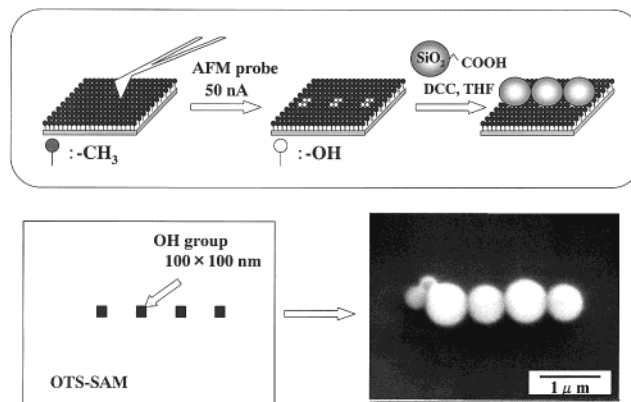


Figure 5. Conceptual process and SEM micrograph of particle arrangement on a patterned SAM modified by AFM lithography.

energy is higher than that of KBr, this chemical shift must have been caused by carbon atoms neighboring bromine atoms.⁴¹ Since bromo groups cannot react directly with silanol groups under the present conditions, the XPS result firmly indicates that carboxyl groups of bromopropionic acid reacted with silanol groups to form possible ester bonds.

Patterning of the Close-Packed Particle Monolayer. SiO_2 particles modified with carboxyl groups were dispersed in water. Octadecyl/amino groups patterned SAM was then immersed in the solution for several minutes (Figure 6). The substrate was rinsed with water and observed with a SEM. SiO_2 particles were observed in silanol regions, selectively forming a close-packed monoparticle layer (Figure 7a). Boundaries between the monoparticle layer and octadecyl region is clearly observable, and a few particles are observed in the octadecyl region. SiO_2 particles modified with carboxyl groups are charged negative, and amino groups of SAM are charged positive in water. Accordingly, particles are attracted to amino groups and form a monoparticle layer. Particles in the solution did not adhere to the monoparticle layer, since both the particles and the monoparticle layer have negative charges and repel each other. Particles were also deposited randomly in some areas (Figure 7b), and this suggests that it is difficult to obtain the pattern of the close-packed particle monolayer in a large area.

Additionally, octadecyl/amino groups patterned SAM was immersed in the solution containing nonmodified SiO_2 particles for several minutes (Figure 8). While SiO_2 particles were observed in silanol region predominantly, the feature edge acuity of the pattern was lower than that of the pattern in which SiO_2 particles modified with carboxyl groups were used. This demonstrates applicability of surface modification of SiO_2 particles with carboxyl

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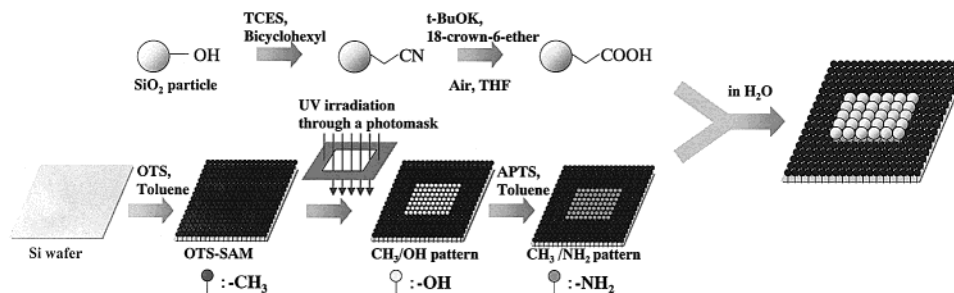


Figure 6. Conceptual process for patterning of the close-packed particle monolayer.

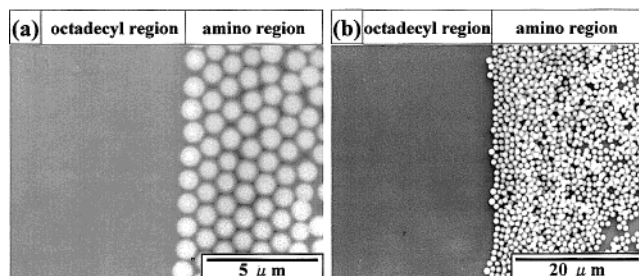


Figure 7. SEM micrographs of (a) a close-packed particle monolayer of SiO₂ particles modified with carboxyl groups formed in the amino region and (b) randomly deposited particles in the amino region.

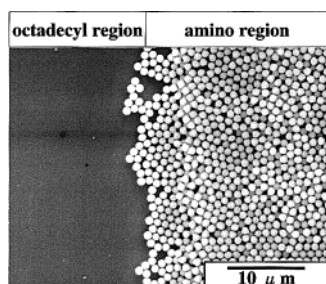


Figure 8. SEM micrograph of randomly deposited SiO₂ particles in the amino region.

groups. Furthermore, octadecyl/silanol groups patterned SAMs were immersed in solutions containing SiO₂ particles modified with carboxyl groups or nonmodified SiO₂

particles for several minutes, respectively. Particles were not adhered to either octadecyl groups or silanol groups. This means that the difference in surface potential between SiO₂ particles modified with carboxyl groups and amino groups of SAM accelerates the adhesion of particles to the amino groups. Particles were attracted and adhered to amino groups predominantly by electrostatic interactions between particles and SAMs, and chemical bonds were not formed because pure water was used as a solution with no condensation agent. The surface of SiO₂ particles modified with carboxyl groups and amino SAM must have changed into -COO^- and -NH_3^+ , respectively, in water to attract each other.

Conclusions

Surface modification of particles and SAM substrates was carried out to realize low-dimensional arrangement of SiO₂ particles. Microsized SiO₂ particles were arranged in silanol or amino regions selectively at room temperature by simply immersing the substrate in the solution. Precise arrangement of particles was achieved to form several kinds of particle wires, though it was difficult to arrange particles in a periodic manner in a large area. The arrangement technique we developed is considered to be promising for its potential applications to the future fabrication of electronic and photonic devices.

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