

Formation and thermal-induced changes of mesostructures in fluorinated organosilicate films

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Abstract

Mesoporous fluorinated organosilicate films were synthesized from tetramethoxysilane and perfluoroalkylsilanes (RO)₃Si-R' under acidic conditions in the presence of cationic surfactant or triblock copolymer by sol–gel spin-coating. The mesoporous fluorinated organosilicate films made from perfluoroalkylsilanes (PFASs) with long perfluoroalkyl chains, which acted as a template, displayed a hexagonal mesostructure with very low concentration of surfactants, regardless of the kind of surfactants. Although most of the surfactants and organic moieties were decomposed above 550 °C, the mesostructure of mesoporous fluorinated organosilicate films was maintained up to 650 °C. After calcination at 550 °C, their composition was similar to that of mesoporous silica films, and the cylindrical mesostructure was changed to less ordered and broken mesostructure when PFASs with long perfluoroalkyl chains were used. However, the cylindrical mesostructure was maintained when PFASs with short perfluoroalkyl chains were used. Therefore, PFASs with long perfluoroalkyl chains acted as a structure directing agent, and the chain length of PFASs affected the formation of mesostructure and thermal-induced mesostructural change. Also, the increase of calcination temperature caused the change of the composition, mesostructure, and optical property in the mesoporous fluorinated organosilicate films.

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1. Introduction

The mesoporous materials having uniformly controlled pore sizes are promising materials in such applications as catalyst supports, sensors, filtration membranes and in a variety of optoelectronic applications [1–3]. Especially, mesoporous silica thin films show application potentials in membrane separations, chemical sensors, optical devices, and electronic devices such as low-*k* dielectric films. Such mesoporous silica thin films can be formed by spin coating or dip coating in what is called evaporation induced self-assembly (EISA) [4–6]. By changing the ratio of surfactant to silica precursor solutions, the silica films were found to acquire lamellar, hexagonal, or cubic mesostructures

[7,8]. Furthermore, organic functional groups can be incorporated in the inorganic network by co-condensation method, yielding specific pore surface properties such as hydrophobicity, optical or electronic activity. Co-condensation of a tetraalkoxysilane and organoalkoxysilane with Si–C bonds was used to introduce organic functionality via sol–gel chemistry [9,10]. The co-condensation method allows a higher organic content and a more homogeneous organic distribution [11]. This method has been readily used for the synthesis of mesoporous organosilicate materials with varying organic functionalization.

Especially, the fluoro-containing groups remained covalently bonded to the mesoporous silica network, yielding materials with potential hydrophobic, adsorbing and optical properties [12]. In particular, the mesoporous fluorinated organosilicate films with low refractive index and high hydrophobicity are applicable for optoelectronic

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devices and low- k dielectrics. The hydrophobicity of the mesoporous films is an important factor in maintaining a reasonably low dielectric constant [13]. Thus, it is expected that the mesoporous fluorinated organosilicate films with high porosity and hydrophobicity might find practical applications in the manufacture of low- k dielectric material. In our previous study, using cationic surfactant (CTACl) it was found that mesoporous fluorinated organosilicate films with short perfluoroalkyl chains could display both hexagonal and cubic mesoporous structures by varying the mole ratio of surfactant to silane, which is similar to the results seen with mesoporous silica films [14]. However, the films with long perfluoroalkyl chains could display only hexagonal mesoporous structures. Furthermore, the role of PFASs with long perfluoroalkyl chains, which act as templates, was examined by changing the concentrations of surfactant across a broad range.

The mesoporous fluorinated organosilicate films with long perfluoroalkyl chain were also formed with triblock copolymer (Pluronic F68). This study examined the possibility of using PFASs with long chain as co-surfactants to form mesoporous organosilicate films, regardless of the kind of surfactant. However, the difference in used templates caused the changes in the range of mesostructure formation. It was found that these changes could result from the different interactions among hydrophilic silica groups, hydrophobic perfluoroalkyl groups and amphiphilic surfactant. Also, the mesostructural change in the films with increasing calcination temperature was studied with XRD and TEM. Moreover, the chain length of perfluoroalkyl group changed the mesostructure with increasing calcination temperature. The composition and optical property as well as the mesostructure of the films was changed with calcination temperature.

2. Experimental section

2.1. Preparation of the mesoporous fluorinated films

Mesoporous fluorinated organosilicate films were synthesized at room temperature from tetramethoxysilane (TMOS, Aldrich) and a functionalized perfluoroalkylsilane (PFAS) in the presence of a cationic surfactant, cetyltrimethylammonium chloride (CTACl, 25% in water, Aldrich) or PEO-PPO-PEO triblock copolymer, Pluronic F68 (PEO₇₇PPO₂₉PEO₇₇, 10% in water, Sigma). The following PFASs (RO)₃Si-R' were used: 3,3,3-trifluoropropyltrimethoxysilane [3-FPTMS, R' = (CH₂)₂CF₃, Fluka], tridecafluoro-1,1,2,2-tetrahydrooctyltriethoxysilane [13-FOTES, R' = (CH₂)₂(CF₂)₅CF₃, Avocado], and heptadecafluoro-1,1,2,2-tetrahydrodecyltriethoxysilane [17-FDTMS, R' = (CH₂)₂(CF₂)₇CF₃, Toshiba]. PFAS was hydrolyzed in distilled water containing 0.01 N hydrochloric acid (HCl, J.T. Baker) as a catalyst for the sol-gel reaction. To avoid unwanted precipitation of hydrophobic organoalkoxysilane, PFAS was reacted with weak HCl. TMOS was added to the hydrolyzed PFAS solution in a molar ratio

of 9:1. The mixed solution was hydrolyzed under acidic conditions and then methanol (CH₃OH, Merck) was added. Finally, CTACl was added to achieve the final reactant mole ratios of 0.9 TMOS:0.1 PFAS:8–16H₂O:0.0005–0.0006HCl:18–30CH₃OH:0.0038–0.25CTACl [14]. When triblock copolymer Pluronic F68 was used as templates, mesoporous silica films were formed over a range of reaction compositions, for example, 1 TMOS:30–50H₂O:0.09–0.11HCl:6–20CH₃OH:0.006–0.01 Pluronic F68 in the molar ratio [15]. However, 17-FDTMS organosilicate mesoporous films with long perfluoroalkyl chains could be grown at room temperature with small contents of templates on substrates when the composition of final gel was 0.9TMOS:0.117-FDTMS:8–16H₂O:0.0005–0.0006HCl:18–30CH₃OH:0.0006–0.0024 Pluronic F68. After the addition of templates, the solution was allowed to react for another 24 h at room temperature to oligomerize. To remove impurities and gas bubbles, the transparent solution was filtered through a 0.22 μm-size Teflon filter. This solution was then deposited onto p-type Si(100) wafers and quartz substrates by spin coating at 2000 rpm for 30 s.

The organosilicate-surfactant composite thin films were pre-dried at 60 °C, followed by 130 °C and then 200 °C for 1 h at each stage, using the increasing temperature to remove the solvent, water, and organic template, respec-

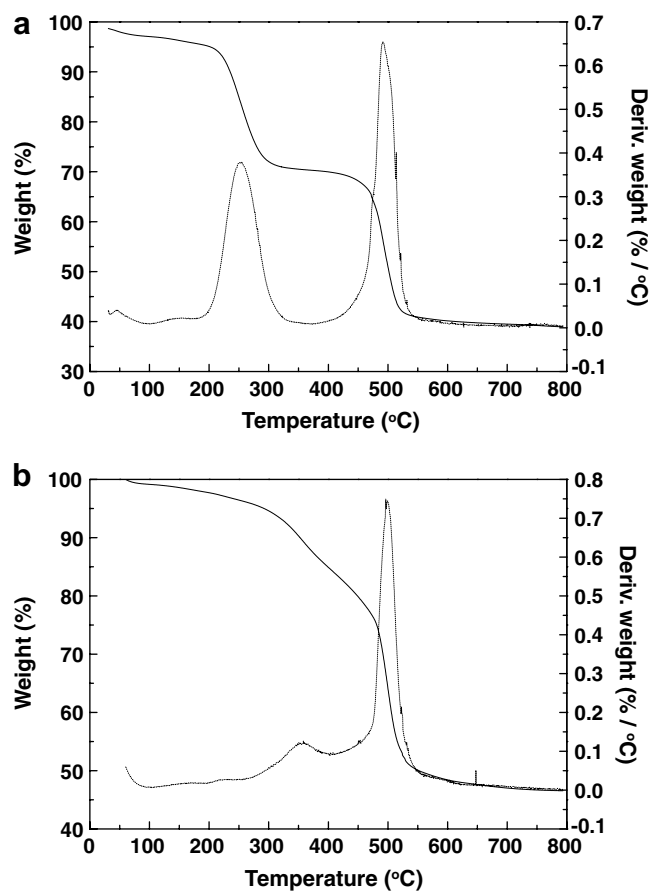


Fig. 1. TGA curves of 17-FDTMS organosilicate materials made from (a) CTACl and (b) Pluronic F68.

tively. Such a cumulative heating procedure that evaporates excess solvents and decomposes surfactants produced structurally stable mesoporous silica films [16]. The pre-dried films were calcined in flowing air at 350 °C to burn off CTACl or at 400 °C to burn off Pluronic F68 for 12 h, with the temperature increased at the rate of 1 °C/min to fully remove the organic template. The final calcination temperature was set from the TGA results (Fig. 1).

2.2. Characterization of the mesoporous fluorinated films

To confirm the ordered structure, the as-synthesized and calcined films, deposited on silicon wafer substrates, were subjected to X-ray diffraction (XRD) measurements with CuK α radiation (40 kV, 80 mA) with a 0.01° step width and 1 s step on a Rigaku D/MAX-RC diffractometer. A JEOL JEM-2000EX electron microscope operating at 200 kV recorded the TEM micrographs of the cross-section of the calcined films. Cross-sectional film samples on Si wafers were prepared by placing films directly onto an epoxy resin substrate. Cross-sectional slices were cut using ultramicrotome and deposited on a copper grid. Film thickness was also measured by scanning electron microscopy (SEM, Philips, XLSFEG). Thermal analyses using thermogravimetric analysis (TGA) were performed under a nitrogen flow using a Dupont Model 2200 thermogravimetric analyzer at a heating rate of 5 °C/min in the temperature range of 30–800 °C. Infrared spectra were collected on a Bruker EQUINOX55 Fourier transform infrared spectrometer in the range of 400–4000 cm⁻¹ with a resolution of 4 cm⁻¹. Quantitative analysis of the mesoporous film was performed by auger electron spectroscopy (AES, SAM 4300) at 5 kV. The contaminant on the sample surface was removed by sputtering of Ar ions before the clean surface was investigated. To quantify the refractive index and thickness of the films at $\lambda = 632.8$ nm for 70° incidence angle, an AXIC SE500 ellipsometer (Sentech) was used.

3. Results and discussion

3.1. Formation of mesostructure using Pluronic F68 in the fluorinated organosilicate films

Triblock copolymer, Pluronic F68 has a known critical micelle concentration (cmc) value of 0.48 g/mol which makes an ordered structure [17]. Typically, bulk mesophases are made when initial surfactant concentration is larger than cmc. However, homogeneous mesoporous films are formed by evaporation-induced self-assembly [6]. Mesoporous silica films in previous research were formed with the Pluronic F68 concentrations under the cmc [15]. However, mesoporous 17-FDTMS organosilicate films were formed when the Pluronic F68 concentration was much lower than the concentration needed to form mesoporous silica films. Fig. 2 shows the XRD patterns of the as-synthesized and the calcined mesoporous fluorinated organosilicate films with different surfactant concentrations. To

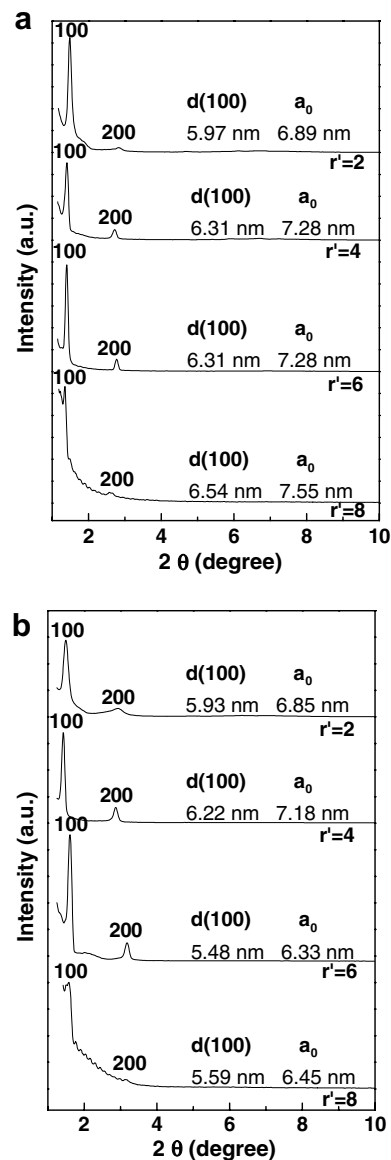


Fig. 2. XRD patterns of 17-FDTMS organosilicate mesoporous films made from Pluronic F68 with different surfactant concentrations (a) as-synthesized and (b) after calcined films. ($r' = [\text{Pluronic F68}]/[\text{TMOS}] \times 1680$.)

avoid decimal numbers and easily compare the surfactant/silanes mole ratios, r' was set to $[\text{Pluronic F68}]/[\text{TMOS}] \times 1680$. These data indicate that hexagonal mesoporous structures exist in all the films formed on the Si substrate. The XRD patterns showed a prominent peak at $2\theta = 1.0\text{--}2.0^\circ$ and some broad peaks at $2\theta = 2.0\text{--}4.0^\circ$ that are characteristic of the hexagonal structure. These were similar to the XRD of hexagonal MCM-41 materials [18,19] (Fig. 2(a) and (b)). According to the XRD patterns, the mesoporous structure is retained even after Pluronic F68 was decomposed by calcination at 400 °C in flowing air for 12 h. The unit cell parameter (a_0) of the hexagonal structure was calculated from $d(100)$ according to $(a_0) = 2d(100)/3^{1/2}$ where $d(100)$ was obtained from the

2θ value of the first peak in the XRD pattern, from $d(100) = \lambda/2\sin\theta$ where $\lambda = 0.15417$ nm for the Cu K α line [20]. The XRD of the calcined mesoporous organosilicate film was qualitatively similar to that prior to calcination except for a decrease in $d(100)$ spacing. The $d(100)$ spacing decreased about 0.04–0.83 nm in calcined samples as opposed to the as-synthesized samples. The unit cell parameter a_0 is equal to the sum of the internal pore diameter and one pore wall thickness [21].

The mesoporous silica films had a mesoporous structure over $r' = 20$ under the same processing conditions with Pluronic F68 (Fig. 3). However, hexagonal mesoporous fluorinated organosilicate films were formed at $r' = 2$ –8. PFASs with a hydrophilic silanol head group and long hydrophobic perfluoroalkyl chains are amphiphilic in nature, as were other templates [22]. Thus, it was reported that PFASs acts as co-surfactants. From previous research, it was confirmed that PFASs also act as a structure directing agent when the reactant is mixed with a small amount of surfactant (CTACl) [14]. PFASs with long perfluoroalkyl chain also acted as templates when the small concentration of template, triblock copolymer (Pluronic F68) was added. Also, the 17-FDTMS fluorinated organosilicate films with long perfluoroalkyl chains assumed a hexagonal mesostructure regardless of Pluronic F68 concentration. This result is caused by the reduction of micellar curvature on the incorporation of PFASs with long perfluoroalkyl chain into the hydrophobic interior of micelle [14].

TGA of the as-synthesized fluorinated samples showed weight losses near 60 °C, 260 °C and 500 °C. These loss peaks indicate the desorption of solvent and decomposition of CTACl and perfluoroalkyl chains, respectively. However, weight loss peak of triblock copolymer, Pluronic F68 appeared near 350 °C. For full decomposition of the surfactant and for protection of decomposition of the perfluoroalkyl chains, the calcination temperature of fluorinated films made from Pluronic F68 was set at around 400 °C. This temperature was taken from the TGA results.

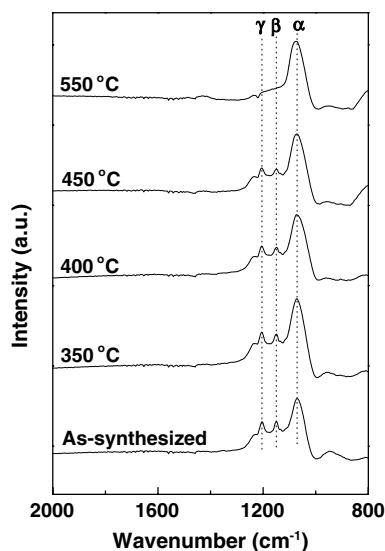


Fig. 4. FT-IR absorption spectra of mesoporous 17-FDTMS organosilicate films made from Pluronic F68. The y -axis shows the relative intensities of the absorption coefficient.

Fig. 4 shows the FT-IR spectra of the as-synthesized mesoporous 17-FDTMS organosilicate film and the samples heated at 350 °C, 400 °C, 450 °C, and 550 °C. The band appearing at around 1072 cm^{-1} (α) was associated with Si-O-Si asymmetric bond stretching [23]. The bands at 1146 and 1206 cm^{-1} were assigned to C-H bending and C-F stretching modes in the perfluoroalkyl chains (β and γ) [24]. Perfluoroalkyl groups appeared to be maintained in the spectra after calcination at 450 °C. However, the calcination at 450 °C allowed the weight loss of perfluoroalkyl groups from TGA results. Thus, to protect the decomposition of perfluoroalkyl groups covalently bonded to Si atoms and form the mesoporous fluorinated organosilicate films, the films were calcined at 400 °C.

After the surfactant was removed, the thickness of the mesoporous fluorinated organosilicate films with CTACl and Pluronic F68 was about 500 nm and 150 nm, respectively. The calcined mesoporous fluorinated organosilicate films had over 90% optical transmission through quartz without cracks. These results showed the application possibility for the optical and electronic applications.

3.2. Mesostructure formation mechanism in fluorinated organosilicate films

Mesoporous organosilicate films were made from 17-FDTMS and two different types of surfactants. Hexagonal mesoporous 17-FDTMS organosilicate films made from CTACl surfactants were formed using 1/50 of the surfactant concentrations necessary for the formation of hexagonal mesoporous silica films (Fig. 3) [14]. To avoid decimal numbers and easily compare the surfactant/silanes mole ratios, r was set to $[\text{CTACl}]/[\text{TMOS}] \times 263$. Hexagonal mesoporous 17-FDTMS organosilicate films made from

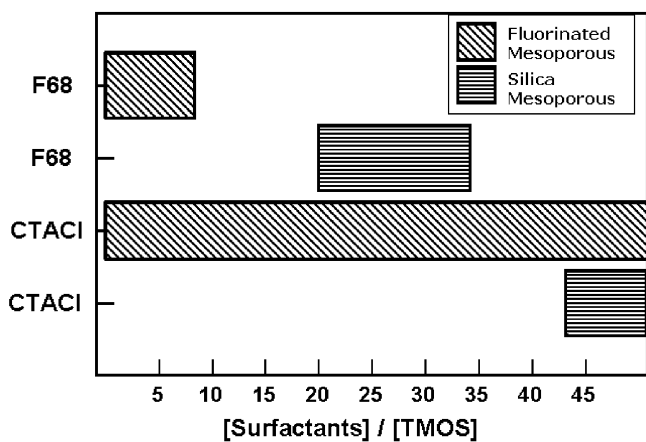
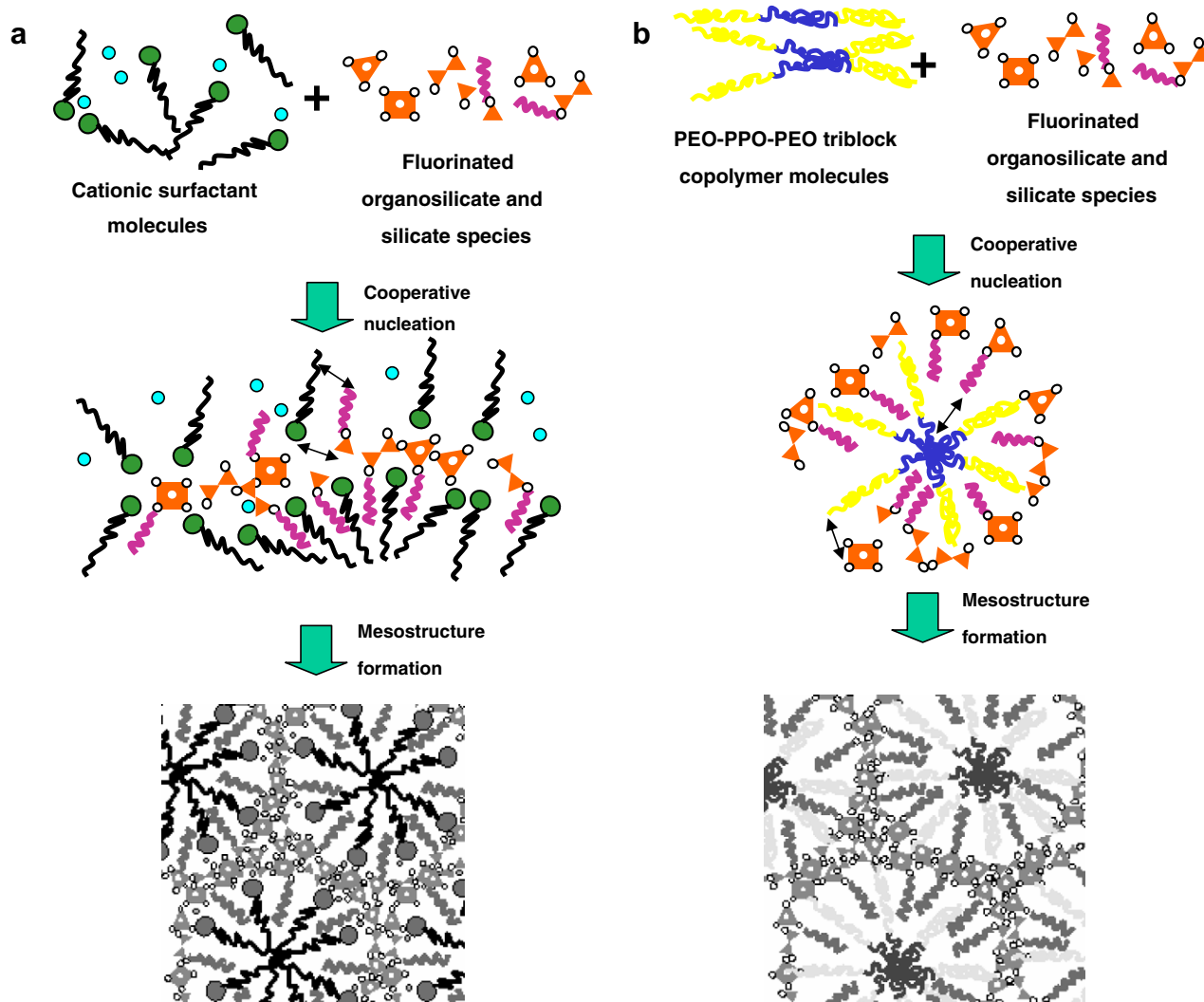


Fig. 3. The mesostructures of products at various $[\text{CTACl}]/[\text{TMOS}]$ ($\times 263$) ratios and $[\text{Pluronic F68}]/[\text{TMOS}]$ ($\times 1680$) ratios.

Pluronic F68 triblock copolymers were formed using 1/20 of the surfactant concentrations necessary for the formation of mesoporous silica films. These results showed that PFASs with long perfluoroalkyl chain acted as templates. However, the fluorinated organosilicate films made from Pluronic F68 were not formed over the r' value when the mesoporous silica films formed. The fluorinated organosilicate mesoporous films made from Pluronic F68 were just formed with the low concentration of Pluronic F68. (Fig. 3) The difference in the mesostructure formation range in the fluorinated organosilicate films made from two different kinds of structure directing agents came from the different micelle formation mechanism. Cationic surfactant, CTACl have electrostatic interaction with anionic silicate and fluorinated organosilicate species and hydrophobic interaction with fluorinated organosilicate species [25,26]. Furthermore, many previous researches for inorganic–organic hybrid mesoporous materials have shown that the organic groups tend toward the pore

(Scheme 1) [27,28]. When the surfactant concentration is very low, the PFASs with long perfluoroalkyl chain act as co-surfactants. When the surfactant concentration increases, the hydrophobic interaction of hydrophobic alkyl chain groups of CTACl and hydrophobic perfluoroalkyl chain groups of PFAS increases [11,29]. Thus, the increase of surfactant concentration caused the decrease of the $d(100)$ spacing value of the films [14].

However, Pluronic F68 is composed of two long hydrophilic chain groups and one hydrophobic chain group between them. Pluronic F68 forms hydrophobic PPO core and hydrophilic PEO shell when a spherical micelle is formed. In the presence of nonionic amphiphilic di-block copolymer templates, there are three possibilities to form mesostructure in the surfactant/inorganic composite materials [30–32]. First, the PEO forms a pure PEO layer at the interface with the hydrophobic block and inorganic blocks (“three-phase system”). Second, the transition state is from “two-phase” structure to “three-phase” structure. Third,



Scheme 1. Formation model of the fluorinated mesoporous organosilicate materials containing PFASs with long perfluoroalkyl chain. The materials are made from (a) cationic surfactants and (b) PEO-PPO-PEO triblock copolymers.

the PEO chains are homogeneously dissolved in the aqueous solution of the inorganic precursor (“two-phase system”). During EISA, evaporation and the incipient stages of siloxane condensation cause correspondingly a change from “two-phase” to “three-phase” structure [33,34]. Also, the long perfluoroalkyl chains tend toward hydrophobic PPO core and two hydrophilic groups co-exist with perfluoroalkyl chain in the mesoporous fluorinated organosilicate films made from Pluronic F68. When the concentration of triblock copolymer template is very low, long perfluoroalkyl chains tend toward to the PPO core and acted as co-surfactants. As triblock copolymer concentration increases, the repulsion of hydrophilic blocks and hydrophobic perfluoroalkyl chain increases rapidly. Therefore, the fluorinated organosilicate mesoporous films made from triblock copolymer were formed when triblock

copolymer concentration was very lower than that of necessary for the formation of the mesoporous silica films made from the same template.

3.3. Thermal-induced change of mesostructure in fluorinated organosilicate films

From our research on the fluorinated organosilicate films made from CTACl or Pluronic F68, the organosilicate films were formed after calcination at 350 or 400 °C. As calcination temperature increased over 450 °C, the perfluoroalkyl chains were decomposed. Thus, the fluorinated organosilicate films with increasing calcination temperature showed their structural and compositional change. First, the XRD patterns of 3-FPTMS and 17-FDTMS organosilicate mesoporous films using CTACl with increas-

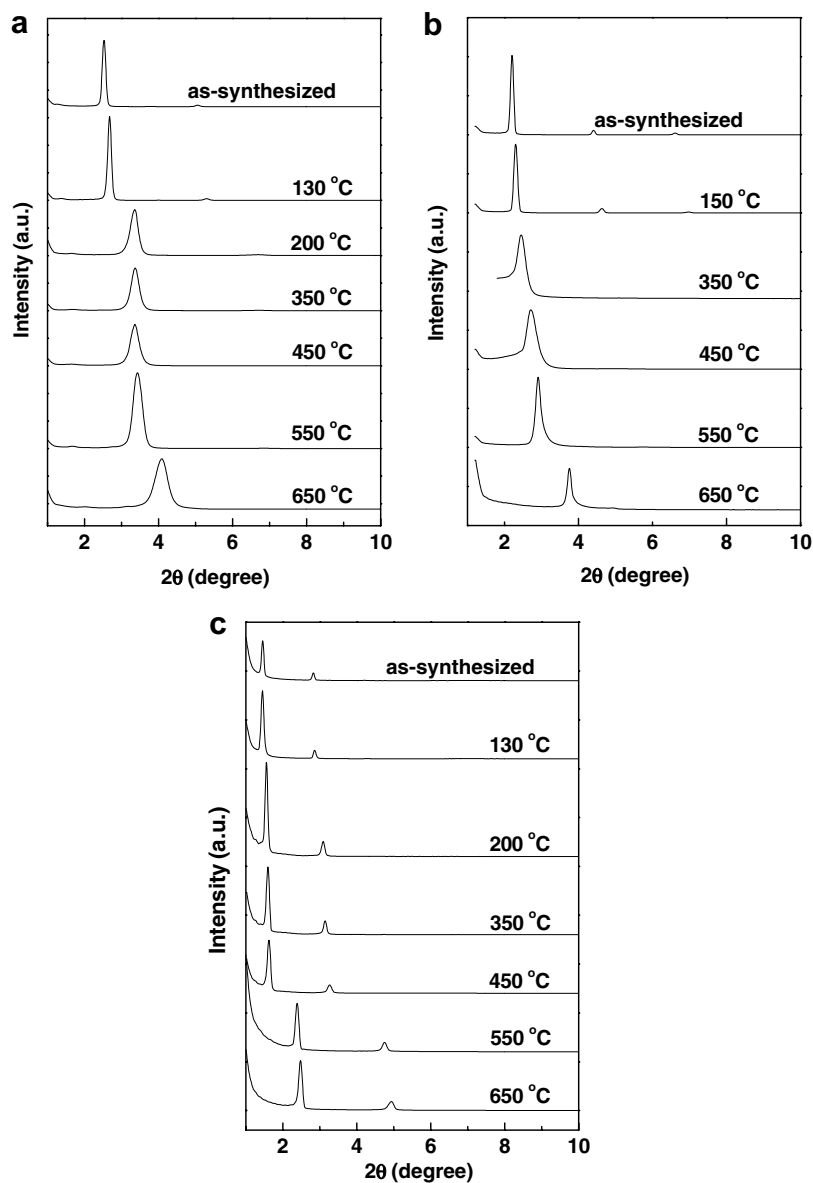


Fig. 5. XRD patterns of organosilicate mesoporous films after being subjected to heat treatment in flowing air for 12 h at increasing temperature using (a) 3-FPTMS/CTACl (b) 17-FDTMS/CTACl and (c) 17-FDTMS/F68.

ing calcination temperature were qualitatively similar to that prior to calcination except for a substantial decrease in $d(100)$ spacing (Fig. 5). The $d(100)$ spacing decreased because of the gradual removal of surfactants and the condensation of silanol groups [16]. The broadening of the $d(100)$ peak on calcination suggests a less structural ordering in the film after the surfactant decomposition. The mesostructures were retained even after the decomposition of perfluoroalkyl groups by calcination at 650 °C in flowing air for 12 h. The mesoporous 3-FPTMS and 17-FDTMS organosilicate films showed a hexagonal mesostructure independent of calcination temperature from the XRD results. The mesoporous 17-FDTMS organosilicate films

made from Pluronic F68 showed similar XRD results with increasing calcination temperature. Because the films were made with low concentration of surfactants, the structure did not shrink despite the decomposition of surfactants. However, the abrupt $d(100)$ spacing change occurred after the calcination at 550 °C because of the decomposition of perfluoroalkyl chain groups.

The ordered structure of the 3-FPTMS calcined film having the short perfluoroalkyl chain group with CTACl was further confirmed by the TEM in Fig. 6. Calcination at 350 °C allowed the removal of surfactant without destroying the organic contents. The films had cylindrical pores with a pore size similar to the mesoporous silica

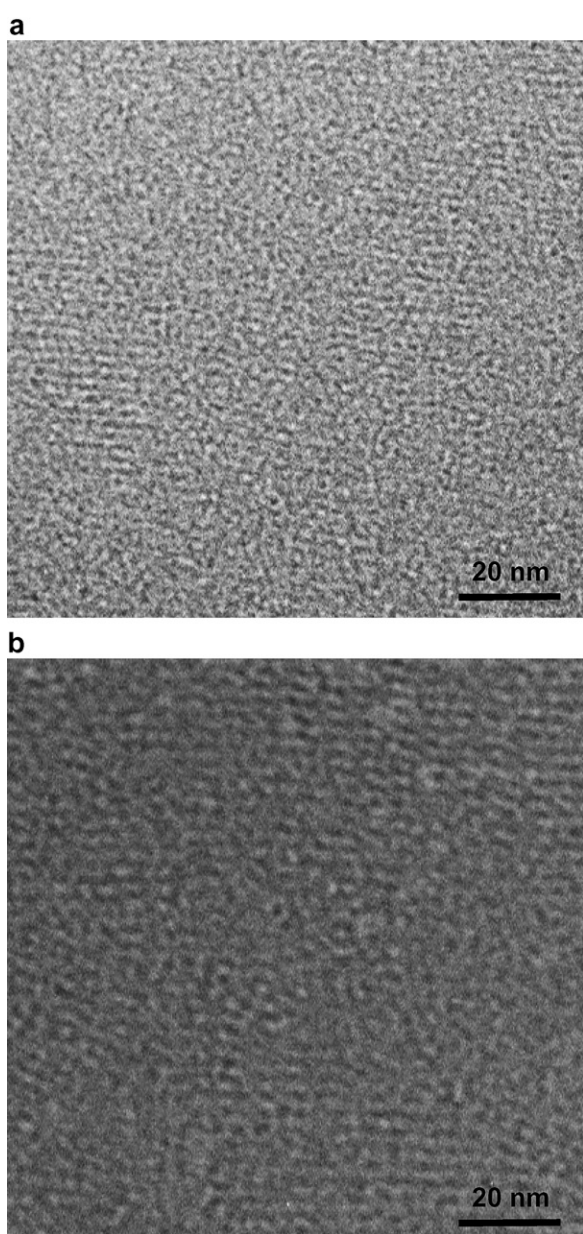


Fig. 6. TEM cross-sectional images of a hexagonal mesoporous 3-FPTMS organosilicate films using CTACl after calcination at (a) 350 °C and (b) 550 °C.

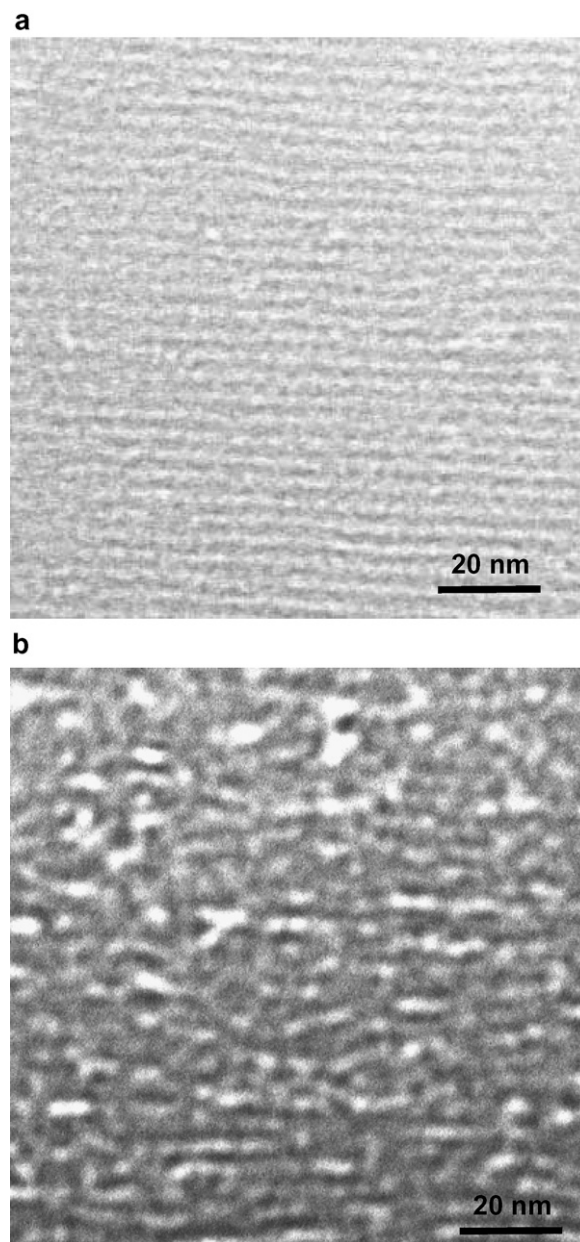


Fig. 7. TEM cross-sectional images of a hexagonal mesoporous 17-FDTMS organosilicate films using CTACl after calcination at (a) 350 °C and (b) 550 °C.

materials made from CTACl. After calcination at 550 °C, the fluoro-containing groups disappeared, and the films with more consolidated silica walls and the same cylindrical pores were formed. In contrast, the ordered structures of the mesoporous films containing 17-FDTMS with CTACl demonstrated the presence of channels after calcination at 350 °C (Fig. 7). TEM confirmed the existence of highly oriented hexagonal mesoporous 17-FDTMS organosilicate films. After calcination at 550 °C, the long fluoro-containing groups disappeared, and a less ordered mesoporous structure was formed. The decomposition of long perfluoroalkyl chains caused the broken mesostructure. The mesostructure of 3-FPTMS organosilicate films were independent of calcination temperature. However, the mesostructure of 17-FDTMS organosilicate films were changed to the less ordered and broken mesostructure with calcination temperature. These findings led us to conclude that the difference in mesostructure after calcination at high temperature resulted from the decomposition of perfluoroalkyl chain with different length at 550 °C.

Fig. 8 shows the changes in the relative atomic ratios of the fluorinated organosilicate mesoporous films made from CTACl or Pluronic F68 after calcination at different temperatures, and they were investigated by auger electron spectroscopy (AES). After calcination at 350 °C, the atomic ratio of carbon to silicon decreased rapidly in both films. The decreases is related to the decomposition of the organic alkyl group of CTACl or the decomposition of the organic PEO and PPO group of Pluronic F68. After calcination at 550 °C, the atomic ratios of carbon to silicon and fluorine to silicon decreased rapidly. The decreases in the relative amounts of carbon and fluorine are related to the decomposition of the organic perfluoroalkyl group. Thus, after calcination at 550 °C, the final composition of fluorinated organosilicate film was similar to pure silica materials. As a result, the mesoporous silica films were formed from the fluorinated organosilicate mesoporous films after calcination at 550 °C. After 550 °C calcination, the final composition of silica material was independent of the length of perfluoroalkyl chain and the templates used but the mesostructure of the fluorinated organosilicate films was dependent on the chain length of perfluoroalkyl groups.

The mesoporous silica materials have a very low refractive index because of their high porosity. However, the fluorinated organosilicate films are expected to have a refractive index much lower than the mesoporous silica materials. The refractive index of the films decreases with fluorine content [14]. Thus, 17-FDTMS organosilicate mesoporous films made from CTACl have the lowest value after calcination at 350 °C (Fig. 9). The films have the fluorine content and high porosity after the surfactant removal. As calcination temperature increases, the refractive index of the films increases owing to the decomposition of perfluoroalkyl chain groups and the condensation of the silanol groups. The mesoporous fluorinated organosilicate films after calcination at 550 °C have higher refractive index than the films after calcination at 350 °C.

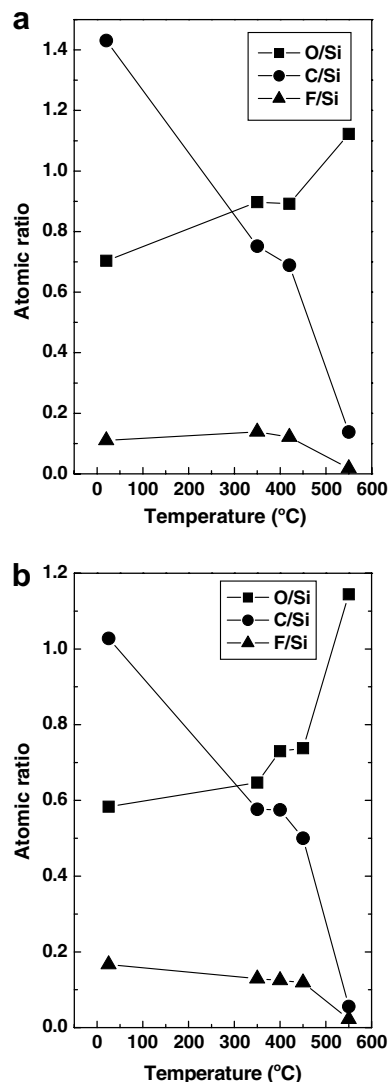


Fig. 8. Auger electron spectroscopy (AES) of the 17-FDTMS organosilicate mesoporous films monitoring the change of relative atomic ratios upon calcinations temperature made from (a) CTACl and (b) F68.

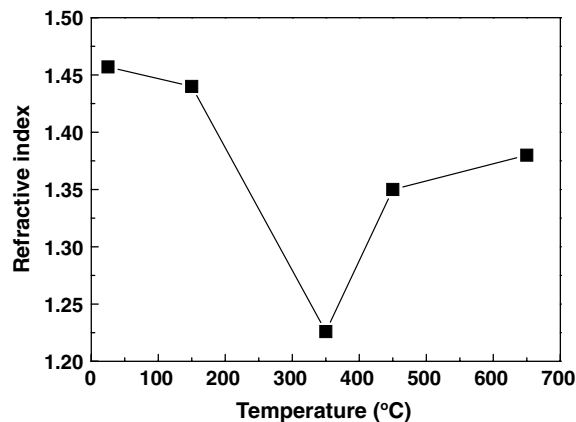


Fig. 9. Change in refractive index of 17-FDTMS organosilicate mesoporous films made from CTACl with calcinations temperature in flowing air conditions.

4. Conclusions

Ordered mesoporous fluorinated organosilicate films were formed with co-condensation of TMOS, PFAS with long perfluoroalkyl chains, and surfactants. Triblock copolymer, Pluronic F68 or cationic surfactant, CTACI were used to form mesostructures. Mesoporous 17-FDTMS fluorinated films with long perfluoroalkyl chains had an ordered hexagonal mesostructure even when 1/20 of the Pluronic F68 concentration needed to form mesoporous silica films was used. Cationic surfactants, CTACI have electrostatic interaction with anionic silicate and fluorinated organosilicate species and hydrophobic interaction with fluorinated organosilicate species. When the surfactant concentration is very low, the PFASs with long perfluoroalkyl chain act as co-surfactants. Unlike the films made from cationic surfactant, the mesoporous films were just formed with low concentration of triblock copolymer because of the increase of repulsion between hydrophobic perfluoroalkyl chain groups and hydrophilic PEO groups with increasing template concentration. PFASs with long chains may be used as co-surfactants in forming the mesoporous organosilicate films. The fluorinated organosilicate mesoporous films maintained mesoporous structure even after calcinations at 650 °C. However, there is a difference in the structural changes of mesostructure after calcinations at 550 °C depending on whether 3-FPTMS or 17-FDTMS is used as the organic source. As temperature increased up to 550 °C, most organic templates and perfluoroalkyl chains were decomposed. Thus, the fluorinated organosilicate films with long perfluoroalkyl chains were changed to the less ordered and broken mesoporous silica films but cylindrical pores of the films with short perfluoroalkyl chains were maintained. Furthermore, the optical property of the fluorinated films can be controlled with calcination temperature. Therefore, the increase of calcination temperature results in the changes of the composition, mesostructure, and optical property in the mesoporous fluorinated organosilicate films.

Acknowledgments

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