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Laser-induced thin film formation from a gaseous mixture of trimethylsilylacetylene and methyl acrylate

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Abstract

Under irradiation with N_2 laser light, gaseous trimethylsilylacetylene (TMeSiA) and a gaseous mixture of TMeSiA and methyl acrylate (MA) produced thin films on an incident optical quartz window of an irradiation vessel. The product yield decreased with increasing partial pressure of TMeSiA. From the analysis of FT-IR spectra and X-ray photoelectron spectra of the deposited films, it was shown that Si–C bond of TMeSiA was cleaved by a two-photon absorption of N_2 laser light to produce trimethylsilyl radical and the silanes, $R(CH_3)Si{=}CH_2$ (R: H_3C–, CH≡C–). Trimethylsilyl radical reacted with MA at C=C and C=O bonds to produce Si–C and Si–O bonds in the film deposited from the gaseous mixture. The silanes were polymerized in each other to produce a thin film from pure TMeSiA vapor. Under irradiation with a medium pressure mercury lamp, a gaseous mixture of TMeSiA and MA produced aerosol particles in addition to the film. Polymeric species of TMeSiA was more involved in the aerosol particles rather than in the film, suggesting that collisionally induced chemical reaction takes place efficiently between excited MA and TMeSiA molecules. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: N_2 laser-induced film formation; Trimethylsilylacetylene; Methyl acrylate; Two-photon process; Silanes

1. Introduction

Under irradiation with N_2 laser light, gaseous methyl acrylate (MA) (2-propenoic acid methyl ester) produced a thin film on an incident optical window by a two-photon process [1]. Laser synthesis of organic thin films was further developed to efficiently synthesize a composite film from a gaseous mixture of MA and carbon disulfide [2,3]. On the other hand, aerosol particles were successfully synthesized from gaseous acrolein (2-propenal) (AC) by a two-photon process under irradiation with N_2 laser light [4], and it was found that AC reacted with foreign gaseous molecules during polymerization reaction to produce composite particles [5–8]. Some organosilicon compounds such as trimethyl(2-propynyloxy)silane (TMPSi) and allyltrimethylsilane (ATMeSi) were incorporated into chemical reactions with AC, and chemical analysis of the sedimentary particles revealed that organosilicon compounds were involved into the aerosol particles as several different chemical species such as $(Si)_n$, SiO_2, and silacyclobutane, depending on the chemical reactivity of the parent organosilicon compounds [9,10].

As one of the reactive organosilicon compounds, gaseous trimethylsilylacetylene (TMeSiA) (ethynyltrimethylsilane) was irradiated with ArF excimer laser light (193 nm), and polymerized at the C≡C bond in the absence of photoinitiators. The chemical vapor deposited (CVD) film did not involve C=C double bonds, and was composed of $-((CH_3)_3Si{-}C{-}C)-$ units [11]. TMeSiA has the longest wavelength absorption band shorter than 200 nm. In order to synthesize a film of TMeSiA under irradiation with N_2 laser light, chemical reaction of TMeSiA must be initiated by a two-photon process or through photochemical reaction of foreign gaseous molecules which absorb 337.1 nm light.

In the present paper, we have prepared a thin film efficiently under irradiation with N_2 laser light using MA as a foreign gas. A gaseous mixture of TMeSiA and MA was irradiated with N_2 laser light, and chemical structure of the deposited film was investigated by FT-IR and X-ray photoelectron spectra (XPS) to reveal chemical reactivity of TMeSiA with MA. Thin film formation from pure TMeSiA vapor by a two-photon process was also studied, and compared to the results of ArF excimer laser light irradiation.

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2. Experimental details

MA (Wako, 98%, stabilized with 0.03% hydroquinone monomethyl ether) and TMeSiA (Tokyo kasei, 98%, G.R. grade) were stored in a refrigerator below 10°C to prevent polymerization. Each of the TMeSiA and MA liquid was degassed repeatedly (three times) by freeze-pump-thaw cycles and distilled under the vacuum immediately before use. To prepare a gaseous mixture of TMeSiA and MA, a fixed amount of MA vapor was collected in a glass tube by freezing with liquid nitrogen through a vacuum line equipped with a capacitance manometer (Edwards Barocel Type 600), and then another fixed amount of TMeSiA vapor was collected over the frozen MA liquid in the glass tube. After thawing them into a mixed liquid, vapor of the mixed liquid composed of TMeSiA and MA was introduced into a cylindrical cell (inner diameter: 35 mm, cell length: 155 or 165 mm) with quartz optical windows on both ends, or into a cross-shaped irradiation cell with four optical quartz windows (inner diameter of long arm: 35 mm, inner diameter of short arm: 20 mm, long arm: 155 mm, short arm: 80 mm). The background pressure of the irradiation cell was $<5 \times 10^{-5}$ Torr (1 Torr = 133.3 Pa) and the leakage into the evacuated cell from the atmosphere was carefully controlled to result in pressure increase lower than 0.5 Torr in a day. The partial pressure of MA in the irradiation cell was determined by measuring UV absorption at 242 nm. The gaseous samples were irradiated with nitrogen gas laser light (Lumonics HE-440, 337.1 nm) at a repetition rate of 12 Hz, or with a medium pressure mercury lamp (Ushio UM-452, 450 W). Laser power was measured with a pyroelectric joulemeter (Gentec ED-500) combined with a synchroscope (Iwatsu SS-7810). FT-IR spectra of deposits were measured with a Nicolet 740 FT-IR spectrometer, and XPS were measured with a Gammadata Scienta ESCA 310 electron spectrometer equipped with rotating anode, monochromator and electron flood gun. Light intensity scattered perpendicularly to the incident monitor (He–Ne laser) light by the aerosol particles which were produced under irradiation with a mercury lamp was measured with a combination of a photomultiplier tube (Hamamatsu 1P28) and a lock-in amplifier (SRS SR-530) through a monochromator (Ritsu MC-10N).

3. Results and discussion

Upon exposure to N_2 laser light at an energy of 2.2 mJ/pulse for 5 h, pure TMeSiA vapor (50 Torr) deposited a transparent thin film on an optical quartz window. The film became thick in the light-exposed area of the quartz window. TMeSiA vapor has the longest wavelength absorption band shorter than 200 nm and its absorption tail extended only up to 230 nm. The fact that TMeSiA has practically no absorbance at 337.1 nm implies that the film formation process was initiated through a photochemical reaction induced by a two-photon absorption. This conclusion was further supported by the observation that pure TMeSiA vapor did not produce any deposit under light irradiation with a medium pressure mercury lamp (12 mJ cm^{-2} s^{-1}) which can induce only a one-photon process in the wavelength region longer than 253 nm.

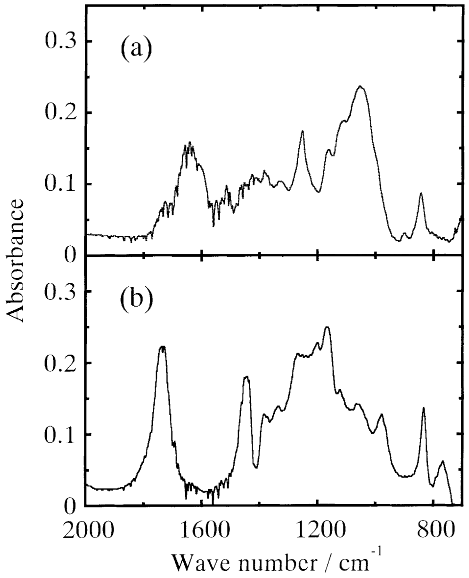


Fig. 1. FT-IR spectrum of the film deposited from: (a) pure TMeSiA vapor at a pressure of 50 Torr; (b) pure MA vapor at a pressure of 50 Torr under irradiation with N_2 laser light at an energy of 2.2 mJ/pulse for 5 h.

FT-IR spectrum of the film deposited under irradiation with N_2 laser light (Fig. 1(a)) showed a strong band at 1050 cm^{-1} and a moderate band at 1650 cm^{-1} besides the bands (αa(HCH) band at 1421 cm^{-1}, bending vibrational band of Si–CH$_3$ at 1250 cm^{-1}, a blend of ν(Si–CH$_3$) and β(SiCH) bands at 837 cm^{-1}) observed with TMeSiA monomer [12]. The 1650 cm^{-1} band was assigned to C=C stretching vibrational band. The stretching vibrational band of the C≡C bond got lost in the deposited film, and this indicated that a part of TMeSiA molecules was polymerized at the C≡C bond without decomposing into fragments. The 1050 cm^{-1} band was not observed in poly(trimethylsilylacetylene) which was synthesized using WCl$_6$-based catalyst and had a conjugated polyene structure [13,14]. Polymerization of TMeSiA was also reported under a glow discharge [15] and under irradiation with ArF excimer laser light at 193 nm [11]. In both cases, IR spectra of the deposited films showed a broad moderate/weak band at ~1045 cm^{-1} assigned to the Si–O stretching vibrational band. In the present case, the 1050 cm^{-1} band was strong in intensity, and can be assigned to Si–CH$_2$–Si stretching band [16,17], indicating that the two-photon process of N_2 laser light dissociated TMeSiA molecule at Si–C bond into trimethylsilyl radical and C$_2$H radical, and that silanes R(CH$_3$)Si=CH$_2$ (R: H$_3$C–, CH≡C–) were produced by disproportionation between two silyl radicals [18,19]. The silanes are known to photopolymerize to

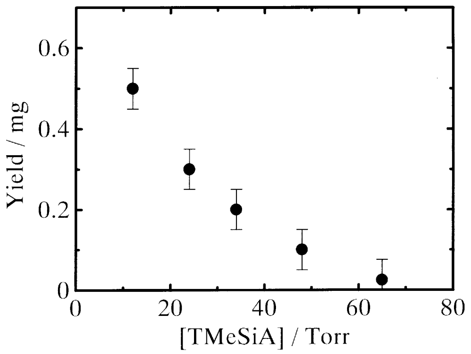


Fig. 2. The yield of the film deposited from gaseous mixtures of TMeSiA (12–65 Torr) and MA (30 Torr) under irradiation with N_2 laser light at an energy of 2.2 mJ/pulse for 5 h.

$-(R(CH_3)Si{-}CH_2)_n-$ polymers in the gas phase to form $Si{-}CH_2{-}Si$ bond [20] (see Eqs. (1)–(4)).

$$(CH_3)_3Si{-}C{\equiv}CH \rightarrow (CH_3)_3Si^{\bullet} + {}^{\bullet}C{\equiv}CH \quad (1)$$

$$(CH_3)_3Si{-}C{\equiv}CH \rightarrow CH_3^{\bullet} + {}^{\bullet}Si(CH_3)_2{-}C{\equiv}CH \quad (2)$$

$$2R(CH_3)_2Si^{\bullet}(R: H_3C{-}, CH{\equiv}C{-}) \rightarrow R(CH_3)_2SiH + R(CH_3)Si{=}CH_2 \quad (3)$$

$$R(CH_3)Si{=}CH_2 \rightarrow (-R(CH_3)Si{-}CH_2-)_n \quad (4)$$

Gaseous MA could efficiently produce a transparent film under irradiation with N_2 laser light by a two-photon process [1]. In order to prepare a composite film from MA and TMeSiA molecules and to elucidate chemical processes in film formation, a gaseous mixture of TMeSiA (24 Torr) and MA (37 Torr) was irradiated with N_2 laser light at an energy of 2.2 mJ/pulse for 5 h. The gaseous mixture produced a transparent thin film on an optical quartz window of the irradiation cell. The yield of the film obtained with different partial pressures of TMeSiA was measured by weighing. The results are shown in Fig. 2. With increasing partial pressure of TMeSiA, the yield decreased rapidly, showing that the efficiency of TMeSiA in film formation was much lower than that of MA.

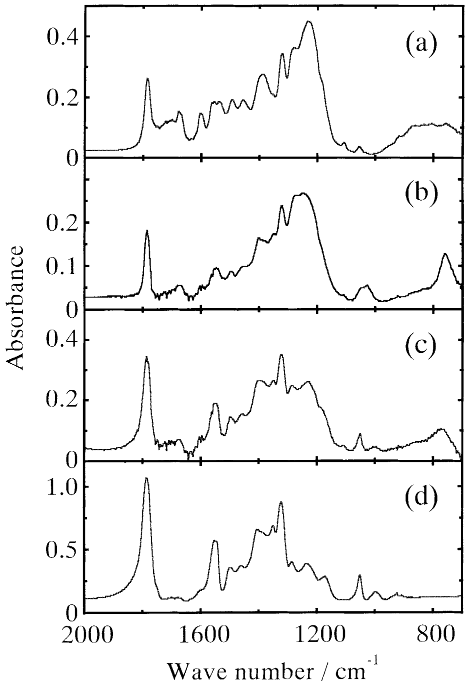


Fig. 3. FT-IR spectra of the film deposited from gaseous mixtures of TMeSiA and MA under irradiation with N_2 laser light at an energy of 2.2 mJ/pulse for 5 h. Partial pressures of TMeSiA and MA are: (a) 65 and 26 Torr; (b) 48 and 30 Torr; (c) 34 and 34 Torr; (d) 12 and 41 Torr; respectively.

FT-IR spectra measured with the films deposited from gaseous mixtures at various molar ratios of TMeSiA and MA are shown in Fig. 3. The spectra could be resolved into two components, i.e. into the absorption bands observed with poly(methyl acrylate) film shown in Fig. 1(b) and those observed with the polymeric film of TMeSiA (Fig. 1(a)). With increasing partial pressure of TMeSiA in the gaseous mixture, polymeric species of TMeSiA was more involved in the film. The 1050 cm^{-1} band shifted to 1072 cm^{-1} only in curve (b) in which the major component was changing from poly(methyl acrylate) to polymeric species of TMeSiA. This may reflect the fact that under this experimental conditions, chemical reaction between TMeSiA and MA molecules became favorable. In curves (a) and (d) where chemical reactions between TMeSiA molecules or MA molecules predominated, polymerization reaction between TMeSiA and MA molecules became less significant.

Under irradiation with a medium pressure mercury lamp (without using any filter) at an exposure energy of 12 mJ/cm^2 s for 2 h, MA vapor (10 Torr) produced sedimentary aerosol particles as a minor product in addition to a transparent thin film on the incident optical quartz window as the major product. The formation of the aerosol particles was detected by measuring the monitor (He–Ne laser) light intensity scattered by the aerosol particles as formed under light irradiation with the mercury lamp. The result is shown in Fig. 4(a). Scattered light was detected in 1 min after light exposure and its intensity increased rapidly to reach its maximum at 2 min. After 5 min, the scattered light intensity decreased gradually until 120 min when the mercury lamp was shut off. Considering the fact that under irradiation with N_2 laser light, MA vapor did not produce any aerosol particles, the short induction period (1 min) to detect scattered light in Fig. 4(a) clearly indicated that MA molecules were efficiently excited at 253.7 nm by one-photon absorption [21,22] and initiated the nucleation reaction to produce aerosol particles. Rapid increase in scattered light intensity in 2 min can be attributed to the increasing number of aerosol particles and also to the growth in size of each particle. At the same time, MA vapor produced the thin film on the incident optical

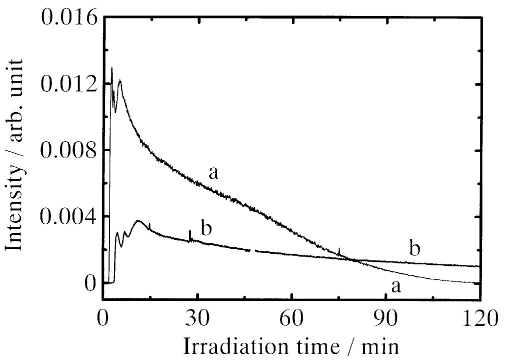


Fig. 4. He–Ne laser light intensity scattered by the aerosol particles produced from: (a) pure MA vapor (10 Torr); (b) a gaseous mixture of TMeSiA (5 Torr) and MA (10 Torr) under irradiation with a medium pressure mercury lamp.

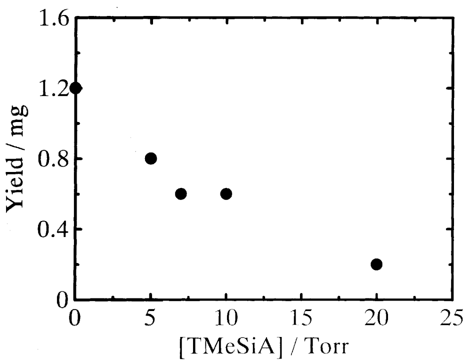


Fig. 5. The yield of the film deposited from gaseous mixtures of TMeSiA (0–20 Torr) and MA (10 Torr) under irradiation with a medium pressure mercury lamp for 2 h.

quartz window. We may assume that once the film was produced on the window, an adsorbed layer of MA molecules was formed on the film, and polymerization reaction in adsorbed layer became much more efficient than the aerosol particle formation [4,23]. Consumption of MA vapor in the film formation process resulted in the decrease of the efficiency of nucleation reaction in aerosol particle formation, thus reducing the number of aerosol particles newly formed. The gradual but finally complete decrease in the scattered light intensity was not observed in the case of acrolein vapor [6] in which aerosol particles were predominantly produced and the scattered light intensity became constant after reaching steady conditions in aerosol particle formation.

FT-IR spectra of the film and of the aerosol particles deposited from pure MA vapor under irradiation with a medium pressure mercury lamp allow us to compare the chemical structure of these materials. The spectrum of the film coincided with the spectrum of the aerosol particles, and also with the spectrum of the film deposited under irradiation with N_2 laser light (Fig. 1(b)), showing that the chemical structures of all the deposits were essentially the same and independent of the excitation conditions.

Under irradiation with the mercury lamp for 2 h, a gaseous mixture of TMeSiA (5 Torr) and MA (10 Torr) produced a transparent thin film on the incident optical window as the major product (yield; 0.8 mg) and sedimentary aerosol particles (yield; 0.1 mg) as in the case of pure MA vapor. The efficient nucleation was observed by measuring scattered light intensity of monitor (He–Ne laser) light as shown in Fig. 4(b). A short but a little longer induction period (3 min) and weaker scattered light intensity than those of pure MA corresponded to the less product yield of the aerosol particles from the gaseous mixture. The product yield of the polymeric film for different partial pressures of TMeSiA was measured by weighing. The results are shown in Fig. 5; the yield decreased with increasing partial pressure of TMeSiA as in the case of N_2 laser light irradiation (Fig. 2). FT-IR spectra of the deposited films and of the sedimentary aerosol particles are shown in Fig. 6A and B, respectively. Polymeric species of TMeSiA was easily detected in the sedimentary aerosol particles, but hardly detected in the deposited films. In the film, polymeric species of TMeSiA was significantly recognized only for the gaseous mixtures containing excess TMeSiA vapor as was shown in spectrum (a) in Fig. 6A. More abundance of polymeric species of TMeSiA in the aerosol particles than in the film strongly suggested that excited MA molecules in the gas phase collisionally induced chemical reaction with TMeSiA molecules, whereas excited MA molecules in the adsorbed layer on the deposited film reacted with poly(methyl acrylate) rather than TMeSiA.To investigate chemical incorporation of TMeSiA in the deposits, XPS of Si 2p, C 1s, and O 1s core levels were measured. The Si 2p spectra of the sedimentary particles and the film deposited from a gaseous mixture of TMeSiA (20 Torr) and MA (10 Torr) under irradiation with a medium pressure mercury lamp are shown in Fig. 7(a) and 7(b), respectively. In each case, Si 2p band can be resolved into two components with binding energies at 100.6 and 102.5 eV, assignable to Si–C and Si–O bonds, respectively. In the sedimentary aerosol particles, the population of the Si–O bond is higher than that of the Si–C bond, whereas the opposite situation is observed in the deposited film. The stoichiometry of the products, $Si_{1.0}\,C_{14}\,O_{10}$ for the particles and $Si_{1.0}\,C_{30}\,O_{12}$ for the film, allows us to estimate the molar ratio of TMeSiA to MA being 1:2.2 for the particles and 1:6.3 for the film, showing that TMeSiA is more contained in the particles than in the film, being in agreement with the FT-IR spectra (Fig. 6).

The Si 2p spectrum of the film deposited from a gaseous mixture of TMeSiA (24 Torr) and MA (37 Torr) under irradiation with N_2 laser light (Fig. 7(c)) revealed that the resolved Si–C and Si–O components are shifted to higher binding energies (101.2 and 103.8 eV), and that both components are equally contributed.

The C 1s spectra (Fig. 8) were identical for the above three samples, and resolved into three components. The strong band at 284.8 eV was assigned to C–H and/or C–C bonds, and moderate bands at 286.3 and 288.6 eV were assigned to C–O bond and C=O and/or COO bonds, respectively. The

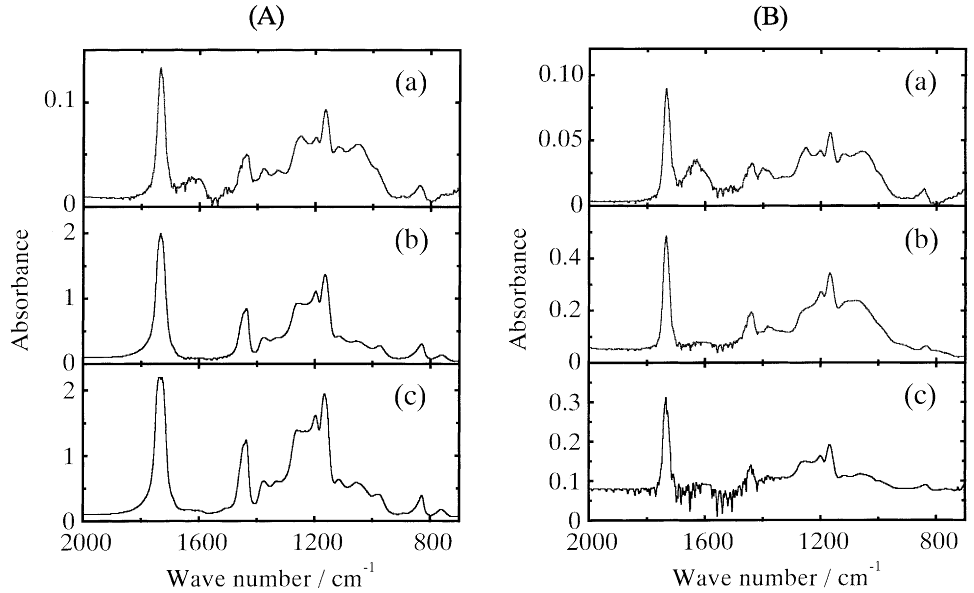


Fig. 6. FT-IR spectra of: (A) the film; (B) the aerosol particles deposited from gaseous mixtures of TMeSiA and MA under irradiation with a medium pressure mercury lamp for 2 h. Partial pressures of TMeSiA and MA are: (a) 20 and 10 Torr; (b) 10 and 10 Torr; (c) 5 and10 Torr.

population of the chemically inequivalent C atoms for all the samples is nearly the same (66, 18, and 16%). The O 1s spectra of the film in Fig. 9 showed two components at 532 and at 533.5 eV with almost equal population which were assigned to C=O and/or C–O–C bonds and C–O(H) bond, respectively.

The XPS data allow to outline the chemical processes of the polymer formation. The energy delivered by two photons of N_2 laser light (7.35 eV) is enough to cleave the Si–C bond (ca. 3.8 eV) [24] and form $R(CH_3)_2Si^\bullet$ and $^\bullet C_2H$ radicals with excess of vibrational energy (Eqs. (1) and (2)). These radicals can initiate a number of polymerization steps. Silyl radicals are known to add to electron-rich sites [25–27]. The $R(CH_3)_2Si^\bullet$ radicals can thus add to both C=C and C=O bonds of MA, and produce radical adducts with Si–C and

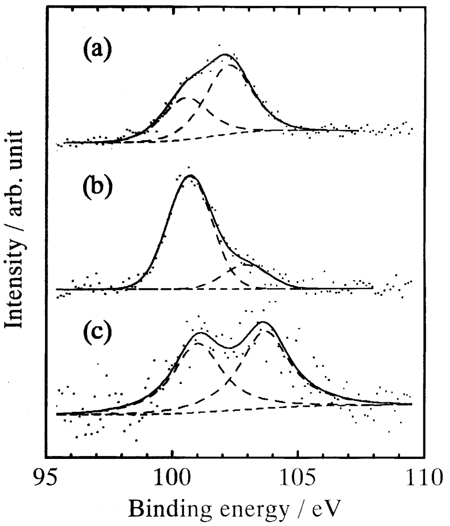


Fig. 7. Si 2p photoelectron spectra of: (a) sedimentary aerosol particles; (b) film deposited from a gaseous mixture of TMeSiA (20 Torr) and MA (10 Torr) under irradiation with a medium pressure mercury lamp for 2 h; (c) film deposited from a gaseous mixture of TMeSiA (24 Torr) and MA (37 Torr) under irradiation with N_2 laser light at an energy of 2.2 mJ/pulse for 5 h.

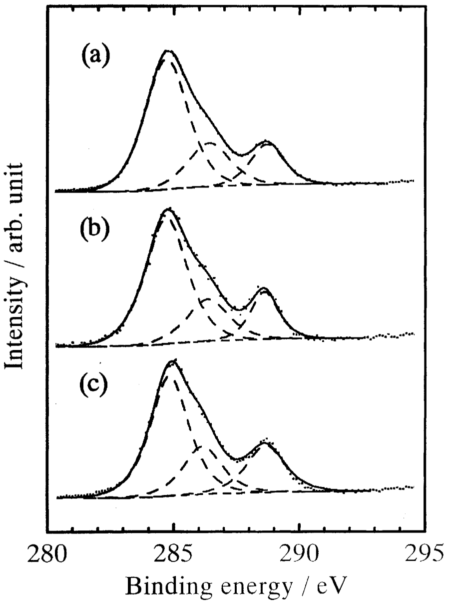


Fig. 8. C 1s photoelectron spectra of: (a) sedimentary aerosol particles; (b) film deposited from a gaseous mixture of TMeSiA (20 Torr) and MA (10 Torr) under irradiation with a medium pressure mercury lamp for 2 h; (c) film deposited from a gaseous mixture of TMeSiA (24 Torr) and MA (37 Torr) under irradiation with N_2 laser light at an energy of 2.2 mJ/pulse for 5 h.

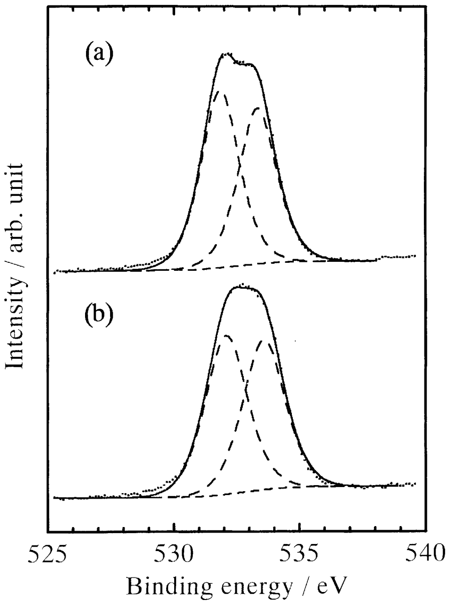
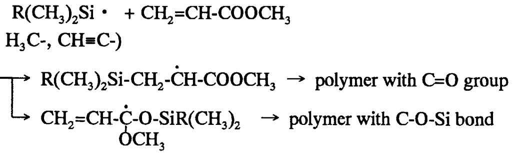


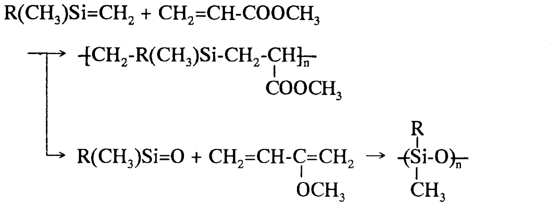
Fig. 9. O 1s photoelectron spectra of: (a) film deposited from a gaseous mixture of TMeSiA (20 Torr) and MA (10 Torr) under irradiation with a medium pressure mercury lamp for 2 h; (b) film deposited from a gaseous mixture of TMeSiA (24 Torr) and MA (37 Torr) under irradiation with N_2 laser light at an energy of 2.2 mJ/pulse for 5 h.

Si–O bonds (Eqs. (5) and (6)) which subsequently produce polymers with C=O group and C–O–Si bond, respectively, as is proved by the XPS data.



$$\mathrm{R(CH_3)_2Si\cdot + CH_2{=}CH{-}COOCH_3}$$
$$\mathrm{(R: H_3C{-},\ CH{\equiv}C{-})}$$
$$\rightarrow \mathrm{R(CH_3)_2Si{-}CH_2{-}\dot{C}H{-}COOCH_3} \rightarrow \text{polymer with C=O group} \tag{5}$$
$$\rightarrow \mathrm{CH_2{=}CH{-}\underset{OCH_3}{\dot{C}}{-}O{-}SiR(CH_3)_2} \rightarrow \text{polymer with C-O-Si bond} \tag{6}$$

The silanes, $R(CH_3)Si{=}CH_2$ (Eq. (3)) can also initiate polymerization reactions with MA [28].



$$\mathrm{R(CH_3)Si{=}CH_2 + CH_2{=}CH{-}COOCH_3}$$
$$\rightarrow \mathrm{-\!\!\left[CH_2{-}R(CH_3)Si{-}CH_2{-}\underset{COOCH_3}{CH}\right]_n\!\!-} \tag{7}$$
$$\rightarrow \mathrm{R(CH_3)Si{=}O + CH_2{=}CH{-}\underset{OCH_3}{C}{=}CH_2} \rightarrow \mathrm{-\!\!\left(\underset{CH_3}{\overset{R}{Si}}{-}O\right)_n\!\!-} \tag{8}$$

Both the silyl radicals and the silanes can produce polymers with Si–C bond and Si–O bond, and reactions in Eqs. (5)–(8) are believed to be effective in the present case. The polymerization can occur both in the gas phase and on the surface of the deposited film.

Under irradiation with a mercury lamp where only MA molecules were photoexcited and initiated polymerization reaction, the C≡C bond of gaseous TMeSiA molecule may be incorporated into the polymerization reaction of MA. Once TMeSiA was involved in polymeric structure, trimethylsilyl group favors to migrate to O atom in C=O group to produce Si–O bond [29]. This migration may occur more easily in aerosol particles than on the surface of the polymeric film. This explains higher population of Si–O bond than that of Si–C bond in sedimentary aerosol particles as revealed from XPS in Fig. 7.

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