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Chemical reactivity of allyltrimethylsilane in UV laser induced aerosol particle formation with acrolein

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Abstract

Under irradiation with N_2 laser light, a gaseous mixture of allyltrimethylsilane (ATMeSi) and acrolein (AC) produced sedimentary aerosol particles with a mean diameter of 0.78 μm. From the analysis of FT–IR spectrum measured with the sedimentary particles, 1,1-dimethyl-1-silacyclobutane and trimethylsilyl group were identified in the sedimentary particles, showing that ATMeSi produced 2-methyl-2-silapropene intermediate by a retroene elimination of propene from ATMeSi, in addition to allyl and trimethylsilyl radicals by a homolytic cleavage of ATMeSi. The nucleation process of the aerosol particles was investigated by measuring monitor (He–Ne laser) light intensity scattered by the aerosol particles as formed under N_2 laser light irradiation. From the dependence of scattered light intensity on the partial pressure of ATMeSi, it was found that the reactive intermediates generated from ATMeSi by a two-photon process of N_2 laser light accelerated the nucleation process of the gaseous mixture.

Keywords: N_2 laser-induced nucleation; Sedimentary aerosol particles; Allyltrimethylsilane; Acrolein; 1,1-Dimethyl-1-silacyclobutane

1. Introduction

Under irradiation with N_2 laser light, gaseous acrolein (2-propenal) (AC) produced sedimentary aerosol particles of polyacrolein by a two-photon process, and reacted with foreign gaseous molecules during the polymerization reaction to result in producing composite aerosol particles [1–3]. In order to prepare novel organosilicon compounds using chemical reactions on the surface of nano- to submicron-meter sized particles, some organosilicon compounds such as trimethyl(2-propynyloxy)silane (TMPSi) and vinyltrimethoxysilane (VTMSi) were incorporated into the aerosol particles of polyacrolein under irradiation with N_2 laser light [4,5]. Chemical analysis of sedimentary aerosol particles by X-ray photoelectron spectroscopy (XPS) and FT–IR spectra revealed that organosilicon compounds were involved into the aerosol particles as several different chemical species, the amounts of which were very much dependent on the chemical reactivity of the parent organosilicon compounds, and that TMPSi which has a C≡C triple bond was incorporated into the polymerization reaction of AC molecules in spite of rather unfavorable character of the triple bond towards the polymerization reaction [6,7].

Allyltrimethylsilane (2-propenyltrimethylsilane) (ATMeSi) can easily dissociate into allyl radical and trimethylsilyl radical thermally and photolytically [8,9], and also produce 2-methyl-2-silapropene intermediate, $(CH_3)_2Si{=}CH_2$ by a retroene elimination of propene from ATMeSi [10,11]. $(CH_3)_2Si{=}CH_2$ was extensively used as a reactive intermediate to induce various chemical reactions of organosilicon compounds [12–15]. This reactive intermediate and the radicals may react with AC molecules during polymerization reactions in aerosol particle formation.

In the present paper, in an attempt to develop novel organosilicon compound in nm to sub-micrometer sized particles and to elucidate the chemical reactivity of ATMeSi, we have undertaken to synthesize composite aerosol particles from a gaseous mixture of ATMeSi and AC under irradiation with N_2 laser light. To distinguish the characteristic behaviors of two-photon process from those of one-photon process, aerosol particles have also been prepared under irradiation with a medium pressure mercury lamp. Nucleation process and propagation process of the aerosol particle formation were investigated by measuring monitor (He–Ne laser) light intensity scattered by the aerosol particles as formed under light irradiation, and chemical structure of

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the sedimentary aerosol particles was studied by measuring FT–IR spectra.

2. Experimental details

AC (Merck, 98%, stabilized with 0.2% hydroquinone) and ATMeSi (Tokyo Kasei, G.R. grade) were stored in a refrigerator below 10°C to prevent polymerization. Each of the ATMeSi and AC liquid was degassed by freeze-pump-thaw cycles and distilled under the vacuum immediately before use. A fixed amount of AC vapor which was introduced into a specific region of a vacuum system was collected into a glass tube by freezing with liquid nitrogen. ATMeSi vapor was then introduced into the 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) through the vacuum system, and finally, AC vapor evaporated from the liquid in the glass tube was introduced into the irradiation cell to prepare a gaseous mixture of ATMeSi and AC. The background pressure of the irradiation cell was less than 8×10^{-5} Torr (1 Torr=133.3 Pa) and the leakage into the evacuated cell from the atmosphere was carefully controlled to be less than 1 Torr in a day. The pressure of the gaseous sample was measured with a capacitance manometer (Edwards Barocel Type 600). The actual partial pressures of ATMeSi and AC in the irradiation cell were determined from the analysis of UV absorption spectrum. Gaseous samples were irradiated with collimated light of a medium pressure mercury lamp (Ushio UM-452, 450W) through a UV-31 and a UVD33S filters or with nitrogen gas laser light (Lumonics HE-440, 337.1 nm) without any focusing lens with a repetition rate of 12 Hz. Laser power was measured with a pyroelectric joulemeter (Gentec ED-500) combined with a synchroscope (Iwatsu SS-5121B). FT–IR spectra of deposits in KBr pellets were measured with a Nicolet 740 FT–IR spectrometer, and scanning electron microscope (SEM) images were taken with a Topcon ABT-32 scanning electron microscope. Light intensity scattered by the aerosol particles floating in the cell was measured with a combination of a photomultiplier tube (EMI 6256S) and a lock-in amplifier (SRS SR-530) by chopping and synchronizing monitor (He–Ne laser) light.

3. Results and discussion

Upon exposure to N_2 laser light at an energy of 1.8 mJ/pulse for 3 h, a gaseous mixture of ATMeSi (12 Torr) and AC (41 Torr) produced sedimentary aerosol particles which deposited on a glass plate placed at the bottom of the irradiation cell with a reproducible sedimentary pattern due to the convection of the gaseous mixture, showing that the particles were produced in the gas phase. The sedimentary particles were white and spherical with a mean diameter of 0.78 μm as is shown in Fig. 1. Under the same experimental conditions, pure AC vapor deposited aerosol particles with a mean diameter of 0.77 μm.

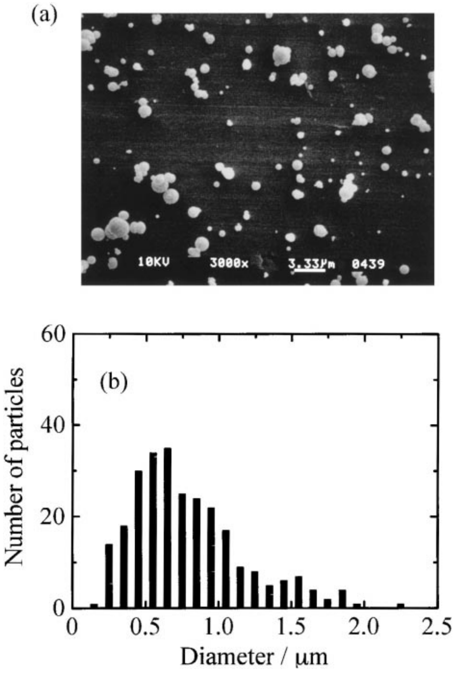


Fig. 1. (a) SEM image of sedimentary aerosol particles deposited from a gaseous mixture of ATMeSi (12 Torr) and AC (41 Torr) under irradiation with N_2 laser light at an energy of 1.8 mJ/pulse for 3 h and (b) particle size distribution therefrom. Original magnification of SEM, 3000×.

ATMeSi vapor has the longest wavelength absorption band shorter than 210 nm and does not absorb the light at 337.1 nm. In order to check whether ATMeSi can initiate nucleation reaction by a two-photon process, pure ATMeSi at a pressure of 41 Torr was irradiated with N_2 laser light at an energy of 1.8 mJ/pulse for 3 h; pure ATMeSi did not produce any deposits, indicating that the aerosol particle formation from the gaseous mixture was initiated through photochemical reaction of AC. As reported previously [2,5], AC molecule dissociated into C_2H_3 and CHO radicals by a two-photon process at 337.1 nm and initiated polymerization reaction to form aerosol particles.

To investigate the nucleation process of the aerosol particles, He–Ne laser light intensity scattered by the aerosol particles which were formed in the gas phase under N_2 laser light irradiation was measured. The result for a gaseous mixture of ATMeSi (12 Torr) and AC (41 Torr) under irradiation with N_2 laser light at an energy of 1.8 mJ/pulse is shown in Fig. 2 (curve a). Scattered light intensity was detected after an induction period of 16 min and reached to its maximum at 67 min. The light intensity fluctuated largely due to the convection of the gaseous molecules inside the irradiation cell. As is shown by curves b–d in Fig. 2, with decreasing N_2 laser intensity, the scattered light intensity decreased and the induction period for detecting the scattered light increased. The dependence on N_2 laser light intensity observed with the gaseous mixture was characteristic to the two-photon

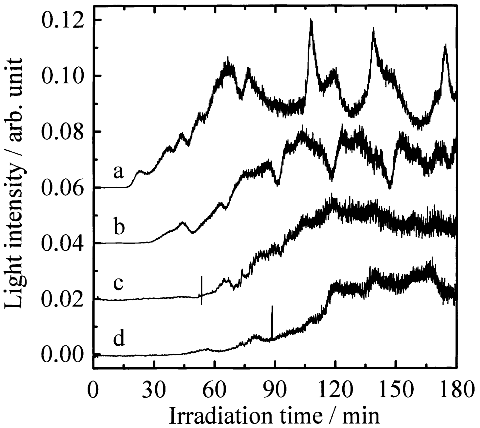


Fig. 2. He–Ne laser light intensity scattered by the aerosol particles produced from a gaseous mixture of ATMeSi (12 Torr) and AC (41 Torr) under irradiation with N_2 laser light at an energy of (a) 1.8; (b) 1.6; (c) 1.3 and (d) 1.0 mJ/pulse. The ordinates are shifted consecutively by −0.02 from (a) to (b), (c) and (d).

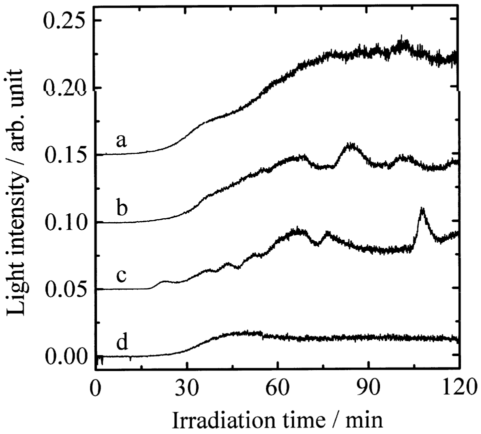


Fig. 4. He–Ne laser light intensity scattered by the aerosol particles produced from a gaseous mixture of ATMeSi and AC (41 Torr) under irradiation with N_2 laser light at an energy of 1.8 mJ/pulse. The partial pressure of ATMeSi was (a) 40; (b) 25; (c) 12 and (d) 0 Torr. The ordinates are shifted consecutively by −0.05 from (a) to (b), (c) and (d).

process observed with pure AC [5], indicating that polymerization of AC is primarily responsible to the nucleation and propagation processes of aerosol particle formation.

From the analysis of SEM images, it was found that the particle size distribution of the sedimentary aerosol particles from the gaseous mixture did not change on N_2 laser light intensity. Taking this into account, the scattered light intensity was thought to be proportional to the number of the aerosol particles, and hence the scattered light intensity integrated for a fixed interval (Y_i) is a measure of the product yield of the aerosol particles. Fig. 3(a) shows the dependence of Y_i integrated for the first 3 h on N_2 laser light intensity. The Y_i value showed quadric behaviors on N_2 laser light intensity, being consistent with the result that the aerosol particles were produced by a two-photon process of AC molecules under irradiation with N_2 laser light.

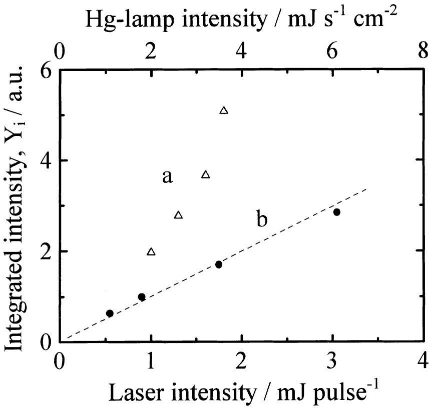


Fig. 3. (a) Dependence of scattered light intensity integrated for the first 3 h against N_2 laser light intensity (△) and (b) dependence of scattered light intensity integrated for the first 2 h against the light intensity of a mercury lamp (●). Aerosol particles were produced from a gaseous mixture of ATMeSi (12 Torr) and AC (41 Torr).

To investigate the effect of addition of ATMeSi molecules on the nucleation process of the aerosol particles, scattered light intensity was measured by varying the partial pressure of ATMeSi; the results are shown in Fig. 4. With increasing partial pressure of ATMeSi, the induction period for detecting the scattered light became shorter (from 18 min (curve d) to 9 min (curve a)), the rate of nucleation (which corresponds to the initial slope of scattered light intensity curve) increased (from $1.8\times10^{-4}\,\mathrm{min}^{-1}$ (curve d) to $2.7\times10^{-4}\,\mathrm{min}^{-1}$ (curve a)), and the rate of propagation (which corresponds to the scattered light intensity under prolonged light irradiation) increased appreciably (≈5 times). Although pure ATMeSi did not produce any deposits, this result clearly showed that ATMeSi accelerated the nucleation and propagation processes in aerosol particle formation under N_2 laser light irradiation.

Under light irradiation at 313 nm for 4 h using a medium pressure mercury lamp through a UV-31 and a UVD33S filters, pure AC vapor (41 Torr) produced sedimentary aerosol particles (yield; 0.2 mg) with a mean diameter of 0.75 μm. Under the same experimental conditions, a gaseous mixture of ATMeSi (12 Torr) and AC (41 Torr) also produced sedimentary aerosol particles (yield; 0.2 mg) with a mean diameter of 0.78 μm (Fig. 5). As in the case under N_2 laser light irradiation, the mean diameters did not change appreciably by adding ATMeSi into AC vapor, indicating that polymerization of AC molecules is mainly responsible to the propagation process of aerosol particle formation. In the gaseous mixture of ATMeSi and AC, propagation reaction of the particles proceeded in the vapor phase under light irradiation. Inside the irradiation cell, aerosol particles were turning around by the convection during light irradiation, and once the particles collided to a glass plate at the bottom and deposited on it, propagation reaction of the deposited particles did not proceed any more. Convectional motion of the aerosol particles under N_2 laser light irradi-

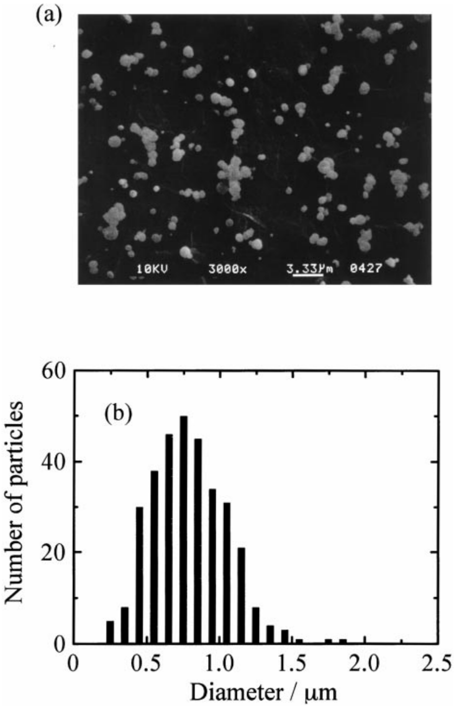


Fig. 5. (a) SEM image of sedimentary aerosol particles deposited from a gaseous mixture of ATMeSi (12 Torr) and AC (41 Torr) under irradiation with a medium pressure mercury lamp at an exposure dose of 6.1 mJ/s cm^2 for 2 h and (b) particle size distribution therefrom. Original magnification of SEM, 3000×.

ation was observed to be a little faster than the one under mercury lamp irradiation. This resulted in shorter propagating time of the particles under N_2 laser light irradiation, but a two-photon process of N_2 laser light could produce active sites of polymerization on the surface of aerosol particles (major component of which is polyacrolein) to increase the rate of propagation. Under the present experimental conditions, these two factors compensated for each other, resulting in producing sedimentary particles with a mean diameter which is almost equal to the one of aerosol particles produced under mercury lamp irradiation.

The monitor (He–Ne laser) light intensity scattered by the aerosol particles which were produced from the gaseous mixture under mercury lamp irradiation was also measured by varying the light intensity of the mercury lamp between 6.1–1.1 mJ/s cm^2; the results are shown in Fig. 6. With increasing light intensity, the induction period became shorter, and the scattered light intensity increased. The scattered light intensity integrated for the first 2 h (Y_i) was plotted in Fig. 3(b) compared with the result obtained under N_2 laser light irradiation. The result indicated that the Y_i value was proportional to the light intensity of the mercury lamp, showing that the nucleation process was initiated by one-photon process (Eq. (1)), being consistent with the fact that AC dissociated into C_2H_3 and CHO radicals at 313 nm under irradiation with a mercury lamp [16,17].

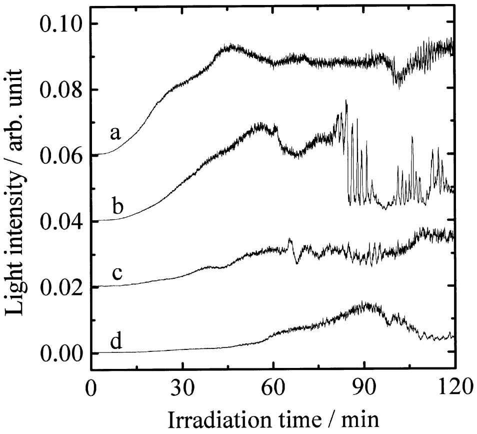


Fig. 6. He–Ne laser light intensity scattered by the aerosol particles produced from a gaseous mixture of ATMeSi (12 Torr) and AC (41 Torr) under irradiation with a medium pressure mercury lamp at an exposure dose of (a) 6.1; (b) 3.5; (c) 1.8 and (d) 1.1 mJ/s cm^2. The ordinates are shifted consecutively by −0.02 from (a) to (b), (c) and (d).

$$\begin{aligned} &CH_2{=}CH{-}CH{=}O \xrightarrow{1h\nu} C_2H_3^{\bullet} + {}^{\bullet}CHO \\ &\quad \rightarrow \quad \text{Nucleation reaction with AC} \qquad (1) \end{aligned}$$

To evaluate the contribution of ATMeSi to the nucleation process, the dependence of the scattered light intensity on the partial pressure of ATMeSi was measured; the results are shown in Fig. 7. Both the induction period for detecting the scattered light (≈28 min) and the initial slope (6×10^{-5} min^{-1}) were rather insensitive to the partial pressure of ATMeSi, in contrast to the result under N_2 laser light irradiation (see Fig. 4). Considering that ATMeSi was not electronically excited at 313 nm by one-photon process, the result in Fig. 7 indicated that electronically unexcited ATMeSi molecule did not contribute to the nucleation pro-

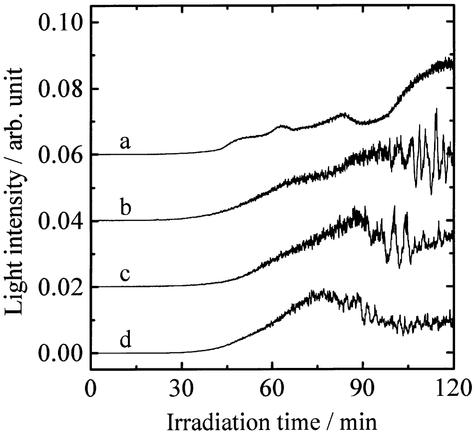


Fig. 7. He–Ne laser light intensity scattered by the aerosol particles produced from a gaseous mixture of ATMeSi and AC (41 Torr) under irradiation with a medium pressure mercury lamp at an exposure dose of 6.1 mJ/s cm^2. The partial pressure of ATMeSi was (a) 20; (b) 15; (c) 5 and (d) 0 Torr. The ordinates are shifted consecutively by −0.02 from (a) to (b), (c) and (d).

cess of aerosol particle formation. Hence, the result that ATMeSi accelerated the nucleation reaction under N_2 laser light irradiation shown in Fig. 4 strongly suggested that ATMeSi was excited by a two-photon process and dissociated into allyl radical and trimethylsilyl radical (Eq. (2)) to additionally initiate the polymerization reaction of AC (Eq. (3)).

$$\begin{aligned}&CH_2{=}CH{-}CH_2{-}Si(CH_3)_3 \xrightarrow{2h\nu} CH_2{=}CH{-}CH_2{}^{\bullet}\\ &\quad + {}^{\bullet}Si(CH_3)_3 \rightarrow \quad \text{Nucleation reaction with AC} \qquad (2)\end{aligned}$$

$$\begin{aligned}&CH_2{=}CH{-}CH{=}O \xrightarrow{2h\nu} C_2H_3{}^{\bullet} + {}^{\bullet}CHO\\ &\quad \rightarrow \quad \text{Nucleation reaction with AC} \qquad (3)\end{aligned}$$

Homolytic cleavage of Si-allyl bond of ATMeSi was reported in gas-phase thermal decomposition, and dissociation energy was estimated to be small (73 kcal/mol) [8]. Under irradiation with N_2 laser light, excitation of ATMeSi by two photons can give enough internal energy to the molecule to induce the photodissociation of ATMeSi to produce trimethylsilyl and allyl radicals.

To characterize the chemical structure of the sedimentary aerosol particles, FT–IR spectra were measured with the sedimentary particles deposited from a gaseous mixture of ATMeSi (12 Torr) and AC (41 Torr) (Fig. 8). For the purpose of comparison, FT–IR spectrum measured with the sedimentary particles deposited from pure gaseous AC under irradiation with a mercury lamp was also shown in Fig. 8(a); the spectrum from pure AC coincided with the spectrum of the sedimentary particles deposited under irradiation with N_2 laser light [1], and can be assigned to polyacrolein which has a cyclic acetal and hemiacetal structure with aldehyde functional group [18–20]. The spectrum of the particles deposited from the gaseous mixture under irradiation with a mercury lamp (Fig. 8(b)) showed new bands at 1243, 839, 765 cm^{-1} in addition to the bands characteristic of polyacrolein in the 800–1800 cm^{-1} region. The band at 1243 cm^{-1} can be assigned to CH_3Si bending vibrational band, and the bands at 839 and 765 cm^{-1}, to a blend of Si-CH_3 stretching and CH_3Si rocking vibrational bands [21–24], indicating that trimethylsilyl group was involved in the sedimentary particles of polyacrolein. Considering the fact that ATMeSi did not significantly decompose into reactive intermediates by homolytic cleavage under mercury lamp irradiation as was evidenced from the result in Fig. 7, ATMeSi may be incorporated into the polymerization reaction of AC through the vinyl polymerization of allyl group.

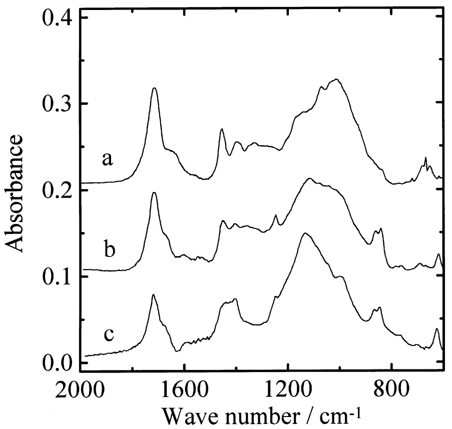
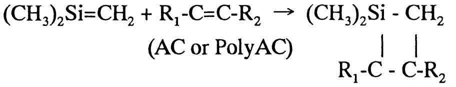


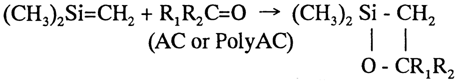
Fig. 8. FT–IR spectra of sedimentary aerosol particles produced from (a) pure gaseous AC (41 Torr); (b) a gaseous mixture of ATMeSi (12 Torr) and AC (41 Torr) under irradiation with a medium pressure mercury lamp and (c) a gaseous mixture of ATMeSi (12 Torr) and AC (41 Torr) under irradiation with N_2 laser light. The ordinates are shifted consecutively by −0.10 from (a) to (b) and (c).

FT–IR spectrum of the sedimentary particles produced under irradiation with N_2 laser light (Fig. 8(c)) showed a strong band at 1130 cm^{-1} and a moderate band at 1410 cm^{-1}, accompanying the intensity decrease of the bands ascribed to trimethylsilyl group and to polyacrolein. The bands observed at 1130 and 1410 cm^{-1} can be assigned to 1,1-dimethyl-1-silacyclobutane structure [25]. As for silaoxetanes, IR spectrum of 1,1-dimethoxy-1,2-silaoxetane was only observed as a photoproduct of (trimethoxysilyl)diazomethane in argon matrix at 10 K, and a tentative assignment of the bands was reported based on an ab-initio calculation [26]. A theoretical calculation on band frequencies of 1,2-silaoxetane was also reported [27]. Although these band assignments were not conclusive, these results do not exclude the possibility that the new bands observed in Fig. 8(c) were assigned to 1,1-dimethyl-1,2-silaoxetane structure. 2-Methyl-2-silapropene intermediate, $(CH_3)_2Si{=}CH_2$ which was produced by a retroene elimination of propene from ATMeSi (Eq. (4)) can easily react with C=C and/or C=O double bonds to form silacyclobutane and silaoxetane structures [10–12,27–29].

$$\begin{aligned}&CH_2{=}CH{-}CH_2{-}Si(CH_3)_3\\ &\quad \xrightarrow{2h\nu} CH_2{=}CH{-}CH_3 + (CH_3)_2\,Si{=}CH_2 \qquad (4)\end{aligned}$$



$$\begin{array}{lll} (CH_3)_2Si{=}CH_2 + R_1\text{-}C{=}C\text{-}R_2 & \rightarrow & (CH_3)_2Si - CH_2 \\ \quad\quad\quad\quad (\text{AC or PolyAC}) & & \quad\quad\;\; | \quad\;\; | \\ & & \quad\;\; R_1\text{-}C - C\text{-}R_2 \end{array} \qquad (5)$$



$$\begin{array}{lll} (CH_3)_2Si{=}CH_2 + R_1R_2C{=}O & \rightarrow & (CH_3)_2\,Si - CH_2 \\ \quad\quad\quad\quad (\text{AC or PolyAC}) & & \quad\quad\;\; | \quad\;\; | \\ & & \quad\quad\; O - CR_1R_2 \end{array} \qquad (6)$$

In aerosol particle formation from the gaseous mixture of ATMeSi and AC under N_2 laser light irradiation, $(CH_3)_2Si{=}CH_2$ intermediate may react with gaseous AC molecule which has a C=C and a C=O double bonds to form the silacyclobutane and/or silaoxetane, followed by polymerization reaction of the other available double bond eventually to form the aerosol particles. Alternatively, $(CH_3)_2Si{=}CH_2$ may react with propagating polyacrolein at the surface of the aerosol particles to form the silacyclobutane and/or silaoxetane structures (Eqs. (5) and (6)).

Considering that C=O stretching band observed at 1718 cm^{-1} decreased its intensity only under irradiation with N_2 laser light, the formation of the silaoxetane structure together with the formation of silacyclobutane structure is favorably suggested. As is well known, the silaoxetanes underwent further chemical reactions to give the trimer and higher oligomers of silanones [27,30]. In the present experimental conditions in which the silaoxetane structure was fixed and isolated on the surface of or inside the aerosol particles, further chemical reactions between the silaoxetanes may possibly be prohibited.

In Fig. 8(b), a slight contribution of 1130 and 1410 cm^{-1} bands was recognized. This may possibly be due to the formation of $(CH_3)_2Si{=}CH_2$ intermediate through hydrogen abstraction from a methyl group by a collision of ATMeSi with reactive radicals generated from excited AC or with AC itself excited under mercury lamp irradiation.

In conclusion, spherical aerosol particles with a mean diameter of 0.78 μm were produced from a gaseous mixture of ATMeSi and AC under irradiation with N_2 laser light and with a mercury lamp. Electronically excited ATMeSi by a two-photon process of N_2 laser light proceeded to the retroene elimination of propene from ATMeSi to produce 2-methyl-2-silapropene intermediate, together with to the homolytic dissociation to produce trimethylsilyl and allyl radicals. These reactive species accelerated the nucleation process in the particle formation and were incorporated into the aerosol particles. 2-Methyl-2-silapropene intermediate was involved as silacyclobutane and/or silaoxetane through the chemical reaction with acrolein.

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