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Laser ablation at 308 nm for the mixture target of 3,4,9,10-perylenetetracarboxylic dianhydride with Co powder — remarkable enhancement on elimination efficiency of anhydride groups

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Abstract

Mixture targets of 3,4,9,10-perylenetetracarboxylic dianhydride (PTCDA) with Co powder were ablated with XeCl laser beams at various fluences. Structures and electric/electronic properties of deposited films and morphology change of PTCDA/Co targets were investigated by scanning electron micrography (SEM), FT-IR, Raman spectroscopies, electron probe microanalyzer (EPMA), and electric conductivity measurements. As for the mixture target of PTCDA and Co powder (diameter 30 nm) ([PTCDA]/[Co] = 1/4), IR spectroscopy showed that fragments without carboxylic dianhydride groups were effectively produced by excimer laser ablation (ELA) at the fluence of more than 1.0 J cm^{-2} pulse^{-1}. Raman spectra for the films prepared by ELA of the PTCDA/Co target at various fluences obviously showed three peaks at 1600, 1380 and 1290 cm^{-1}, characteristic for polyperinaphthalene (PPN). The film prepared by ELA of PTCDA/Co target at a fluence of 1.0 J cm^{-2} pulse^{-1} on a substrate at 20°C consisted of small particles with various diameters ranging from 10 to 100 nm. Only an extremely small amount of Co was detected in the film. Bamboo shoot-like projections could be observed on the PTCDA/Co target surface after irradiation. Co content at the summit was a little bit larger than that at the mountainside. Annealing at 350°C resulted in the increase in electric conductivity up to 1.0×10^{-4} S cm^{-1}, two orders of magnitude higher compared with that before annealing. For the film annealed at 350°C, the presence of a sharp peak at 1290 cm^{-1} and complete disappearance of 1750 and 1780 cm^{-1} peaks related to the side groups of PTCDA indicate the formation of better-developed PPN structure. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Laser ablation; Perylenetetracarboxylic dianhydride (PTCDA); Polyperinaphthalene film; Effect of Co

1. Introduction

Laser ablation is recognized as an excellent method to prepare inorganic functional films such as high-temperature superconductors and ferromagnetic films [1–3]. In contrast, only a few attempts have so far been made on the application of the method to organic compounds. One may believe that laser ablation is not suitable for the synthesis of organic compounds with functional groups because too much excess energy decomposes almost all organic materials into small fragments consisting of atoms and/or small molecules. It is difficult to reproduce some specific organic compounds from such small fragments.

However, one great advantage of laser ablation rests in its wavelength selectability. Making good use of this merit, combining the good points in organic materials that we can design and actually synthesize the molecules with absorption bands in desired region, development of novel laser chemistry such as selective reaction can be expected. In this sense, it may safely be said that laser ablation method possesses the latent potential for application to processing and synthesis of polymers and organic compounds. Actually, we have already demonstrated in a study of pulsed laser deposition (PLD) of polyacrylonitrile (PAN) that the structure of the deposited films could be controlled by selecting ablation wavelength and fluence, i.e. we could obtain films with original PAN structure, ones without cyano group, side group of PAN, and ones consisting of amorphous carbon [4,5]. Furthermore, in a study of ablation of 3,4,9,10-perylenetetracarboxylic dianhydride (PTCDA), one of perylene-related compounds, we could obtain the PTCDA film, films partially possessing an

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organic compound, tetraiodoallene, or polyperinaphthalene (PPN), one of the low-dimensional conducting polymers, by selecting ablation conditions such as wavelength, fluence, substrate temperature and ambient vapor species [6,7].

PPN is expected to be a novel material for microelectronic devices, by its characteristic band structure as well as high chemical and heat resistances [8–10]. In addition, it is pointed out that this material is a promising one for an anode electrode for lithium ion rechargeable batteries which have now attracted a great deal of public attention [11–13]. However, fragile property of PPN prepared with the conventional method called vapor-phase polymerization as developed by Murakami et al. [14] prevents us from applying this material to such microelectronic devices. So many research groups have a fierce competition with each other to develop the method to prepare films of this material [15,16]. In recent years, particularly, preparation of PPN films by laser ablation has attracted a great deal of attention [17,18].

We have demonstrated that amorphous organic semiconducting thin films with PPN component named polyperinaphthalenic organic semiconductor (PPNOS) [18] were prepared by ELA of PTCDA, with a 308 nm (XeCl) beam on a substrate at 300°C, although the film possessed the structural defects such as radicals and depletion of hydrogen atoms. These films showed electric conductivity ranging from 10^{-1} to 10^{-2} S cm^{-1}. Although this method is one of the most superior one to obtain PPN at present, extremely low yield of “naked” perylene skeletons without side groups is a problem to be solved immediately. In our previous paper [19], for the purpose of obtaining PPN more effectively, mixture of PTCDA and metal powder (PTCDA/M) (M = Co, Ni, Fe or Al) was employed as a target to be found that the yield of “naked” perylene skeletons without side groups was increased by improving the efficiency of elimination of side groups of PTCDA. These results are significant not only because this method is profitable for effective preparation of PPN but also because it has a possibility to open up a new paradigm for laser chemistry on organic materials.

In this paper, narrowing an argument down to ELA of a mixture of PTCDA and Co powder, we investigate this peculiar phenomenon in detail and apply this method to prepare better-defined PPN films.

2. Experimental details

The experimental set-up was basically the same as reported previously [19]. A mixture of PTCDA and Co powder (PTCDA/Co) with a molar ratio [PTCDA]/[Co] of 1/1, 1/4 or 1/6, was compressed into a pellet (ca. 10 mm in diameter and 2 mm in thickness) and used as a target. PTCDA and Co powder were purchased from Aldrich and Nilaco, respectively. Two kinds of Co powder with different diameters (1–2 μm and 30 nm) were employed. Co powders were sufficiently mixed with PTCDA powder in a mortar to disperse homogeneously. Laser ablation of PTCDA/Co target was performed for 2–3 h in a reaction chamber evacuated below 10^{-3} Torr with a 308 nm (XeCl) pulsed beam of an excimer laser (Lambda Physik 105E) at a repetition rate of 5 Hz. The pulse duration is ca. 20 ns. Deposited films were prepared on quartz or KBr substrates located at a distance of 3–4 cm from the target. The substrate temperature (T_s) was kept at 20°C during deposition. The laser beam was focused on the PTCDA/Co target with a quartz lens. The fluence of the laser beam was varied from 0.25 to 2.0 J cm^{-2} pulse^{-1}. During deposition, the target was spun with a motor, preventing a laser beam from concentrating on a fixed spot. In the case of the observation of morphology change of the target surface after laser irradiation, 1500 shots of focused laser beam were radiated on a fixed spot. After the film was deposited at a T_s of 20°C, substrate temperature was increased gradually up to 350°C in order to probe the annealing effect on the film structure and electric conductivity.

Structure of the deposited films and morphology change of PTCDA/Co targets were investigated by scanning electron micrography (SEM), FT-IR and Raman spectroscopies and electron probe microanalyzer (EPMA). Electric conductivities were measured with a conventional four-probe method.

3. Results and discussion

3.1. Film formation by ELA of mixture target of PTCDA with Co powder

During ablation of a PTCDA/Co target, ablated species expanded forward conically. The base of the cone was orange-colored, similarly to the ablation of neat PTCDA target. On the contrary, the top was bright white, which was not found for a neat PTCDA target. The emission intensity of ablation plume decreased with increasing Co content. Significant decrease in the emission intensity was observed on repetitive laser irradiation for the ablation of PTCDA/Co target with [PTCDA]/[Co] = 1/4 and 1/6.

As for the Co powder with a diameter of 30 nm, ELA of a PTCDA/Co target ([PTCDA]/[Co] = 1/1) at various fluences ranging from 0.25 to 1.0 J cm^{-2} pulse^{-1} led to a film similar to the evaporated film of PTCDA, as judged by IR spectroscopy. On the other hand, quite different and intriguing results were obtained for the film prepared by ELA of PTCDA/Co targets ([PTCDA]/[Co] = 1/4 and 1/6). For [PTCDA]/[Co] = 1/4, FT-IR spectroscopy was performed for the films prepared at various fluences. The spectra of the films prepared at fluences of 0.5, 1.0 and 1.5 J cm^{-2} pulse^{-1} are shown in Fig. 1 together with that of a PTCDA evaporated film. In the spectrum for the film prepared at 0.5 J cm^{-2} pulse^{-1}, peaks at 1750 and 1780 cm^{-1} related to the side groups of PTCDA monomers can be observed though peak intensity ratio [1780/1750] is different from that for PTCDA evaporated film. The intensity of these peaks decreases gradually with increasing fluence. At more than 1.0 J cm^{-2} pulse^{-1}, it is quite low as shown in

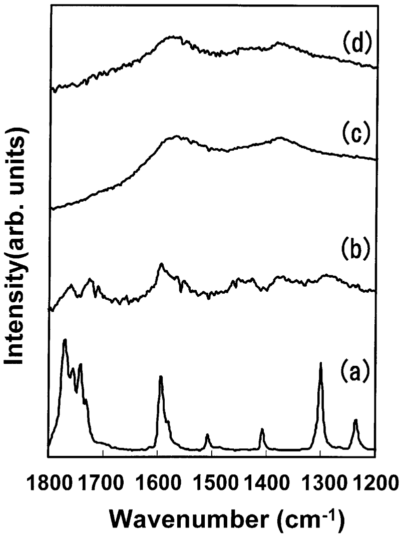

Fig. 1. FT-IR spectra of the films prepared by ELA of the PTCDA/Co target at fluences of 0.5 (b), 1.0 (c) and 1.5 (d) J cm^{-2} pulse^{-1} together with that of a PTCDA-evaporated film (a).

Fig. 1. This suggests that fragments without carboxylic dianhydride groups are effectively produced by ELA at more than 1.0 J cm^{-2} pulse^{-1}. It may safely be said that this is one of the hyperthermal phenomenon where the high-intensity laser beam takes part in, considering from the strong dependence of the extent of elimination of side groups on the fluence. This is supported by a result of a simple experiment that evaporation of PTCDA/Co at 360°C allowed us to obtain just a PTCDA film. These peaks completely vanished for the film prepared by ELA of the PTCDA/Co target ([PTCDA]/[Co] = 1/6) at a fluence of 1.0 J cm^{-2} pulse^{-1}. Quite low deposition rate due to small amount of fragments, however, which are judged from weak emission of ablation plume as mentioned above, prevented us from preparing the film effectively with this target. So, we performed the following experiments using PTCDA/Co targets with [PTCDA]/[Co] = 1/4. Contrary to the Co powder with the diameter of 30 nm, for the powder with diameter of 1–2 μm, the efficiency of elimination of the anhydride groups was quite low. Fig. 2 shows the FT-IR spectrum of a film prepared by ELA of the mixture target of PTCDA/Co powder with the diameter of 1–2 μm at a fluence of 1.0 J cm^{-2} pulse^{-1}. Considerable amount of the side groups remained in the film. This result must be one of the clues for elucidation of effect of Co on elimination of side groups of PTCDA. Hereafter, we used the mixture target of PTCDA with Co powder with a diameter of 30 nm.

Although the FT-IR measurement gives us the important information on elimination of the side group of PTCDA, it is of no avail for obtaining knowledge of the structure of the main body of PTCDA because enhancement of background around 1000–1500 cm^{-1}, which is often observed in various amorphous carbon materials, conceals the finger print region

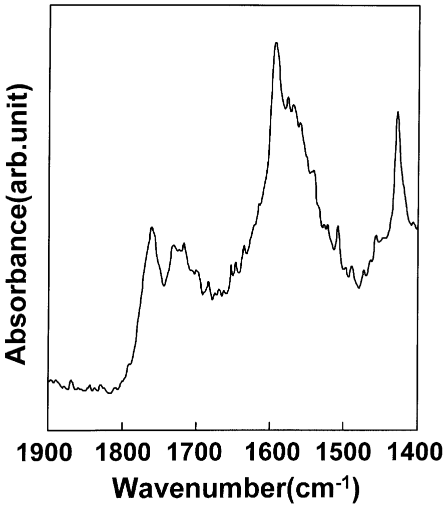

Fig. 2. FT-IR spectrum of a film prepared by ELA of PTCDA/Co powder with the diameter of 1–2 μm at a fluence of 1.0 J cm^{-2} pulse^{-1}.

of PTCDA. Raman spectroscopy is very useful for this purpose. Fig. 3 shows the Raman spectra for the films prepared by ELA of the PTCDA/Co target at various fluences. Each spectrum obviously shows three peaks at 1600, 1380 and 1290 cm^{-1}. Each peak position is completely consistent with that for PPN prepared by Murakami et al. [14]. Here both peaks at 1600 and 1380 cm^{-1} are due to condensed aromatic rings. It is well known that the intensity of the latter peak depends on the size of condensed aromatic rings, decreasing with increasing the size [20,21]. The peak at 1290 cm^{-1} is due to in-plane C–H bending in perylene structure [14]. The full width at half maximum (FWHM) of the peak at 1290 cm^{-1} for each spectrum is smaller than that of the film prepared previously by ELA of PTCDA target at an appropriate fluence on a substrate at 300°C, indicating that the perylene structure is not so cruelly damaged compared with

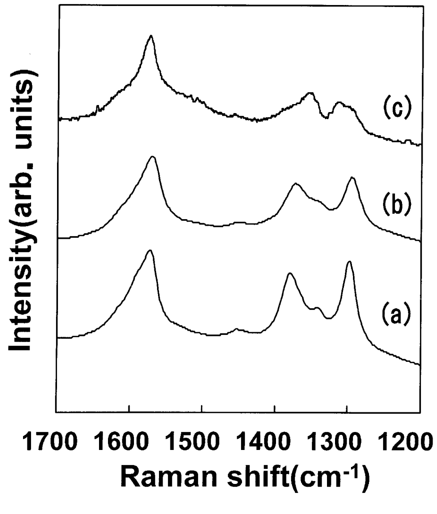

Fig. 3. Raman spectra for the films prepared by ELA at fluences of 0.5 (a), 1.0 (b) and 1.5 (c) J cm^{-2} pulse^{-1}.

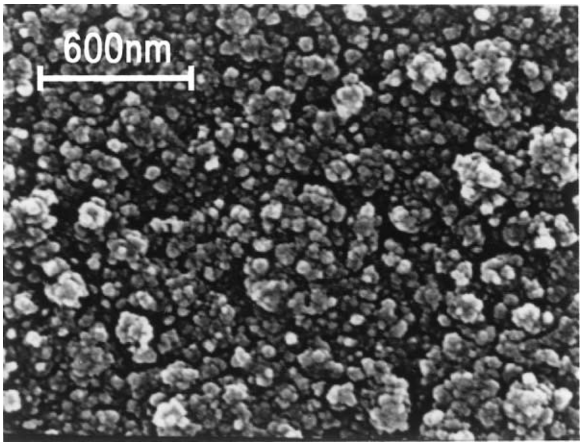

Fig. 4. Surface morphology of a film prepared on a substrate at 20°C by ELA at a fluence of 1.0 J cm^{-2} pulse^{-1}.

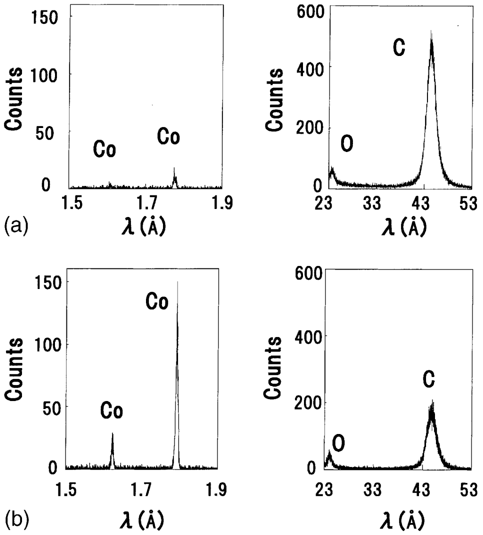

Fig. 5. EPMA results for carbon, oxygen and Co in a film prepared by ELA at a fluence of 1.0 J cm^{-2} pulse^{-1} on a substrate at 20°C (a) together with those of PTCDA/Co target ([PTCDA]/[Co] = 1/4) (b).

our previous study. Considering from the fact that the peak almost completely vanished for the film prepared by ELA of PTCDA at more than 1.0 J cm^{-2} pulse^{-1} on a substrate at 300°C [7], it is surprising to note that the peak at 1290 cm^{-1} is detected for the film prepared even at 1.5 J cm^{-2} pulse^{-1}, though it is a little bit broadened. This fact is quite interesting since it means that Co irradiated with XeCl excimer laser beams not only acts as a catalysis improving elimination of anhydride groups of PTCDA but also preserves perylene structure from damaging by high-intensity laser beam.

Fig. 4 shows the surface morphology of a film prepared by ELA of PTCDA/Co target at a fluence of 1.0 J cm^{-2} pulse^{-1} on a substrate at 20°C. It consists of small particles with various diameters ranging from 10 to 100 nm. The morphology is not so different from that of the films prepared by ELA of PTCDA [18]. According to observation of section morphology of the film, particles were closely packed. EPMA results for carbon, oxygen and Co in a film prepared by ELA of PTCDA/Co target at a fluence of 1.0 J cm^{-2} pulse^{-1} on a substrate at 20°C are shown in Fig. 5 together with those of PTCDA/Co target ([PTCDA]/[Co] = 1/4). Extremely small amount of Co is detected in the film, compared with the target. Here, considering the fact that thickness of the film is much thinner than that of the target, it should be noted that direct comparison of the peak intensities of Co would not be wise because of the large penetration depth over the order of micrometers. Therefore, the small quantity of Co was comcluded by comparison of peak intensity ratios of Co against carbon between the film and the target. According to the analysis, Co content is reduced to at least less than 10% of that in the target, though unevenness of the distribution is recognized to some extent. The result indicates that almost all the Co remains in the target or possibly floats in a reaction chamber as organometallic compounds. We preliminarily ascertained that pellets of neat Co powder did not get ablated by ELA with a 308 nm beam at a fluence less than 2.0 J cm^{-2} pulse^{-1}. Co detected in the film was probably due to getting a lift in PTCDA ablated with large translation energy from the target. Although such a contamination of Co may be an obstacle to form pure PPN from practical point of view, it should be admirable at present in the point that Co content in the film is limited to less than 10% of that in the target regardless of simultaneous ablation. Oxygen is detected in the film to some extent. This is probably due to the adsorption of oxygen and/or H_2O in the air after leaking in addition to the small amount of PTCDA possessing anhydride groups fully or partially remaining without elimination.

3.2. Morphology and component change of target surface of PTCDA/Co after laser beam irradiation

Hereafter, let us focus our attention on the morphology change of a PTCDA/Co target surface after laser beam irradiation. Fig. 6 shows the SEM images of the surface of PTCDA/Co target after 1500 pulses irradiation at fluences of 0.5, 1.0 and 1.5 J cm^{-2} pulse^{-1} together with that before irradiation. Bamboo shoot-like projections can be observed on each surface after irradiation. By increasing the fluence, they become sharp and trace of molten material appears at the top of each projection. EPMA results for carbon, oxygen and Co at the summit and the mountainside (spots A and B in Fig. 6(c), respectively) of a project formed by ELA of PTCDA/Co target at a fluence of 1.0 J cm^{-2} pulse^{-1} are shown in Fig. 7. Co content against that of carbon at the summit is a little bit larger than that at the mountainside, suggesting that radiation of the laser beam melts Co powder to precipitate at the summit. It is intriguing to note the content of oxygen at the summit is also larger. It is of no doubt that almost all the oxygen detected never stems from

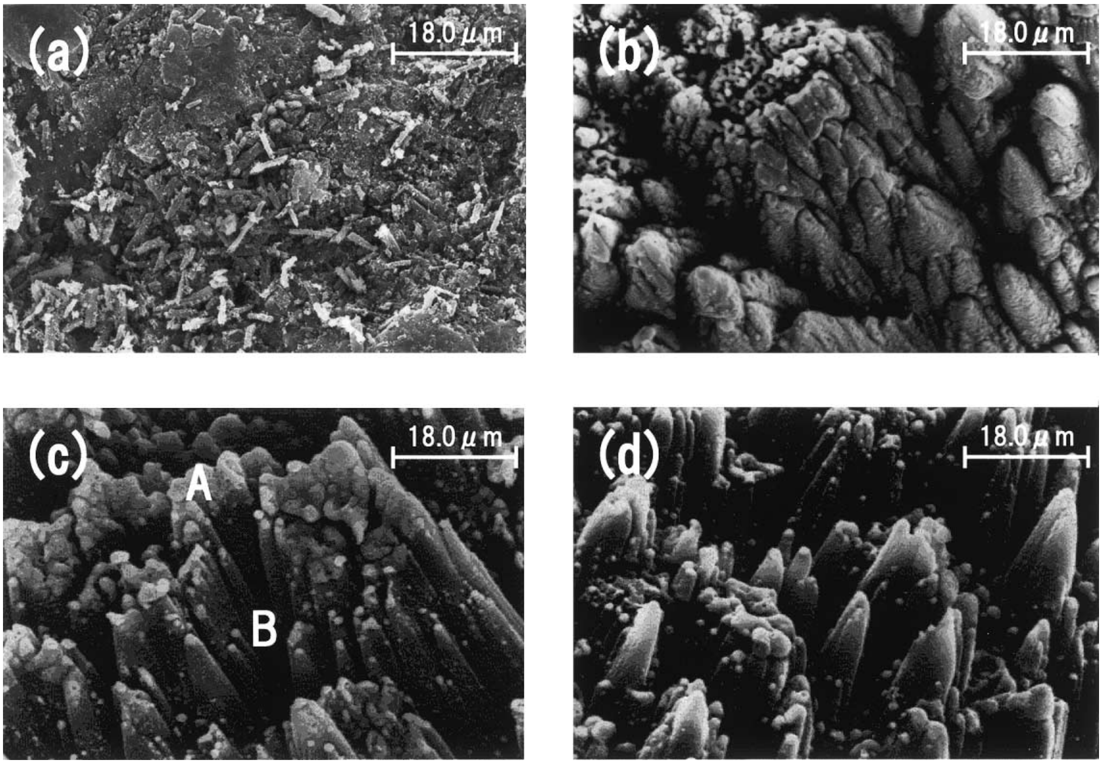

Fig. 6. SEM images of the surface of PTCDA/Co target after 1500 pulses irradiation at fluences of 0.5 (b), 1.0 (c) and 1.5 (d) J cm^{-2} pulse^{-1} together with that before irradiation (a).

oxidation of Co due to the reaction of oxygen in the chamber, but from anhydride groups of PTCDA, considering from the result of irradiation of a pellet of Co powder with XeCl laser beam where only a quite little bit of the oxygen detected.

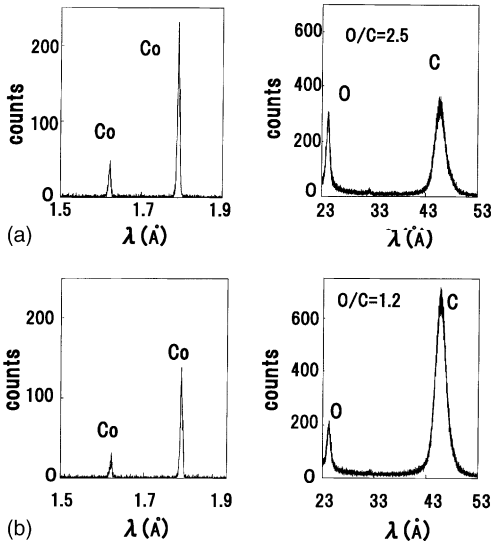

Fig. 7. EPMA results for carbon, oxygen and Co at the summit (a) and the mountainside (b) (spots A and B in Fig. 6(c), respectively) of a project formed by ELA at a fluence of 1.0 J cm^{-2} pulse^{-1}.

Drastic decrease in the emission intensity with increasing the number of laser shot mentioned above seems to ascribe to precipitation of the molten material with high Co content which is hard to ablate at a low fluence as we applied.

3.3. Annealing effect on structure and electric conductivity of the film

Let us go back to the subject of the structure of deposited film prepared by ELA of PTCDA/Co. Considering from the results of FT-IR and Raman spectroscopies, species possessing well-defined perylene structure without anhydride groups must be deposited in great quantity. We call these species perylene radicals tentatively in this paper, though the direct evidence for the existence of this species is not obtained experimentally at present. The electric conductivity of the film by ELA of a PTCDA/Co target prepared on a substrate at 20°C was 1.0×10^{-4}–1.0×10^{-5} S cm^{-1} in an evacuated chamber. Immediately after leaking of reaction chamber, however, abrupt decrease in the conductivity down to less than 1.0×10^{-6} S cm^{-1} was observed. This suggests that perylene radicals do not recombine each other sufficiently and that π-conjugated systems do not develop well. The relatively high conductivity of this film in an evacuated chamber may be due to surviving radicals in the film.

For the purpose of formation of PPN with the longer π-conjugated ladder, annealing at various temperatures was performed for this film. Fig. 8 shows the change in electric

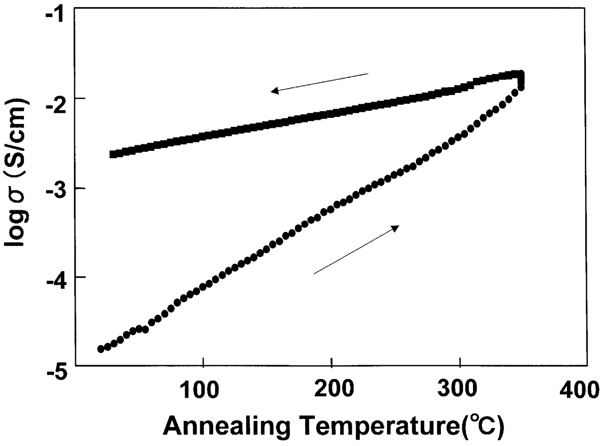

Fig. 8. Change in electric conductivity by changing substrate temperature between 20 and 350°C in vacuum.

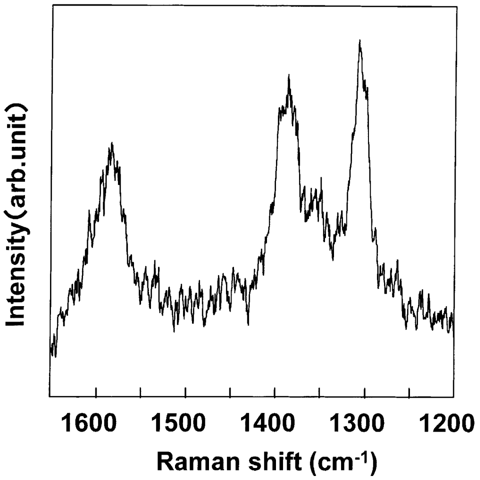

Fig. 9. Raman spectrum for a film prepared by ELA at a fluence of 1.0 J cm^{-2} pulse^{-1} and annealed at 350°C.

conductivity by changing substrate temperature between 20 and 350°C in vacuum. The film prepared on a substrate at T_S of 20°C was heated up to 350°C in 2 h and then continue heating when kept at that temperature for 30 min. Fair annealing effect can be recognized. Annealing at 350°C resulted in increase in electric conductivity from 1.0×10^{-5} to 1.0×10^{-3} S cm^{-1}, two orders of magnitude higher compared with that before annealing. In addition, after annealing, little decrease in the conductivity was observed when the reaction chamber was leaked. After 30 min annealing, temperature dependence of the electric conductivity seems to be fitted better by the Mott–Davis ($\log \sigma$ vs. $T^{-1/4}$) plot [22] than by the Arrhenius ($\log \sigma$ vs. T^{-1}) plot, suggesting that the electric conduction arises from variable range hopping of carriers. For further clarification of the mechanism, electric conductivity of the films annealed at various T_S will be measured over wider range of temperature in the near future.

Fig. 9 shows a Raman spectrum for a film annealed at 350°C. It is found that the sharp peak at 1290 cm^{-1} still remains. Table 1 lists the electric conductivities at room temperature measured in air and FWHM values of the peak at 1290 cm^{-1} for the films prepared by ELA under various conditions, in comparison with that for PPN prepared with vapor-phase polymerization method by Murakami et al. [14]. For the films annealed at 350°C, a sharp peak at 1290 cm^{-1} and complete disappearance of IR peaks at 1750 and 1780 cm^{-1} (not shown) suggest the formation of better-defined PPN, though the conductivity of 1.0×10^{-3} S cm^{-1} is a little bit lower compared with that of PPN by Murakami et al. [14], or our film prepared on a substrate at 300°C by ELA of PTCDA [18]. Improvement can be expected by minute control of annealing conditions.

Table 1
Electric conductivity at room temperature measured in air and FWHM values of the peak at 1290 cm^{-1} for the films prepared by ELA under various conditions, together with that for PPN prepared with vapor-phase polymerization method by Murakami et al. [14]

Sample	Target	Sub. Temperature (°C)	Fluence (J cm^{-2} pulse^{-1})	FWHM at 1290 cm^{-1} (cm^{-1})
A	PTCDA/Co	20	0.5	20
B	PTCDA/Co	20	1.0	22
C	PTCDA/Co	20	1.5	37
D	PTCDA/Co	20–200[a]	1.0	22
E	PTCDA/Co	20–350[b]	1.0	23
F	PTCDA/Co	300	1.0	23
G[c]	PTCDA	300	0.25	26
H[d]	PTCDA	300	1.0	–[f]
PPN[e]				22

[a] Annealed at 200°C after prepared at 20°C.
[b] Annealed at 350°C after prepared at 20°C.
[c] Estimated from Ref. [18].
[d] Estimated from Ref. [7].
[e] Estimated from Ref. [14].
[f] Not detected.

4. Conclusion

PTCDA/Co mixture targets were ablated with XeCl laser beams at various fluences ranging from 0.5 to 2.0 J cm^{-2} pulse^{-1}. Structures and electric/electronic properties of deposited films, together with the morphology change of PTCDA/Co targets were investigated by SEM, FT-IR and Raman spectroscopies, EPMA, and electric conductivity measurements. As for the mixture target ([PTCDA]/[Co] $= 1/4$) using Co powder with a diameter of 30 nm, IR spectroscopy showed that carboxylic dianhydride-free fragments were effectively produced by ELA at more than 1.0 J cm^{-2} pulse^{-1}. The efficiency of elimination of anhydride groups was quite low for the powder with the 1–2 μm diameter. Raman spectra for the films prepared by ELA of the PTCDA/Co target at various fluences showed apparently three peaks at 1600, 1380 and 1290 cm^{-1} completely consistent with that for PPN prepared by Murakami et al. [14]. The peak at 1290 cm^{-1} was detected for the film prepared even at a fluence as high as 2.0 J cm^{-2} pulse^{-1}. This finding is quite interesting since it means that Co irradiated with XeCl excimer laser beams not only acts as a catalysis assisting elimination of anhydride groups of PTCDA but also preserves perylene structure from damaging by high-intensity laser beam. The film prepared by ELA of a PTCDA/Co target at a fluence of 1.0 J cm^{-2} pulse^{-1} on a substrate at 20°C consisted of small particles with diameters ranging from 10 to 100 nm, not so different from that of the films prepared by ELA of PTCDA. Only an extremely small amount of Co was detected in the film. Bamboo shoot-like projections could be observed on each target surface of PTCDA/Co after irradiation. As laser fluence was increased, they became sharper and trace of molten material appeared at the top of each projection. Co content at the summit was a little bit larger than that at the mountainside. The electric conductivity of the film by ELA of a PTCDA/Co target prepared on a substrate at 20°C was 1.0×10^{-4}–1.0×10^{-5} S cm^{-1} in an evacuated chamber. Immediately after leaking of reaction chamber, however, abrupt decrease in the conductivity down to less than 1.0×10^{-6} S cm^{-1} was observed. Fair annealing effect can be recognized. Annealing at 350°C resulted in an increase in electric conductivity from 1.0×10^{-5} up to 1.0×10^{-3} S cm^{-1}. In addition, after annealing, little decrease in the conductivity was observed when the reaction chamber leaked. For the films annealed at 350°C, the presence of a sharp peak at 1290 cm^{-1} with FWHM comparable to that of PPN prepared by Murakami et al. [14] and complete disappearance of IR peaks at 1750 and 1780 cm^{-1} for the film suggest the formation of a better-defined PPN.

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