Thermal treatment effects on N-alkyl perylene diimide thin-film transistors with different alkyl chain

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The authors report that thermal treatment effect on various N,N'-dialkyl-3,4,9,10-perylene tetracarbxylic diimides [PTCDI-Cn, alkyl–dodecyl (n=12), butadecyl (n=14), octadecyl (n=18)] thin-film transistors (TFTs) depends on the substituted alkyl chain length. It is clearly demonstrated that there are two kinds of molecular movements during the thermal treatment on PTCDI films; molecular rearrangement in the same layer and molecular migration from the lower layer to the upper layer. The former is directly related to the grain growth and can be controllable by applying an external electric field. The latter is also related not only to the grain growth but also to the formation of cracks between grains. These two movements show opposite dependence on the alkyl chain length during the thermal treatment; the former is more active in longer alkyl chain, but the latter in shorter one. However, they also have opposite effect to TFT performance, and PTCDI films with longer alkyl chains have great advantage on TFT performance for the thermal treatment. Consequently, PTCDI-C18 TFTs show the highest electron mobility as large as 1.2 cm^2/V s after the thermal treatment at 140 °C. © *2010 American Institute of Physics*. [doi:10.1063/1.3525997]

I. INTRODUCTION

Organic thin-film transistor (OTFT) has attracted considerable interests because it has strong potential for applications to flexible, large-area, and low-cost electronic devices, and plenty of reports were published in recent years.[1–13] To realize such applications, it is strongly desirable to use OTFTs as a part of complementary circuits, and which needs both of p-type and n-type TFTs with similar performance.[14–18] There have been many reports of p-type OTFTs with very high performance, but only a few reports for n-type ones. This imbalance between p- and n-type OTFTs comes from both of the intrinsic and the extrinsic property of organic materials, and many intensive researches were pumped into developing high-performed n-type OTFT by elucidating the basic transport mechanism, by controlling the surface state of electrode, by synthesizing new materials, and so on.[14–25]

Perylene diimide derivatives are one of the most important n-type organic materials because of their relatively strong electron affinities and easily controllable properties by changing the substituent on the imide N atoms or on the perylene backbone. N,N'-dialkyl-3,4,9,10-perylene tetracarboxylic diimide (PTCDI-Cn), especially, is very promising n-type materials with a high electron mobility, and studied intensively from the first stage of researches on OTFTs.[14–17,26–39] For example, Horowitz *et al.* have reported the first n-type organic TFT made of N,N'-diphenyl PTCDI 14 yr ago.[26] After 6 yr, Malenfant *et al.* have reported a N,N'-dioctyl PTCDI TFT with fairly high electron mobility as high as 0.6 cm^2/V s.[27] Jones *et al.* have synthesized core-cyanated PTCDI with fluorinated alkyl chains, and reported an air-stable TFT with electron mobility of 0.64 cm^2/V s.[28] Chesterfield *et al.* have reported strikingly high electron mobility in N,N'-dioctyl PTCDI TFT of up to 1.7 cm^2/V s through exquisite control of deposition condition,[29] and after that many research groups have poured their efforts to develop high-mobility n-type PTCDI OTFTs by modifying the molecular structure with core-substitution or with changing the substituent on the imide N atoms, by controlling deposition condition, by optimizing device structure such as the surface state of dielectrics and electrode contact, and so on.[30–39]

On the other hand, many of OTFTs have shown a large enhancement in carrier mobility through the thermal treatment during film deposition[29–31,33–37,40–44] and/or after film formation.[44–51] Materials with long alkyl chains, especially, have shown drastic morphological change after the thermal treatment, which lead to large change in the performance of TFT.[29–31,37,42–45] Attached long alkyl chains added highly mobile nature to the molecular backbone, and this highly mobile nature makes it possible to change the film morphology greatly with a thermal treatment at a rather low temperature. For the thermal treatment during the film deposition, the effect of the thermal treatment is widely known to be related to the density of nucleation sites, film growth rate, and the surface free energy of substrates.[44] For the thermal treatment after film deposition, however, the detailed mechanism is not so much studied yet although it seems very much simple

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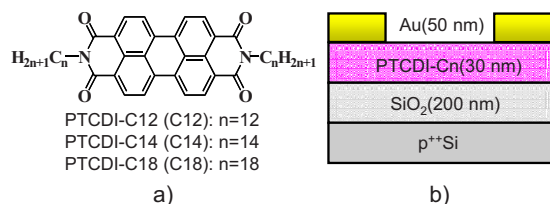a)Electronic mail: musubu@shinshu-u.ac.jp.

FIG. 1. (Color online) (a) Molecular formulae of the various PTCDIs used in this study, and (b) a schematic illustration of a top-contact PTCDI TFT structure.

FIG. 2. (Color online) DSC curves of various PTCDIs used in this study.

comparing with that of the thermal treatment during film deposition because there is not nucleation step nor film growth step.

Our research group have already reported that N,N′-ditridecyl PTCDI (PTCDI-C13) TFTs exhibited very high electron mobility of 2.1 cm^2/V s by the thermal treatment at 140 °C after film deposition.[52] The thermal treatment improved the crystallinity and the grain size of the deposited film which caused the drastic increase in electron mobility. At higher treatment temperature (200 °C), the grains grown more largely and the crystallinity was also improved, but the electron mobility of the film was lowered. We have concluded that this decrease mainly due to the cracks between grains which was clearly appeared in AFM observation. If it is possible to control the grain growth without such cracks, we can get much higher device performance with larger grains. To achieve this purpose, it is strongly desirable to elucidate the relation between the temperature of the thermal treatment and the film morphology change in various PTCDI films with different alkyl chain length.

In this paper, we report thermal treatment effect on various PTCDI TFTs with different alkyl chain length. It is clearly shown that there are two kinds of molecular movements during the thermal treatment; molecular rearrangement in the same layer and molecular migration from the lower layer to the upper layer. These two kinds of molecular movements show opposite dependence on the alkyl chain length for the thermal treatment and also opposite effect on TFT performance. Consequently, PTCDI with longer alkyl chains is more favorable for enhancement of TFT performance by the thermal treatment.

II. EXPERIMENTAL

We have used three kinds of PTCDIs; N,N′-didodecyl (C12), N,N′-dibutadecyl (C14), N,N′-dioctadecyl (C18) PTCDI. Their molecular formulae are shown in Fig. 1. All of them were synthesized with modified methods following literatures.[53] The phase transition temperatures of them were deduced from the differential scanning calorimetry (DSC) data measured with a SII DSC6200.

Figure 1 also shows a schematic illustration of the device structure of top-contact PTCDI TFTs used in this study. The devices were prepared on heavily-doped p-type silicon wafers with thermally grown SiO$_2$ surface (200 nm in thickness) which acted as a gate electrode and a gate dielectric (specific capacitance of 18 nF/cm^2), respectively, PTCDI films with thickness of 30 nm were deposited on nontreated SiO$_2$ surface by thermal evaporation technique at the depo-
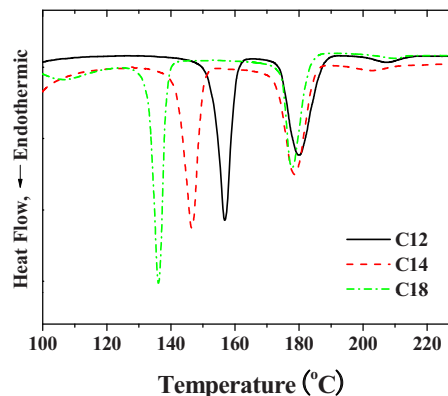
sition rate of 0.3 Å/s. The substrate temperature was not controlled intentionally. Au was thermally evaporated on the PTCDI film through a shadow mask to form the source-drain electrodes. The channel length and width were 100 μm and 2 mm, respectively.

TFT characteristics were measured in a vacuum (below 5×10^{-4} Pa) at room temperature with an Agilent Technologies B1500A semiconductor device analyzer. After the first measurement the devices were transferred to a vacuum oven and treated for 1 h at a controlled temperature (120–220 °C), and then slowly cooled down to room temperature with keeping the vacuum. The devices were exposed to the atmosphere in short before and after the thermal treatment. After the thermal treatment, the second measurement of TFT characteristics was carried out again under the same condition with the first measurement. We have also tried to apply only a gate bias during the thermal treatment with the same semiconductor device analyzer in another evacuated temperature controllable probe station system.

The surface morphologies of films were observed with a Seiko SPA-400 atomic force microscope (AFM) with a tapping mode. PTCDIs films for the AFM observation were prepared with the same condition of those for the electrical measurement.

III. RESULT AND DISCUSSION

Figure 2 shows the DSC curves of PTCDIs. According to the literatures, the peaks near 140–160 °C of DSC data represent the phase transition of PTCDIs between two crystalline phases.[54] These data show us that the phase transition temperature of PTCDIs is shifted to lower temperature as increasing the alkyl chain length, from 156.8 °C for C12 to 136.1 °C for C18, which is a general phenomenon in alkyl chain substituted molecules.[54–56] The long alkyl substituents add highly mobile nature to a rigid perylene skeleton. As the alkyl chain length become longer the mobile nature of molecules becomes stronger,[57,58] which results in the phase transition temperature shift to lower temperature. This result means that PTCDIs with longer alkyl chains are more easily influenced by heat treatment than shorter ones, and again means that the morphology of those films can be changed largely at a lower temperature.
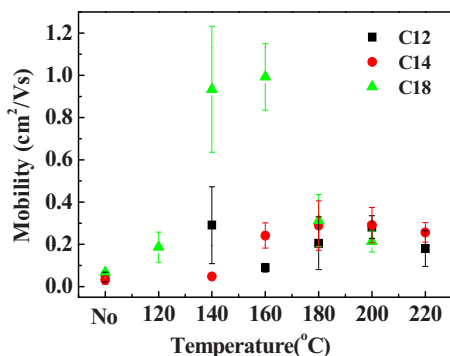
FIG. 3. (Color online) Change in the electron mobility of various PTCDI TFTs with the thermal treatment.

FIG. 5. (Color online) XRD patterns of nontreated and thermal treated C18 films at 140 and 160 °C.

We have checked the change in the field effect electron mobility in the saturation regime of various PTCDI TFTs by the thermal treatment, and summarized in Fig. 3. Before the thermal treatment, the electron mobility of all the PTCDI TFTs was not so much different; about $0.03-0.07$ cm^2/V s. With the thermal treatment, the mobility was increased largely in all the PTCDI TFTs; up to 0.4 cm^2/V s for C12 and C14, and even 1.2 cm^2/V s for C18. The mobility of C18 TFTs, especially, increased abruptly at 140 and 160 °C. This increase is very similar to another PTCDI TFTs such as PTCDI-C13.[52] The main reason for the mobility increase by the thermal treatment was considered to be the morphological change in the films. The same analysis can be applied to the above results when we consider the surface morphology of the C18 films as shown in Fig. 4. It is very clear that the grain size was enlarged by the thermal treatment, and grains grown more largely at 160 than 140 °C. This grain growth resulted in the reduction in grain boundaries where the electron transport is delayed or trapped, and hence the increased carrier mobility. Grain growth by thermal treatment was also observed in x-ray diffraction (XRD) data as shown in Fig. 5. Before thermal treatment, we can see somewhat broad (001) peak and only very small (003) peak. By thermal treatment, the width of (001) peak was decreased and (002) and (003) peaks are increased largely. These results indicate that the grain size and the crystallinity of C18 film were improved by thermal treatment, which is identical with the results of the mobility change and the AFM observation.

This kind of grain growth is considered to be occurred by the molecular rearrangement. The molecular rearrangement is derived by the molecular interaction, and the dipole moment of molecules may be a key factor for this interaction. It is well known that imide has a large dipole moment, and it is also reported that PTCDI has very large polarizabilit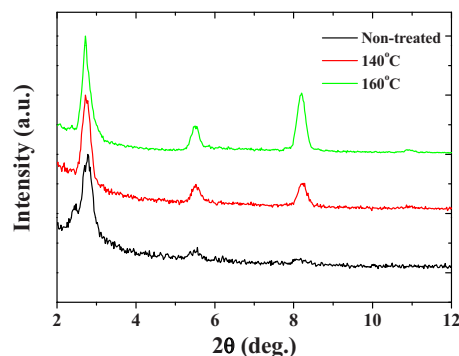y, as large as >1000 au along the long axis of the molecule.[59] These kinds of molecules with large dipole units and large polarizability can be affected by external forces such as an electric field.[60] By applying an electric field on the substrate during film deposition, the grain growth can be changed; enhanced or restrained. Actually, the grain growth at 160 °C was restrained by applying −100 V gate voltage during thermal treatment. AFM image of that film show us that the grain size is much smaller than that of only thermally treated film without applying bias, as shown in Fig. 6, and is almost the same with that of a nontreated film [Fig. 4(a)]. It seems that applying +100 V gate voltages did not affect on the grain size, but the space between grains were enlarged and the number of upper layer was also increased which resulted into the increase in the roughness of the film [Fig. 6(c)]. This result strongly supports that the dipole moment of molecules is a key factor for the grain growth in thermally treated films, and the grain size and the morphology of films can be controlled by an external forces such as an electric field.

It is noticeable that the electron mobility of C18 film increased very largely by the thermal treatment at 140 and 160 °C, but those of C12 and C14 films a little, as arranged in Fig. 3. This difference can be explained by considering the film morphology too. From AFM images of C12, C14, and C18 films treated at 160 °C shown in Fig. 7, we can see two important points. The first is the grain size of films, and the second is the existence of cracks. The grain size of C18 film is smaller than that of the other films. Therefore, we can estimate that the electron mobility of C18 film will be lower than those of the other films. But the results are not the case. For that reason, we have paid attention to the existence of cracks. There are many cracks between grains in C12 and C14 films, but no such cracks in C18 film. Those cracks can affect the electron transport significantly, and lead to lowering the electron mobility as described for PTCDI-C13 films.[52]
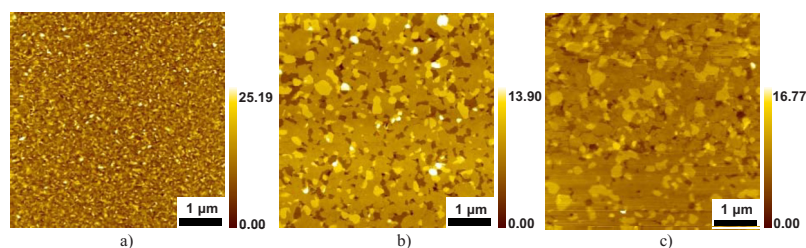

FIG. 4. (Color online) Surface morphology of (a) non-treated, (b) 140 °C thermal treated, and (c) 160 °C thermal treated C18 films obtained by AFM observation.
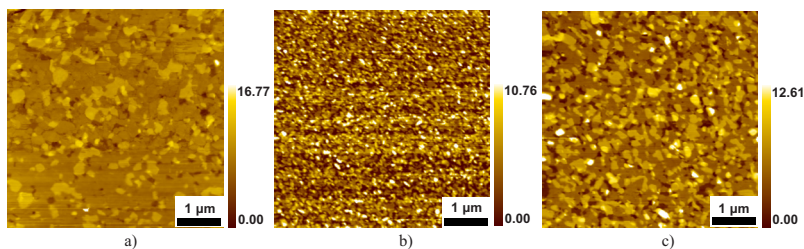
FIG. 6. (Color online) AFM surface images of 160 °C thermal treated C18 films (a) without applying a gate voltage, (b) with applying −100 V gate voltage, and (c) with applying +100 V gate voltage.

The most reasonable cause of appearing cracks after the thermal treatment may be the difference of thermal expansion between organic films and the Si substrate.[37,52] Organic materials usually have much higher thermal expansion coefficient (α) than that of Si, and that difference will bring out internal stresses in organic films deposited on Si substrate during the thermal treatment, especially during cooling process. PTCDI films will expand more largely than Si substrate during heating process and PTCDI molecules will move and rearrange their position so as to reduce such internal stresses. During cooling process, PTCDI films will contract more largely than Si substrate, but it is not easy to reduce internal stresses by molecular rearrangement over a wide range because thermal energy is not high enough. Instead, PTCDI molecules will contract within their domains which lead to appearing cracks between grains.

In our case, however, this mechanism cannot explain sufficiently why many cracks are observed in C12 and C14 films after the thermal treatment at 160 °C but not in C18 film. Generally, thermal expansion coefficient (α) will be increased with longer alkyl chain. Therefore, α of C18 film may be higher than C12 and C14, and cracks will be observed more abundantly in C18 film than C12 and C14 films. The above result, however, was completely opposite. Similar cracks are appeared in C18 film after thermal treatment at 180 °C and higher temperature. This result means that there is/are another factor(s) for producing cracks between grains.

We have focused on the area of the upper layer of films. C12 and C14 films show somewhat large grains of the upper layer but C18 much smaller ones. We can infer an interesting process of the grain growth of the upper layer during the thermal treatment from comparing the morphology of films treated at 140 and 160 °C. Figure 8 shows us representative AFM surface images of C14 films thermally treated at 140 and 160 °C. By increasing the treating temperature from 140 to 160 °C, the number and the area of cracks between grains are increased. At the same time, it is also very clear that the area of the upper layer was increased. This result strongly supports that PTCDI molecules may migrate from the lower layer to the upper layer, and this migration can enhance the formation of cracks. A similar observation for

α-sexythiophene molecules was reported by Dinelli *et al.*[48] This mechanism can explain why many cracks were observed in C12 and C14 films after the thermal treatment at 160 °C but not in C18 film. Generally, molecular interaction becomes stronger with increasing alkyl chains and C18 may have stronger molecular interaction than C12 and C14. The strong molecular interaction between C18 molecules can effectively hinder the molecular migration from the lower layer to the upper layer, and this can reduce or interrupt the formation of cracks. Actually, similar cracks with C12 and C14 films were observed in a C18 film after thermal treatment at 180 °C and higher temperature, and the electron mobility was decreased abruptly as shown in Fig. 3. Therefore, we can conclude that the migration of PTCDI molecules from the lower layer to the upper layer is one of the important causes of the formation of cracks.

As mentioned in the first part of this section, the mobile nature of PTCDI molecules comes from the substituted alkyl chains, and as the alkyl chain length becomes longer the mobile nature of molecules becomes stronger. Therefore, the molecular rearrangement during the thermal treatment will be more vigorous in C18 molecules than C12 and C14, which is represented by the shift in the phase transition temperature to lower temperature as increasing the alkyl chain length. However, the migration of molecules from the lower layer to the upper layer is completely opposite; weaker with longer alkyl chains. Important thing is that these two kinds of molecular movements during the thermal treatment— molecular rearrangement in the same layer and molecular migration from the lower layer to the upper layer—can work together more beneficially to TFT performance in PTCDI molecules with longer alkyl chains. As the substituted alkyl chains become longer the molecular rearrangement in the same layer will be more active at the same temperature, and will lead to larger grain growth, hence higher TFT performance. Simultaneously, the molecular migration from the lower layer to the upper layer will be suppressed, which results in fewer cracks between grains, hence higher FET performance too. Much higher electron mobility of C18 films after the thermal treatment at 140 and 160 °C than C14 and C12 can be explained by these two molecular movements.
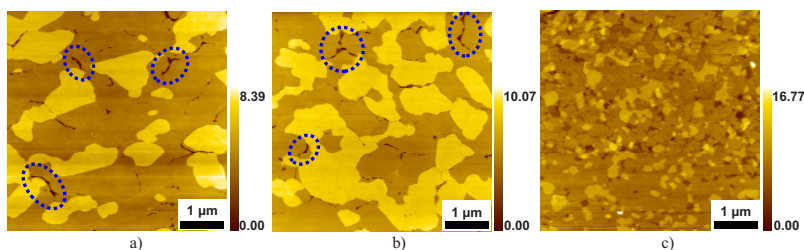

FIG. 7. (Color online) AFM surface images of (a) C12, (b) C14, and (c) C18 films thermally treated at 160 °C. Marked spots with dotted-circles show deep cracks between grains.
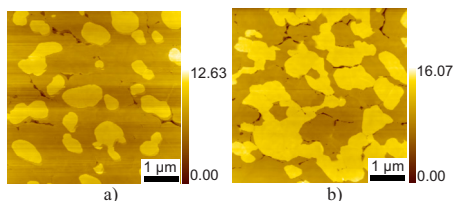
FIG. 8. (Color online) AFM surface images of C14 films thermally treated at (a) 140 °C, and (b) 160 °C. Not only the cracks but also the grain sizes of the upper layer of film were clearly increased by the thermal treatment at 160 °C comparing with those of film treated at 140 °C.

IV. CONCLUSION

In this study, we have clearly shown that there are two kinds of molecular movements during the thermal treatment on alkyl substituted PTCDI films; molecular rearrangement in the same layer and molecular migration from the lower layer to the upper layer. The former comes from the mobile nature of PTCDI molecules which is caused by the substituted alkyl chains, and is directly related to the grain growth. It is also shown that this molecular rearrangement can be controllable by applying an external electric field. The latter is related to the formation of cracks between grains. These two movements show opposite dependence on the alkyl chain length during the thermal treatment; the former is more active in longer alkyl chain, but the latter in shorter one. However, grain growth and the formation of cracks, hence those two molecular movements, have opposite effect on the TFT performance of PTCDI films, and PTCDI films with longer alkyl chains have more advantage on enhancement of TFT performance by the thermal treatment. Consequently, PTCDI-C18 TFTs show the highest electron mobility as large as 1.2 cm^2/V s. This electron mobility is fairly high value, but still lower than that of PTCDI-C13, 2.1 cm^2/V s. There may be other effects of alkyl chains on TFT performances such as odd-even effect of alkyl chains, and it remains to be studied.

ACKNOWLEDGMENTS

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