

Structural Characterization of Mixed-Stack Charge Transfer Films of 2-Octadecyl-7,7,8,8-tetracyanoquinodimethane and 5,10-Dimethyl-5,10-dihydrophenazine Prepared by the Langmuir–Blodgett Technique and Donor Doping. 3. Thickness Dependence of Thermal Behavior Investigated by Ultraviolet–Visible–Near-Infrared and Infrared Spectroscopies and X-ray Diffraction

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Thermal behavior of mixed-stack charge transfer (CT) films prepared by 5,10-dimethyl-5,10-dihydrophenazine ((Me)₂P) doping of Langmuir–Blodgett (LB) films of 2-octadecyl-7,7,8,8-tetracyanoquinodimethane (octadecyl-TCNQ) has been investigated by using X-ray diffraction and ultraviolet–visible–near-infrared (UV–vis–NIR) and infrared (IR) spectroscopies. Temperature-dependent changes in the UV–vis–NIR and IR spectra reveal that donors (D, (Me)₂P) dedope from the CT complex, resulting in a restoration of acceptors (A, octadecyl-TCNQ) to their neutral state in the temperature range 70–90 °C. The dedope temperature was determined to be 80, 84, 86, and 88 °C for the 1-, 3-, 7-, and 11-layer CT films, respectively, by monitoring an α_{g} C≡N stretching band of the TCNQ chromophore in the temperature-dependent IR spectra. From room temperature to 60 °C, the molecular arrangement and orientation of D and A in the well-ordered multilayered CT complex seem to be thermally stable because the X-ray diffraction pattern and UV–vis–NIR and IR spectra are nearly unchanged. The dedoping begins in the temperature range 60–70 °C, but it proceeds rather slowly for the multilayered CT films. Dramatic structural changes take place in the region of 70–90 °C, where the X-ray diffraction pattern and characteristic absorption bands of the CT complex in the UV–vis–NIR and IR spectra disappear. The thickness-dependent thermal behavior may be attributed to the longitudinal interactions between the sheetlike multilayered microcrystals as well as to the interaction between the first layer and a CaF₂ substrate.

Introduction

In the preceding papers,^{1–3} the structure, molecular orientation, morphology, and annealing effects have been reported for the mixed-stack charge transfer (CT) complex films of 2-octadecyl-7,7,8,8-tetracyanoquinodimethane (octadecyl-TCNQ) doped by 5,10-dimethyl-5,10-dihydrophenazine ((Me)₂P). The present paper, as a successive report, discusses the thermal behavior of the mixed-stack CT films investigated by X-ray diffraction and ultraviolet–visible–near-infrared (UV–vis–NIR) and infrared (IR) spectroscopies. An exploration of thermal behavior of the two-dimensional CT films is of great importance because of the following reasons. First, the thin films may exhibit thermal behavior different from that of crystals because of their two-dimensional characteristics. Second, it provides an indication of the effects of physical aging. Furthermore, the study may yield significant ramifications for the service life of microelectronic devices utilizing this type of the CT complex film.

We previously studied the thermal behavior of LB films of octadecyl-TCNQ^{4–6} and mixed-stack CT films of octadecyl-TCNQ doped with 3,3',5,5'-tetramethylbenzidine (TMB).^{7,8} For the former the order–disorder phase transition and annealing effects were investigated by atomic force microscopy (AFM)

and ultraviolet–visible (UV–vis) and IR spectroscopies.^{4–6} These studies elucidated that the thermal behavior of the LB films depends upon the number of layers.^{4–6} For example, the phase transition temperature increases with it. It was also found that the annealing effects on the structure and morphology of the LB films of octadecyl-TCNQ are largely different between the one- and multilayer films.⁶

Thermally induced structural and morphological changes in the CT films of octadecyl-TCNQ and TMB were explored by UV–vis–NIR and IR spectroscopies and AFM. Again, a strong dependence of the changes on the number of layers was noted. The temperature-dependent IR spectra revealed that the dedope temperature was 128, 134, 144, and 146 °C for the 1-, 3-, 7-, and 11-layer CT films of octadecyl-TCNQ and TMB, respectively.⁷ The progressive thermal process of the 1-layer CT complex film changes into a sudden variation one as the number of layers increases. Studies of the annealing effects show that a rearrangement process of needle-like microcrystals takes place but molecular arrangement remains unchanged if the CT films are annealed below their dedope temperatures. In contrast, the donor molecules dedope from the CT complexes and the acceptor molecules are restored from the CT state to the neutral one if the CT films are heated above their dedope temperatures. These fundamental investigations of the LB films of octadecyl-TCNQ, as well as the mixed-stack CT films of octadecyl-TCNQ doped with TMB, have provided plentiful reference information for the present experimental study of a new type of mixed-stack CT complex films.

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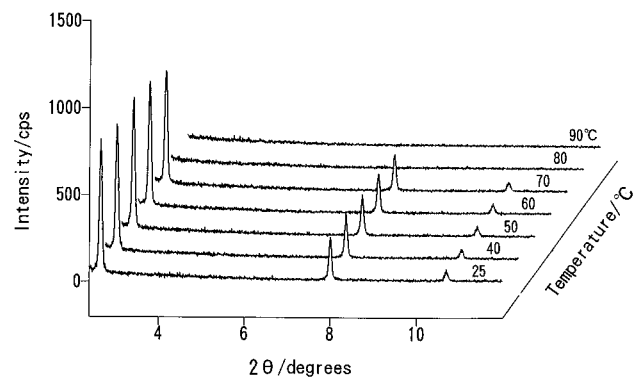


Figure 1. X-ray diffraction patterns of an 11-layer mixed-stack CT film of octadecyl-TCNQ doped by (Me)₂P measured at 25, 40, 50, 60, 70, 80, and 90 °C.

Experimental Section

Sample Preparation. The procedure for the preparation of the mixed-stack CT films of octadecyl-TCNQ and (Me)₂P was described in detail in the preceding paper.¹ In the experiments presented in this paper, the 1-, 3-, 7-, and 11-layer CT films were prepared on CaF₂ substrates by (Me)₂P doping of LB films of octadecyl-TCNQ in the vapor phase for 6, 12, 20, and 24 h, respectively, to ensure complete formation of the CT complexes.

Spectroscopy and X-ray Diffraction. The instruments employed for the UV–vis–NIR and IR spectroscopic measurements and X-ray diffraction experiments were the same as those described in the first paper.¹ The apparatus and sample-handling technique for measuring the UV–vis–NIR and IR spectra and X-ray diffraction patterns at elevated temperatures were reported previously.⁴

Results

X-ray Diffraction. Figure 1 shows temperature-dependent X-ray diffraction patterns of an 11-layer mixed-stack CT film of octadecyl-TCNQ doped with (Me)₂P prepared on a CaF₂ plate. The measurements were carried out over a temperature range from room temperature to 90 °C at an increment of 10 °C. Peaks at $2\theta = 2.7^\circ$, 8.0° , and 10.7° are assigned to the first-, third-, and fourth-order diffractions in the CT film at room temperature.¹ The diffraction pattern illustrates that a well-oriented arrangement of D (donor) and A (acceptor) molecules exists in the highly ordered multilayered structure, which consists of the bimolecular layers. This was further verified by the morphological features observed for the 11-layer mixed-stack CT film by AFM measurements (see the preceding paper).² It can be observed in Figure 1 that the diffraction pattern is unchanged until 70 °C but vanishes completely above 80 °C. This indicates that the 11-layer CT film is stable below 70 °C, but it undergoes a dramatic structural change from 70 to 80 °C. It can be concluded that the molecular arrangement and orientation of D and A in the multilayered CT complex remain almost unchanged until 70 °C but the multilayered structure vanishes if the CT film is heated to 80 °C.

Ultraviolet–Visible–Near-Infrared Spectroscopy. Parts a and b of Figure 2 show temperature-dependent UV–vis–NIR absorption spectra of 1- and 11-layer mixed-stack CT films of octadecyl-TCNQ and (Me)₂P prepared on CaF₂ plates, respectively. The temperature was raised at an increment of 10 °C over the temperature range 30–90 °C. The spectra of the 1- and 11-layer CT films measured at 30 °C show absorption bands similar to those of the 7-layer CT film reported in the first paper.¹ A band at 1730 nm in the NIR region is assigned to the CT excitation between octadecyl-TCNQ and (Me)₂P,

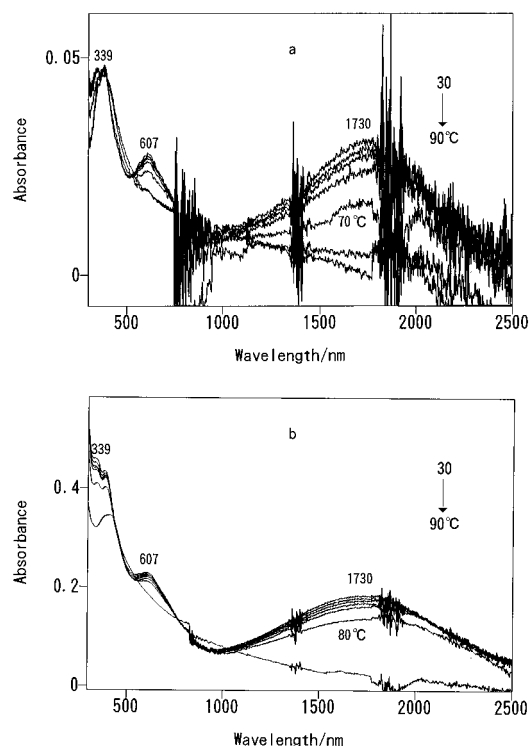


Figure 2. UV–vis–NIR absorption spectra of (a) 1- and (b) 11-layer mixed-stack CT films of octadecyl-TCNQ doped by (Me)₂P measured over a temperature range 30–90 °C at an increment of 10 °C.

while two bands at 339 and 607 nm are attributed to localized electronic transitions of octadecyl-TCNQ and (Me)₂P, respectively.^{1,9}

Thermally induced spectral variations of the CT complex films of octadecyl-TCNQ doped by (Me)₂P depend on the number of layers. First, the CT excitation band at 1730 nm decreases gradually in the spectrum of the 1-layer CT film while that of the 11-layer CT film shows only a small decrease below 70 °C. Marked spectral changes can be observed for both the 1- and 11-layer CT films from 70 to 90 °C, where the intensities of the CT band decrease abruptly. Second, the dedope temperature of the CT films shows a thickness dependence. Not only the CT excitation bands but also the intramolecular excitation bands of D and A disappear at 80 and 90 °C for the 1- and 11-layer CT films, respectively. The disappearance of the characteristic bands of CT films indicates the decomposition of the mixed-stack CT complex. A new band appears at 360 nm in the spectra measured above the dedope temperature, suggesting that the remaining part contains only the neutral octadecyl-TCNQ moiety.^{4,5} The temperature-dependent UV–vis–NIR spectra of the three- and 7-layer films show that their dedope temperatures are in the range 80–90 °C. More accurate estimation of the dedope temperature can be made from the analysis of their IR spectra.

Infrared Spectroscopy. The temperature dependence of an IR spectrum of the 7-layer mixed-stack CT film is presented in Figure 3a. Band frequencies and assignments of the spectrum were reported in the first paper.¹ It can be seen from Figure 3a that notable spectral changes occur between 80 and 90 °C for all the bands. In this temperature range the characteristic bands of the CT complex vanish, showing that the donor molecules dedope from the CT complex film.

To determine the dedope temperature more accurately, temperature-dependent spectral changes in the C≡N stretching band region were measured from 80 to 90 °C at an increment of 2 °C as shown in Figure 3b. As discussed in the first paper,¹

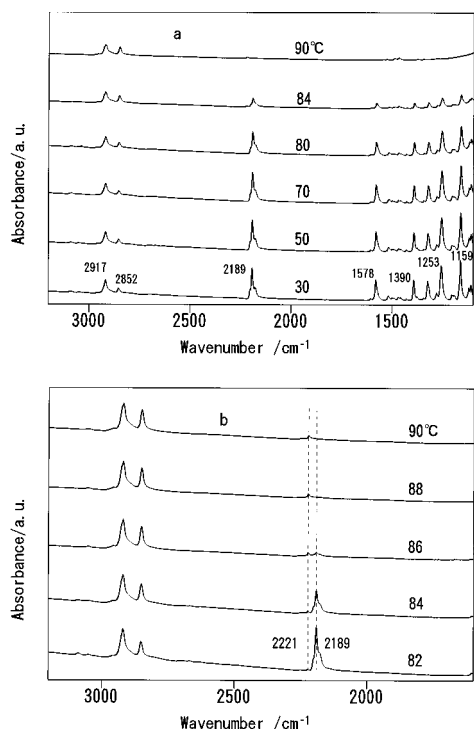


Figure 3. (a) IR transmission spectra of a 7-layer mixed-stack CT film of octadecyl-TCNQ doped by $(\text{Me})_2\text{P}$ at various temperatures from 30 to 90 °C. (b) Enlargement of the CH_2 and $\text{C}\equiv\text{N}$ stretching band region measured in the temperature range 80–90 °C.

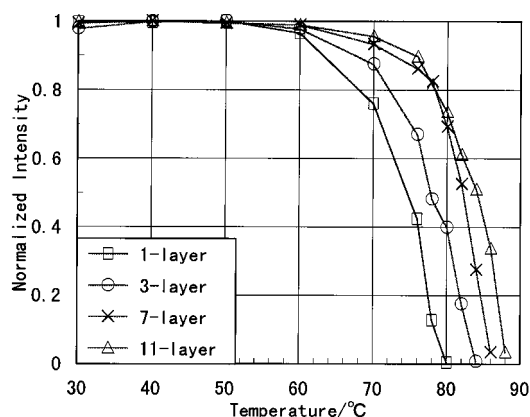


Figure 4. Temperature dependence of the normalized intensity of an a_g $\text{C}\equiv\text{N}$ stretching band of TCNQ chromophore for the 1-, 3-, 7-, and 11-layer mixed-stack CT films of octadecyl-TCNQ doped by $(\text{Me})_2\text{P}$.

bands at 2189 and 2221 cm^{-1} are assigned to an a_g $\text{C}\equiv\text{N}$ stretching mode of the TCNQ chromophore in the CT complex and a b_{1u} $\text{C}\equiv\text{N}$ stretching mode of its neutral state, respectively. At 86 °C, the a_g band at 2189 cm^{-1} almost disappears while the b_{1u} band at 2221 cm^{-1} becomes dominant. The corresponding spectral changes are observed at 80, 84, and 88 °C for the 1-, 3-, and 11-layer CT complex films, respectively. Based on these results, the thermally induced dedope temperature is determined to be 80, 84, 86, and 88 °C for the 1-, 3-, 7-, and 11-layer CT complex films of octadecyl-TCNQ doped by $(\text{Me})_2\text{P}$, respectively. It becomes apparent that the dedope temperature increases with the number of layers in the CT complex films.

The normalized intensity of the a_g $\text{C}\equiv\text{N}$ stretching band (2189 cm^{-1}) is plotted as a function of temperature for the 1-, 3-, 7-, and 11-layer mixed-stack CT films in Figure 4. It becomes clear that the thermal behavior of the CT films shows dependency on the number of layers. The intensity of the a_g $\text{C}\equiv\text{N}$ stretching band changes little for all the films until 60 °C. This

means that the CT complex films are stable irrespective of the number of layers if the temperature is kept below 60 °C. For the 1-layer CT film, the intensity decreases rapidly from 60 to 80 °C. However, in the cases of the 7- and 11-layer CT films, the intensity decrease is rather slow until 70 °C, and above 70 °C it is accelerated. It seems that the dedoping of $(\text{Me})_2\text{P}$ from the CT complex takes place at higher temperature by about 10 °C in the 7- and 11-layer CT films than in the 1-layer film. The intensity of the a_g $\text{C}\equiv\text{N}$ band shows a very similar temperature-dependent variation between the 7- and 11-layer films, indicating that the thickness-dependency of the thermal behavior becomes less obvious if the number of layers is more than 7. The temperature-dependent change for the 3-layer film is between those of the 1- and 11-layer CT films. Figure 4 clearly shows that the decomposition of the CT films does not occur at a certain temperature but takes place progressively in a high-temperature region. In contrast to the a_g $\text{C}\equiv\text{N}$ stretching band, the b_{1u} $\text{C}\equiv\text{N}$ stretching band at 2221 cm^{-1} , arising from the neutral species of octadecyl-TCNQ, begins to appear at 70 °C and becomes dominant at 80 °C for the 1-layer film. Again, the b_{1u} band of the 7- and 11-layer films shows an intensity variation similar to that of the 1-layer film, but the changes occur approximately 10 °C higher in the former. The gradual disappearance of the band at 2189 cm^{-1} , together with the progressive increase in the band at 2221 cm^{-1} , indicates that the donor molecules sublime from the CT complex in the dedope process of the CT films. Therefore, the dedope temperature defined above corresponds to the temperature where the dedoping of donor molecules from the CT films is completed.

Bands at 2918 and 2852 cm^{-1} in Figure 3a are due to CH_2 antisymmetric and symmetric stretching modes of the hydrocarbon chain of octadecyl-TCNQ in the CT state, respectively.^{1,4,6} The relationship of these two bands to the packing pattern of the hydrocarbon chain will be discussed in detail in the next section. In Figure 5a, the normalized intensity of the CH_2 symmetric stretching band is plotted against temperature for the 1-, 3-, 7-, and 11-layer CT films. Comparison of Figure 5a with Figure 4 reveals that the CH_2 stretching band undergoes a thermal behavior similar to that of the a_g $\text{C}\equiv\text{N}$ stretching band for all the films. The intensity increase in the CH_2 symmetric stretching band suggests that the molecular orientation of the hydrocarbon chain in the films changes during the dedope process. Furthermore, the frequency of this band also changes with temperature as shown in Figure 5b. The shift of the CH_2 symmetric stretching band takes place in two steps; the band shifts from 2852 to 2850 cm^{-1} in the first step, while in the second it shifts from 2850 to 2848 cm^{-1} . The temperatures at which the shifts happen obviously increase with the number of layers. The intensity increase and wavenumber shift of the CH_2 symmetric stretching band indicate that not only the molecular orientation but also the packing pattern of the hydrocarbon chain undergo remarkable variations in the course of the dedope.

Discussion

Thermal Behavior of Mixed-Stack CT Films of Octadecyl-TCNQ Doped by $(\text{Me})_2\text{P}$. The thermally induced structural changes in the mixed-stack CT films of octadecyl-TCNQ and $(\text{Me})_2\text{P}$ may be discussed for three temperature regions. In the first region from 25 to 60 °C, the X-ray diffraction pattern of the 11-layer CT film is nearly unchanged. The CT excitation band at 1730 nm of both the 1- and 11-layer CT films shows only a slight intensity decrease in this temperature region. The a_g $\text{C}\equiv\text{N}$ stretching band of the TCNQ chromophore shows almost no intensity change for the 1-, 3-, 7-, and 11-layer CT

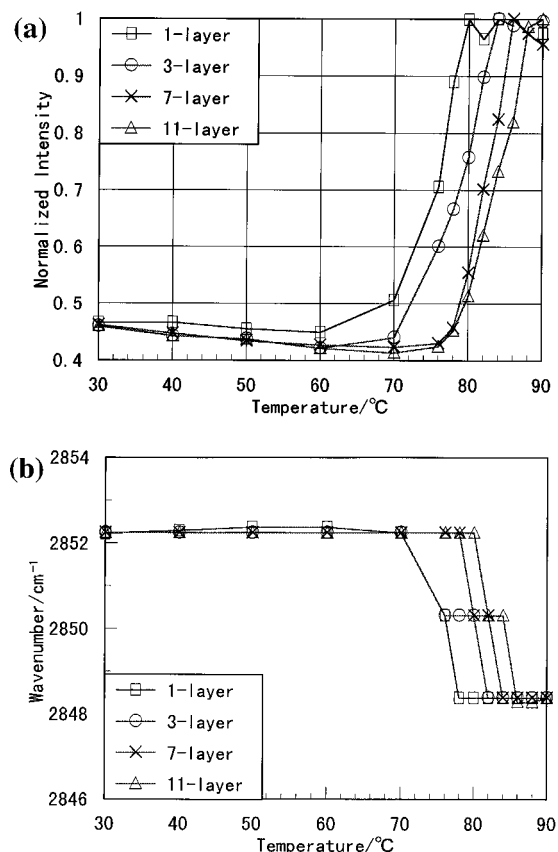


Figure 5. Temperature dependences of the (a) normalized intensity and (b) wavenumber of a CH₂ symmetric stretching band of the hydrocarbon chain for the 1-, 3-, 7-, and 11-layer mixed-stack CT films of octadecyl-TCNQ doped by (Me)₂P.

films. Based upon these observations, it can be concluded that the molecular orientation and arrangement of D and A and the multilayered microcrystal domains consisting of bimolecular layers in the CT films are thermally stable below 60 °C irrespective of the number of layers.

The dedope of (Me)₂P from the CT complex begins around 60 °C, but it proceeds rather slowly from 60 to 70 °C for the multilayer films. For example, in the temperature region of 60–70 °C the intensity of the *a_g* C≡N stretching band decreases considerably for the 1-layer CT film but only slightly for the 11-layer film (Figure 4). The changes in the UV–vis–NIR spectra in the same temperature range are also considerable and slight for the 1- and 11-layer films, respectively (parts a and b of Figure 2). These observations indicate that the dedope process occurs earlier in the 1-layer film than in the 11-layer film.

From 70 to 90 °C, the dedope proceeds rapidly accompanied by striking structural changes. The *a_g* C≡N stretching band in the IR spectra and the CT excitation band in the NIR region decrease, indicating the rapid decomposition process of the CT complex. The well-ordered multilayered structure in the films no longer exists, judging from the disappearance of the X-ray diffraction peaks. The thickness dependence of the decomposition is clearly recognized by the dedope temperature determined by observing the *a_g* and *b_{1u}* C≡N stretching modes of the TCNQ chromophore.

The thickness dependence of thermal behavior of the mixed-stack CT films may be explained by referring to the morphological features provided by the AFM measurements.^{2,3} Longitudinal interactions between the sheets of layers in the crystalline multilayered CT films may become a resistance force to the dedope process. In contrast, the donor molecules seem

more readily to dedope from the 1-layer CT film because of the lack of such interactions.

Several experimental pieces of evidence enable us to explore the decomposition mechanism. For example, the disappearance of the characteristic bands of the CT complex in the UV–vis–NIR and IR spectra and the appearances of the bands due to neutral octadecyl-TCNQ species lead us to conclude that the donor molecules (Me)₂P dedope completely from the CT complex at the dedope temperature, resulting in a restoration of acceptor molecules (octadecyl-TCNQ) from the CT state to neutral one. Moreover, the fact that the AFM image of the CT film annealed at 90 °C resembles closely that of an LB film of octadecyl-TCNQ gives morphological evidence for the decomposition mechanism of the CT films.

Thermal Behavior of Hydrocarbon Chains in the Mixed-Stack CT Films. The appearances of the CH₂ antisymmetric and symmetric stretching bands at lower wavenumbers (ca. 2918 and 2848 cm⁻¹, respectively) indicate highly ordered conformation of the hydrocarbon chains.^{10–17} For instance, in the IR spectra of the LB films of octadecyl-TCNQ they were observed at 2918 and 2848 cm⁻¹, respectively.^{4–6} Based on this observation, we concluded that the alkyl chains in the LB films of octadecyl-TCNQ assume a trans-zigzag conformation. Their AFM images and X-ray diffraction patterns indicate that the hydrocarbon chains are highly ordered and take an interdigitated pattern inside the bilayer microcrystal platelet domains.^{5,18}

The CH₂ antisymmetric and symmetric stretching bands of the hydrocarbon chain appear at 2922 and 2852 cm⁻¹, respectively, in the spectra of the mixed-stack CT films of octadecyl-TCNQ doped by TMB.^{5,6} It means that the interactions among the hydrocarbon chains are weak and that at least the highly ordered interdigitated pattern of the hydrocarbon chains does not exist.⁶ The CH₂ symmetric band shifts from 2852 to 2854 cm⁻¹ in the temperature range between 80 and 90 °C, indicating the increase in the conformation disorder, i.e., gauche conformers in the chain.⁵

In the case of the mixed-stack CT films of octadecyl-TCNQ and (Me)₂P investigated in the present study, the CH₂ antisymmetric and symmetric stretching bands of octadecyl-TCNQ appear at 2918 and 2852 cm⁻¹, respectively. The frequency of the symmetric stretching band is higher than that for the LB films of octadecyl-TCNQ. Therefore, it seems that the hydrocarbon chains are separated by the insertion of the donor molecules into the LB films and that they contain some gauche conformation. However, the interdigitated pattern probably still remains because the AFM images of the CT films of octadecyl-TCNQ and (Me)₂P clearly show evidence for the bilayer structure.

The CH₂ symmetric band shifts from 2852 to 2848 cm⁻¹ in the temperature range 70–90 °C, where the dedope process of the CT complex occurs. The downward shift might be an indication of realignment and/or rearrangement of the hydrocarbon chains toward a more compact and tight packing pattern. The realignment and rearrangement happen because the loss of donor molecules should create free volume when the donors dedope from the CT films. The downshift is also dependent on the number of layers and proceeds in concert with the decomposition of the CT films. The change in the orientation of the hydrocarbon chains is also worthy of note. The intensity of the CH₂ symmetric band increases considerably for all the films from 70 to 90 °C as shown in Figure 5a while that of the antisymmetric band is nearly temperature independent. This indicates that the orientation of hydrocarbon chains become tilted with respect to the substrate surface from the original orientation of being parallel to the surface.¹

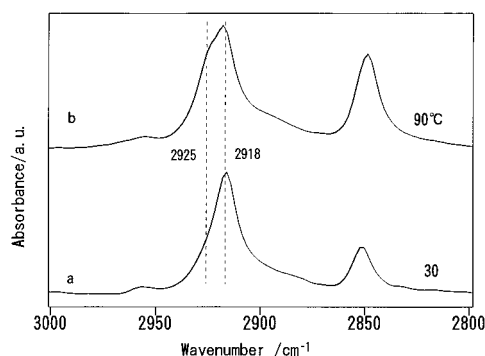


Figure 6. Enlargement of the CH_2 stretching band region in IR spectra of a 7-layer mixed-stack CT film of octadecyl-TCNQ doped by $(\text{Me})_2\text{P}$ measured at (a) 30 and (b) 90 °C, respectively.

There is one more important factor that must be considered in regard to the thermal behavior of hydrocarbon chains. As reported previously, the hydrocarbon chains experience a phase transition from 80 to 90 °C in the LB films of octadecyl-TCNQ⁴ and the mixed-stack CT films of octadecyl-TCNQ doped with TMB.⁷ Figure 6 compares the CH_2 stretching band region for the spectra measured at room temperature and 90 °C. Note that the band at 2918 cm^{-1} becomes broad with the appearance of a shoulder near 2925 cm^{-1} at 90 °C. This implies that a small portion of the hydrocarbon chains becomes disordered while the majority is still highly ordered. It seems likely that the ordered and disordered hydrocarbon chains coexist in the temperature region of 80–90 °C.

An interesting and unusual phenomenon has been observed for the thermally induced shift of the CH_2 antisymmetric and symmetric stretching bands. The shift of these two bands shows an opposite tendency. The antisymmetric and symmetric bands shift upward and downward, respectively, during the decomposition process of the CT films. To the best of our knowledge, it is the first time that this phenomenon has ever been observed in any kind of organic LB film. It indicates that the antisymmetric and symmetric bands reflect different features of thermally induced structural changes in the hydrocarbon chains although they come from the same alkyl chains.

Conclusions

The thermal behavior of the mixed-stack CT films of octadecyl-TCNQ doped with $(\text{Me})_2\text{P}$ has been discussed in this paper based upon the temperature-dependent X-ray diffraction patterns and UV-vis-NIR and IR spectra. The following

conclusions can be reached for the mechanism of the thermally induced decomposition of the 1-, 3-, 7-, and 11-layer mixed-stack CT complex films.

(i) The donor molecules $(\text{Me})_2\text{P}$ dedope from the CT complex, resulting in a restoration of acceptor molecules (octadecyl-TCNQ) from the CT state to the neutral one. This process occurs gradually in a temperature range from 70 to 90 °C but does not happen at a certain temperature.

(ii) Thermally induced structural changes in the mixed-stack CT films strongly depend upon the number of layers. For example, the dedope temperature, at which the CT complex decomposes completely, increases with the number of layers.

(iii) The orientation and structure of the hydrocarbon chains of TCNQ chromophores change largely during the dedope process. The longitudinal interactions within the layered platelet microcrystals as well as the interaction between the first layer and the substrates may play an important role in the thermal behavior of the CT films.

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