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Ultrathin polycrystalline 6,13-Bis(triisopropylsilylethynyl)-pentacene films

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Ultrathin (<6 nm) polycrystalline films of 6,13-bis(triisopropylsilylethynyl) pentacene (TIPS-P) are deposited with a two-step spin-coating process. The influence of spin-coating conditions on morphology of the resulting film was examined by atomic force microscopy. Film thickness and RMS surface roughness were in the range of 4.0–6.1 and 0.6–1.1 nm, respectively, except for small holes. Polycrystalline structure was confirmed by grazing incidence x-ray diffraction measurements. Near-edge x-ray absorption fine structure measurements suggested that the plane through aromatic rings of TIPS-P molecules was perpendicular to the substrate surface. © 2014 American Vacuum Society. [<http://dx.doi.org/10.1116/1.4904063>]

I. INTRODUCTION

Recently, pentacene has attracted attention as a promising material for organic-based electronic devices because of its good carrier mobility.^{1–3} However, molecules like pentacene with high carrier mobilities are generally insoluble in common solvents, which severely complicate their processing. In response, 6,13-bis(triisopropylsilylethynyl) pentacene (TIPS-P) [Fig. 1(a)] was developed to have good solubility in organic solvents without sacrificing high mobility.^{4,5} The most critical factor in maintaining carrier mobility of TIPS-P is the formation of the crystalline phase, which has good π – π overlap of pentacene aromatic rings.^{6,7} In published studies, simple methods for forming TIPS-P crystalline phases include common solution methods, e.g., rubbing, solution-casting, and drop-casting on an inclined substrate.^{4–7} Some group have tried mixing TIPS-P with other components, like graphene flakes, to modify the crystallization and performance of TIPS-P films.⁸ A high hole mobility of 0.8 – 4.6 cm² V^{–1} s^{–1} was reported for a TIPS-P transistor fabricated from a film deposited by the solution-shearing method.⁸ However, these methods require additional processing that is not fully compatible with mass production. For this reason, they are less desirable for practical device applications that demand simple, low-cost fabrication.

From the perspective of performance, thin films on thin insulators are advantageous. The standard methods for forming organic electronics require high operational voltages because they use thick dielectric barriers at the gate electrode. High voltages, in turn create a thick conduction channel between 10 and 50 nm, which requires films with similar thickness for optimum performance.^{9–12} Commercial electronics require lower voltages for compatibility and power requirements. If the voltages are lowered, the active channel

becomes only nanometers thick right at the dielectric surface. This requires very high-quality ultrathin films. A recent report even observed a dramatic enhancement of mobility in ultrathin organic semiconductor films on substrates with a thick insulating polymer coating.¹³ This effect should be maintained with a thinner dielectric. Thus, it is desirable to prepare high quality ultrathin organic films on a thin insulating barrier of silicon dioxide or an insulating monolayer using a method that is compatible with traditional semiconductor processes. In this study, we examined the possibility of fabricating ultrathin TIPS-P films directly on the smooth surface of native oxide on bare silicon and silicon coated with self-assembled monolayers using a simple spin-coating method.

Ultrathin TIPS-P films were fabricated using only spin-coating with a two-step process on bare Si, Si modified with *n*-octyltriethoxysilane (OTS) self-assembled monolayers, and Si modified with Langmuir–Blodgett (LB) monolayers of stearic acid. All samples were characterized with respect to surface morphology and atomic/electronic structure using various measurement techniques, including spectroscopy with synchrotron radiation.

II. EXPERIMENT

A solution of TIPS-P was prepared by dissolving 1 mg of TIPS-P (C₄₄H₅₄Si₂, > 98.0%, Tokyo Chemical Industry Co., Ltd., Japan) in 1 ml of chlorobenzene (C₆H₅Cl, > 97%, Sigma-Aldrich). An *n*-type Si(100) substrate (resistivity = 0.011–0.015 Ω cm, Samuco Co.) with native oxide (bare Si) was treated with UV-ozone cleaning for 10 min to remove organic contaminants. To prepare the OTS [CH₃(CH₂)₁₇SiCl₃, > 90%, Sigma-Aldrich]/Si and stearic acid [CH₃(CH₂)₁₆COOH, > 98.5%, Sigma-Aldrich]/Si samples, further treatment was performed following UV-ozone cleaning. For OTS treatment, the bare Si was submerged in 5 mM OTS solution in toluene at room temperature for 5 min. After removal, the sample was

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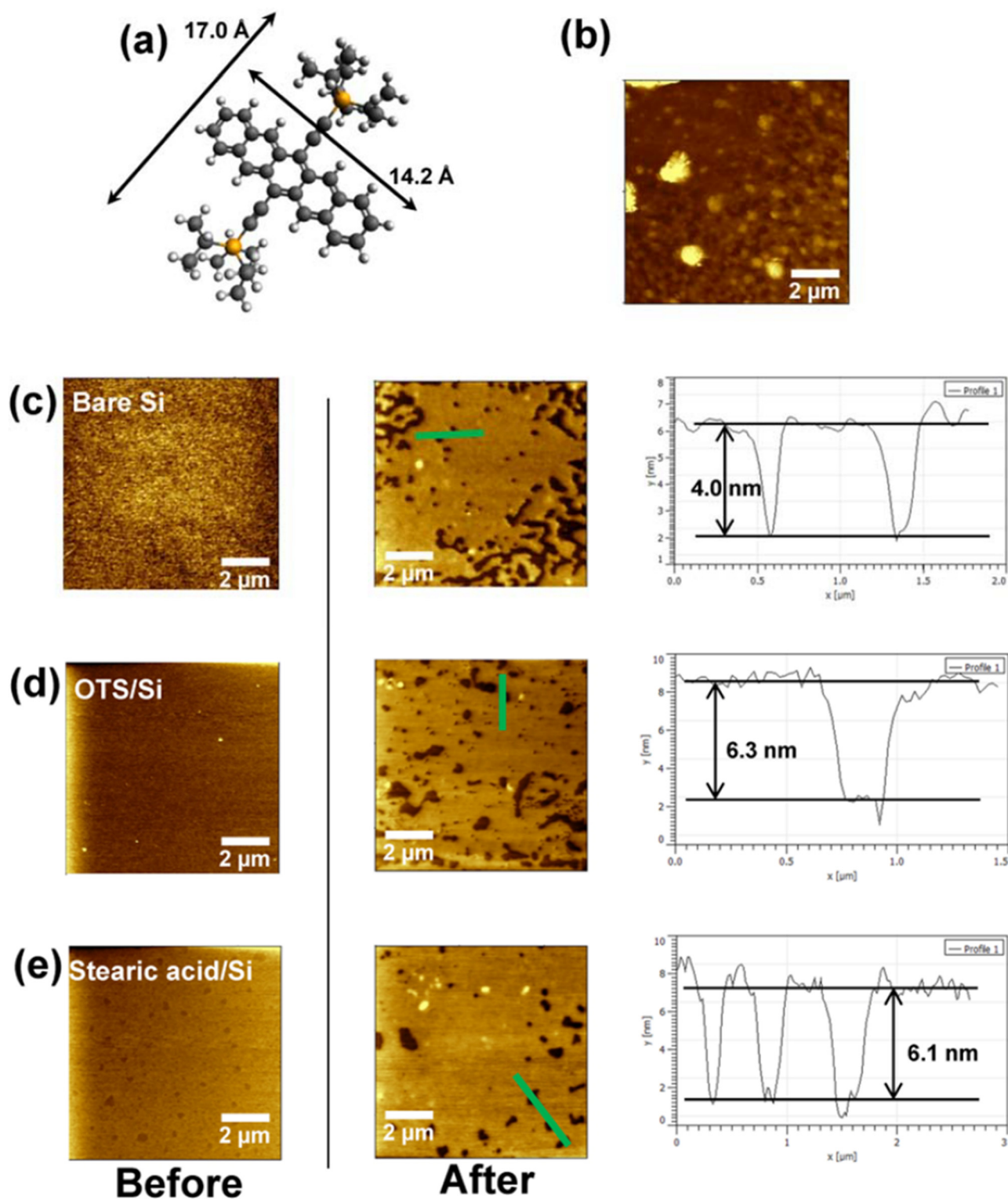


FIG. 1. (Color online) Ultrathin polycrystalline TIPS-pentacene films formed by spin-coating with a two-step process. (a) TIPS-P structure with calculated molecular distances (b) surface morphology coated with a single spin-coating condition (5 krpm for 30 s). AFM images showing surface morphologies and profiles before and after deposition of TIPS-P on (c) native oxide, (d) OTS-treated surface, and (e) stearic acid-coated surface. Images are $5 \times 5 \mu\text{m}$. The RMS of each surface is 0.29, 0.5, and 0.4 nm, respectively. Heights are around 4.0, 6.3, and 6.1 nm between TIPS-P and the substrate in the depth profile. The dark regions and golden regions represent bare substrate and TIPS-P-coated regions, respectively.

rinsed in pure toluene. After rinsing, it was heated on a hot-plate at 120°C for 10 min under ambient conditions to dry the sample and cross-link the OTS monolayer. Finally, we ultrasonicated the sample in ethanol for 5 min to remove residual

physisorbed OTS silane monomers or oligomers. To form a monolayer of stearic acid on a Si substrate, we submerged the sample in a clean water phase in an LB trough (MicroTroughXS, Kibron Inc.), spread a 0.5 mg/ml stearic

acid solution in chloroform on the water phase and allowed the chloroform to evaporate. We compressed the LB layer to a pressure of 6 mN/m, and then withdrew the sample from the LB trough while maintaining constant surface pressure. The TIPS-P thin films were formed on bare Si (sample S1), OTS/Si (sample S2), and stearic acid/Si (sample S3) substrates using a programmable spin-coater (MS-A100, MIKASA Company). The two-step spin-coating process for samples S1, S2, and S3 was performed in ambient environment and consisted of a low speed step with 500 rpm for 5 s, followed by a high speed step with 5000 for 60 s. The solvent evaporates readily at high spin speeds, so the films were completely dry after two-step spin-coating. To assess surface morphology, each TIPS-P sample was examined with atomic force microscopy (AFM, MFP-3D Asylum Research) in tapping mode, using an Al reflex-coated tip (Tap190Al-G, Budget Sensors Company) with a resonance frequency of 190 kHz and a spring constant of 48 N/m (256×256 pixels). To investigate crystallinity and crystal orientation of sample S1, grazing incidence x-ray diffraction (GIXD) measurements were conducted at PLS-II 9 A U-SAXS beamline (wavelength $\lambda = 1.112 \text{ \AA}$) at the Pohang Accelerator Laboratory. Two-dimensional GIXD patterns were acquired from a $\sim 2 \times 2 \text{ mm}^2$ area of sample S1. Since the beam size at the sample position was about $70 \text{ (V)} \times 400 \text{ (H)} \mu\text{m}^2$, irradiating a sampling area was $\sim 0.4 \text{ (W)} \times 2 \text{ (L)} \text{ mm}^2$; the beam was scanned across the surface to analyze the full $2 \times 2 \text{ mm}^2$ area. With a sample-to-detector distance of $\sim 238 \text{ mm}$, this small sampling area ensured negligible peak broadening ($<1\%$) arising from the sample size. Samples were mounted on a seven-axis motorized stage with the incident angle of the x-ray beam set to $\sim 0.16^\circ$. Diffraction angles were calibrated with a pre-calibrated sucrose crystal (Monoclinic, P21, $a = 10.8631 \text{ \AA}$, $b = 8.7044 \text{ \AA}$, $c = 7.7624 \text{ \AA}$, $\beta = 102.938^\circ$).¹⁴ For x-ray photoelectron spectroscopy (XPS), we used unmonochromated MgK α (1253.6 eV) and an EA125 energy analyzer with single channeltron (Omicron Nanotechnology). The energy resolution of this analyzer was 0.9 eV. Chemical states of sample S1 were measured by acquiring O 1s, C 1s, and Si 2p core-level spectra. Binding energies were calibrated against the Au 4f_{7/2} level (84.0 eV).¹⁵ Near-edge x-ray absorption fine structure (NEXAFS) spectroscopy study was conducted at 8A1 undulator-radiation (U7) beamline at the PLS-II using synchrotron radiation.¹⁶ We measured the C K-edge of sample S1 with angles of normal, 30° , 45° , and 60° between the incident beam and sample. NEXAFS measurement and normalization were performed with a total electron yield and gold grid, respectively.

III. RESULTS AND DISCUSSION

In general, it is difficult to obtain a thin, high-coverage film using only one-step spin-coating with TIPS-P [Fig. 1(b)] during solution-based fabrication. When a film on a Si substrate was prepared with spin-coating at 5000 for 30 s, the surface roughness of this film was 15.8 nm in $5 \times 5 \mu\text{m}^2$ [Fig. 1(b)]. However, with an additional spreading spin-

coating step, a thin, high-coverage film is achievable [Figs. 1(c)–1(e)].

In order to better analyze the films formed by TIPS-P, computational simulations can help to understand the underlying molecule. A TIPS-P molecule has orthogonal molecular dimensions of $1.70 \times 1.42 \text{ nm}$ [Fig. 1(a)]. Similar sizes were obtained using force field optimization and also with *ab initio* calculations. Force field optimization employed a set of functions named MMFF94, developed by Merck Research Laboratories.¹⁶ For *ab initio* calculations, augmented split valence basis sets using double zeta functions with added polarization functions were used for all atoms.^{17,18} Split valence means that the basis set that describes the valence electrons on each atom includes more than one kind of basis function. For example, double zeta basis sets include functions to represent both radially symmetrical orbitals, which correspond to *s* orbitals, and also axially symmetrical orbitals, which correspond to *p* orbitals. The polarization functions allow for asymmetry to be represented on each atom. Since the double zeta represents both *s* and *p* orbitals, it is adequate for all the atoms in the TIPS-P molecule. For a TIPS-P film in which molecules are oriented with the long axis perpendicular to normal (*vide infra*), a film with a height of 4–6 nm would consist of 2–4 monolayers of TIPS-P. Control of monolayer thickness to this level is typically only possible with vacuum deposition.¹⁹

The coverage of samples S1 (TIPS-P/Si), S2 (TIPS-P/OTS/Si), and S3 (TIPS-P/stearic acid/Si) were 75.0%, 77.6%, and 80.3%, respectively. The coverage is maintained over the sample surface based on both large-area AFM images and also optical images [Figs. S1–S4].²⁰ The coverage is better than some Langmuir–Blodgett monolayers with ultrathin films and almost as good as films formed by spin-coating larger, more-soluble molecules onto hydrophobic monolayers.^{21–23} It is reasonable to expect that automation and environmental control from a commercial process would further improve coverage while simultaneously reducing run-to-run variation. The dark regions and golden regions in AFM images represent bare substrate and TIPS-P-coated regions, respectively. Prior to deposition of TIPS-P with two steps, surface roughness was calculated across a $5 \times 5 \mu\text{m}^2$ area for each of the three substrates. These were 0.09 nm for bare Si with the native oxide, 0.37 nm for OTS/Si, and 0.35 nm for stearic acid/Si (Table I). After deposition, surface roughnesses increased to 0.6, 1.1, and 0.8 nm for samples S1, S2, and S3, respectively (excluding holes and islands).

To obtain structural information, specifically with regard to the crystalline phase, we performed GIXD measurements on sample S1, which has a surface coverage $>90\%$. The white box highlights some representative peaks from our TIPS-P ultrathin film, shown in the form of a line scan to (a). Even though the film was only an average of two or three layers thick, diffraction spots were clearly observable [Fig. 2(a)]. Without a high-degree of crystallinity in such an ultrathin film, diffraction peaks could not have been observed. From these measurements, we conclude that the two-step

TABLE I. RMS of TIPS-P based thin films.

$5 \times 5 \mu\text{m}^2$ area	Two-step			
	Before TIPS-P deposition	Without holes and islands	All area (Coverage)	One-step [Fig. 1(b)]
TIPS-P/Si	0.09 nm (± 0.02) (bare Si with native oxide)	0.60 nm (± 0.06)	1.62 nm (± 0.10) (75.0 %)	15.8 nm (± 0.2)
TIPS-P/OTS/Si	0.37 nm (± 0.05)	1.10 nm (± 0.09)	1.84 nm (± 0.11) (77.6%)	
TIPS-P/Stearic acid/Si	0.35 nm (± 0.05)	0.80 nm (± 0.08)	1.25 nm (± 0.09) (80.3 %)	

spin-coating process forms TIPS-P films with a polycrystalline structure similar to traditional thicker organic electronic films.²⁴ Even with the axis of the TIPS-P molecules oriented perpendicular to the surface, in-plane rotations can produce grain boundaries between grains.

The sample on bare silicon (sample S1) was measured by XPS to provide additional confirmation that the films are ultrathin and to confirm that the TIPS-P has not reacted. Core-level spectra for O 1s, C 1s, and Si 2p from the ultrathin film on bare Si were determined and binding energies of O and C 1s core-level peaks were measured to be 533.4 and 285.5 eV, respectively [Figs. 2(b) and 2(c)]. There is only one C 1s peak, which is consistent with TIPS-P. In the Si 2p core-level spectrum, two peaks displayed binding energies of 103.0 and 99.7 eV. In order to analyze the spectra in more

detail, we fitted Si 2p core-level peaks using Doniach–Sünjic curves convoluted with Gaussian distribution of 0.5 eV full-width at half-maximum.²⁵ Background due to inelastic scattering was subtracted by the Shirley (or integral) method.²⁶ We found three chemical states of pure Si-Si (99.7 eV), Si-C (100.8 eV), and SiO₂ (102.8 eV) bonding [Fig. 2(d)]. Si-Si and SiO₂ states originated from Si substrate and the native oxide (typically 1–2 nm thick), respectively. Since the probing depth of XPS is on the order of 10 nm with an exponential decay in the probability of electrons escaping, the TIPS-P film thicknesses of 4–6 nm are roughly correct. The Si-C peak originates from the Si atoms in TIPS-P.²⁷ XPS was not performed on the other samples because Si 2p and C 1s spectra would be confounded by photoelectrons from OTS or stearic acid.

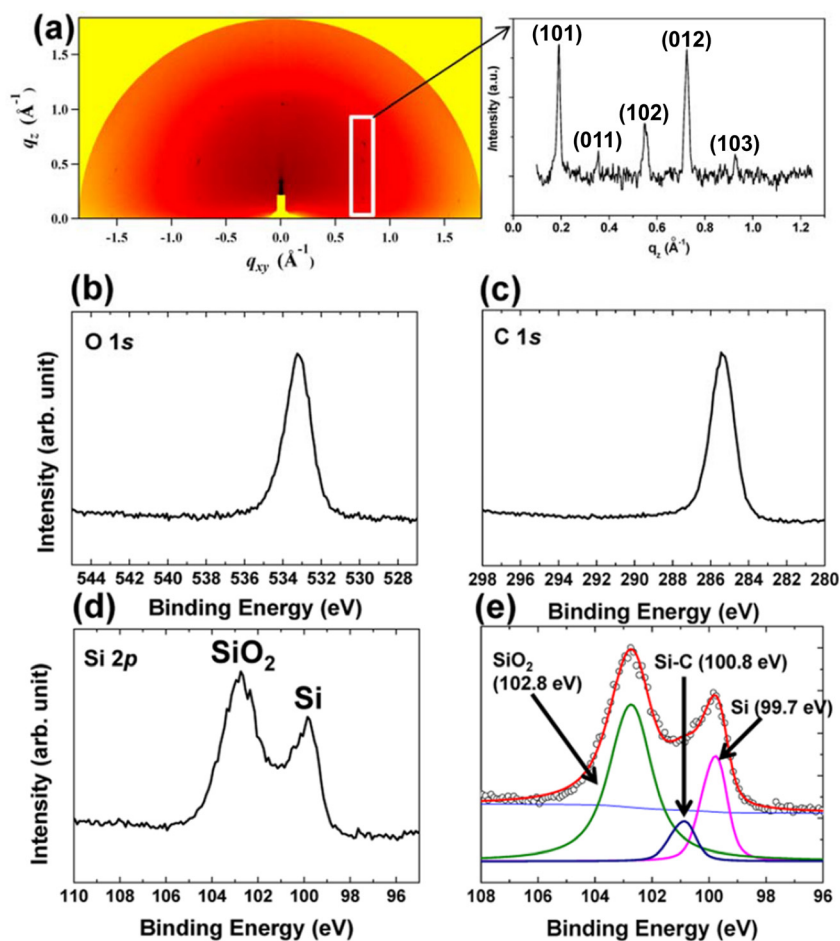


FIG. 2. (Color online) Results from GIXD and XPS analysis of ultrathin TIPS-pentacene films. (a) GIXD data with the extracted region showing the (101), (011), (102), (012), and (103) peaks from a TIPS-P ultrathin film. (b) XPS spectra of O 1s, (c) C 1s, and (d) Si 2p core-level regions for TIPS-P on native oxide and (e) curve fitting data of Si 2p core-level spectrum with chemical states of SiO₂, Si-C, and pure Si.

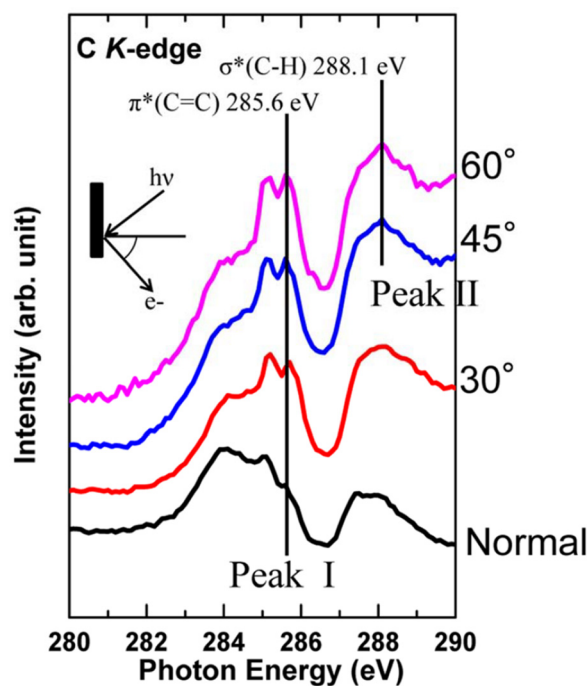


Fig. 3. (Color online) NEXAFS data from ultrathin TIPS-P on a native oxide substrate were obtained with angles of 30°, 45°, and 60° from the surface normal. The $\pi^*(\text{C}=\text{C})$ peak at 285.6 eV and the $\sigma^*(\text{C}-\text{H})$ peak at 288.1 eV are identified as peaks I and II, respectively. The inset shows the angle between the electron extraction and the surface normal.

To study the orientation of TIPS-P molecules in the ultrathin crystalline film on the substrate surface, the C *K*-edge of sample S1 film was measured by angle-dependent NEXAFS (Fig. 3). We observed two significant peaks (I and II), the intensity of which changed with the incident angle of the synchrotron beam. The absorption photon energies of I and II were 285.6 and 288.1 eV, respectively. I was $\pi^* \text{C}=\text{C}$ resonance, while II was a $\sigma^* \text{C}-\text{H}$ resonance peak.^{27–29} With an incident angle of 0°, peak I was muted. As the incident angle increased, however, the intensity of peak I increased significantly, reaching an apparent maximum at 60°. On the other hand, the σ^* resonance (peak II) appeared in the spectrum starting at an incident angle of 45°. We assigned peak I to the pentacene core and peak II to the $\text{CH}_3\text{-Si-CH}_3$.^{27–29} The increase of peak I and II intensities with increasing incident angle suggests that the spin-coated TIPS-P in the crystalline phase stands upright on the Si substrate.

IV. SUMMARY AND CONCLUSIONS

In summary, we spin-coated TIPS-P on a Si substrate with native oxide. The ultrathin films of TIPS-P/Si, TIPS-P/OTS/Si, and TIPS-P/stearic acid/Si demonstrated thicknesses (RMS) of 4.0 (0.3), 6.3 (0.5), and 6.1 (0.4), respectively. These films have a crystalline phase that appears to consist of

TIPS-P molecules standing with the plane of the molecules perpendicular to the Si surface. We present evidence that ultrathin films composed of crystalline TIP-P were successfully fabricated using a simple, two-step, spin-coating method

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