Revealing molecular conformation–induced stress at embedded interfaces of organic optoelectronic devices by sum frequency generation spectroscopy

Zhongwu Wang1, Honghen Lin2, Xi Zhang3, Jie Li4, Xiaosong Chen5, Shuguang Wang1, Wenbin Gong1, Hui Yan6, Qiang Zhao7, Weibang Lv2, Xue Gong2, Qingbo Xiao2, Fujin Li2, Deyang Ji1, Xiaotao Zhang1, Huanli Dong3, Liqiang Li1,2,6*, Wenping Hu1,3,6

Interface stresses are pervasive and critical in conventional optoelectronic devices and generally lead to many failures and reliability problems. However, detection of the interface stress embedded in organic optoelectronic devices is a long-standing problem, which causes the unknown relationship between interface stress and organic device stability (one key and unsettled issue for practical applications). In this study, a kind of previously unknown molecular conformation–induced stress is revealed at the organic embedded interface through sum frequency generation (SFG) spectroscopy technique. This stress can be greater than 10 kcal/mol per nm² and is sufficient to induce molecular disorder in the organic semiconductor layer (with energy below 8 kcal/mol per nm²), finally causing instability of the organic transistor. This study not only reveals interface stress in organic devices but also correlates instability of organic devices with the interface stress for the first time, offering an effective solution for improving device stability.

INTRODUCTION

Organic optoelectronics has attracted attention for decades and is becoming the emerging industrial economic growth point (1–5). Heterogeneous interfaces in organic optoelectronic devices are the most complex and critical components that decide the device performance and the fate of device stability, so considerable efforts are devoted to characterize and understand the properties [such as chemistry (6), molecular packing (7), and energy level (8)] of embedded interfaces (9,10). However, until now, mechanical stress property has not been investigated at the interface of organic optoelectronic devices because no valid method is available for detection. It is well known that interface mechanical stress plays a vital role in many failures and reliability problems (11–14) of conventional inorganic optoelectronic devices and emerging perovskite solar cell and two-dimensional materials (15–18). The unrevealed interface stress in organic interface directly leads to the unknown relationship between stress and device stability. This has been a long-standing problem in this field.

To address the aforementioned problems, in this study, we use sum frequency generation (SFG) vibrational spectroscopy as an effective technique for detecting the mechanical stress at the molecular nanointerface embedded in organic field-effect transistors (OFETs), one key component of organic circuits. By monitoring the conformational change (bending and restretching) of the buried alkyl chain through SFG, we reveal a type of molecular conformation–induced interface stress, which may induce molecular disorder of organic semiconductor layer and finally lead to device instability (for the schematic process, see movie S1). Applying a unique perspective, we obtain results that explain the intrinsic origin of OFET instability, which is a long-standing and unresolved problem in this field, thereby providing hopeful possibilities for improving device stability.

RESULTS

Model devices and property characterization

Representative model devices for the embedded interface between an organic semiconductor/gate insulator are depicted in Fig. 1 (A to C), in which a self-assembly monolayer (SAM) is inserted between the organic semiconductor and gate insulator to form a high-compatibility interface (19–26). This model device suffers from serious stability problems (20,21,27–31). Figure 1D shows the shelf-life stability of pentacene OFETs with different octadecyl trichlorosilane (ODTS) densities (Materials and Methods and fig. S1). OFETs with high-density ODTS (H-ODTS) exhibit poor stability, and the device mobility undergoes a drastic decrease of two orders of magnitude within 3 months (violet curve in Fig. 1D and fig. S2A) (20,21,27–31). However, OFETs with medium-density ODTS (M-ODTS) exhibit high stability (blue curve in Fig. 1D), whereas those with low-density ODTS (L-ODTS) exhibit even better stability with very small mobility fluctuation (orange curve in Fig. 1D and fig. S2B). Furthermore, it is found that the effect of the ODTS density on the mobility is independent of the initial mobility of pentacene OFETs (fig. S3).

It is always believed that chemical decomposition is the main cause for device degradation; however, this is invalid in our case because the device stability and ultraviolet-visible (UV-vis) dependence under H-, M-, and L-ODTS show opposite trends (Fig. 1, D and E, and fig. S4; detailed discussion in section S4). X-ray diffraction (XRD) measurements show that pentacene film on a
H-ODTS surface undergoes a substantial phase transition from poly-crystalline to disordered (Fig. 1F, top), which may cause device instability. However, this phase transition does not occur for devices with L-ODTS because pentacene film remains ordered even after storage (Fig. 1F, bottom), indicating its stability in this case. This distinct difference of aggregation state was further confirmed by grazing-incidence wide-angle x-ray scattering (GIWAXS; fig. S5) and atomic force microscopy (AFM; fig. S6) measurements. As pentacene films and OFETs with different ODTS densities were fabricated in the same batch and stored under identical conditions, the differences in device stability should have other intrinsic origins. Considering the pentacene/SAM interface, it is essential to elucidate the role of the embedded interface.

**SFG measurements**

SFG vibrational spectroscopy is a second-order nonlinear optical technique that can provide information on the composition and molecular orientation at the interface (Fig. 2A, working principle and spectral analysis in section S7, and figs. S7 to S10) (32–40). Hence, it was applied for investigating our model device. Figure 2C shows the raw SFG signals of pristine SAMs with high, medium, and low densities. The polarization combination was set to SSP (S-polarized SFG signal generated from the incident S-polarized visible and P-polarized infrared (IR) beams; for details, see section S7) to probe the vibrational modes, with the transition dipole components oriented predominantly perpendicular to the surface. A strong nonresonant (NR) background was observed in all the cases, which may be related to the intraband transitions of the substrates and pentacene layer. For a clear view, we replotted the resonant contribution alone in Fig. 2D according to the fitting results (fitting details are described in section S7). A H-ODTS monolayer on silicon surface provides two strong bands in the CCH vibrational regions (violet curve in Fig. 2D): one centered at approximately 2877 cm⁻¹ corresponding to the CH₃ symmetric stretch (Fig. 2B) and the other at approximately 2941 cm⁻¹ arising from its Fermi resonance (39). The CH2 symmetric and asymmetric stretching (ss and as, respectively, in Fig. 2B) modes display very weak signals at approximately 2853 and 2916 cm⁻¹, respectively (32, 41–43). The dominance of the CH₃-ss mode in the SFG SSP spectra indicates that the alkyl chains are predominantly in an all-trans configuration with high ordering, i.e., they are oriented normally to the surface with the CH₃ groups pointing outside (39–41), as schematically shown in Fig. 2E (top) and fig. S13A. This is further confirmed by the SFG results at other polarization combinations (section S7) (44). In contrast, M- and L-ODTS yield different SFG spectra (Fig. 2, C and D, blue and orange curves) compared to the H-ODTS. The CH3-ss intensities decrease dramatically compared to that of H-ODTS, suggesting the orientation disordering of CH3 groups (Fig. 2F, left, and fig. S13C). Meanwhile, the CH2-ss mode at approximately
2855 cm⁻¹ and the CH₂-as (Fig. 2B, right) mode at approximately 2915 cm⁻¹ exhibit increased intensities, indicating the occurrence of gauche defects within the alkyl chains (Fig. 2F, left, and fig. S13C) (41).

In literature, the ratio of the intensity between the CH₃-ss and CH₂-ss modes has often been applied to describe the ordering degree of alkyl chains (41, 45, 46). Here, we define the ordering parameter as the ratio of the oscillator strength (A), i.e., |A(CH₃-ss)|/|A(CH₂-ss)| (41). The ordering parameter values for pristine H-, M-, and L-ODTS are approximately 5.73, 1.56, and 0.96 (corresponding to the data at zero day in Fig. 3A and Table 1, respectively), indicating large discrepancies in the ordering or conformation of alkyl chains.

**Stress at theODTS SAM/pentacene interface**

After the deposition of pentacene, the H-ODTS SAM film exhibits obvious SFG changes (Fig. 3, C and D, green curves). The drastic decrease of the ordering parameter (A(CH₃-ss)/A(CH₂-ss)) compared to pristine H-ODTS (Fig. 2D) suggests that the deposition of pentacene induces substantial trans-gauche transitions in the alkyl chains (Fig. 2E, middle, and fig. S13, A and B). The ordering parameter decreases to as low as 0.1 (corresponding to the violet triangle data at day 0 in Fig. 3B and Table 1) at this stage from the initial value of 5.73, indicating that pentacene deposition induces drastic ODTS disordering (movie S1). The increase in gauche defects in densely packed alkyl molecules upon deposition of a superimposing layer has been witnessed in other systems (39, 47, 48). The trans-gauche conformational change of the CH₂-CH₂ segments implies the bending down of the alkyl chain of ODTS (Fig. 2E, middle), which may cause interface stress. This is a type of previously unidentified and special interface stress in organic systems, different from the well-known stresses in inorganic systems induced by heterogeneous film and film edge, thermal process, lattice mismatch, and misfit dislocation (14, 17, 49–52).

As the storage time increases, the CH₃-ss band (2877 cm⁻¹) becomes more apparent (Fig. 3, C and D). An obvious increase around 2875 cm⁻¹ can be visualized in the SFG spectra after the sample is stored for a certain period; spectral fitting shows that the ratio of the vibration amplitude of CH₃-ss/CH₂-ss gradually increases to approximately 0.6 in the first 10 days (violet triangle in Fig. 3B). This result indicates that the CH₃ groups of certain parts of the alkyl chains repoint out to the surface during storage (Fig. 2E, bottom) and that the structure of the alkyl chain recovers slowly from the initial disordering induced by pentacene deposition to ordering. On
the other hand, even after considerable time, the intensity of the CH$_3$-ss band at approximately 2875 cm$^{-1}$ remains weaker than that of the pristine H-ODTS layer, suggesting that only a small part of the alkyl chains is restretched with the CH$_3$ groups extending outward (Fig. 2E, bottom). The conformational change (restretching) of the alkyl chain may serve as the driving force of the phase change of pentacene film from ordering to disordering (Fig. 2E, bottom). Probably, the bended ODTS alkyl chains by pentacene deposition are in a constrained and thermodynamically unstable state and yield prominent stress at the ODTS SAM/pentacene interface, which tends to release through the restretching of the bended alkyl chain. This produces sufficient energy to induce the shift of pentacene molecules beyond the balance position, gradually disturbing the ordering of pentacene film (movie S1). This is further confirmed through theoretical simulation (Fig. 4 and section S9).

For the L-ODTS sample, the ordering parameter decreases from the initial value of approximately 1.0 (Fig. 3A) to approximately 0.3 (corresponding to the orange square data at day 0 in Fig. 3B and Table 1) after pentacene deposition, suggesting that deposition slightly increases the disorder degree of the ODTS layer.

Table 1. ODTS ordering parameter and OFET mobility.

<table>
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<tr>
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<th>Ordering parameter (A$<em>{CH3}$/A$</em>{CH2}$)</th>
<th>Mobility (cm$^2$/Vs)</th>
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<tr>
<td></td>
<td>Pristine ODTS</td>
<td>OSC deposition</td>
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<tr>
<td>Pentacene</td>
<td>H-ODTS</td>
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<td>L-ODTS</td>
<td>0.96</td>
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<td>VOPc</td>
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<td>DPA</td>
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Fig. 3. SFG results of the ODTS/pentacene samples. (A) Evolution of the ordering parameters (A$_{CH3}$/A$_{CH2}$) of pure H-, M-, and L-ODTS with the storage time. (B) Evolution of the ordering parameter (A$_{CH3}$/A$_{CH2}$) of H-, M-, and L-ODTS/pentacene with the storage time. (C) Raw data of the SFG spectra of H-ODTS/pentacene samples under different storage time. (D) Fitted data of several representative SFG spectra of H-ODTS/pentacene samples under different storage time. (E) Raw data of the SFG spectra of L-ODTS/pentacene samples under different storage time. (F) Fitted data of several representative SFG spectra of L-ODTS/pentacene samples under different storage time.
However, during storage, there are no obvious changes in the SFG spectra (Fig. 3, E and F), and the ratio of $|A_{CH_3-ss}|/|A_{CH_2-ss}|$ remains low at approximately 0.3 with small fluctuations (orange square in Fig. 3B), suggesting that there are no obvious changes in the conformation of L-ODTS during storage (Fig. 2F and fig. S13, C and D). In the L-ODTS layer, the packing of alkyl chains is loose and random, and most of the alkyl chains may lie down on the substrate. As the deposition of pentacene slightly increases the disordering of L-ODTS without forming prominent stress, ODTS does not undergo considerable structural changes during storage (Fig. 2F and fig. S13, C and D). As a result, the pentacene film on the L-ODTS surface exhibits good structural and electrical stability, in contrast to the H-ODTS sample. The structural state of M-ODTS is similar to the L-ODTS, as proven by SFG characterization (fig. S10).

To demonstrate the intrinsic influence of the interface stress on device stability, we altered the storage conditions of devices and continuously measured their electrical performance and SFG spectra. As shown in fig. S11, the evolution of the SFG spectra and mobility of samples in vacuum or at different temperatures display the same trend as that stored in air. Comparing to the pentacene film stored at room temperature (RT) (Figs. 1D and 3B), raising the storage temperature causes faster evolution of SFG spectra and mobility for the H-ODTS/pentacene, while it maintains stable for the L-ODTS/pentacene. This fact can be explained by that the release of interface stress is accelerated at a higher temperature because of the increased kinetic energy of the interfacial molecules. All these data clearly indicate that the phase transition of pentacene film on H-ODTS stems from internal factors rather than external factors.

Theoretical simulation of the interface conformational change

SFG measurements demonstrate that pentacene deposition may induce bending of the ODTS alkyl chain, corresponding to the formation of interface stress. To quantitatively understand the above process, the energy during the bending of alkyl chains is calculated through a molecular dynamic simulation (53). The bending degree is characterized by the vertical distance ($d$) between the end methyl (CH$_3$) and adjacent methylene (CH$_2$) groups (Fig. 4A, inset); the energy increases linearly from single-digit to tens of kilocalorie per mole per square nanometer with a decrease in $d$, i.e., a larger bending degree leads to greater interface energy. It is to be noted that for a particular molecule, the alkyl chain would more likely bear only few gauche defects rather than adopt this continuous bending conformation as shown in Fig. 2E (middle). Nevertheless, it represents the statistically averaged case, where the closer the C-C unit is to the interface, the greater is the formed interface stress. We further calculated the energy for the disordering of pentacene molecules in the crystal (54). As displayed in Fig. 4B, when a molecule is rotated by 180°, the maximum disturbance energy is approximately 8 kcal/mol per nm$^2$. In actual cases, the energy for disordering an organic semiconductor should be less than the maximum disturbance energy because it is unnecessary for molecules to rotate by such a large angle to achieve a disordered state.

These theoretical results indicate that if the bending degree is sufficient, then the resulting interface stress energy may overcome the disturbance energy, resulting in the disordering of pentacene. This process is further simulated as shown in Fig. 4C (section S9). The configurations of the ODTS SAM and pentacene in fig. S13A are set as the initial state. After performing molecular dynamics simulation, it is determined that the pentacene layer on a H-ODTS surface becomes disordered and that parts of the bended ODTS restretch (Fig. 4, C to E, and figs. S13B, S14B, and S15), whereas the ordering of pentacene layer does not show clear changes on a L-ODTS surface (figs. S13, C and D, and S15).

Generality of the interface stress at semiconductor/SAM interface

The generality of the interface stress between an organic semiconductor and SAM is examined in two ways: changing the organic semiconductor or changing the SAM. Initially, three representative organic semiconductors, vanadyl phthalocyanine (VOPc), dinaphtho[2,3-b:2′,3′-f][thieno[3,2-b] thiophene (DNTT), and 2,6-diphenylanthracene (DPA) are selected (Fig. 1A). As shown in Fig. 5A and Table 1, the device stability dependence of VOPc is similar to that of the
pentacene, i.e., higher ODTS density worsens the stability, while the degree of mobility decay (approximately 50%, black curve in Fig. 5A) is less than that of pentacene devices. After VOPc deposition, the ordering parameter \( A_{\text{CH}_3/\text{CH}_2} \) of H- and L-ODTS/VOPc with the storage time, which is higher than that of the pentacene system (0.1) (Fig. 3B and Table 1). This indicates that VOPc deposition induces less disordering than that of pentacene, i.e., smaller interface stress. During storage, the ordering parameter \( A_{\text{CH}_3/\text{CH}_2} \) of VOPc increases from the initial 0.6 to 0.9 (Fig. 5B), whereas that of the pentacene system increases from 0.1 to 0.7 (Fig. 5A and Table 1). This establishes that VOPc OFETs with H-ODTS release smaller interface stress, which may be responsible for lesser mobility degradation. In a L-ODTS system, the ordering degree remains at the same level during storage (red curve in Fig. 5B), indicating that there is no obvious change in the interface stress; hence, the device stability is high. DNTT and DPA OFETs with H-ODTS show mobility decrease of approximately 27 and 5% during storage, respectively, suggesting better stability than pentacene and VOPc OFETs (Fig. 5C and Table 1). From the SFG spectra, the ordering parameter of H-ODTS after deposition of DNTT and DPA reduces to approximately 0.8 and 1.0 (Fig. 5D and Table 1), respectively, indicating a smaller bending degree of the alkyl chain (i.e., less interface stress) compared to the pentacene and VOPc systems. The ordering parameter shows a slight increase during storage (Fig. 5D and Table 1), implying that small interface stress is released during storage. The comparative OFET and SFG results of the four types of organic semiconductors indicate that smaller interface stress is beneficial for better device stability.

Second, the effect of different SAMs on the structure and stability of pentacene OFETs is examined (Fig. S18A). It is clear that OFETs with shorter alkyl chains [octyltrimethoxysilane (C8TS), propyltrimethoxysilane (C3TS), and hexamethyldisilane (C1DS)] are more stable than those with ODTS (C18). In general, the shorter the alkyl chain, the better is the stability. The SFG results of C3TS/pentacene and C8TS/pentacene are displayed in Fig. S18 (B and C); C1TS/pentacene does not show useful SFG signals due to the lack of methylene groups. The ordering of C3TS/pentacene is higher than C8TS/pentacene (Fig. S18D), establishing the weaker bending of C3TS compared to C8TS. Furthermore, the ordering parameters of C3TS/pentacene and C8TS/pentacene remain stable during storage, indicating structural stability of these two systems. These results suggest that shorter alkyl chains yield weaker interface stress and, therefore, better OFET stability.

**DISCUSSION**

Interface stress has not been investigated in organic optoelectronic devices before mainly because (i) it is thought that organic compound has intrinsic flexibility and thus might not form prominent interface stress; (ii) no proper method is used to probe the embedded interface of organic devices. In this work, SFG was demonstrated as an effective technique for detecting the structural and mechanical properties at organic interface. The molecular conformation evolution (bending and restretching) at the embedded interface is clearly disclosed, which corresponds to the formation and release of interface stress. This work demonstrates that flexible alkyl chain may form stress at the embedded interface, which was sufficiently high to induce the disordering of the organic semiconductor layer (movie S1) and thus leads to the device instability. This study may overturn the conventional understanding of device instability that is generally ascribed to chemical degradation of organic compounds and thus presents a unique aspect for expounding the long-standing and unsettled instability problem in organic optoelectronics, paving pioneering avenues to improve device stability by reducing the interface stress.

**MATERIALS AND METHODS**

**Materials and instruments**

Four batches of pentacene were purchased from Sigma-Aldrich and Fluka. VOPC, DNTT, and DPA were purchased from Shanghai Daeyeon Chemicals. ODTS, C8TS, C3TS, and C1DS were purchased from Sigma-Aldrich. Acetone, chloroform, and ethanol (high-performance liquid chromatography) were obtained from Aladdin Industrial Corporation. All these compounds were used as received. The UV-vis absorption spectra were obtained using a Cary Series UV-vis-NIR spectrophotometer, and the AFM measurements were obtained using a Vecco Dimension 3100. XRD measurements in the \( \theta-2\theta \) scan mode were carried out using a Bruker D8 ADVANCE diffractometer. The static water contact angle was obtained using the DCAT 21 tensiometer.

**Preparation of substrates with different-density ODTS**

Substrates with H-ODTS were prepared in a vacuum of 0.1 MPa at 120°C for 140 min. Lower temperature and lesser time were
used to obtain M- and L-ODTS. These OTDS substrates were acquired at 70°C for 50 min and 40°C for 20 min, respectively. After modification, the substrates were washed by sonication in acetone, chloroform, and ethanol, respectively. The static water contact angles of H-, M-, and L-ODTS were approximately 101°, 66°, and 44°, respectively (fig. S1).

Device fabrication and characterization
Highly doped silicon wafer and a 300-nm thermally oxidized SiO₂ layer (C₅ ≈ 10 nF cm⁻²) were used as the gate and insulator, respectively. Pentacene films of 30 to 50 nm were thermally deposited under a vacuum of 10⁻³ Pa at a rate of approximately 0.05 Å s⁻¹. Gold was deposited as the source and drain electrodes. The OFETs were characterized using the Keithley 4200-SCS in a probe station system at dark in air conditions. The field-effect characteristics were calculated using Eq. 1 in the saturation regime

$$ I_{DS} = C_1 \mu W/2L (V_{GS} - V_T)^2 $$

where $I_{DS}$, $V_{GS}$, $\mu$, and $V_T$ are the source-drain current, gate-source voltage, mobility, and threshold voltage, respectively; $C_1$ is the dielectric capacitance; and $W$ and $L$ are the channel width and length, respectively. During the lifetime test, all the devices were stored under dark in air. The temperature range was 15° to 35°C, and the humidity was 25 to 55%. To ensure the shelf-life results, four batches of pentacene were used for device fabrication and characterization, and eight people reproduced the OFET experiments with three types of vacuum equipment at three locations (Tianjin, Suzhou, and Beijing).

SFG measurements
We used a picosecond SFG spectrometer laser system built by EKSPLA, operating at 50 Hz with a copropagating configuration. The pulse width was approximately 25 ps. The visible wavelength was fixed at 532 nm, and the IR pulse was tunable in the 1000 to 4300 cm⁻¹ range. The incident angle was 60° for the visible beam and 55° for the IR beam. The SFG signal was collected at around 59.5° in the reflection geometry within a small range depending on the IR wave number (2800 to 3000 cm⁻¹ in the experiment). The spectrum was recorded at 2 cm⁻¹ increments per scan and averaged more than 200 laser pulses per point. The energy of the visible beam was typically less than 100 mJ, whereas that of the IR beam was less than 200 mJ; no obvious photodamage or heat effect was observed throughout the study. The polarization combination was typically SSP during the experiment. More details are available in the Supplementary Materials.

Molecular dynamics simulation
SAM systems were constructed by directly connecting the carbon alkyl chains with the silica oxide substrate in a well-ordered arrangement. In this study, two different density SAM systems (4.3 and 1.4 count/nm²) were built by deleting a certain number of alkyl chains on the silica oxide substrate. The configuration including pentacene molecular in the model SAM was formed by placing the pentacene monolayer in the yx axes directions of the SAM substrate as shown in Fig. 2 (E and F) and fig. S13.

Molecular dynamics simulations were performed with leapfrog time integration and LINear Constraint Solver bond constraint, and the interatomic interactions were described by the all-atoms optimized potentials for liquid simulations force field. All the simulations were initialized by minimizing the energy of the initial configurations with the steepest descent method, followed by 5-ns simulation under canonical ensemble (NVT) for each SAM system with a time step of 0.5 fs. During all the simulations, the silicon oxide substrate was considered rigid, while the alkyl chains were set to be flexible. Downward forces with different magnitudes were applied to the methyl groups of the high-density SAM to bend the ODTS molecules, and the energy variation was recorded to evaluate the SAM deformation energy. The energy needed to disorder the herringbone pattern of pentacene and other molecules (DPA and DNTT) was determined by calculating the energy barrier for twisting a single molecule in the monolayer structure.

SUPPLEMENTARY MATERIALS
Supplementary material for this article is available at http://advances.sciencemag.org/cgi/content/full/7/16/eabf8555/DC1

REFERENCES AND NOTES

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