

Initial gas exposure effects on monolayer pentacene field-effect transistor studied using four gallium indium probes

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ABSTRACT

In this study, we report that the electrical transport property in monolayer pentacene is highly sensitive to a small amount of exposure to gaseous molecules of oxygen, nitrogen, and argon, by using independently-driven four gallium indium (GaIn) probes. Liquid metal GaIn probes have been used as non-destructive conductivity electrodes for monolayer OFET films in a vacuum chamber. We carried out the fabrication of monolayer pentacene and the four-probe measurement without exposure to atmospheric air. Four GaIn probes were used *in situ* to measure conductivity in the channel separately from the overall characteristics including contact resistance at the electrode probes. The results demonstrate that oxygen exposure of 1 L (10^{-6} Torr s) reduces mobility to 8% of the original value in the monolayer film, and the reduction is irreversible, i.e., mobility does not recover its original value after the evacuation of gaseous molecules. Physisorption of gaseous molecules is involved in the gas exposure effects, because chemically inert gases of nitrogen and argon also reduce the mobility. Taking these findings together with the results of photoelectron spectroscopy and atomic force microscopy measurements, we assume that the reduction is attributed to the physisorption at grain boundaries formed by the coalescence of monolayer islands.

1. Introduction

Interactions of gaseous molecules with organic field effect transistors (OFETs) play a crucial role in the electronic transport property. Chemical reactions, such as oxidation, are a key issue for degradation of long-term stability of organic devices [1–3]. Physical adsorption can also modify electronic states of OFETs. Gaseous molecules diffuse into OFET film through defect sites including grain boundaries, resulting in a change in the film structure and the formation of a gap state, which leads to a shift of the HOMO band to a lower binding energy [4–6]. The diffusion of gaseous molecules is reversible, i.e., the molecules desorb from the film after evacuation of the gas atmosphere [5]. This kind of reversible effect is sometimes used to enhance device performance. In order to enhance mobility to the best value, OFET devices are often kept in inert gases for a few days [7,8]. Furthermore, charge transfer between adsorbed molecules and OFET film is important in particular for sensor applications [9–11]. Adsorption of acceptor/donor molecules of NO_2 and NH_3 generates holes/electrons in the film, which lead to changes in the threshold voltage, drain current, and mobility [12–16]. It has been reported that monolayer pentacene film is highly sensitive to these acceptor/donor molecules due to grain boundary dominated two-dimensional transport [14,15,17]. Since these gas interactions not

only degrade device performance but also enhance charge transport properties [18], control and understanding of the effects are necessary for OFET devices.

These gas exposure experiments were performed under ambient conditions, in other words, a large number of gaseous molecules were exposed on the OFET film surface. On the other hand, little is known about the initial adsorption of gaseous molecules, due to the difficulty of fabricating electrical contacts on OFET film in a vacuum chamber without destroying the OFET film. In this study, we report that the electrical transport property in monolayer pentacene is highly sensitive to a small amount of exposure to gaseous molecules of oxygen, nitrogen, and argon, by using four independently-driven gallium indium (GaIn) probes. We carried out the fabrication of monolayer pentacene and the four-probe measurement *in situ*. We reported that liquid metal GaIn probes, which have often been used for tunnel transport on self-assembled monolayers [19,20], can be used as non-destructive conductivity electrodes for OFET films in a vacuum chamber [21]. Four GaIn probes were used to measure conductivity in the channel separately from the overall characteristics including contact resistance at the electrode probes. As reported previously, monolayer pentacene is more sensitive to gas exposure than multilayer films [14,15].

The present results demonstrate that a small amount of gas exposure

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highly reduces mobility in the monolayer film, and the reduction is irreversible, i.e., mobility does not recover its original value after evacuation of gaseous molecules. Furthermore, we carried out atomic force microscopy (AFM) and photoelectron spectroscopy, in order to investigate the effects of gas exposure on the structure and the electronic state of monolayer pentacene.

2. Experimental

Four GaIn probes were fabricated by attaching a GaIn droplet with diameters ranging from 100 to 300 μm on the apex of each Au-coated W tip in air [21]. A highly doped n-type Si(100) wafer covered with 180-nm-thick thermal oxide (Electronics and Materials Co., Ltd.) was cleaned by ultrasonication in acetone and ethanol, followed by cleaning in piranha solution ($\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2 = 3:1$) at 90°C for 10 min. Then the GaIn probes and the sample were installed in a vacuum chamber ($\sim 10^{-6}$ Pa) equipped with a four-probe system [22]. Pentacene was deposited on the sample from a quartz Knudsen cell through a shadow mask (1 mm \times 1 mm) at room temperature. 1.5-nm-thick (1 ML) or 4.5-nm-thick (3 ML) pentacene film was deposited with a deposition rate of 0.1 $\text{\AA}/\text{s}$ using a quartz microbalance.

The four GaIn probes were contacted on the pentacene film with a linear probe alignment at a probe distance of 300 μm under optical microscope observation (Fig. 1(a)). Electrical contacts between the probes and the pentacene film were detected by monitoring the AC current on each probe with applying AC bias on the gate substrate. A schematic of the four-probe $I - V$ measurements is shown in Fig. 2(b). The outer two probes were used as source and drain electrodes, and the inner two probes (c1 and c2) were used to measure the voltage drop in the channel. We used a feedback circuit to keep the channel potential V_{c1} at ground level during the $I - V$ measurements. In this setup, the gate-to-channel bias is kept constant even when change in contact

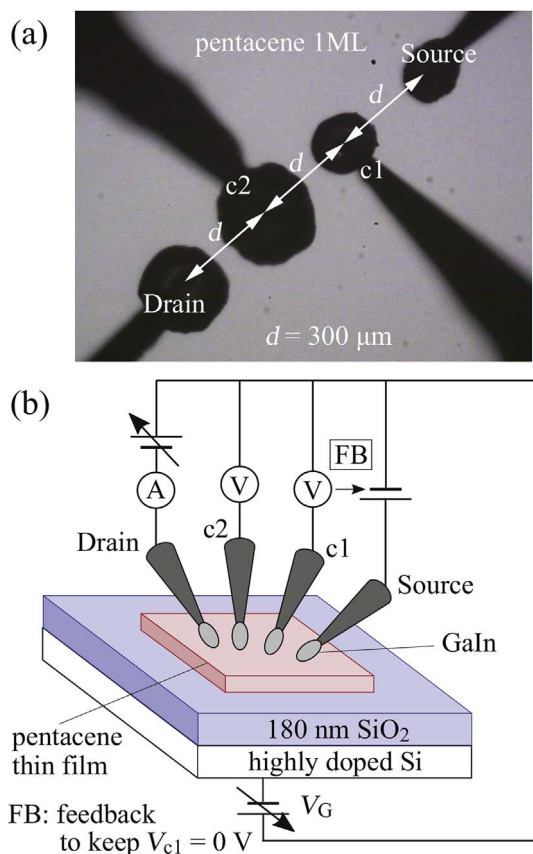


Fig. 1. (a) Optical microscope image of GaIn probes contacted with 1 ML pentacene. (b) Schematic of the electrical circuits.

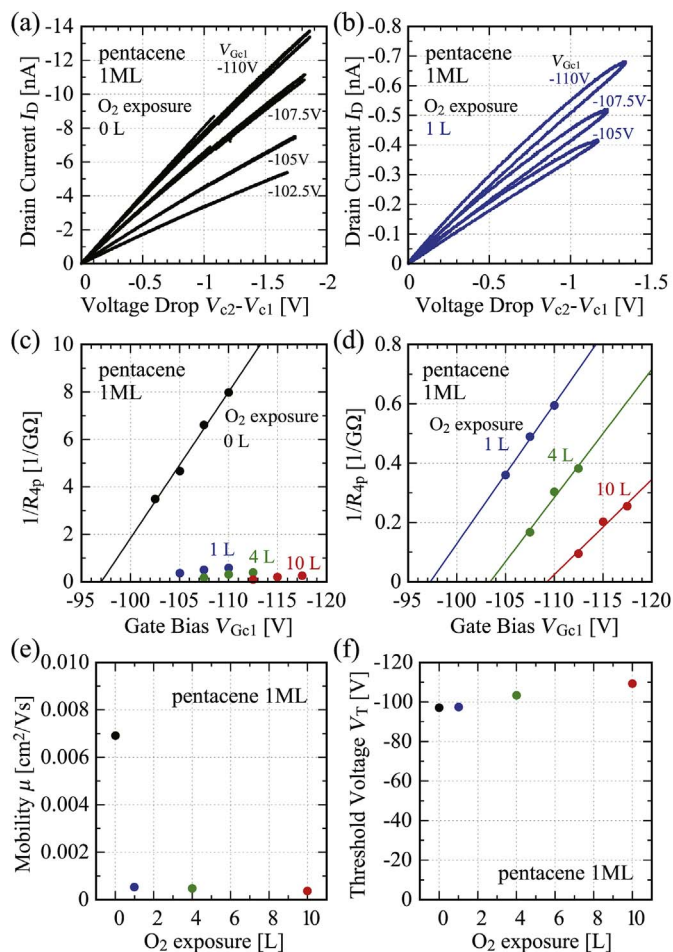


Fig. 2. Four-probe $I - V$ curves (a) before and (b) after O_2 exposure measured with the probe alignment shown in Fig. 1(a). (c) and (d) Inverse of four-probe resistance $1/R_{4p}$ as a function of gate to channel bias V_{Gc1} . (e) Mobility and (f) threshold voltage calculated by Eq. (1).

resistance at the source and drain electrodes occurs during the experiments [22]. After the $I - V$ measurements, the sample was exposed to gaseous oxygen, nitrogen, or argon through a variable leak valve. All $I - V$ measurements were performed at room temperature in high vacuum, i.e., gaseous molecules were evacuated before the $I - V$ measurements, except the highest exposure on 3 ML pentacene in which $I - V$ was measured in O_2 atmosphere. The GaIn probes were kept in contact with the film during the exposure, thus, the GaIn-pentacene interfaces were not exposed to gaseous molecules. During the experiments, the gas qualities were confirmed by a quadrupole mass spectrometer.

High resolution X-ray photoelectron spectroscopy (XPS) and ultraviolet photoelectron spectroscopy (UPS) measurements were performed in an ultrahigh vacuum (UHV) chamber equipped with a hemispherical electron energy analyzer (Scienta SES200) at BL-13B of the synchrotron light source Photon Factory, KEK, Japan (PAC 2015-S2-008). All XPS and UPS measurements were performed at room temperature in UHV. The XPS and UPS spectra were collected using photon energies of 650 eV and 50 eV under normal emission conditions, and the overall instrumental resolutions (monochromator plus analyzer) were 120 meV and 20 meV, respectively. The zero-binding energy (BE) was taken at the Fermi edge of a tantalum film. In order to prevent charge-up of the sample, a n-type Si(111) wafer with a 5 \AA -thick native oxide was used instead of the thermal oxide. A Si wafer was first cleaned in piranha solution, then it was etched in hydrofluoric acid (5% HF), and finally a thin oxide layer formed in piranha solution. Details of the native oxide

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