Fabrication and Electrical Characterizations of Field-Effect Transistors with a piece of C\textsubscript{60} Nano-Whisker

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Abstract. C\textsubscript{60} nano-whiskers (C\textsubscript{60} NWs) have been synthesized by a method of liquid-liquid interfacial precipitation using a system of C\textsubscript{60}-saturated \textit{m}-xylene and isopropl alcohol, and a C\textsubscript{60} nano-whisker based field-effect transistor (C\textsubscript{60} NW-FET) has been fabricated by a manipulator with use of a glass micro-capillary. The transport of the C\textsubscript{60} NW-FET exhibits \textit{n}-channel normally on properties and their carrier mobility is estimated to be 2.5 \times 10^{-2} \text{ cm}^2/\text{V}\text{s} in vacuum at room temperature.

1. Introduction

Recently, many approaches to the solution-based growth of C\textsubscript{60} crystals have been reported in the literature on carbon fullerene devices. Of particular interest here, needle-like C\textsubscript{60} structures have been obtained by slowly evaporating solutions of C\textsubscript{60} in 1,2-dichloroethane (DECAN) at room temperature [1], or of toluene-containing C\textsubscript{60} [2]. Fibrous C\textsubscript{60} “nano-whiskers” (NWs) have been obtained by a method of liquid-liquid interfacial precipitation (LLIP) [3,4]. The NWs exhibit a single-crystal structure and have sub-micrometer diameters and more than several tens-micrometer lengths. Moreover, hollow C\textsubscript{60} NWs which are called C\textsubscript{60} “nano-tubes” have also been found [5,6]. Both of C\textsubscript{60} NWs and C\textsubscript{60} nano-tubes are confirmed that these fibers are composed of C\textsubscript{60} molecules by Fourier-transform infrared (FTIR) spectra [6,7]. In spite of their potential importance for carbon-based nanoelectronics, there have been only a few reports of the electrical properties of these NWs so far.

Field-effect transistors (FETs) based on thin films of organic molecules have recently been studied by many groups [8,9]. In this field, C\textsubscript{60} thin-film transistors have been found to show \textit{n}-channel enhancement-type properties, whose highest mobilities are 0.1 \sim 0.5 \text{ cm}^2/\text{V}\text{s} [10-12]. During the fabricating process, it has been popular to use a heating [13,14] or a hydrophobic treatment [15] to enhance molecular accumulation, and large organic crystals are thereby obtained. In another approach, the channel size should be reduced to a scale comparable to the crystal grains [16] because the granular film by high-vacuum thermal deposition prevents the achievement of the mobility.
In this report, we present the FET characteristic of a single C\textsubscript{60} NW that is a single-crystal structure synthesized by LLIP. The FET shows $n$-channel normally on properties, and the room-temperature carrier mobility is estimated to be $2.5 \times 10^{-2}$ cm$^2$/Vs. Our results demonstrate that C\textsubscript{60} NW are an important candidate for high mobility FETs based on organic materials.

2. Experimental procedure

The C\textsubscript{60} NWs used in this study were synthesized by LLIP, using a system of C\textsubscript{60}-saturated $m$-xylene and isopropyl alcohol. The C\textsubscript{60}-saturated $m$-xylene solution was placed in a glass bottle, and then isopropyl alcohol was gently added so as to realize a liquid-liquid interface between the saturated $m$-xylene solution and the isopropyl alcohol. For promoting the NW growth, the bottle was capped and left undisturbed at $\sim 7$ °C for 7 days. For fabricating C\textsubscript{60} NW-FET, we used a heavily-doped ($5 \times 10^{-3}$ cm$^{-1}$) Si wafer as a substrate and as a back gate, and it has insulating top layer of a thermally-grown SiO\textsubscript{2} (500 nm). A source and a drain electrodes of Ti/Au (10/20 nm) were fabricated on the surface of SiO\textsubscript{2} by photo-lithography and lift-off process. The device had a channel length of 5 $\mu$m and a piece of the C\textsubscript{60} NW was used to bridge the two electrodes by use of a three-dimensional manipulator with a glass micro-capillary under a high-resolution optical microscope. In order to remove intercalated oxygen and solvents in the C\textsubscript{60} NW, the device was annealed for about 24 hours at $\sim 440$ °C and $\sim 10^{-6}$ torr. After annealing, the FET characteristics was measured without exposing to air. Moreover, we investigated gas exposure effects of the resistance of C\textsubscript{60} NW by introducing N\textsubscript{2} and O\textsubscript{2} gas into the vacuum chamber.

3. Results and discussion

Typical size of C\textsubscript{60} NWs obtained by the LLIP were less than 0.3 $\mu$m in diameter and more than several tens micrometer in length. These NWs are generally straight or slightly curved, with an almost constant diameter along their growth axis. The finest NWs were found to be about 20 nm in diameter, confirmed by atomic-force-microscope (AFM). From both scanning-electron-microscope (SEM) and AFM observations, the NWs exhibit a hexagonal cross section, which can be confirmed clearly in figure 1(a). In figure 1(a), the line profile of C\textsubscript{60} NW shows a flat at around the top due to the hexagonal cross section.

![Figure 1](image_url)

**Figure 1.** (a) SEM image showing typical C\textsubscript{60} NWs obtained by LLIP. The inset shows the typical line profile of the C\textsubscript{60} NW by AFM. The dotted line is guide for eyes. (b) SEM image showing the C\textsubscript{60} NW bridging between the two electrodes in the C\textsubscript{60} NW-FET device.

Before the annealing of C\textsubscript{60} NW-FET, no evidence of FET characteristic was observed. However, after 24 hours annealing at $\sim 440$ K and $\sim 10^{-6}$ torr, we observed the FET operation. In several reports related to C\textsubscript{60} NWs, structure of C\textsubscript{60} NWs exhibit a hexagonal crystal structure (P6\textsubscript{3}, $a = 23.7$ Å, $c = 10.0$ Å) due to intercalation of $m$-xylene molecules among C\textsubscript{60} molecules [17,18]. Recently, it is reported that the crystal structure of the C\textsubscript{60} NW changes to fcc structure by the evaporation of solvent.
(m-xylene) in air [7]. For annealing our device, solvent molecules (m-xylene) in the C₆₀ NW were evaporated as well. Therefore, after annealing, this device has exhibited FET performance because of evaporating almost solvent molecules and as a consequent crystal change. The C₆₀ NW still remain the shape without sublimation even at 440K, since usual sublimation temperature of C₆₀ is higher than about 573K under ~10⁻⁶ torr.

Figure 1(b) shows the SEM image of our C₆₀ NW bridging between the two electrodes. Source-drain current (I₈D) vs source-drain voltage (V₈D) characteristics (I₈D-V₈D) of the C₆₀ NW-FETs at several back-gate voltages (V₈G) are shown in figure 2(a), which show n-type FET properties. Gate-voltage characteristics (I₈D-V₈G) is shown in figure 2(b). The threshold voltage (Vₖ) is about 10 V, and the field-effect mobility (µ) can be estimated from a following relation [19],

\[ I_{SD} = \left( \mu WC_0/L \right) \left( V_G - V_T - V_{SD}/2 \right) V_{SD} \]  \hspace{1cm} (1)

where \( W \) and \( L \) are the channel width and the length of the device, respectively, and are estimated to be 5 \( \mu \)m and 400 nm from the SEM observation as in figure 1(b). \( C_0 \) is a capacitance per unit area of the gate dielectric. Here, the C₆₀ NW bridging the electrodes does not touch the SiO₂ surface layer directly, and is suspended with the height of about 30 nm from the surface due to the thickness of these electrodes according to the AFM measurement. Therefore, if we can assume a compound electrostatic capacitance due to the SiO₂ layer and the gap space, the mobility is estimated about \( 2.5 \times 10^{-2} \text{ cm}^2/\text{Vs} \).

![Figure 2](image-url)

**Figure 2.** (a) \( I_{SD}-V_{SD} \) characteristics of the C₆₀ NW-FET device at several gate voltages. (b) \( I_{SD}-V_{G} \) characteristics of the C₆₀ NW-FET device at \( V_{SD} = 20 \text{ V} \).

In figure 2(a), the \( I_{SD} \) does not exhibit a saturation even up to \( V_G = 40 \text{ V} \). However, many papers have reported such a saturation region in C₆₀ thin-film FET [10-12]. One of the possible reasons is because the C₆₀ NW was only put on the electrodes, therefore, the contact resistance between C₆₀ NW and the electrodes became high inevitably. It may play an important role to prevent the saturation of our device. In order to improve the field-effect mobility of the C₆₀ NW-FETs, we must firstly reduce such a contact resistance. Probably, a use of a low work-function metal electrodes would be effective to realize a good ohmic contact [20].

Moreover, we have observed a change of the resistance in annealed C₆₀ NW under N₂ and O₂ gas. We have introduced O₂ gas into the vacuum chamber just after introducing N₂ gas. Although the resistance is no change in case of introducing N₂ gas, the resistance suddenly increases about two orders in case of O₂ gas. A similar behavior on those gas purgings have been observed in C₆₀ thin-film FET [21], which also indicates that electrical properties of C₆₀ NW is bad for oxygen absorption as well as C₆₀ thin-film.
4. Summary
In summary, C\textsubscript{60} NWs have been synthesized by LLIP and have been utilized as a channel of a FET structure. We fabricate C\textsubscript{60} NW-FETs by using manipulator with a glass micro-capillary. After annealing of the device, the C\textsubscript{60} NW-FET exhibites \textit{n}-channel normally on properties, and the carrier mobility is estimated to be $2.5 \times 10^{-2}$ cm$^2$/Vs under vacuum at room temperature. Furthermore, we inspect the effect of gas exposure for C\textsubscript{60} NW. The resistivity has no change in N\textsubscript{2} gas while it suddenly increases in O\textsubscript{2} gas.

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