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Weak localization in bilayer graphene with Li-intercalation/desorption

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Abstract

We performed *in-situ* electrical transport measurements for bilayer graphene grown on SiC(0001) substrate, Li-intercalated bilayer graphene, and after that desorbing Li atoms by heating. Bilayer graphene after desorbing intercalated Li atoms showed a higher resistivity and different behavior in magnetoconductance compared to pristine bilayer graphene. We observed the weak localization of carriers at low temperatures in all the three samples and analyzed the experimental results with the extended Hikami–Larkin–Nagaoka equation to investigate the transport properties. The result shows that the magnetoconductance of pristine bilayer graphene is described by the AB stacking structure model and the phase breaking scattering is dominated by the electron–electron scattering. The intra-valley scattering occurs most frequently probably due to dopants in SiC substrate. However, in Li-desorbed graphene, the magnetoconductance can be described by neither AB nor AA-stacking model, suggesting the coexistence of domains with several different stacking structures.

Keywords: graphene, weak localization, quantum coherent transport, intercalation, atomic layer, Berry phase, stacking structure

 Supplementary material for this article is available [online](#)

(Some figures may appear in colour only in the online journal)

1. Introduction

Graphene, a single atomic sheet of graphite, has been a target of intensive studies because of the novel properties arising from the notable two-dimensional (2D) and massless nature of carriers therein [1–3]. The effects of quantum interference in 2D electron systems, such as weak localization (WL) and weak antilocalization (WAL), have been intensively studied because they provide rich information on the electronic transport property [4, 5]. In particular, exfoliated monolayer and bilayer graphene and monolayer graphene grown on SiC substrate have been used as a platform for observing such various novel phenomena [6–13]. Previous studies clarified a

substantial contribution from the electron–electron interaction and WL to the resistivity and its sub-micrometer coherence lengths. The transport property of bilayer graphene grown on SiC substrate, however, has not been sufficiently investigated whereas it is abundant in novel phenomena such as 2D superconductivity [14, 15].

Graphite intercalation compounds (GICs) show various novel properties such as superconductivity and magnetism. GICs are widely used for technological application like an electrode of Li-ion battery and chemical catalysis [16]. Recently, graphene intercalation compounds, the thinnest limit of GICs, are attracting much attention as a platform for 2D superconductivity and electrochemical devices [14,

15]. Li-intercalated graphene has been actively investigated in these days for application to Li-ion nano-battery [17, 18]. However, the basic process of Li intercalation and de-intercalation is still unclear, in particular, in the relation to the electronic transport property.

In this paper, we report an *in situ* electrical transport measurement study of bilayer graphene grown on SiC substrate to investigate the change in the transport properties upon Li-intercalation and desorption (de-intercalation). We found that the temperature and magnetic-field dependences of the resistivity in bilayer graphene are well explained in terms of the WL effect. We observed that the conductivity was remarkably enhanced upon Li-intercalation, while it was drastically reduced upon desorption of Li to the value much lower than that of pristine bilayer graphene. The observed WL effect in Li-desorbed sample cannot be explained with the same model as used in pristine bilayer graphene, implying a significant structural change during the Li-intercalation/desorption process.

2. Experimental

Figures 1(a)–(c) show RHEED patterns of pristine, Li-intercalated and Li-desorbed bilayer graphene, respectively. Pristine bilayer graphene (called S_1 hereafter) was prepared on an *n*-type Si-rich 6H-SiC(0001) substrate by direct heating up to 1550 °C under 1 atm argon atmosphere in an ultrahigh vacuum (UHV) chamber. By precisely controlling the heating temperature and the duration time, we succeeded in fabricating bilayer graphene [17]. The number of graphene sheets was confirmed by observing the band dispersions near *K* point in the Brillouin zone by angle-resolved photoemission spectroscopy (ARPES) [17]. After a short-time exposure to air, the bilayer graphene sample was transferred to another UHV system equipped with a reflection high-energy electron diffraction (RHEED) apparatus, Li evaporator, and a resistance measurement system [19]. As seen in figure 1(a), after heating up to 500 °C under UHV to remove contaminants adsorbed on the surface, the RHEED pattern from S_1 clearly exhibited the graphene's 1×1 and the buffer layer's $(6\sqrt{3} \times 6\sqrt{3})R30^\circ$ spots. Deposition of Li atoms was carried out using a Li dispenser (SAES Getters) at room temperature under UHV (3×10^{-10} Torr). The resultant sample (S_2) showed a RHEED pattern of $(\sqrt{3} \times \sqrt{3})R30^\circ$ characteristic of Li-intercalated bilayer graphene (figure 1(d)) [14, 17] with no trace of $(6\sqrt{3} \times 6\sqrt{3})R30^\circ$ spots. The third sample (S_3) was prepared by heating S_2 sample at 900 °C in UHV to desorb the Li atoms until the $(\sqrt{3} \times \sqrt{3})R30^\circ$ pattern disappeared and the $(6\sqrt{3} \times 6\sqrt{3})R30^\circ$ pattern from the buffer layer recovered as shown in figure 1(c).

The transport measurements were performed by the *in situ* four-point probe (4PP) technique in a UHV system (Unisoku USM-1300S) without exposing the samples to air [19]. The 4PP consists of four copper wires of 100 μm in diameter, aligned on a line with the probe spacing of ca. 200 μm . The sheet resistance R_s was obtained by the 4PP dc current-voltage measurement by using the dual configuration method

to avoid the data scattering due to the error in the probe spacing [19–22].

3. Results and discussion

Figure 2(a) shows the temperature dependence of conductance for S_1 – S_3 . The conductance of S_2 (Li-intercalated one) is ~ 3 times higher than that of S_1 (pristine), and it is remarked that S_3 (Li-desorbed one) exhibits lower conductance than S_1 , although the RHEED pattern of S_3 looks similar to that of S_1 . The increase in conductance from S_1 to S_2 is due to doping of electrons by intercalated Li atoms. As seen in figure 2(b), we performed the same experiment by using another sample, and then found a scattering in the conductance for S_2 from sample to sample, while the conductance of S_1 and S_3 was well reproducible. This scattering in conductance value in S_2 may be due to difference in the amount of Li atoms intercalated in and/or adsorbed on graphene. However, the carrier density of S_2 is at least ~ 16 times larger than that of S_1 estimated from the band dispersion by ARPES [17]. According to the result of low-energy electron microscopy (LEEM), Li-intercalation induces wrinkles/cracks and defects in graphene [23], which implies that the Li-intercalation process causes additional scattering centers for carriers.

We also found that the conductance of S_3 is lower than that of S_1 . To reveal the origin of this difference, we compared the conductance of S_1 with that of S_1 after only heating at 900 °C without Li-intercalation/desorption. As a result, we found that there was little difference between the two samples (figure 1(c)), suggesting that the process of Li-intercalation and desorption should play a major role to induce the irreversible change in the conductance from S_1 to S_3 .

Figure 2(d) shows the temperature dependence of conductance of S_1 – S_3 . When lowering the temperature from 30 K, the conductivity of S_1 at first shows a gradual increase till ~ 7 K and then rapidly decreases at low temperatures as seen in figure 2(d). This rapid decrease of conductance is a characteristic behavior of localization. In the case of S_2 and S_3 , on the other hand, a decrease of conductance appears to start at a higher temperature at around 30 K although the reduction speed is not so rapid as S_1 . This difference suggests that S_2 and S_3 have stronger localization than S_1 .

Now we discuss in details the conductivity of pristine bilayer graphene (S_1) in relation to the WL and WAL effects [12, 13]. The WL occurs when an electron travels a closed path clockwise and anti-clockwise simultaneously to interfere constructively with itself, while the WAL is a result of destructive interference of these two time-reversal processes [24–26]. Whereas the electron is scattered elastically many times during these processes, the phase coherence of wave function is preserved within a finite distance called the phase breaking length L_ϕ . Then, such phase coherence is broken by the inelastic scattering. As a result, the electron forms a standing wave around the closed path within L_ϕ , leading to the decrease (increase) of the electrical conductance due to the WL (WAL). In the case of graphene, WL and WAL can be better explained with three characteristic lengths: the phase

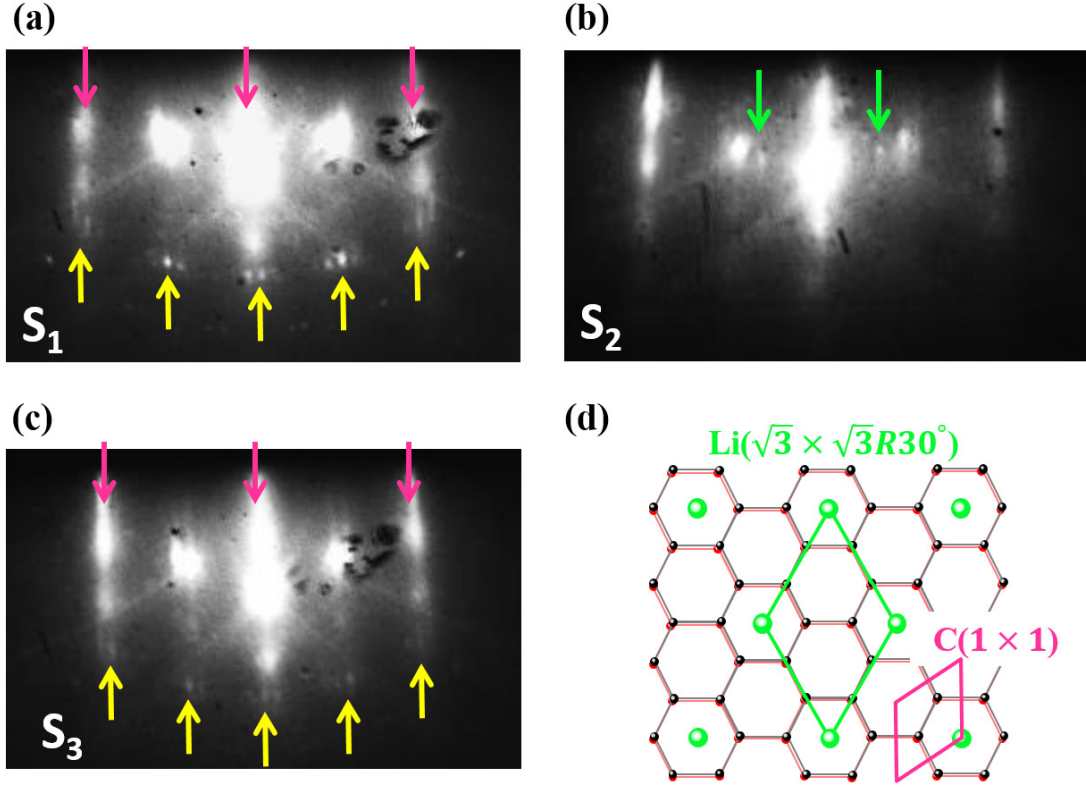


Figure 1. RHEED patterns of (a) pristine bilayer graphene grown on SiC(0001) (S_1), (b) Li-intercalated bilayer graphene (S_2), and (c) bilayer graphene heated at 900 °C after Li-intercalation to desorb Li atoms (S_3). Pink, yellow and green arrows indicate the (1×1) pattern of graphene, the $(6\sqrt{3} \times 6\sqrt{3})R30^\circ$ pattern of the buffer layer, and the $(\sqrt{3} \times \sqrt{3})R30^\circ$ pattern of intercalated Li atoms, respectively. (d) Schematic view of atomic arrangement of Li-intercalated bilayer graphene in AA stacking. Green balls are Li atoms intercalated between graphene layers. Pink and green rhombuses are unit cells of graphene and Li intercalated graphene, respectively.

breaking length L_ϕ , the inter-valley scattering length L_i , and the intra-valley scattering length L_* as shown in figure 3(a). There are two kinds of well-defined stacking structures for bilayer graphene, which are called as AB stacking and AA stacking (figures 3(b) and (c)). Magnetic field dependence of conductivity in AB and AA stacking bilayer graphene is described by the extended Hikami–Larkin–Nagaoka (HLN) equation as equation (1) [9–11, 27, 28] and equation (2), respectively;

$$\Delta\sigma_{AB} = \sigma(B) - \sigma(0) = \frac{e^2}{\pi h} \left[F\left(\frac{B}{B_\phi}\right) - F\left(\frac{B}{B_\phi + 2B_i}\right) + 2F\left(\frac{B}{B_\phi + B_i + B_*}\right) \right], \quad (1)$$

$$\Delta\sigma_{AA} = \sigma(B) - \sigma(0) = \frac{2e^2}{\pi h} \left[F\left(\frac{B}{B_\phi}\right) - F\left(\frac{B}{B_\phi + 2B_i}\right) - 2F\left(\frac{B}{B_\phi + B_i + B_*}\right) \right]. \quad (2)$$

In both equations, the first and second terms describe the WL behavior and $F(x) \equiv \log x + \Psi\left(\frac{1}{2} + \frac{1}{x}\right)$, where Ψ is a digamma function. B_ϕ , B_i , and B_* are defined as $B_{\phi, i, *} \equiv \hbar / (4eD\tau_{\phi, i, *}) = \hbar / (4eL_{\phi, i, *}^2)$, where D is the diffusion coefficient and $\tau_{\phi, i, *}$ is the scattering time for the phase

breaking scattering (τ_ϕ), the inter-valley scattering (τ_i), and the intra-valley scattering (τ_*), respectively. The factor 2 on the right-hand side in equation (2) comes from double Dirac cones in AA stacking bilayer graphene [29, 30]. Equation (1) has factor 1 because AB stacking bilayer graphene possesses a parabolic band. Namely, the factor depends on the number of channels. The third term in both equations (1) and (2) represents an opposite sign each other corresponding to the Berry phase. When the Berry phase is 2π for AB stacking bilayer graphene, the third term has a positive sign indicative of WL [9–11, 27, 28]. In bilayer graphene with AA stacking which has the linear band dispersion, the Berry phase is π , resulting in the negative sign in the third term related to WAL. Generally, the third term has much less contribution than the first and second terms in equations (1) and (2) because of the small intra-valley scattering length (L_*) [9].

Figure 3(d) shows the change in conductance $\Delta\sigma$ of S_1 upon applying the perpendicular magnetic field B . The conductance increases with increasing the magnetic field at all temperatures ($T = 1\text{--}15$ K). This B dependence is explained in terms of the time-reversal-symmetry breaking in WL, that is, the phase of electron is changed with increasing the magnetic field due to the Aharonov–Bohm effect, leading to breaking of the localization (constructive) interference. Black solid curves in figure 3(d) are the results of numerical fitting with equation (1). We find a good agreement, in particular, in the range of low magnetic field (below ~ 60 mT) between experiments

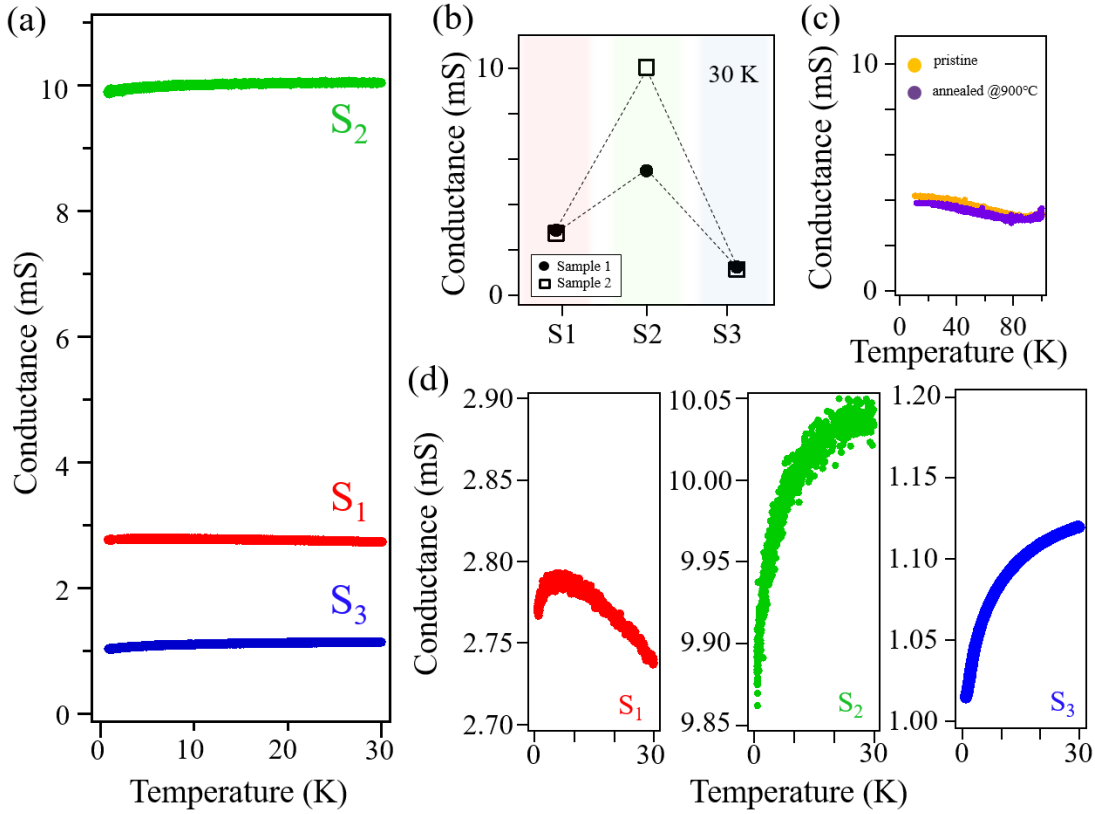


Figure 2. (a) Temperature dependences of conductance among S_1 , S_2 and S_3 . (b) Comparison of conductance at 30 K in S_1 , S_2 and S_3 states between two independent samples. A scattering in conductance value is seen in S_2 state. (c) Comparison of temperature dependence of conductance between pristine (S_1) and that after just heating up to 900 °C without Li intercalation. (d) Conductance of S_1 – S_3 at low temperatures in an expanded scale.

and fittings, which suggests the valid applicability of AB stacking model to pristine bilayer graphene (S_1). We have derived parameters B_ϕ , B_i , and B_* , together with the corresponding scattering times τ_ϕ , τ_i , and τ_* , by fitting the curves, and summarize the results in table 1.

The temperature dependences of $L_{\phi,i,*}$ are plotted in figure 3(e). The L_ϕ depends on temperature T as $L_\phi \propto T^{-P/2}$. When P is close to 1 (2), the phase breaking is caused by the electron–electron interaction (the electron–phonon interaction) [31–33]. In S_1 , the L_ϕ was fitted with $P = 0.83$, as seen in figure 3(e), indicating that the phase breaking is dominated by the electron–electron interaction in pristine bilayer graphene grown on SiC.

Figure 3(f) was derived by fitting curves in figure 3(d) using equation (2) with the AA stacking form. The L_ϕ was fitted with $P = 0.46$, which, however, is much smaller than the theoretical value (1 or 2). Thus, it is suggested that the AA stacking model is not suitable for pristine bilayer graphene (S_1), consistent with the theoretical calculation result that the AB stacking structure is energetically stable than AA stacking for bilayer graphene [34].

S_2 also shows the WL behavior as seen in the supplementary data (figure S1) (stacks.iop.org/JPhysCM/30/305701/mmedia), but the change of magnetoconductance is too small to fit accurately by using equation (1) or equation (2). This may be due to the carrier doping by intercalated Li [17], and thus we have performed a fitting to S_2 by the original HLN

equation to obtain only the L_ϕ . The fitting result shows that $L_\phi = 170 \pm 20$ nm at 1 K, which is relatively smaller than the value of pristine bilayer graphene ($L_\phi = 588 \pm 106$ nm). This difference suggests the increase of phase breaking scattering by Li-intercalation.

Figure 3(g) shows the change in conductance $\Delta\sigma$ of S_3 upon applying the surface-normal magnetic field B . The B dependence can be also interpreted as breaking of the localization (constructive) interference. On the other hand, the B range of increasing conductance is larger than that of S_1 by at least one order of magnitude, meaning the increase in population of scatterers. The B dependence of S_3 was analyzed by equations (1) and (2). Each result is shown in figures 3(h) and (i), respectively. Theoretically, since the inter and intra-valley scatterings originate from the impurity/defect scattering, the $L_{i,*}$ should be temperature-independent. However, L_i shows a non-negligible temperature dependence in both figures 3(h) and (i). Furthermore, the L_ϕ was fitted by $L_\phi \propto T^{-P/2}$ as the solid curves in figures 3(h) and (i), and the obtained P values are 0.27 and 0.22, respectively. These values are too small compared with the theoretical one (1 or 2). All these results indicate that S_3 cannot be described by either pure AB or AA stacking model. This implies that the Li-intercalation/desorption process induces a change in the stacking structure from the AB stacking in pristine bilayer graphene to a mixed domains of different stacking structures including twisted stacking structure [35].

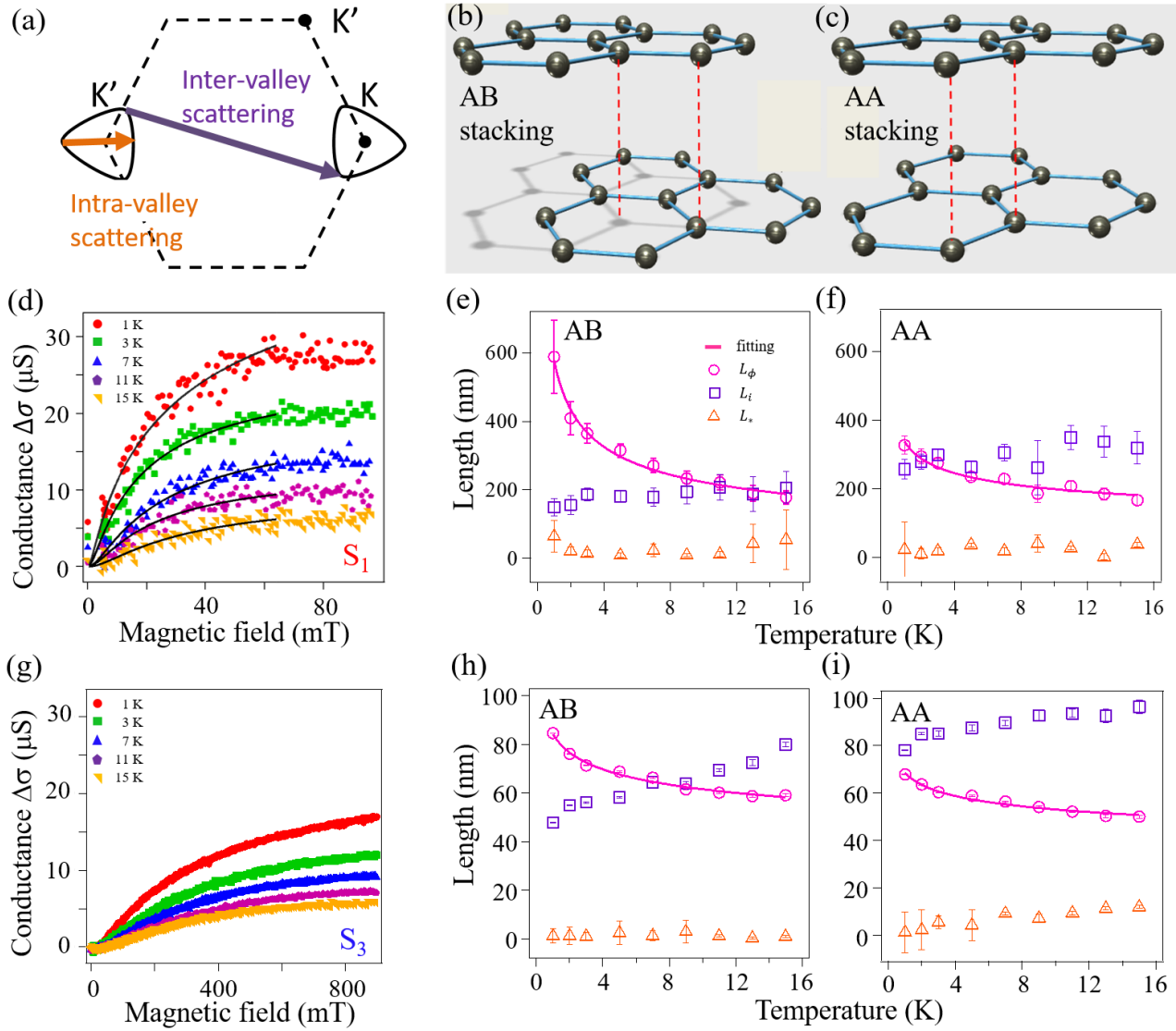


Figure 3. (a) Schematic view of inter- and intra-valley scattering paths on the Fermi surfaces in bilayer graphene. (b) and (c) AB and AA stacking sequence in bilayer graphene. (d) Magnetic field dependence of conductance at various temperatures for S_1 . Black solid lines are fitting curves with equation (1). (e) and (f) Temperature dependences of the scattering lengths in S_1 which were derived by equations (1) and (2), respectively, i.e. the phase breaking scattering (L_ϕ), the inter-valley scattering (L_i), and the intra-valley scattering (L_*) in S_1 . Solid curve is a fitting with $L_\phi \propto T^{-P/2}$. (g) Magnetic field dependences of conductance at various temperatures for S_3 . (h) and (i) Temperature dependences of the scattering lengths in S_3 which were derived by equations (1) and (2), respectively.

Next, we discuss the detail of transport properties in pristine bilayer graphene on SiC(0001) substrate. In table 1, we compare the present results derived by equation (1) with previous reports [12, 13]. $\tau_{\phi,i,*}$ of graphene grown on SiC (two right columns) are smaller than those of exfoliated graphene (two left columns). Here, we discuss the relationship between the scattering time of carriers and its mechanism. First, we focus on τ_* for the intra-valley scattering in which the scattering occurs within each valley (figure 3(a)). In all cases in table 1, τ_* is much smaller than τ_ϕ and τ_i , meaning that the intra-valley scattering occurs most frequently. This type of scattering with smaller changes in momentum can be induced by weaker scattering potentials compared with the inter-valley scattering. For the case of monolayer graphene grown on SiC, it is reported [13] that the intra-valley scattering can be induced by donors in the SiC substrate. Actually, the SiC wafer used

as a substrate in our study has a high nitrogen dopant density of $\sim 1 \times 10^{18} - 1 \times 10^{19} \text{ cm}^{-3}$. The Thomas–Fermi screening length is estimated to be 0.25–0.37 nm by taking into account the carrier density of degenerate semiconductor substrate [36]. The dopant density on the surface and the average distance between the nearest dopants in our SiC was estimated to be $\sim 3.7 \times 10^{10} - 2.5 \times 10^{11} \text{ cm}^{-2}$ and 11–29 nm, respectively. As shown in figure 3(e), the intra-valley scattering length L_* (which is theoretically independent of temperature) is 10–64 nm, which is comparable to the distance between the dopants on the SiC surface. Therefore, as one of the possible explanations, we infer that the nitrogen dopants in SiC substrate act as the main scatterers for the intra-valley scattering.

Then, let us discuss the inter-valley scattering time τ_i of (monolayer and bilayer) graphene grown on SiC. In our case, it is much smaller than that of exfoliated graphene [12]. This

Table 1. Comparison of scattering times, τ_φ , τ_i and τ_* at 3 K, among monolayer and bilayer graphene prepared by exfoliation or on SiC substrate.

| | Monolayer (exfoliated) [12] | Bilayer (exfoliated) [12] | Monolayer (SiC) [13] | Bilayer (SiC) (present study) |
|---|-----------------------------|---------------------------|----------------------|-------------------------------|
| Carrier density (10^{12} cm^{-2}) | 1.5 | 1.5 | 0.4 | 5.8 |
| τ_φ (ps) | 12 | 34 | 5.3 | 2.1 |
| τ_i (ps) | 18 | 47 | 1.0 | 0.55 |
| τ_* (ps) | 0.08 | 3 | 0.01 | 0.004 |

means that graphene grown on SiC has more defects than the exfoliated graphene. Such defects may produce strong potentials enough to induce the inter-valley scattering [12].

On the other aspect about the intra-valley scattering and inter-valley scattering, we also need to consider the carrier density in each sample because scattering events are proportional to the carrier density in accordance with Fermi's golden rule [37]. In table 1, bilayer graphene on SiC naturally has the carrier doping due to the charge transfer from the buffer layer, which is larger than that of the exfoliated graphene. This relation can explain that the intra and inter-valley scattering times of bilayer graphene on SiC are smaller in table 1.

Finally, we comment on the stacking structure of S_3 by comparing with *ex situ* measurements of transport of pristine bilayer graphene and Li-desorbed bilayer graphene under high magnetic field [38]. Our previous Shubnikov–de Haas (SdH) study has revealed that the Berry phase is nearly π indicative of the AA stacking after desorbing intercalated Li atoms, while pristine bilayer graphene has nearly 2π -Berry phase. This is not fully consistent with the present study in which the W(A)L analysis shows that the Berry phase of Li-desorbed bilayer graphene is neither π nor 2π , suggesting a mixture of several different stacking structures of AB, AA, and twisted stacking structures [39]. The SdH oscillations are in general more contributed by domains having higher carrier mobility and lower carrier density, while the W(A)L phenomena show a averaged property of all domains. Such a difference should be considered when interpreting the experimental results with different methods. It would be necessary to clarify the microscopic structure by future experiments.

4. Summary

We performed *in situ* electrical transport measurements for pristine bilayer graphene grown on SiC(0001), Li-intercalated bilayer graphene, and that after desorbing the intercalated Li atoms. In all the three samples, we observed the increase of conductance at low temperatures upon applying the surface-normal magnetic field, which indicates the WL of carriers. By analyzing the data with the extended HLN equation, we have revealed the followings; (i) the magnetoconductance of pristine bilayer graphene is explained well by the AB stacking model, (ii) the phase breaking scattering is dominantly caused by the electron–electron scattering in a low temperature region (<15 K), (iii) dopants in the SiC substrate may cause a frequent intra-valley scattering in pristine bilayer graphene, and (iv) the Li-intercalation/

desorption process produces additional scatterers and modifies the stacking structure. To investigate the irreversible structural change upon Li-treatments, further studies with a high spatial resolution such as scanning tunneling microscope are desired.

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