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Preparation and Characterisation of Exfoliated Graphene for Quantum Resistance Metrology

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Abstract

Exfoliated graphene samples have been prepared for use in quantum resistance metrology. Good progress is recently made in achieving contact resistances to graphene of less than 50 Ω. Details are presented on the handling and measurement of graphene samples.

Introduction

In recent years, graphene is one of the leading scientific topics in research on two-dimensional electron gas (2DEG) transport. What sets graphene apart from other 2DEG systems is that the electrons in this one-atom thick layer of carbon atoms behave relativistic and follow the Dirac-equation [1].

Graphene is not only interesting from a fundamental point of view, but also can be applied as a quantum resistance standard [2],[3]. Unlike Si-mosfet ant AlGaAs 2DEG devices used so far as quantum resistance standards, graphene also shows the quantum Hall effect at *high* temperatures, up to room temperature [4]. Although the accuracy at this elevated temperature is insufficient for metrological applications, still a good quantisation at 4 K [3] already greatly facilitates the industrial applicability of graphene as quantum resistance standard.

In this paper we present our experience in preparation and handling of graphene samples, as well as measurement results of contact resistances.

Sample preparation

Graphene samples are obtained by cleavage of graphite on the surface of a Si/SiO_2-wafer. After cleavage, the best flakes (shape and size) are selected by optical microscopy [5] and AFM. Gold markers on the surface, separated by 14 μm, are used to align contacts made by e-beam lithography as accurately as possible to the graphene sample. The actual contacts are made by evaporation of Cr/Au.

Fig. 1 shows a typical flake with inner space of 20 x 10 μm^2. The contacts are made as large as possible, especially for the source and drain, in order to have a large overlap with the flake and thus a low contact resistance.

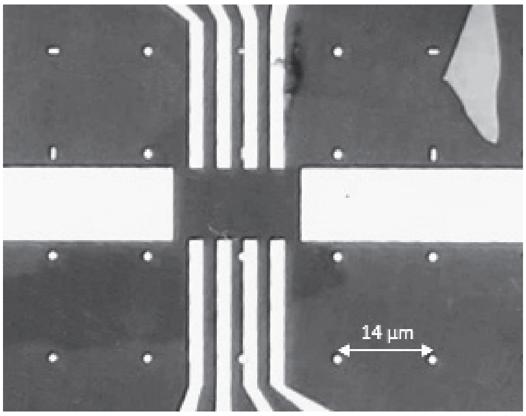

Fig. 1: Bilayer exfoliated graphene sample with Cr/Au contacts in a Hall bar geometry.

Sample handling

Graphene samples are extremely fragile and therefore a structured way of handling them once they have been fabricated is very important. As with metallic single electron devices, it is crucial to protect them against discharges and voltage changes. One of the critical operations is the mounting of samples in the measurement insert, so especially then all contacts and insert wiring must ground to avoid unintended discharging over the sample. Similarly, the use of low noise equipment and an LC-filter on the current wires is important during characterization and precision measurements.

After an initial basic test of the sample at room temperature, the sample is flushed with ^{4}He gas and subsequently annealed in vacuum in order to remove adsorbants from the sample surface that give rise to initial doping of the graphene layer. It is our experience that best metrological properties and most symmetric behavior for electron and hole doping are achieved for samples having their charge neutrality point near zero gate voltage. Our present annealing temperature of 170 °C, instead of the previous experimentally limited temperature of 115 °C, has

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improved the required annealing time with a factor of two, to typically a few hours.

It appears important to cool down the graphene samples slowly. At the HFML, samples are annealed while mounted in the tail of the vacuum-loading ^{3}He insert and the thermal mass of the insert assures slow changes in temperature. However, in the VSL top-loading ^{3}He insert special measures have to be taken to prevent too sudden temperature changes of the sample. Cooling down too quickly affects the contacts and can break the sample as shown in Fig. 2.

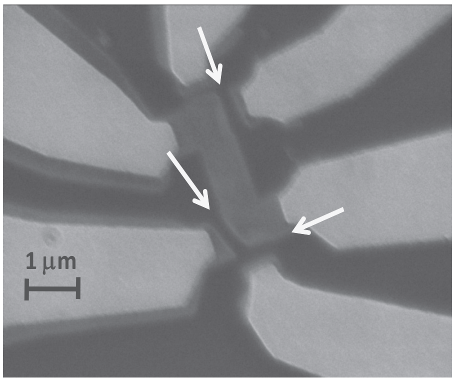

Fig. 2: Broken contacts (arrows) after too fast cool down of an exfoliated graphene sample.

Contact resistance

One of the major challenges in producing samples suitable for precision quantum Hall measurements is the realization of reliable, low ohmic contacts. Guidelines for Si MOSFET and conventional AlGaAs samples indicate that the contact resistance should be less than 100 Ω for quantum Hall resistance measurements at the level of a few ppb [6].

In our samples, we typically find a significant variation of contact resistances [2], measured in a 3-wire measurement in the middle of the quantum Hall plateau, ranging from 30 Ω to a few kΩ. Moreover, this resistance differs for electrons or holes. This is illustrated in Fig. 3 where the contact resistance of two current contacts in an exfoliated graphene sample is given. A dependence on the current is seen and the more high-ohmic contact does not behave entirely symmetric for positive and negative currents. There apparently is a non-ideal coupling between the Cr/Au metal contacts and the exfoliated graphene. Recently, contact resistances as low a 1.5 Ω have been obtained on graphene epitaxially grown on SiC [3], where likely surface preparation and the large contact area (10^4 μm^2) play a crucial role. Also one of our latest exfoliated samples showed good contact resistances of less than 50 Ω for all contacts, where each contact has approximately 10 μm^2 overlap with the graphene flake.

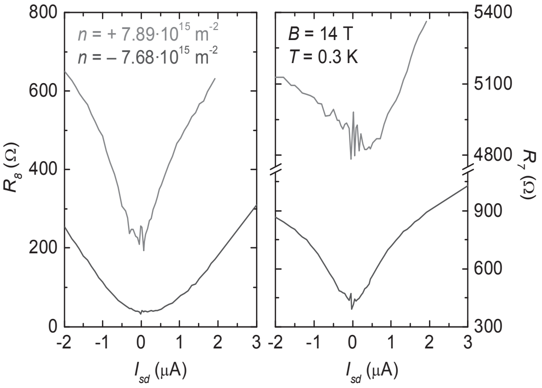

Fig. 3: Contact resistance of two current contacts of an exfoliated graphene sample for electron and hole doping (upper and lower curves respectively).

Outlook

Graphene offers useful perspectives for a quantum Hall resistance standard at elevated temperatures. However, the preparation of high quality samples suitable for metrological applications is not trivial. Especially making reliable, low ohmic contacts to the graphene still is a challenge. Present work is focused on further improvement of contact resistances, mainly by increasing the contact area and chemically cleaning the flake properly after each development step. This should allow more accurate precision measurements [2],[3].

References

[1] A.K. Geim et. al., The rise of graphene, Nature Materials **6**, 183-191 (2007)

[2] A.J.M. Giesbers et. al., Quantum resistance metrology in graphene, *Appl. Phys. Lett.* **93**, 222109 (2008).

[3] A. Tzalenchuk et. al., Towards a quantum resistance standard based on epitaxial graphene, Nature nanotechnology (2010).

[4] K.S. Novoselov et. al., Room-temperature quantum Hall effect in graphene, *Science* **315**, 1379 (2007).

[5] P. Blake et al., Making graphene visible, *Appl. Phys. Lett.* **91**, 063124 (2007).

[6] F. Delahaye and B. Jeckelmann, Revised technical guidelines for reliable dc measurements of the quantized Hall resistance, Metrologia **40**, 217 (2003).