

ConceptGraphene

New Electronics Concept: Wafer-Scale Epitaxial Graphene

Small or medium-scale focused research project

WP3 Charge transport devices

Deliverable 3.7 “Report on metrological measurements on the QHR standard, device #2”

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TABLE OF CONTENTS

Deliverable Summary	3
1. Introduction	5
1.1. Previous work	5
1.2 Fabrication	5
2. Metrological characterisation	6
2.1 Magnetotransport measurements	6
2.2. Resistance vs transport current.	7
2. 3. Coupling between the longitudinal and Hall resistance.	8
2.4. Contact resistance.	9
3. Conclusions.....	9
4. References.....	10

Deliverable Summary

A prototype device – a 100 Hall bar array for implementation of $R_K / 200 \approx 129$ Ohm standard – to be produced in the integration WP2. Metrological measurements aimed to resolve small deviations from the quantised value in any of the individual Hall bars thus characterising the scalability of the technology in a massively parallel fashion. The Hall bar arrays are much more than just a demonstrator – they would be extremely useful for metrology if implemented with necessary accuracy. They could be used for direct calibration of low-ohmic resistors in practical metrology.

1. Introduction

1.1. Previous work

The relevant previous deliverable reporting fabrication of large-scale arrays of Hall bars is Deliverable 2.3 “Report on Device Integration”. In that Report we summarized the technology for fabrication of graphene arrays with multiple layers of metallization. We concluded that at the time of writing and with the available magnetic fields it was not possible to reach the quantum Hall regime in the whole array due to high carrier density in the sample.

1.2 Fabrication

The quantum Hall resistance sample #2 was fabricated at Chalmers from a Linkoping film. The previously reported technology was used, but with adjustment on fabrication parameters to facilitate lower, controllable carrier density and low contact resistance.

Specifically:

- The device was examined using the optical technique described in ¹ and the placement of the array elements was optimised to exclude bilayer patches crossing the Hall bars.
- The thickness of the photochemical gate and the exposure to UV was optimised to decrease the carrier density, while maintaining uniformity.

One of the resulting 100-element arrays is shown in Figure 1.

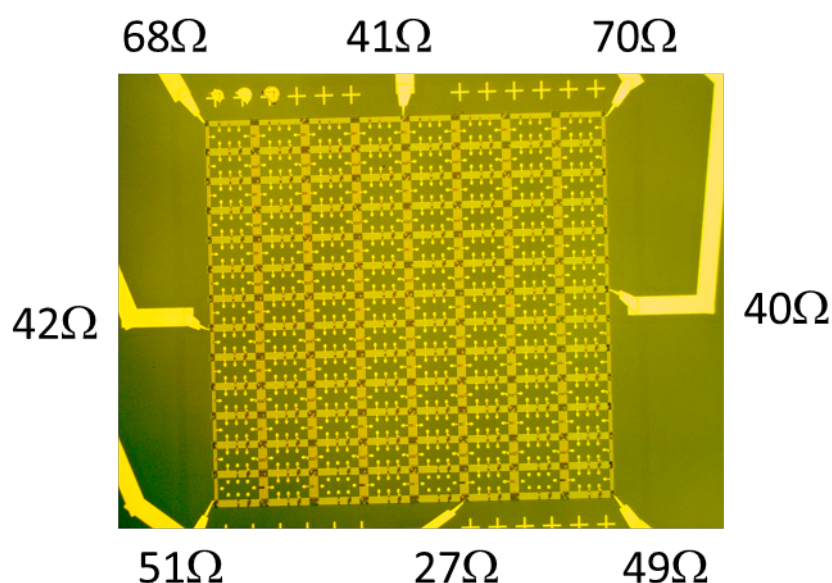


Figure 1. Optical micrograph of a hundred-element array.

Preliminary electrical characterisation was carried out at Chalmers immediately post-production. Other measurements were carried out at NPL in the setup described in Deliverable 3.3: Report on metrological measurements on the Prototype QHR standard, prototype #1. Overall three hundred-element arrays were measured in 5 separate experiments. One of the arrays was measured 3 times with UV exposure in between to adjust the carrier density.

2. Metrological characterisation

2.1 Magnetotransport measurements.

Measurements at Chalmers showed that the Hall resistance in 3 out of 4 arrays was quantised with the accuracy between 10^{-3} and 10^{-5} immediately post-production (Figure 2). This result encouraged us to begin the series of metrological measurements at NPL.

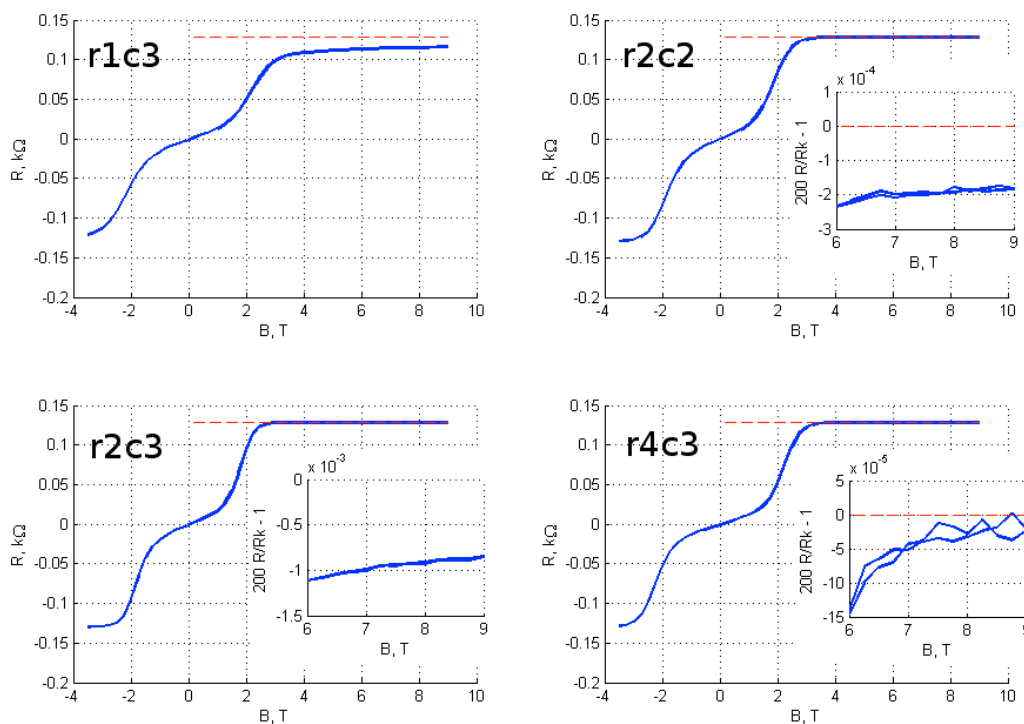


Figure 2. Hall resistance of four arrays immediately post-production. The dashed line is $R_k/200$.

On transfer of the samples to NPL we observed the carrier density in the array r4c3 to be significantly higher than that measured at Chalmers immediately post-production. The high carrier density translated in the high onset field of the Hall plateaux, so that quantisation conditions could not be met even at the highest available field of 14 T. Accordingly, the residual longitudinal resistance of 1.8Ω was measured at the maximum field of 14 T compared with 0 ± 10^{-5} measured at Chalmers (Figure 1).

It was decided to send the sample back to Chalmers to perform additional UV exposure reducing the carrier density. On return of the sample to NPL the measured carrier density was indeed considerably reduced resulting in wide plateaux in the Hall resistance beginning at a few tesla. However, the measured residual longitudinal resistance has increased to 7Ω (Figure 4).

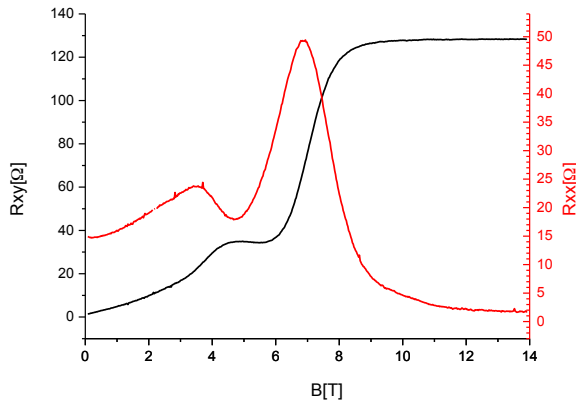


Figure 3. Hall and longitudinal resistance of the array r4c3 (round 1).

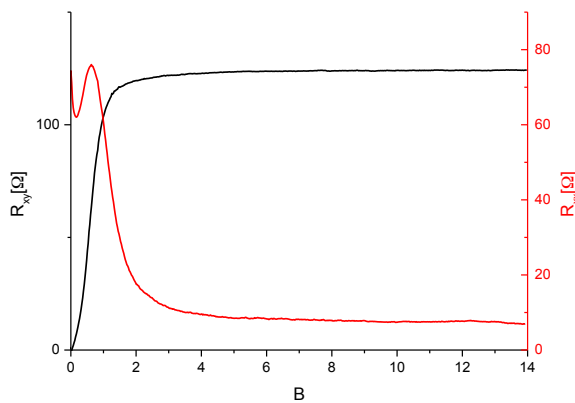


Figure 4. Hall and longitudinal resistance of the array r4c3 (round 2 - after additional UV exposure).

2.2. Resistance vs transport current.

In all cases we have observed a small gradual increase in the residual longitudinal resistance as a function of current without any indication of breakdown of any of the array elements up to several hundred microampere. Accordingly the Hall resistance showed a smooth proportional change. We concluded that the dependence is due to the heating effect of current.

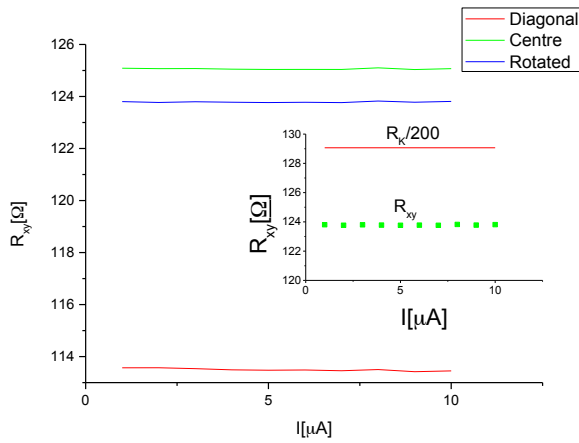


Figure 5. Hall resistance of different pairs of contacts. The diagonal pair has the largest longitudinal contribution. Dependence on current is weak.

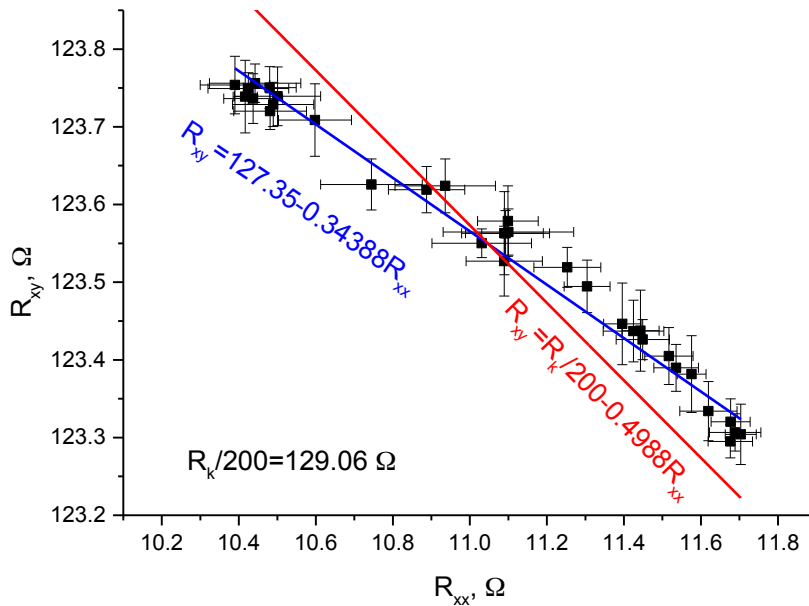


Figure 6. Coupling between the longitudinal resistance and Hall resistance.

2. 3. Coupling between the longitudinal and Hall resistance.

The coupling between R_{xx} and R_{xy} was studied on the same array after an additional thermal cycle. This thermal cycle resulted in a further increase in the residual resistance at maximum field up to about 10 Ω compared to the previous two measurements.

The coupling was measured by varying the temperature in the cryostat while simultaneously measuring R_{xx} and R_{xy} at 1 μA . Figure 6 shows the dependence of R_{xy} on R_{xx} . The error bars on the resistance values were determined from multiple multiple measurements at each temperature. The uncertainty largely results from temperature drifts.

It was previously observed that the deviation of R_{xy} from the quantised value linearly depends on R_{xx} for individual Hall bars in both graphene and conventional semiconductors². Assuming this holds for

the arrays, we linearly fit the experimental dependence. The slope of the fit $dR_{xy}/dR_{xx} = -0.34$ is similar to the values obtained in measurement on a single Hall bar³. Extrapolating the measured dependence to $R_{xx} = 0$, from the intercept we find that $R_k/200 - R_{xy} = 1.7 \Omega$. In other words, the array is not accurately quantised even in the limit of zero dissipation.

2.4. Contact resistance.

The contact resistances were measured in the maximum magnetic field, where R_{xx} reaches its smallest value. For instance, to evaluate the resistance of the drain contact (D in Figure 7), the measuring current (I_{DS}) is passed through contacts D and S and the potential difference between contacts D and, for instance, 1 is measured. The second contact used (here contact 1) is a contact at the same nominal potential (plus a small contribution $I_{SD}R_{xx}$ because of the non-zero resistance) as the contact under test (here D), taking into account the direction of the magnetic flux density vector B . This method provides a measurement of $R_L + R_C$ where R_L is the resistance of the lead attached to the contact under test and R_C the contact resistance.

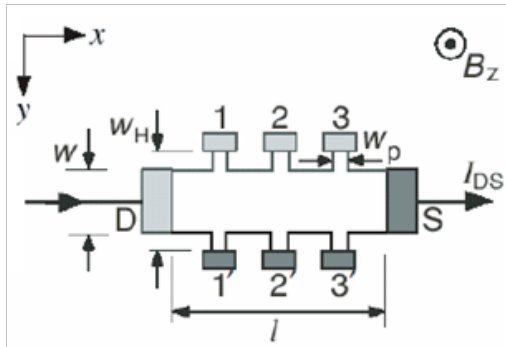


Figure 7. Device(array) with three pairs of Hall-voltage contacts. For the magnetic field pointing out of the sample in the z direction, the drain contact D and the Hall potential contacts 1–3 are at the same potential.

Contact resistance below 100Ω should contribute an error below 1 ppm on the quantisation of the array. In the case of the array, we can only assess the resistance of the external contacts and not all interconnects. The external contact resistances are shown in figure 1. All of them are below 100Ω . Assuming similar values in the interconnects the metrological requirements are narrowly satisfied.

3. Conclusions.

Graphene Hall bar arrays with 100 elements and multiple interconnects have been fabricated and characterised. This landmark shows the progress in technology achieved in the project. However, the quantisation accuracy necessary for metrological use of these arrays have not been achieved in a reproducible way, although accuracy of 10 ppm has been shown in the arrays immediately post-production. The 10 ppm level is array compares very well with that of GaAs arrays which have been under development for many years and still suffer from reliability issues, specifically in terms of contact resistances. The instability of arrays is probably related to the non-uniformity of the polymer bilayer used for photochemical gating. Such instability has not been seen in individual Hall bars. The contact resistance in the array was within the acceptable level, though smaller values would be desirable.

It is anticipated that arrays with larger physical dimensions would alleviate these problems, reducing the non-uniformity of the polymer layers and reducing the contact resistance.

4. References

¹ T. Yager et al., *Nano Lett.* **13**, 4217–4223 (2013)

² T.J.B.M. Janssen et al., *Rep. Prog. Phys.* **76**, 104501 (2013)

³ T.J.B.M. Janssen et al., *Metrologia* **49**, 294–306 (2012)