Transport of NIST Graphene Quantized Hall Devices and Comparison with AIST Gallium-Arsenide Quantized Hall Devices

Dean G. Jarrett[*], Takehiko Oe[†], Randolph E. Elmquist[*], Nobu-hisa Kaneko[†],
Albert F. Rigosi[*], Bi-Yi Wu[*,§,‡], Hsin-Yen Lee[*,⊥], Yanfei Yang[*,‡]

[*] National Institute of Standards and Technology, 100 Bureau Drive, Stop 8171, Gaithersburg, MD, 20899, USA
dean.jarrett@nist.gov

[†]National Metrology Institute of Japan, National Institute of Advanced Industrial Science and Technology,
Tsukuba, Ibaraki, 305-8563, Japan

[‡]University of Maryland, Joint Quantum Institute, College Park, MD, 20742, USA

[§]Graduate Institute of Applied Physics, National Taiwan University, #1 S.4, Roosevelt Road, Taipei 10617, Taiwan

[⊥]Theiss Research, La Jolla, CA 92037, United States

***Abstract* — Two graphene quantized Hall resistance (QHR) devices made at the National Institute of Standards and Technology (NIST) were hand carried to the National Institute for Advanced Industrial Science and Technology (AIST) and compared to a GaAs QHR device and a 100 Ω standard resistor. Measurement of the 100 Ω resistor with the graphene QHR devices agreed within 5 x 10^{-9} of the 100 Ω resistor GaAs measurements. After the initial measurements, the carrier density of the graphene devices was adjusted at AIST to restore the device properties to operate at low magnetic fields of 4 T to 6 T.**

***Index Terms* — quantized Hall resistance, graphene, cryogenic current comparator, standard resistor, carrier density.**

I. INTRODUCTION

The development of graphene-based quantized Hall resistance (QHR) standards [1] over the past decade has provided pathways for proliferation of QHR standards beyond National Metrology Institutes (NMI) and a few primary standards laboratories [2]. Robust graphene samples that are resistant to changes in carrier density are essential. The storage of devices in an inert gas (argon) environment at the National Institute of Standards and Technology (NIST) has been able to protect graphene devices from these shifts. We have been able to adjust the carrier density multiple times to devices exposed to air for extended periods of time and are pursuing techniques to prepare graphene samples that are not subject to shifts in the carrier density such as sealing of devices or chemically treating them.

A sealed chip header in a thirty-two pin package has been developed as one strategy and was considered for this comparison of NIST epitaxial graphene QHR devices with the National Institute for Advanced Industrial Science and Technology (AIST) gallium arsenide (GaAs) QHR devices. The AIST GaAs devices for this comparison were fabricated by Laboratoires d'Electronique Philips (LEP) [3] and mounted on TO-8 headers. The thirty-two pin package was not ready in time and would have required additional work to modify the probe connections at AIST. It was decided for this first comparison to use the TO-8 package for the graphene devices and hand-carry the samples in a small vacuum canister backfilled with argon. The transport canister consisted of a metal-glass-metal tube of diameter 2.54 cm and length of 7.62 cm with KF-25 flanges on the ends, metal end caps, clamps, and O-rings. The transport canister was a vacuum part and provided the necessary sealing to keep the argon gas in and ambient air out during transportation of two graphene QHR devices. The metal-glass-metal tube also provided a transparent window if necessary for inspection at customs or airport security screening without opening the transport canister.

Prior to preparing the two graphene QHR devices for travel, they were characterized at NIST and showed resistance quantization in the i=2 plateaus ($R_{K\text{-}90}/2$ = 12906.4035 Ω) at magnetic fields of 4 T to 6 T. After characterization, the graphene QHR devices and transport canister parts were placed in a glove box filled with argon gas for several hours to a day before being assembled inside the glove box. The assembled transport canister and graphene QHR devices were then hand-carried from NIST to AIST.

II. MEASUREMENT SYSTEM AND INITIAL GRAPHENE MEASUREMENT

Having arrived at AIST with the devices, they were stored in the sealed transport canister for a week until the cryostat could be prepared for measurement. Both graphene devices were placed in the dual sample probe shown in Fig. 1 for initial characterization. These devices were then cooled to about 0.5 K using a ^{3}He wet refrigerator with a 15 T / 17 T superconducting magnet in AIST for precision measurements. Magnetic fields up to 15 T were adequate for the graphene and GaAs devices.

978-1-5386-0974-3/18/$31.00 ©2018 IEEE

A cryogenic current comparator bridge, similar to the system described in reference [4], was used for these measurements. For the comparison of these devices via a 100 Ω standard resistor, the winding ratio of 2065:16 was used. For the 1:1 comparison of QHR devices, 2065:2065 was used. In these comparisons, the balanced voltages were about 0.27 V and 0.35 V, respectively.

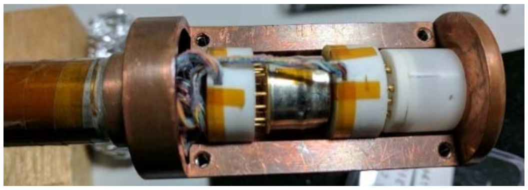

Fig. 1. Two QHR devices shown in the dual probe at AIST. One of the NIST graphene QHR devices is in the metal cap to the left and a AIST GaAs QHR device is in the plastic cap to the right. Both devices are mounted on TO-8 headers.

During the initial testing, the graphene devices were found to be functional but there was a change in the carrier density for both devices, requiring a higher magnetic field of at least 10 T for each device to be quantized. We believe that the devices may not have been in the argon filled glovebox for long enough prior to assembly of the transport canister. We were still able to use both devices and measured a 100 Ω resistor with a difference of 4.4 x 10^{-9} between the two measurements, which agreed with the predicted value for that resistor as shown in Fig 2, within the expanded uncertainty ($k = 2$) of 2.4 x 10^{-8}. The applied magnetic field was 12.0 T.

III. Graphene Adjustment and GaAs Measurement

The probe was removed from the cryostat and one of the graphene devices was replaced with an AIST GaAs QHR device as shown in Fig. 1. Both graphene QHR devices were briefly removed from the probe for inspection, after which a higher magnetic field of 12 T was required to quantize the graphene QHR device due to a second change in the carrier density. The direct comparison of the QHR in GaAs and graphene showed an absolute difference of 1.8 x 10^{-9} at 11.1 T. By using the procedure developed at NIST, the carrier concentration was lowered to restore the device properties that allow operation at lower magnetic fields [5]. The process involved exposing the samples to nitric acid vapors and then heating the device in vacuum while monitoring the longitudinal resistance R_{xx} until the value measured at NIST was reached. The procedure was applied to both devices, then the lower magnetic field in the $i = 2$ plateau of the two graphene QHR devices reduced to 5 T and 8 T, respectively. The measurement of the 100 Ω resistor with the graphene and GaAs devices in the same cryostat showed a difference of 4.3 x 10^{-9} for both devices as shown in Fig. 2, within the expanded uncertainty ($k = 2$) of 2.4 x 10^{-8}. The applied magnetic field was 10.2 T in both cases which was the center of the $i = 2$ plateau of GaAs device. The graphene device for this measurement (G19) had longitudinal resistances of 0.1 mΩ and 0.4 mΩ and contact resistances < 0.3 Ω, which were measured by the 3-terminal method.

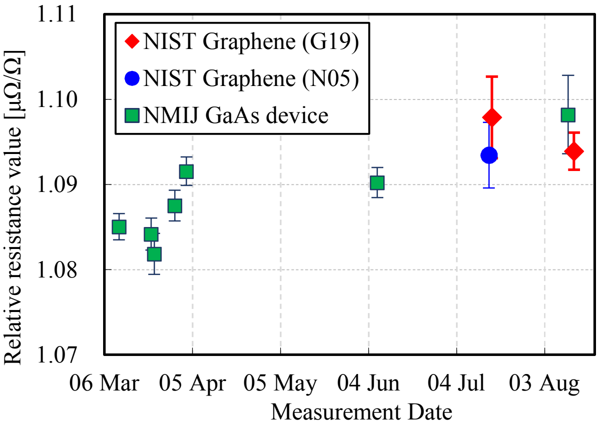

Fig. 2. First direct comparison of a 100 Ω standard resistor with two graphene QHR devices and one GaAs QHR device. Green square markers represent the historical and recent measurements using the AIST GaAs device as the standard. The red diamond and blue circle markers represent the measurements made by using the graphene QHR devices as standards. Error bars are the 1 σ standard deviation of the measurements.

IV. Conclusion

Graphene devices were transported and compared to GaAs QHR devices by measuring a 100 Ω standard resistor, with a difference of 4.3 x 10^{-9}, well within the expanded uncertainty ($k = 2$) of 2.4 x 10^{-8}. The experiment also allowed us to successfully adjust the graphene device carrier density at AIST by using the method developed at NIST. Future comparisons are planned with improvements to the transport process and to use a dry cryostat at AIST, with fewer time and liquid helium constraints in the future.

References

[1] Y. Yang, L.I. Huang, Y. Fukuyama, F.H. Liu, M.A. Real, P. Barbara, C.T. Liang, D.B. Newell, and R.E. Elmquist, “Low Carrier Density Epitaxial Graphene Devices On SiC” *Small*, Vol. 11, No. 1, pp. 90-95, 2015.

[2] F. Delahaye, T. J. Witt, R. E. Elmquist, and R. F. Dziuba, “Comparison of the quantum Hall effect resistance standards of the NIST and the BIPM,” *Metrologia*, Vol. 37, 173-176, 2000.

[3] F. Piquemal, G. Genevès, F. Delahaye, J.-P. André, J.-N. Patillon, and P. Frijlink, “Report on a Joint BIPM-EUROMET Project for the Fabrication of QHR Samples by the LEP,” *IEEE Trans. Instrum. Meas.*, Vol. 42, No. 2, pp. 264-268, April 1993.

[4] J. Kinoshita, K. Inagaki, C. Yamanouchi, K. Yoshihiro, S. Kawaji, N. Nagashima, N. Kikuchi, and J.-I. Wakabayashi, “Self-Balancing Resistance Ratio Bridge Using a Cryogenic Current Comparator,” *IEEE Trans. Instrum. Meas.*, Vol. 38, No. 2, pp. 290-292, April 1989.

[5] C. Chuang, Y. Yang, S. Pookpanratana, C. A. Hacker, C. Liang, R. E. Elmquist, “Chemical-doping-driven crossover from graphene to “ordinary metal” in epitaxial graphene grown on SiC,” *Nanoscale*, Vol. 9, pp. 11537-11544, 2017.