Non-invasive Microwave Method for Extended Electrical Measurements on Graphene

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Abstract

As graphene grows in significance for commercial applications it is becoming rather urgent to solve a number of problems associated with industrial scale-up. Most urgent is the requirement for graphene standardization methods, especially for electrical properties. A second missing element in the required developments towards large scale mass-production of high quality graphene is a quick and accurate method of quality control of the electrical properties of graphene which may be applied in, or close to, the graphene growth process. In this paper we discuss the need for graphene measurement standards while introducing a non-contact method using microwave resonance which we believe solves many of the problems of on-line production quality control.

The method which we are developing at NPL is based on a *microwave dielectric resonator* system. The graphene to be analysed is brought into the near-field region surrounding the dielectric and the perturbation of the resonator's centre frequency and linewidth are both measured. We describe the technique, estimate its accuracy and future developments. In essence it relies on three distinct factors. First, although graphene has a high 3D conductivity the 2D sheet resistance of graphene is comparable with the impedance of free space $((\mu_0/\varepsilon_0)^{1/2}$. Second, a monolayer or even a few-layer thick sample of graphene does not have a significant attenuation effect on electric fields which are parallel to its surface. Third, as a diamagnet, its relative permeability can be assumed to be close to unity.

We have shown that it is possible to convert this technique by a method of substitution, into an accurate and fast method for deriving sheet resistance (or equivalently, 2D conductivity) without the need for patterning or making contacts. We measure first the centre frequency and Q factor of the dielectric resonator on its own, then with a sample of graphene, on a non-conducting substrate, placed nearby and finally with an identical bare substrate in the same position. The presence of the graphene produces a change in Q value (see fig. 1).

These measurements are sufficient to provide accurate determination of the graphene sheet resistance [1]. The reproducibility of our measurements is at the level of a few percent. From comparison of the same measurements made with our microwave technique and conventional van der Pauw measurements on the same samples it is clear that the absolute accuracy is better than 10%. Table 1 indicates a comparison between the two methods. Agreement is generally very close but note the two methods sample the graphene with somewhat different weighting. We have compared measurement on a range of graphene samples grown by chemical vapour deposition (CVD), high temperature decomposition of SiC and reduction of graphene oxide, having a sheet resistance range of some four orders of magnitude.

In a recent development we have also demonstrated how we may use an extension of the same technique to measure the graphene mobility and carrier density without the need of electrical contacts. The NPL patented method [2] uses a different microwave mode from the one used for sheet resistance measurements and also requires the application of a modest d.c. magnetic field (~ 0.3T).

For a metal or semi-conductor enclosed within a microwave cavity, with purely diagonal conductivity tensor, the influence of the electric field vector at the conductor's surface will induce current flow which is parallel to the surface electric field. The main influence on the Q factor of the enclosing cavity is to reduce it, reflecting the additional Joule heating arising from the $\sigma.E$ local heating. In the presence of a d.c. magnetic field B the conductivity tensor takes on off-diagonal terms (σ is the surface conductivity and E is the local electric field). This leads to a small amount of additional dissipation (remember that generally $\sigma_{xy} << \sigma_{xx}$) but, more importantly from the point of view of this discussion, the electric field will induce a flow of current orthogonal to the other component and to itself. The orthogonal current pattern of flow over the surface will act as a *radiator* for the orthogonal degenerate microwave mode. Detection of the magnetic field dependent amplitude of the radiated power into this orthogonal mode allows determination of the mobility and carrier density. The details of this derivation will be presented in a later publication.

Previously microwave cavity methods have been used to determine the Hall coefficient and hence the mobility of small semiconducting samples. The underlying process is to use a high Q copper cavity (often an ESR spectrometer is used) in which a small semiconducting sample is included. Two advantages apply to our method. First, since we are looking at graphene samples the total volume is extremely small, even though the cross-sectional area may be comparable with that of the microwave resonator. Further, the large area makes calculation and calibration simpler and more accurate. Finally we use a dielectric microwave resonator to which the graphene sample is coupled. Thus there is no first order contribution form the resonator conductivity since it is very close to zero.

More details of recent experimental results from these two methods will be presented, including scanned data over a large area of CVD graphene transferred onto PET substrate which is 300mmx200mm in size.

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References

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Figures

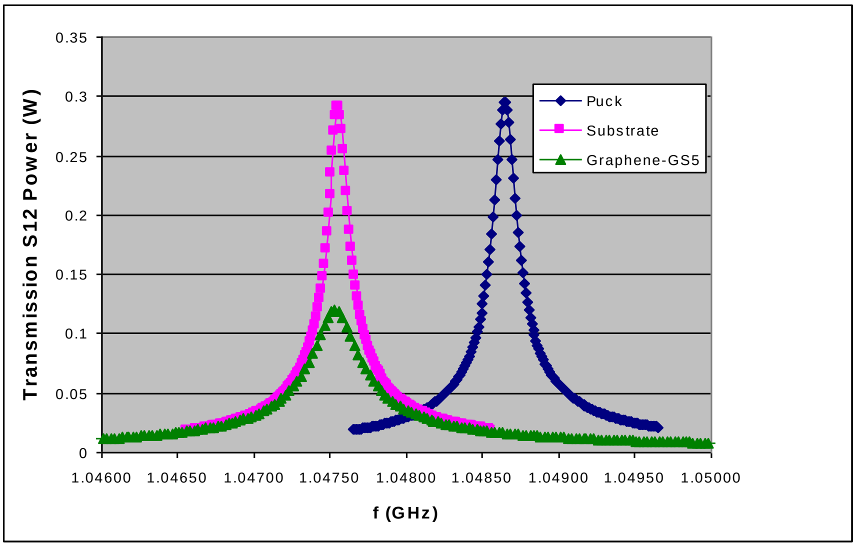

Fig.1 TE010 microwave resonance in a 12mm diameter single crystal sapphire resonator. Blues diamonds show response of empty resonator. Pink squares shows response with a bare quartz substrate. Green triangles when graphene coated substrate in same position. Note the large shift in frequency for both bare quartz and graphene coated quartz. But the linewidth only changes due to graphene.

Table 1. Summary of measurement results of 4 nominally identical monlayer CVD graphene samples.

Sample	Sheet resistance by microwave measurement (Ω)	Sheet resistance by van der Pauw measurement (Ω)
S12	1062.0±32	961.2±1.4
S21	518.0±6.0	546.1±0.9
S22	604.0±12.0	558.2±0.6
S32	788.3±4.6	610.2±0.6