Physica E 56 (2014) 431–434

Contents lists available at ScienceDirect

Physica E

journal homepage: www.elsevier.com/locate/physe

Spatially resolved electrical characterisation of graphene layers by an evanescent field microwave microscope

Andrew Gregory [a], Ling Hao [a,*], Norbert Klein [b], John Gallop [a], Cecilia Mattevi [b], Olena Shaforost [b], Kevin Lees [a], Bob Clarke [a]

[a] National Physical Laboratory, Hampton Road, Teddington, TW11 0LW, UK
[b] Imperial College London, South Kensington Campus, London, SW7 2AZ, UK

HIGHLIGHTS

- Scanning microwave microscope results on thin film graphene samples.
- Reduced graphene oxide on silica.
- Graphene grown by chemical vapour deposition, transferred to silica.

ARTICLE INFO

Article history:
Received 1 June 2012
Received in revised form
3 October 2012
Accepted 3 October 2012
Available online 11 October 2012

ABSTRACT

An evanescent field microwave microscope has been developed at the National Physical Laboratory. This instrument has multiple applications and has been developed to allow *traceable* measurements of local complex permittivity, unlike most other microwave scanning microscopes. In this paper we describe basic operation of the microscope and show measurements on graphene samples produced at Imperial College. The microscope obtains images by raster scanning of a wire probe in 'contact mode'. Of particular interest to the graphene community is the possibility of being able to scan over large areas (up to 4×4 mm^2), and to be able to measure actual values of surface resistance without a requirement for metal contacts. As an ultrathin semimetal, a graphene layer being placed in the evanescent field of the probe is expected to behave like a lossy dielectric material, its microwave loss tangent is proportional to its conductivity. Employing a high Q dual mode re-entrant cavity as host resonator and a spherical metal probe of 180 μm diameter, we found that spatial variations of the conductivity of graphene can be clearly resolved.

1. Introduction

The Near-Field Scanning Microwave Microscope (NSMM) is an instrument for measuring complex permittivity on micron scales. Several groups have published designs for NSMM systems [1–8]. Most are based on a microwave resonator that is coupled to a metal wire probe. Specimens placed at the tip of the probe causes a loading effect on the resonator and the complex permittivity of specimens is computed from measured shifts in *Q*-factor and resonant frequency. This paper describes an NSMM that has been developed at the National Physical Laboratory (NPL) for traceable measurement and outlines a promising application for characterising graphene samples on a micrometre scale.

* Corresponding author. Tel.: +44 2089436296.
E-mail address: Ling.hao@npl.co.uk (L. Hao).

2. Construction of the NSMM

The NPL NSMM allows the cavity resonator and wire probe assembly to be exchanged to enable use over a range of frequencies, and to allow different designs and sizes of probe to be used. For the results presented in this paper a dual mode re-entrant cavity is used, which allows measurements to be made nearly simultaneously at microwave frequencies differing by a factor of 10. The schematic of this cavity is shown in Fig. 1. In addition a dielectric resonator design may be employed, see Fig. 2, which also shows the overall layout of the NSMM. The advantage of using a dielectric resonator is that a high *Q*-factor is obtained, in this case up to 3000, which improves sensitivity, while a disadvantage is that there is normally only one suitable resonant mode, so operation at multiple frequencies is not usually possible. The resonator uses a dielectric puck of single-crystal yttrium aluminium garnet (YAG) inside a copper cavity. It resonates at approximately 3.8 GHz.

1386-9477/$ - see front matter Crown Copyright © 2012 Published by Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.physe.2012.10.006

The overall NSMM system includes scanning motors, approach control electronics, computer control and acoustic shielding of the entire system. In the measurements presented in this paper, the probe is made from tungsten wire 0.125 mm diameter and is positioned co-axially in a small hole in the metal cavity. The probe has a spherical tip (diameter 0.18 mm) that was formed by electro-discharge machining (EDM) [9]. EDM probe tips have minimal surface roughness, which is important in this application. It is desirable to use a spherical tip to be consistent with the published electrostatic model [1] of NSMM tip–specimen interaction. The dimensions of the cavity were chosen on the basis of an electromagnetic field simulation using Microwave Studio [10] to ensure that an adequate amount of energy could be coupled out of the resonator by the probe. A TM mode is used since this ensures that power can be coupled out of the top of the cavity. Axial symmetry has been preserved in this design in order to improve the calculability of the system. In the long-term this should allow a full-wave model to be established for modelling complex permittivity, in place of the electrostatic analysis that is used presently – work on such modelling is on-going. Further details of the microscope and full-wave model will be given in a future extended publication.

In order to obtain repeatable measurements, careful control of the separation between the probe tip and the specimen are required during the calibration process. In this work a tuning fork method based on a shear-force interaction [11,12] is used to provide probe–specimen separation control [13]. This makes use of a (nominally) 32.7 kHz quartz-crystal watch tuning fork

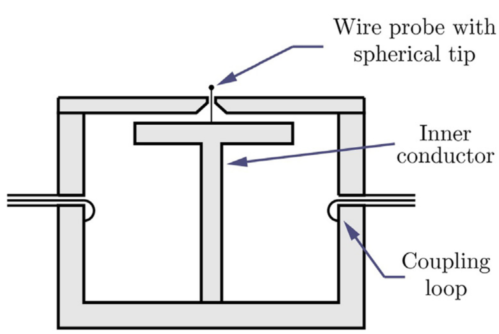

Fig. 1. Schematic of dual mode re-entrant cavity used in this work.

(IQD type XTAL002995). The wire probe is mechanically coupled to one prong (tine) of the tuning fork using a de-laminated strip of mica (approx. dimensions $2 \times 0.5 \times 0.03\ \text{mm}^3$). When the separation between the probe tip and the specimen is below approximately 50 nm, a lateral force is exerted, the origin of which is still open to debate [14,15]. This regime is conventionally referred to as ‘contact mode’. The force causes an increase in the frequency of the tuning fork resonance that can be detected as a change in impedance by means of a simple bridge circuit driven at a constant frequency [12]. The size of the observed change depends critically on the geometry of the tuning fork, mica strip and probe assembly. The optimum geometry was determined experimentally.

3. Experimental results on graphene

The properties of single- and few-layer graphene thin films are of great interest to a wide ranging community of researchers [16–19]. Several different growth methods have been developed already, some of which are applicable to wafer-scale film preparation. There remains considerable variability in the quality of films prepared, and even when identical methods are used the film properties between successive batches may be quite different. Here we report one of the first attempts (but see also [20,21]) to use microwave impedance measurements to analyse the properties of graphene thin films. The successful development of a non-contacting quality assessment method which also provides spatial resolution of electrical properties will have many applications. Graphene samples have a conductivity which ranges from close to that of a metal to values which are orders of magnitude lower, reflecting less perfect crystallinity and inadvertent doping by impurities and defects. The NSMM senses the permittivity of the area of the sample close to the probe wire tip and the imaginary component reflects the conductivity of the graphene sample. Simple dielectric theory predicts that the imaginary part of the graphene permittivity ϵ_g” is connected to the conductivity σ by the relationship

$$\epsilon''_g = \frac{\sigma}{2\pi f \epsilon_0}$$

where f is the microwave frequency at which the measurement is made and ϵ_0 is the permittivity of free space. Thus, by measuring

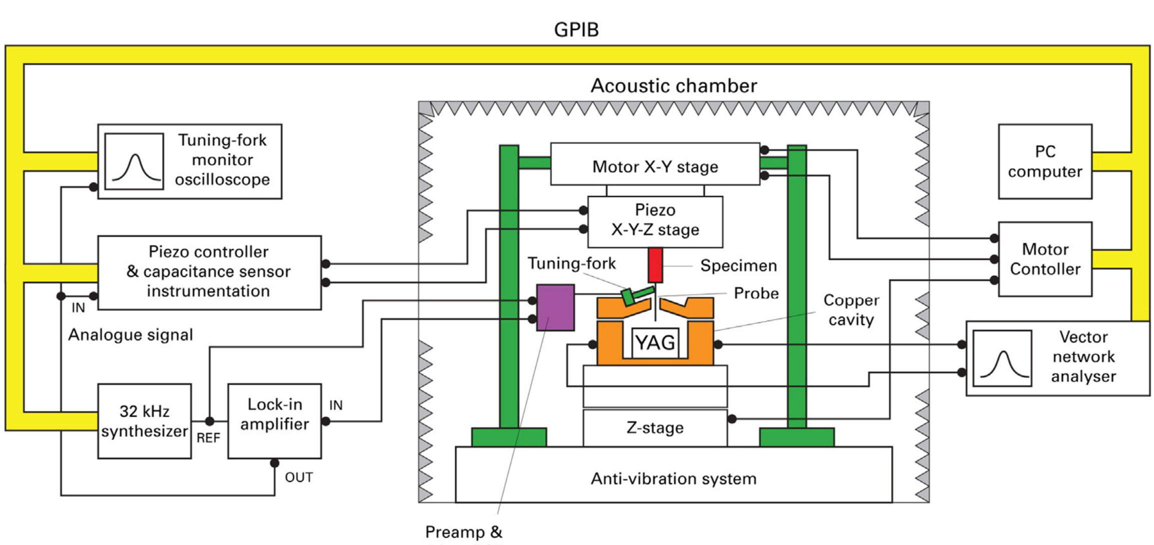

Fig. 2. Overall view of NSMM system showing the alternative dielectric resonator in place within the entire system.