ConceptGraphene

New Electronics Concept: Wafer-Scale Epitaxial Graphene

Small or medium-scale focused research project

WP2 Characterization and Integration

Deliverable 2.4 “Mapping of properties of a large (50 mm) wafer of graphene”

Main Authors:

R. Yakimova, V. Darakchieva, A. Tzalenchuk, S. Kubatkin, T. Löfwander

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LIST OF CONTRIBUTORS

Partner	Acronym	Laboratory Name	Name of the contact
1(coordinator)	CTHA	Chalmers Tekniska Hoegskola AB	Tomas Löfwander
3	NPLML	NPL Management Limited	Alexander Tzalenchuk
6	LIU	Linkopings universitet	Rositza Yakimova
8	TUC	TU Chemnitz	Thomas Seyller

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Deliverable Summary

We have applied large-scale spectroscopic ellipsometry mapping with a spot of 30μm which allows us to correlate film thickness, the parameters of the main transition energy associated with a van-Hove singularity in the density of states, free-charge carrier scattering and interface layer properties of epitaxial graphene. With this technique, a thickness map on a coarse grain scale can be made of a large area wafer of 50 mm diameter. Results are compared with LEEM images. Major parts of this was already reported in Deliverable report D1.3, but is repeated here for completeness of this report on mapping.

We have made electrical measurements by utilizing a dielectric resonator technique and compared different materials delivered from Linköping and Erlangen/Chemnitz. With this technique we can get an express measurement of the surface resistance of the wafer.

By scanning probe microscopy we can go into a small area (10x10 μm^2) and locally study the thickness inhomogeneity.

Through our first measurements, being a combination of these techniques, we can study the thickness inhomogeneity of wafers from the mm scale down to the sub-micron scale. The final goal is to find a suitable express protocol for characterization of graphene wafer quality.

1. Large scale mapping with ellipsometry

Spectroscopic ellipsometry mapping from 1.25 eV up to 5.45 eV was performed with an M2000 rotating compensator ellipsometer from J. A. Woollam Co. The measurements are carried out with a microspot of ~30 μm on a circular area with a diameter of 5 mm or on the whole sample size. The SE data are analyzed using dialect function model accounting for the electronic and free-carrier contributions [1]. The samples were modeled as consisting of a substrate, interface layer, graphene and surface roughness layer (Fig.8). In the view of the 3C substrate promises, graphene grown on such substrates is presented here.

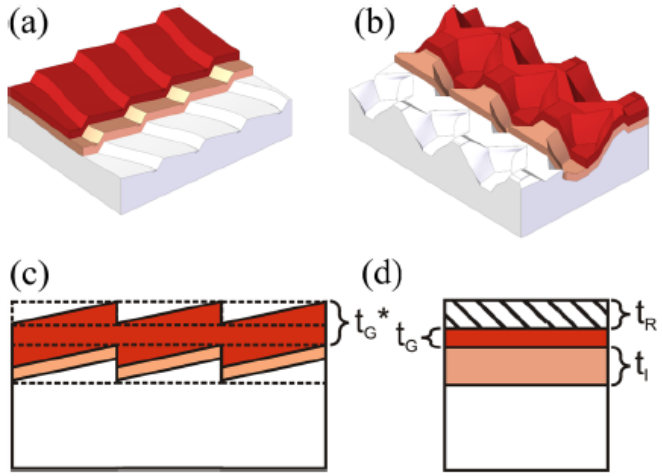

Figure 1 : Illustration of typical EG grown on Si- (a) and C-face (b) of SiC substrates. (c) illustrates a cross-section of EG/substrate showing typical surface steps. Dotted lines indicate the parallel planes assumed by the ellipsometer. The thickness t_G^* is the true thickness of graphene. (d) illustration of the final optical model with effective ellipsometer thickness parameters.

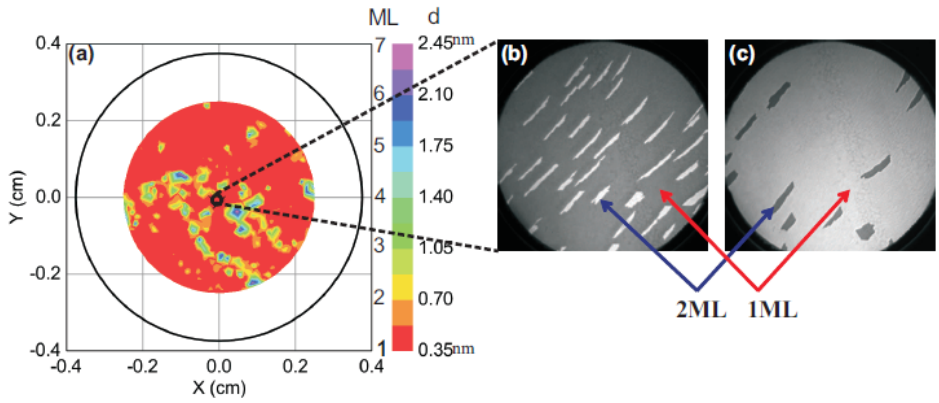

Figure 2 : (a) EG thickness map from SE, (b) and (c) low-energy electron microscopy images. The EG is grown on the Si-face of a polished 3C-SiC(111) substrate.

The SE data analysis renders important information on the parameters of the main van Hove singularity transition at ~ 4.5 eV, the carrier scattering time and interface layer properties. We find that the amplitude of the main transition decreases and the energy position is blue-shifted with decreasing the number of MLs, which is in agreement with our recent ultraviolet to vacuum ultraviolet SE study using macroscopic beam spot [*A. Boosalis, T. Hofmann, V. Darakchieva, R. Yakimova and M. Schuber, APL* ***101****, 011912 (2012)*].

The maps (not shown here) further reveal that low scattering time corresponds to the thicker islands. The reduction of scattering time indicates lower mobility across these areas, while nice 1 to 2 ML graphene surrounding it has high scattering times (high mobilities) and good graphene-like band energy.

We have recently shown that the formation of larger area monolayer graphene stronly depends on the substrate restructuring produced by energetically driven process of step bunching. This is characteristic of vicinal surfaces and takes place during graphene growth due to the unintentional miscut of the substrates and/or to surface rougness. As a result of this study it was shown that different SiC polytypes require tuning of graphene growth conditions [*G. Reza Yazdi, Remigijus Vasiliauskas , Tihomir Iakimov , Alexei Zakharov , Mikael Syva¨ ja¨ rvi , Rositza Yakimova, Growth of large area monolayer graphene on 3C-SiC and a comparison with other SiC polytypes, Carbon (2013) in press*].

2. Express control of surface resistance

The surface resistance of a whole graphene sample can be measured by using a contactless microwave technique, where the properties of a high-Q dielectric resonator are perturbed by the presence of a graphene film. The presence of the graphene perturbs both the centre frequency and the losses of the resonator. The measured data may be interpreted in terms of the real and imaginary components of the permittivity, and by calculation, the conductivity and sheet resistance of the graphene. The method has great sensitivity and dynamic range. The set-up is shown in Figure 3.

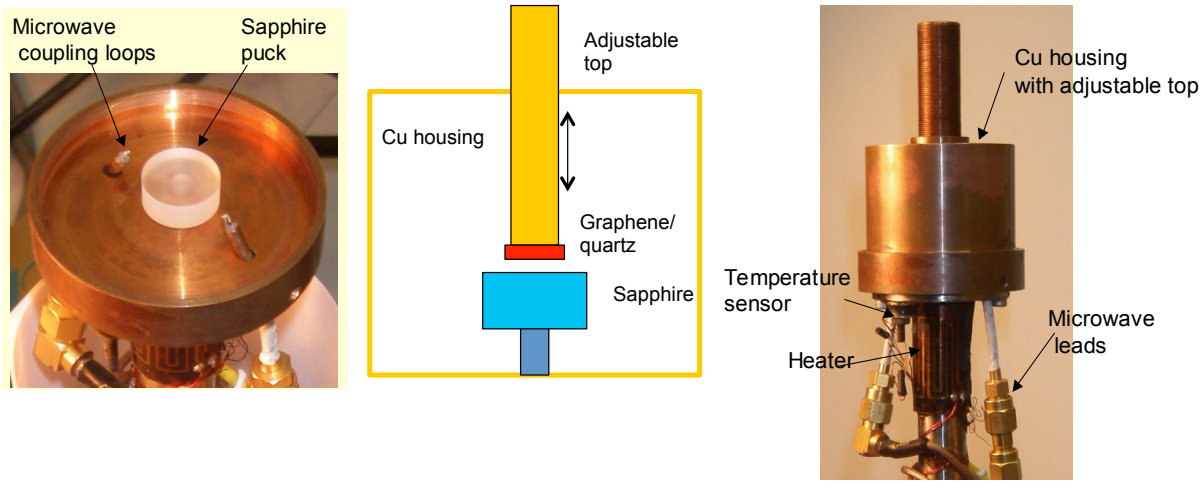

Figure 3 : Contactless microwave method to determine the surface resistance of graphene samples in production environment.

In Table 1 we show results of measurements at NPL, where two samples of SiC graphene from Linköping were measured and compared to other graphene samples. It shows consistently better quality of the epitaxial material. The difference in the parameters of the two samples (#266 and 267) is explained by the processing history of the two samples, see section 4.

Table 1 : Surface resistance results.

Sample	Graphene thickness (nm)	f_0 (GHz)	Δf (MHz)	$\Delta w_g - \Delta w_s$ (MHz)	Conductivity σ (S/m)	Sheet resistance R_s ($\Omega/\square$)
1-layer reduced GO	0.4	10.5504	1.169	0.0243	4.82×10^4	48222
1-layer CVD	0.4	10.4596	140.7	10.91	1.92×10^5	13038
1-layer SiC (266)	0.4	10.5619	7.463	47.30	2.03×10^7	61.7
1-layer SiC (267)	0.4	10.5600	5.057	73.38	4.64×10^7	26.97

In Table 2 we show first results of a comparison of the materials from Linköping and Erlangen/ Chemnitz. In order to get the absolute values of conductivity for the samples from Erlangen/ Chemnitz we need to measure the influence of a representativebare substrate, the work is in progress.

Table 2 : Preliminary comparison of samples from Linkoping and Erlangen.

Sample	f0, GHz	Δf, MHz	Q		Comment
Linkoping 266	10.5619	7.463	1415	Low σ	Contaminated
Linkoping 267	10.5600	5.057	2089	High σ	Clean, low n
Erlangen N60	10.5653	2.320	4554	Highest σ	Clean, high n
Erlangen P6	10.5920	8.030	1319	Low σ	Hydrogenated

3. Scanning probe microscopy

Scanning probe microscopy has been utilized at NPL to simultaneously map the topography and the surface potential on the very fine scale (a few micron window). The experimental set-up is shown in Figure 4. The result, as shown in Figure 5, is that we can distinguish regions of graphene with different number of layers.

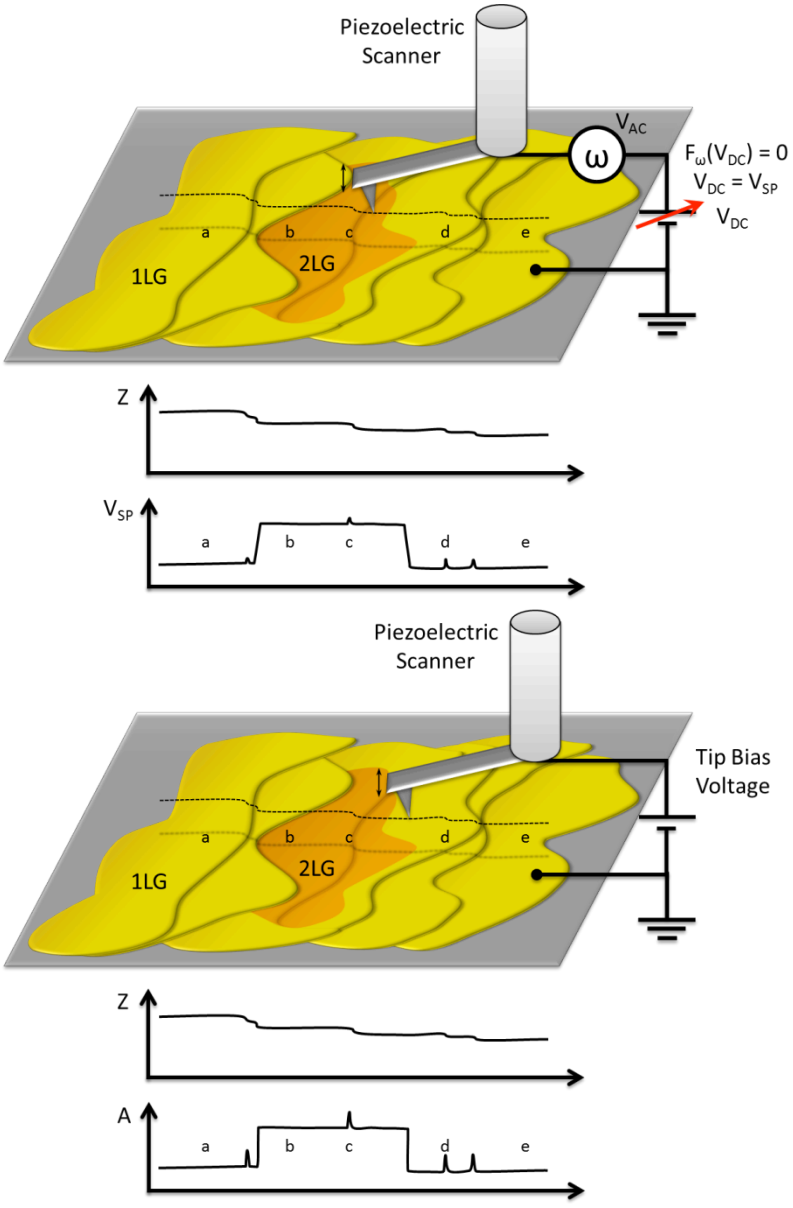

Figure 4 : Scanning probe microscopy. Top panel scanning Kelvin probe mode, lower panel electrostatic force mode.

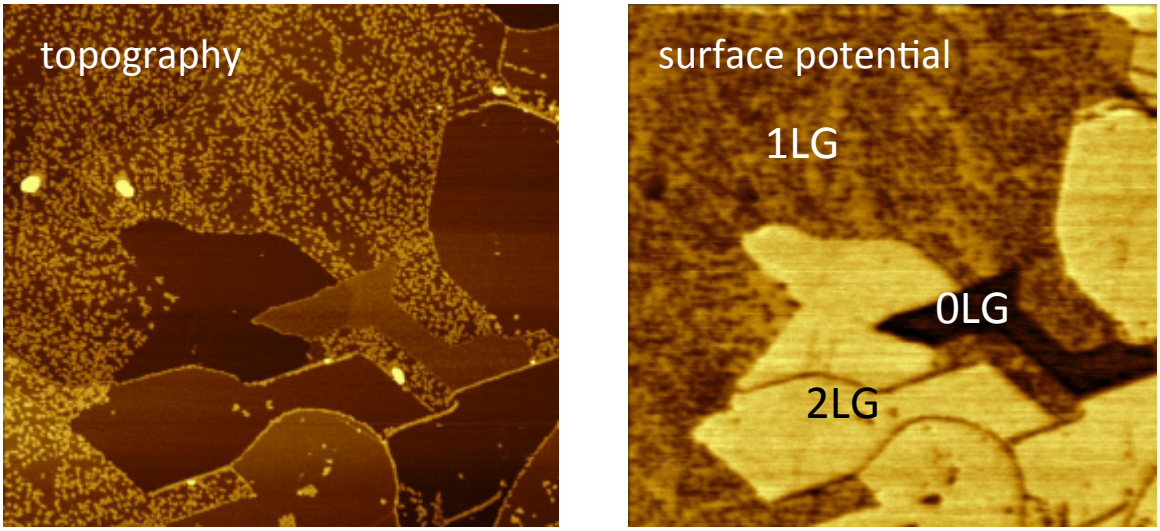

Figure 5 : Simultaneous measurements of topography and surface potential.

4. Influence of contamination

For studies of the residues of lithographic resist used in device fabrication, we have utilized secondary ion mass spectroscopy (SIMS) to compare unprocessed and processed graphene. The experiment shows that polymer indeed remains on the processed sample, which may degrade device performance. Oils, fatty acids or phthalates are found in both samples. In addition, Irgafos168 is found in both samples. We attribute this to the plastic box and the sticky tape holding the samples in place during travel between Linköping, Chalmers, and NPL. The recommendation is to use special Fluoroware ® boxes that will not contaminate the samples.

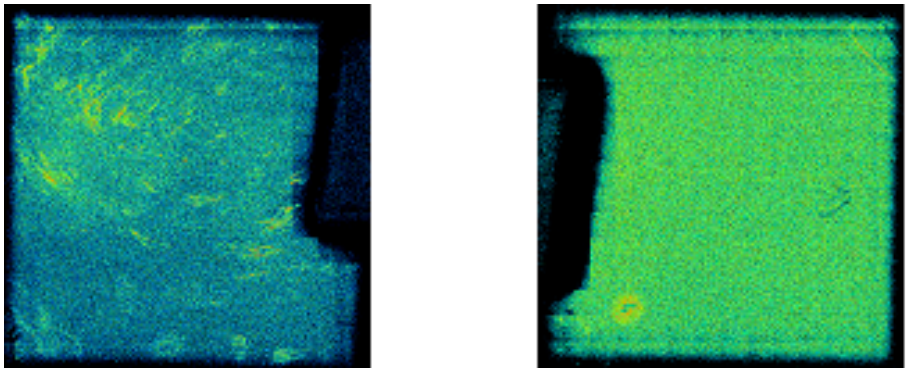

Figure 6 : Contamination maps of the two G-SiC samples (#266, processed, left and #267, untreated, right) obtained with SIMS.