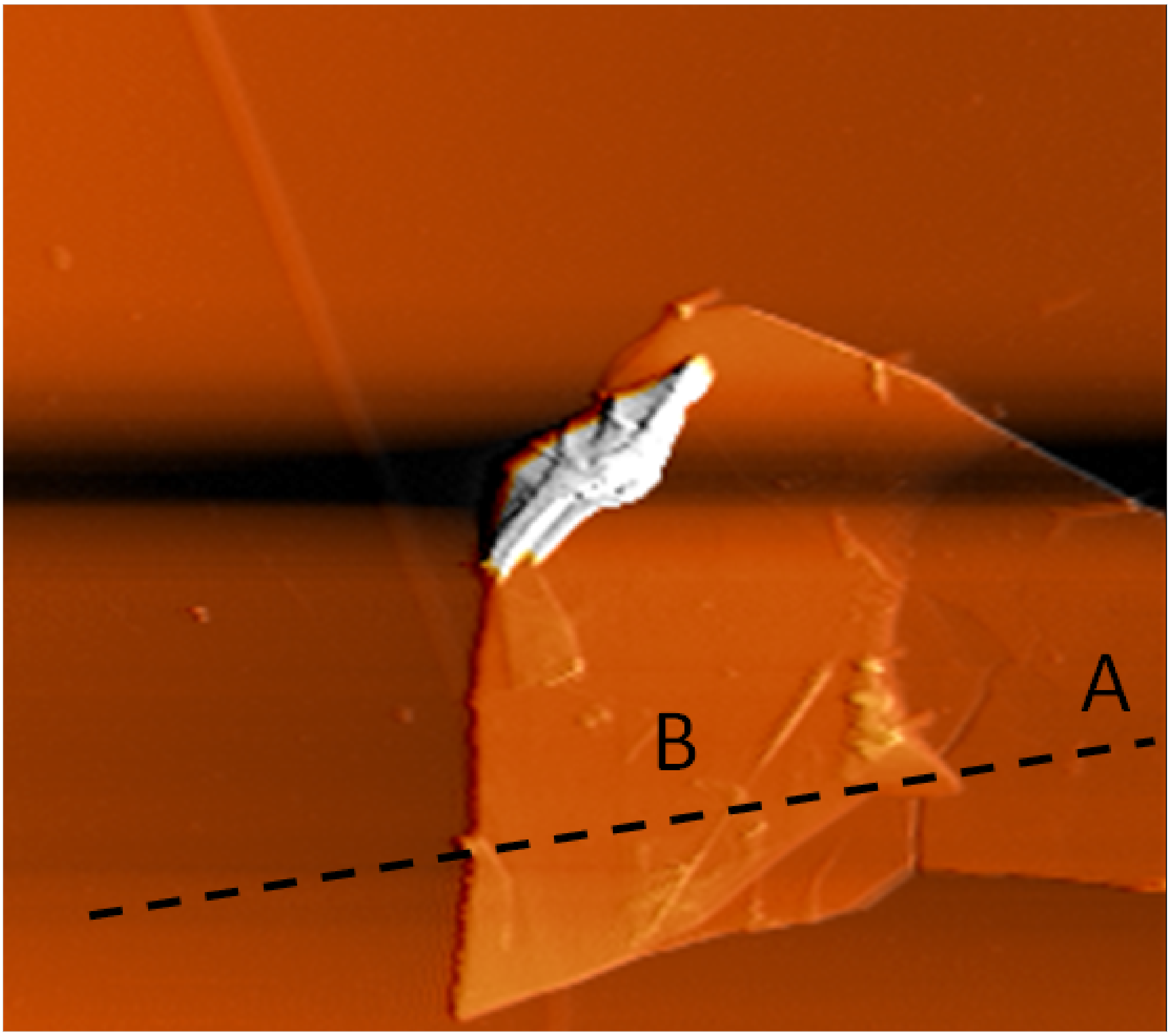

Graphene Characterization by Correlation of Scanning Electron, Atomic Force and Interference Contrast Microscopy

We make it visible.

Graphene Characterization by Correlation of Scanning Electron, Atomic Force and Interference Contrast Microscopy

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Sample: Stack of graphene layers on silicon wafer with native (2 nm) SiO_2

Instrumentation

Light Microscope:	Axio Imager.Z2m with TIC, Objective LD EC Epiplan-Neofluar 100x / 0,75 DIC, 5x camera converter, 550 nm filter, AxioCam HRc, Software: AxioVision with Commander, AutomeasurePlus, Shuttle&Find, and ConfoMap
SEM/AFM:	Merlin Compact with energy selective backscatter detector (In-Lens Duo) and AFM Option
Sample holder:	CorrMic Mat, to mark and recover a particular position with different microscopes (LM, EM and AFM)

Introducing

Graphene has high conductivity for electrons in the atomic plane, which makes graphene a promising candidate as a material for electronic circuits, i.e. field effect transistors (FET), and for MEMS[1]. Quality control in production of graphene devices requires characterization of the electronic properties, e.g. the lattice structure of graphene may be analyzed by transmission electron microscopy (TEM). EM and AFM are time consuming when looking for graphene flakes or for defects in graphene in large sample areas. Monochromatic bright field microscopy is suitable in fast finding tiny graphene flakes in large areas, if the graphene is on a thin optically resonant film[2,3,4] or if the graphene is on a conductive substrate, i.e. on native SiO_2-layer (typical 2 nm thickness) on silicon[5], which is investigated in the following. Interference contrast microscopy[6] measures the phase shift upon reflection on the sample. By means of the optical model, the phase shift can be converted into height or other optical parameters of the materials of layers and substrate.

A special realization of interference contrast is total interference contrast (TIC), which has been introduced in reflected light microscopy by Carl ZEISS in the year 2003. The relationship of predecessors of TIC and white light interferometer (WLI) of Michelson and Mireau type are discussed in Ref.[6]. Today’s WLI employs a mirror in the so called Mireau-objective to reflect a wave with reference phase shift. In TIC the function of the mirror is taken over by the sample surface next to the object to be profiled[5]. Consequently TIC has some advantages with respect to WLI: TIC does not require expensive Mireau-objectives; TIC can use standard microscope objectives, which offer higher numerical aperture and consequently higher lateral resolution; Mechanical stabilization of the interferometric paths is not required in TIC, but in WLI. A thickness profile across single and bilayers of graphene on 300 nm SiO_2 has been measured with 0.03 nm height resolution by TIC[5]. Moreover ref.[5] reports most of the following measurements.

Measurements

The layer-stack of graphene is clearly visible and consequently easily found in bright field (fig. 1 a). The visibility is similar with crossed circular polarizers (fig. 1 b). In bright field the thicker, the more the color of the graphene flake differs from the background color. In crossed circular polarization the brightness of the flake increases with the graphene layer thickness. Once the desired flake is found in the field of view of the light microscope, the position on the sample holder is stored by the software "shuttle & find". To relocate the same position with SEM / AFM, the sample holder is mounted in the vacuum chamber of the SEM. Pushing one software button, the desired flake is automatically moved into the field of view. By means of a suitable bias voltage on the filtering grid in front of the energy selective backscatter detector (In-Lens Duo), the height variation across the graphene layers (fig. 2) is resolved: Most backscattered electrons (BE) are generated within the silicon substrate and the oxide layer. The amount of those BE, which are lost in graphene, increases with the height of the graphene layers. AFM (fig. 3) measured the height of the layer stack in positions A: 9 nm and B: 13.5 nm in vacuum. The heights in A and B calibrate the height scale of the phase profile (fig. 4, measured by TIC along the dashed line). With an optical model describing graphene layers on 2 nm SiO_2 on silicon substrate, the complex refractive index N = 3.9+9.2i and the dielectric constant $\varepsilon = -67+72\iota$ of the graphene layers are achieved from the height calibration. Even the optical conductivity $\sigma(\omega) \equiv -\iota\omega\varepsilon(\omega)$ can be obtained from the dielectric constant for the optical frequency ω.

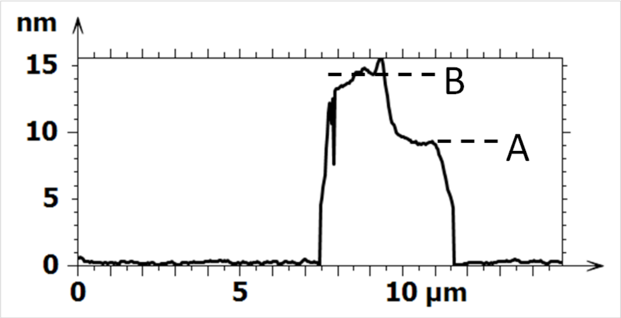

Figure 4 *interference contrast microscopy: phase profile along dashed line, converted into height of graphene stack*

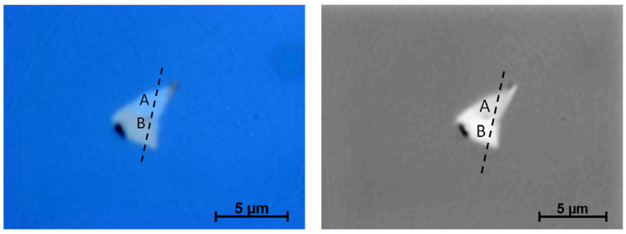

Figure 1 *Graphene layer stack observed in BF (a) and with crossed circular polarization (red color channel, b). Optical phase profile (fig. 4) measured along dashed line*

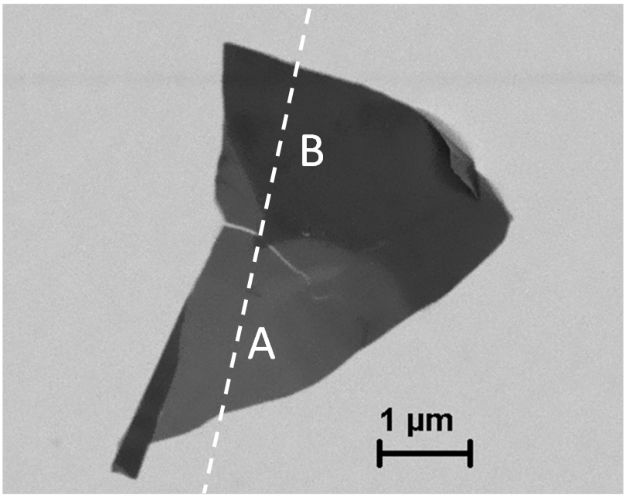

Figure 2 *SEM of graphene layer stack on conductive silicon with 2 nm native* SiO_2 *with In-lens Energy selective Backscatter-detector (EsB), EHT 1.5 kV, grid bias 1.2 kV, optical phase profile (fig. 4) measured along dashed line*

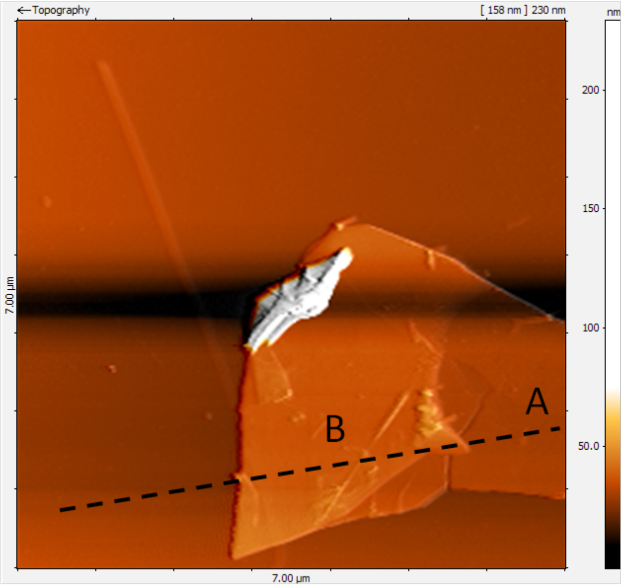

Figure 3 *height map by AFM*

Results

The refractive index and the conductivity of graphene on 2 nm SiO_2 is much different from suspended graphene layers and from graphene on 300 nm thick SiO_2 (with typical N=2.0+0.5i[7]). The reason for this difference is conductivity induced by the electric field of the substrate, silicon in our experiment. In other words: graphene has different optical and electronic properties dependent from the substrate. These properties are important for most applications of graphene. Consequently quality control of graphene devices must characterize thickness and opto-electronic properties of graphene. Correlative Microscopy of SEM / AFM and TIC is the solution. Moreover this method is applicable to study electronic properties of thin films in general without electric contacts.

Conclusion

With the correlation of SEM / AFM and light microscopy (bright field and total interference contrast) one easily localizes and measures height variations in graphene layers. This method allows to control the quality of graphene coatings by a measurement of refractive index, extinction, and conductivity on any, conductive or isolating, substrate.

References:

[1] A. H. Castro Neto, F. Guinea, N. M. R. Peres, K. S. Novoselov, and A. K. Geim, Rev. Mod. Phys. 81, 109 (2009)

[2] P. Blake, E. W. Hill, A. H. C. Neto, K. S. Novoselov, D. Jiang, R. Yang, T. J. Booth, and A. K. Geim, Appl. Phys. Lett. 91, 063124 (2007)

[3] Z. H. Ni, H. M. Wang, J. Kasim, H. M. Fan, T. Yu, Y. H. Wu, Y. P. Feng, and Z. X. Shen, Nano Lett. 7, 2758 (2007)

[4] Y. Y. Wang, R. X. Gao, Z. H. Ni, H. He, S. P. Guo, H. P. Yang, C. X. Cong, T. Yu, Nanotechnology 23, 495713 (2012)

[5] M. Vaupel, A. Dutschke, U. Wurstbauer, F. Hitzel, A. Pasupathy, Topography, complex refractive index, and conductivity of graphene layers measured by correlation of optical interference contrast, atomic force, and back scattered electron microscopy, J. Appl. Phys. 114, 183107 (2013)

[6] H. Beyer, Theorie und Praxis der Interferenzmikroskopie, Akademische Verlagsgesellschaft Geest & Portig K. – G., Leipzig, 1974

[7] U. Wurstbauer, C. Röling, U. Wurstbauer, W. Wegscheider, M. Vaupel, P.H. Thiesen, D. Weiss, Appl. Phys. Lett. 97, 231901 (2010)

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