

# THz Near-Field Nanoscopy of Graphene Layers

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**Abstract**— Far-field terahertz imaging is limited by diffraction to low resolutions in the 50  $\mu\text{m}$  range. On the other hand, near-field optical nanoscopy is a recent technique that shows permittivity contrasts at the nanoscale. We present here images of graphene layers on  $\text{SiO}_2$  obtained by scattering scanning near-field nanoscopy at 2.5 THz that show high contrasts.

## I. INTRODUCTION

Electromagnetic waves at THz frequencies exhibit strong interactions with matter, in particular with electrons and oscillating electric or magnetic dipoles: phonons, plasmons, molecular rotations or vibrations. Consequently THz waves can be used for material characterization, unfortunately diffraction limits the resolution to a few 10's  $\mu\text{m}$ . Characterization of free carriers in graphene thanks to THz would be particularly useful if it can be done at the nanoscale and without contact. Mid-infrared (MIR) and visible scanning probe near-field nanoscopy has been demonstrated [1,2] and extended to the THz range [3]. Graphene layers have been investigated in the MIR range using nanoscopy [4], here we demonstrate the first observation of a graphene layer at THz frequencies thanks to this technique.

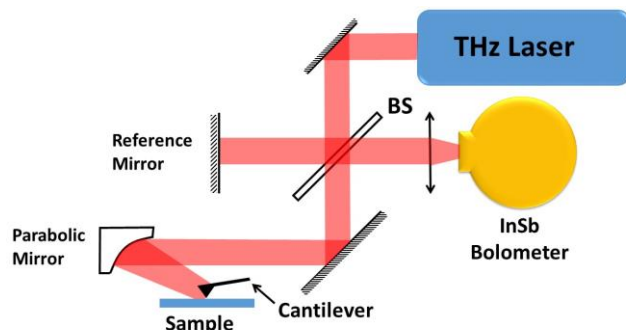


Fig. 1. THz s-SNOM optical set-up (BS: beam splitter).

Fig. 1 shows the experimental set-up. It is based on an atomic force microscope (AFM) coupled with an apertureless scanning near-field optical microscope (SNOM). The optical source is a  $\text{CO}_2$ -pumped THz gas laser generating the 2.5 THz line of  $\text{CH}_3\text{OH}$ . The beam is split into a reference arm and to a sample arm by a mylar beamsplitter (BS). In the sample arm, the beam is focused thanks to a parabolic mirror on a metallized silicon cantilever oscillating at  $\Omega = 250$  kHz. The scattered light is collected by the parabolic mirror, combined with the reflected beam of the reference arm and focused in an InSb hot electron bolometer. Demodulation is done at  $\Omega$  and harmonics of  $\Omega$ . The sample is scanned and AFM topography informations are simultaneously recorded with optical signals.

## II. SAMPLE

The sample is a monolayer of graphene grown by chemical vapor deposition (CVD) on a copper foil and transferred on an oxidized Si wafer with an improved wet chemical transfer process [5] (see Fig. 2). The  $\text{SiO}_2$  thickness is 300 nm.

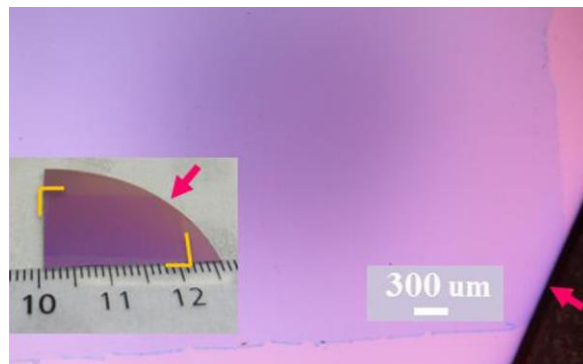


Fig. 2. Optical microscopy image of the graphene monolayer transferred on  $\text{SiO}_2/\text{Si}$  substrate (image of the full sample is shown in the inset).

The graphene layer was then locally patterned thanks to resist spin-coating, optical lithography and  $\text{O}_2$  plasma etching [5].

## III. RESULTS

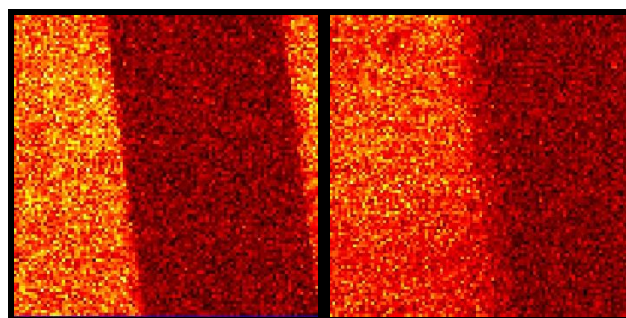


Fig. 3. SNOM images of the graphene layer at 2.5 THz (magnitude of second harmonic signal). Left image:  $20 \times 20 \mu\text{m}^2$  scan showing that a strip of graphene was etched (darker area). Right image:  $3 \times 3 \mu\text{m}^2$  zoom of the left area.

Fig. 3 shows SNOM images of a region where the graphene layer was etched. More intense  $2\Omega$  signals are detected on the graphene and less intense on  $\text{SiO}_2$  (darker central area visible on the left image). The right image shows a zoom of the transition between graphene and  $\text{SiO}_2$  and shows that the signal does not fall abruptly (transition region  $\approx 300$  nm).

We attribute this contrast to the high conductivity of the graphene layer. The sheet resistivity of this sample was measured by 4-probe experiments to be  $\sim 800 \Omega/\text{square}$ .

Comparisons with AFM topography images show that the gradual transition is not due to the radius of curvature of the tip (< 50 nm). A region of lower conductivity is perhaps present close to the etched region. Plasmon fringes have also been observed close to the edges of graphene microcrystals and grain boundaries in the MIR range [4].

In conclusion, we demonstrate that THz-SNOM can be used to image a monolayer of graphene at nanoscale resolution.

#### ACKNOWLEDGMENT

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#### REFERENCES

- [1] Y. De Wilde, F. Formanek, and L. Aigouy, "Apertureless near-field scanning optical microscope based on a quartz tuning fork," *Review of Scientific Instruments*, vol. 74, p. 3889, 2003.
- [2] F. Keilmann and R. Hillenbrand, "Near-field microscopy by elastic light scattering from a tip," *Phil. Trans. Roy. Soc. London*, vol. A362, p. 787, 2004.
- [3] A. J. Huber, F. Keilmann, J. Wittborn, J. Aizpura, and R. Hillenbrand, "Terahertz near-field nanoscopy of mobile carriers in single semiconductors nanodevices," *Nano Letters*, vol. 8, no. 11, p. 3766, 2008.
- [4] Z. Fei, A.S. Rodin, W. Gannett, S. Dai, W. Regan, M. Wagner, M.K. Liu, A.S. McLeod, G. Dominguez, M. Thiemens, Antonio H. Castro Neto, F. Keilmann, A. Zettl, R. Hillenbrand, M.M. Fogler, and D.N. Basov, "Electronic and plasmonic phenomena at graphene grain boundaries," *Nature Nanotechnology*, vol. 8, p. 821, 2013.
- [5] G. Deokar, J. Avila, I. Razado-Colambo, J.L. Codron, C. Boyaval, E. Galopin, M.C. Asensio, and D. Vignaud, "Towards high quality CVD graphene growth and transfer," *Carbon*, vol. 89, p. 82, 2015.