

# Near-field Imaging and Nano-FTIR Spectroscopy using Synchrotron Radiation from the Metrology Light Source (MLS)

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**Introduction:** Near-field optical microscopy (SNOM) is a versatile technique for imaging of nanoscale structures with a spatial resolution significantly below the diffraction limit. IR-SNOM measurements performed with laser sources provide spatially resolved information about the optical properties of the sample at discrete wavelengths. However, the spectra acquisition is time consuming since it requires repeated measurements at different wavelengths, the number (hence spectral resolution) of which is also limited. Thus near-field FTIR spectroscopy using broadband radiation such as from thermal sources or electron storage rings is desirable.

## Samples:

- \* E3: 6H-SiC (0001) oriented (Si-face), vanadium doped, semi-insulating
- \* G033, G068: H-etching of substrates like E3 [2] resulting in step-bunching; subsequently graphitized [3] (thermal decomposition), resulting in 60% monolayer coverage (n-type with densities  $>10^{12}\text{cm}^{-2}$ ), as well as some bi-layer formation
- \* D01: 6H-SiC, undoped

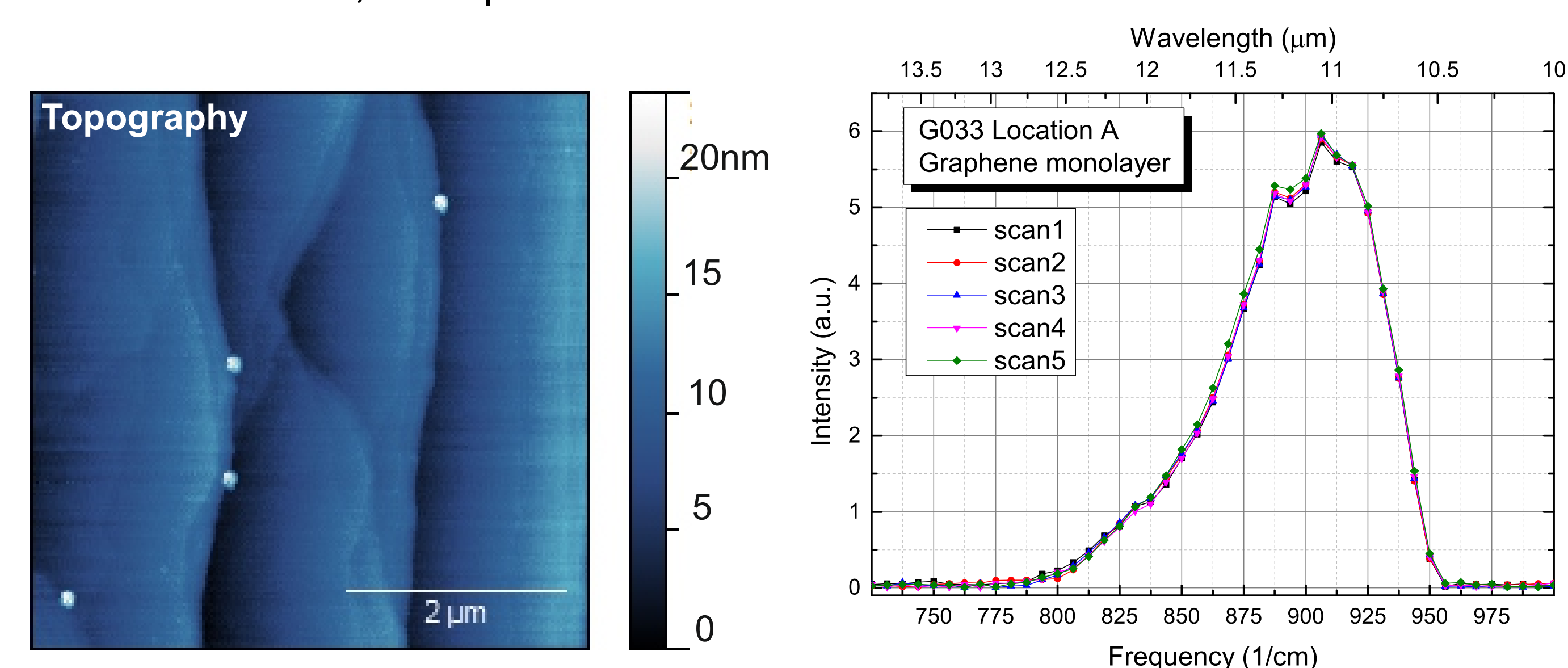


Fig 1: AFM image showing topography of G033 sample

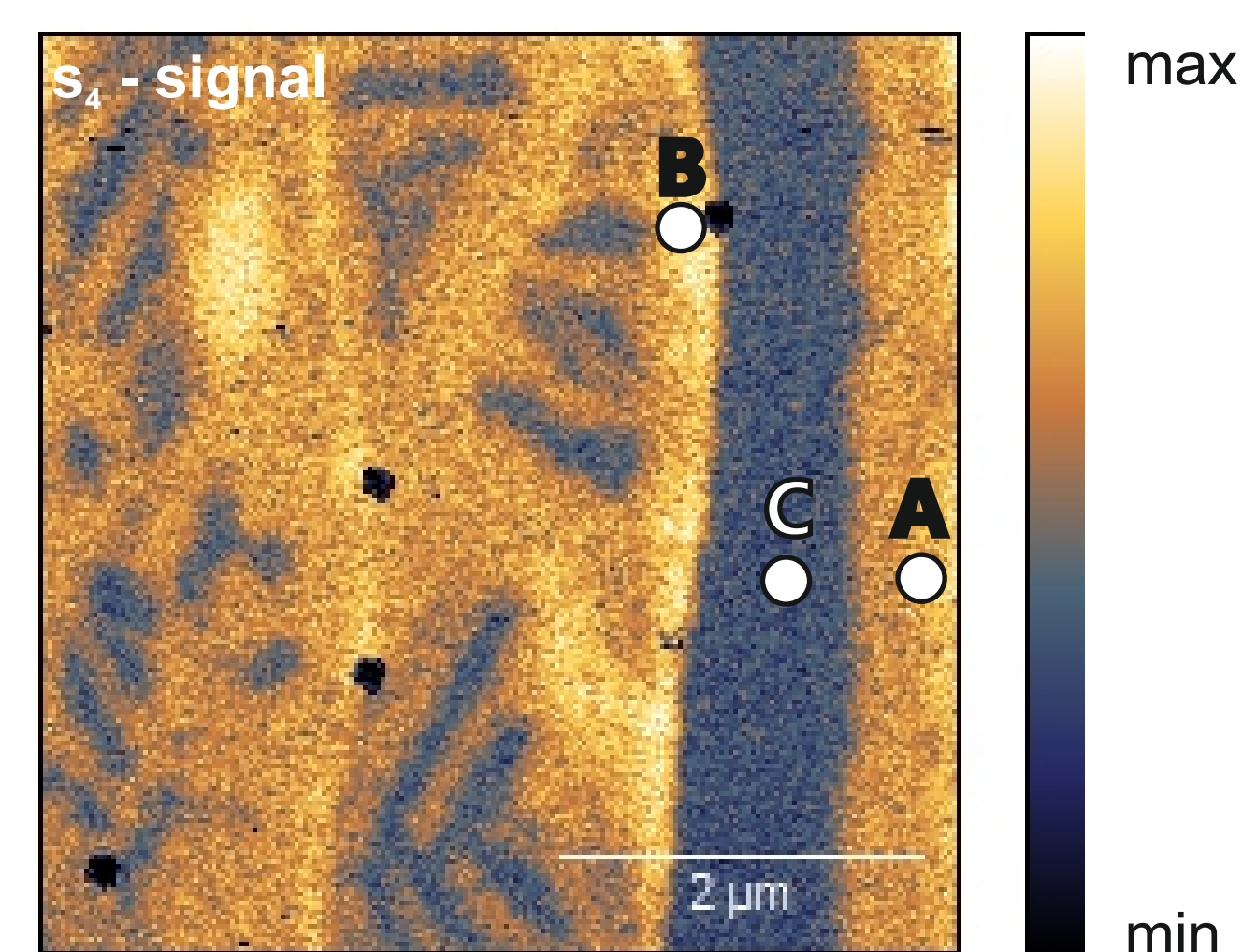


Fig 2: 4th harmonic signal intensity  $s_4$  corresponding to Fig 1 using broadband IR excitation; Location A and B corresponds to SiC covered by mono- and bilayers, respectively. C shows the uncovered SiC surface.

## Laser based near-field imaging

Imaging of G068 sample as function of laser wavelength using 4th harmonic signal intensity  $s_4$

- \* Depending on wavelength uncovered SiC (marked in Fig. 9), graphene or steppedges dominate
- \* Contrast inverses compared to broadband imaging (see also Fig. 7)

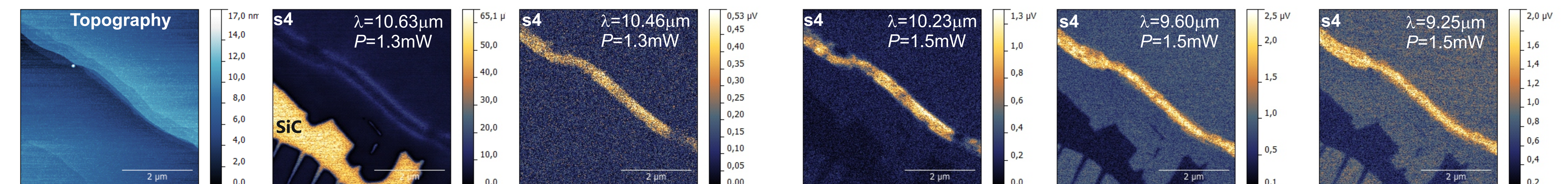


Fig 9 Corresponding AFM (a) and laser based SNOM images (b-f), recorded at different laser wavelengths; Laser power measured at interferometer entrance is indicated;

## References:

- [1] P. Hermann *et al.*, Optics Express **21**, 2913 (2013)  
[2] J. Robinson *et al.*, ACS Nano **4**, 153 (2009)

In this work we couple a scattering-type IR-SNOM to the IR-beamline at the MLS [1]. Compared to conventional thermal sources the use of synchrotron radiation provides several major advantages such as (i) a higher brilliance, (ii) pulsed radiation in the ps range, and (iii) polarized radiation. In the following we analyse graphitized 6H-SiC to demonstrate high-resolution imaging and nano-FTIR spectroscopy by using a broadband synchrotron radiation source, and compare to laser based near-field imaging at different wavelengths. We find significant differences in the contrast mechanisms and indications of strong plasmon reflections at SiC step-edges.

## Nano FTIR Measurements, using 4<sup>th</sup> harmonic:

- \* Enhanced emission of graphene wrt. pure SiC surface due to plasmon-phonon coupling [4]. Note opposite effect in laser imaging, see Fig. 7
- \* Spectra vary for doped and undoped SiC samples, Fig. 8
- \* Weak influence of graphitization (pure SiC region on G033) wrt. starting material (E3), see Fig. 8
- \* Almost no difference between bilayer and monolayer graphene, Fig. 8

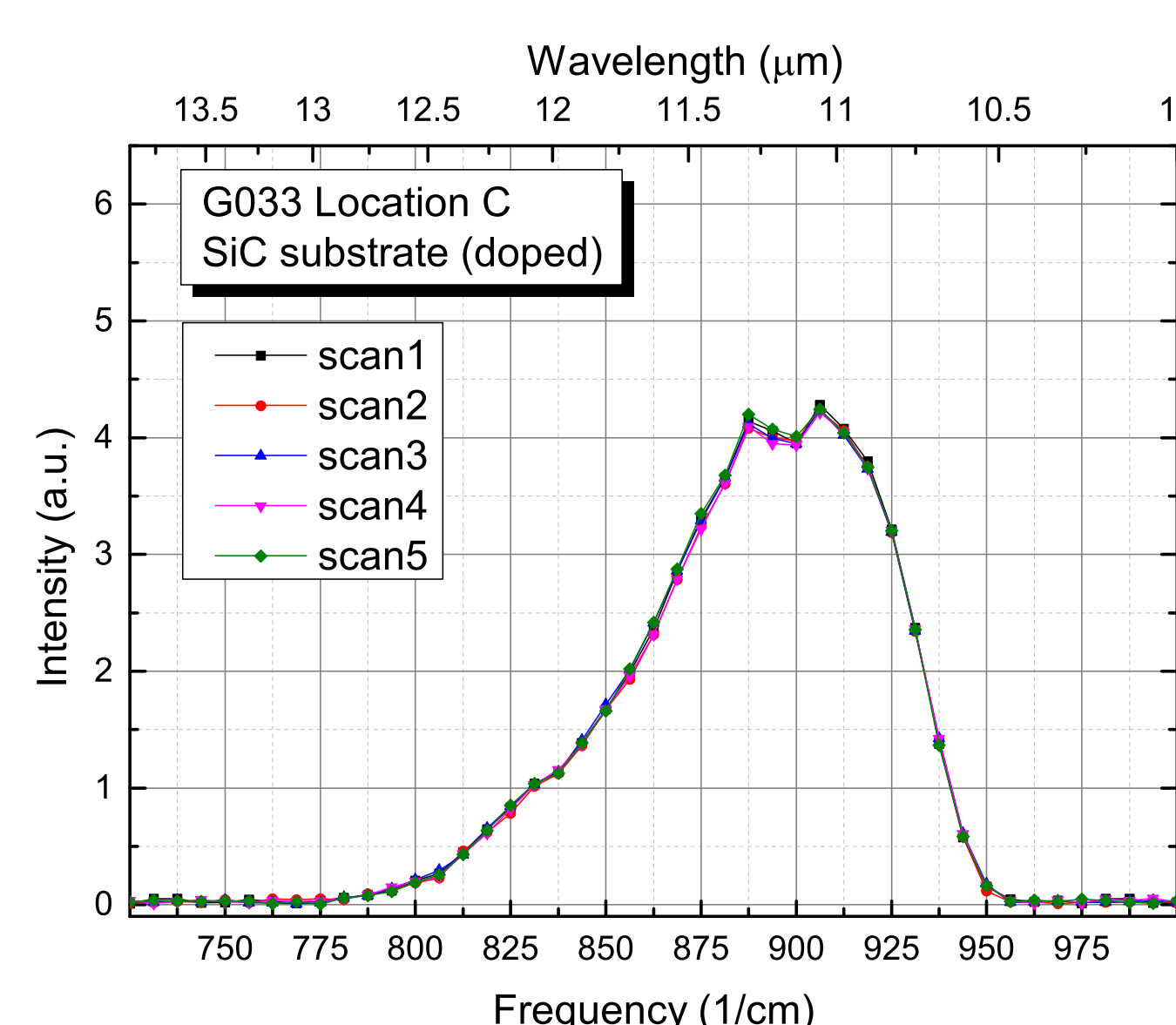


Fig 3: nano FTIR spectrum at location A on G033, storage ring current  $I_s = 117\text{mA}$ .

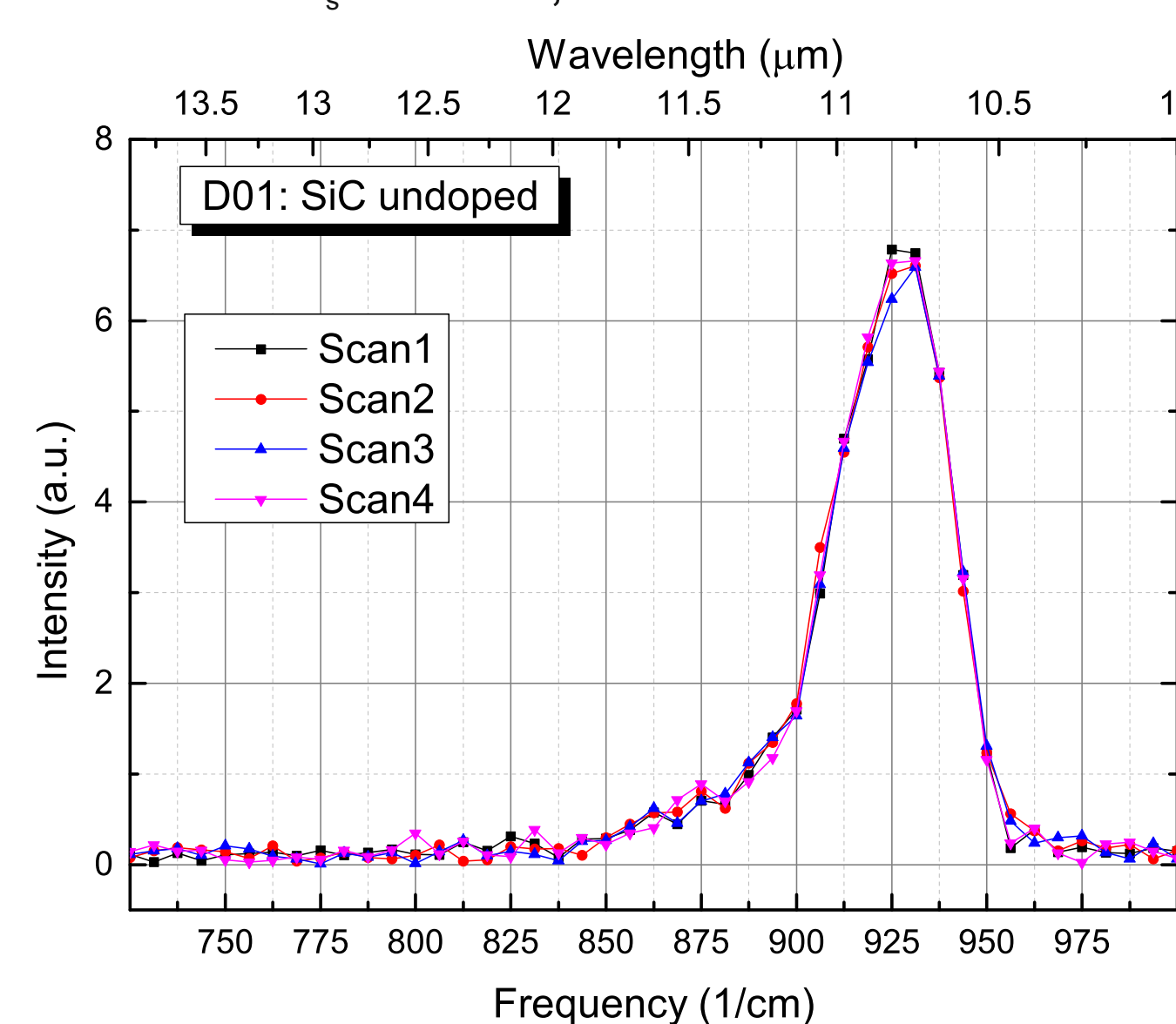


Fig 5: Spectrum at C on G033, storage ring current  $I_s = 121\text{mA}$ ;

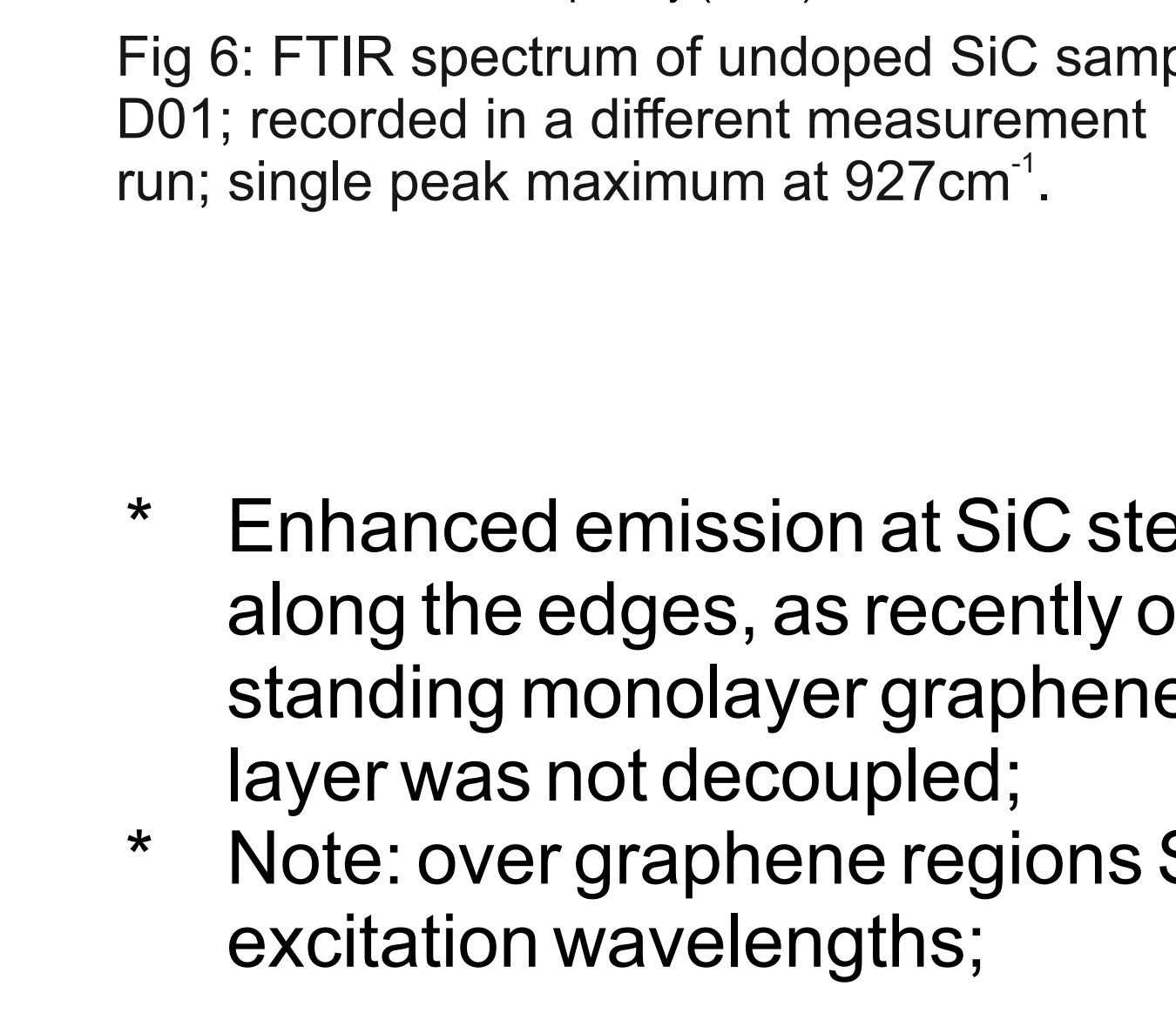


Fig 6: FTIR spectrum of undoped SiC sample D01; recorded in a different measurement run; single peak maximum at  $927\text{cm}^{-1}$ .

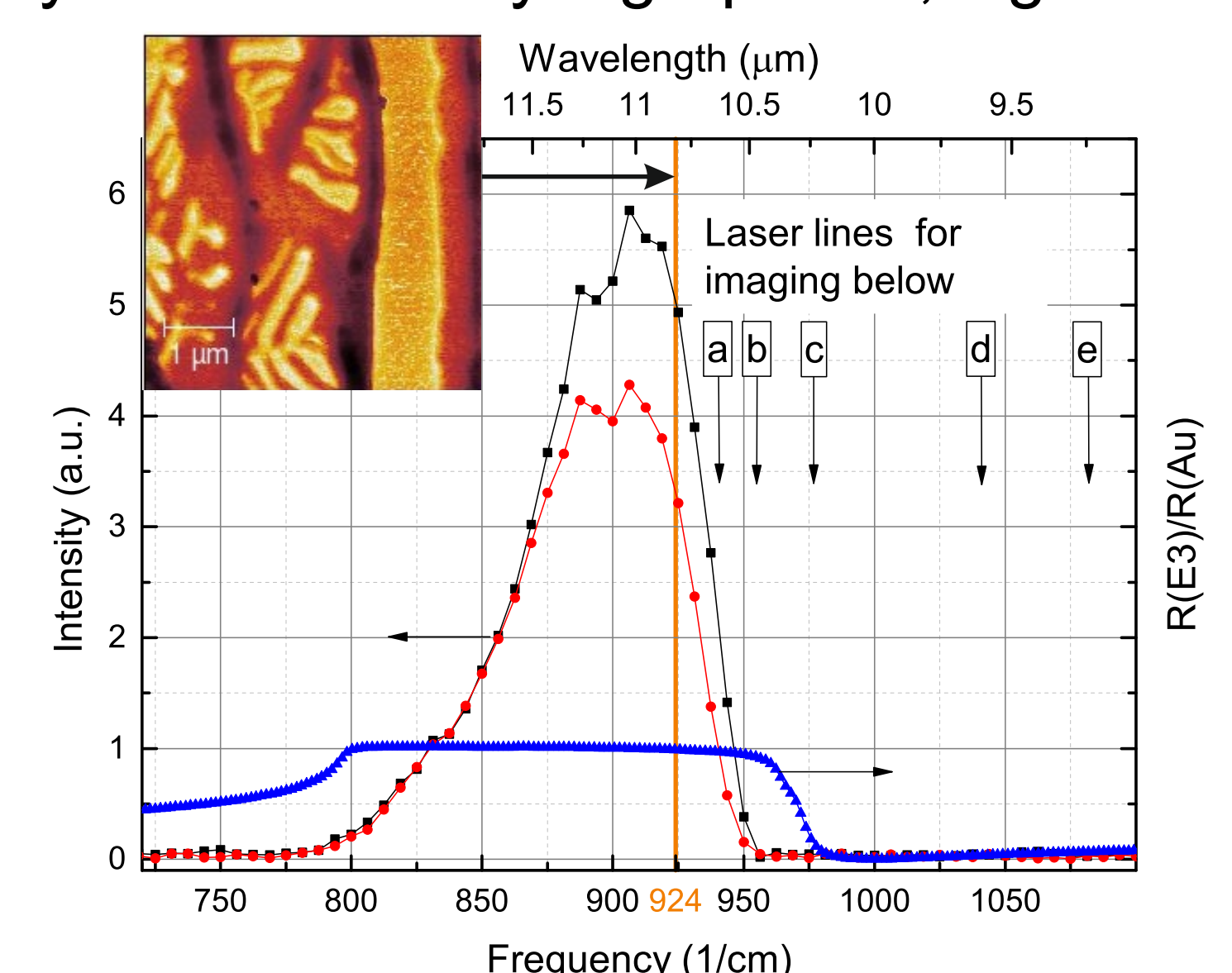


Fig 7: Spectra at A (black) and C (orange); far-field reflectivity spectrum shown by the blue triangles

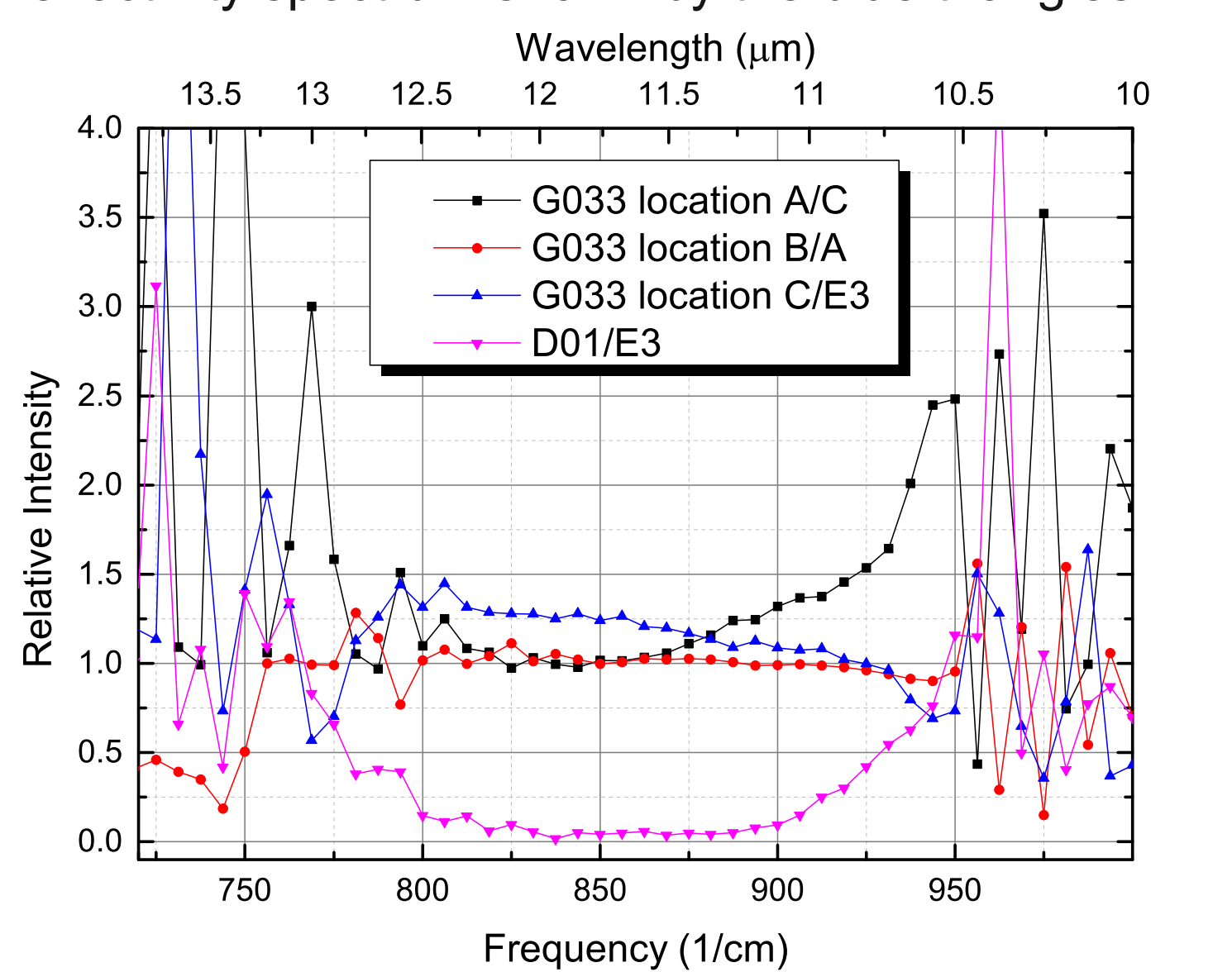


Fig 8: Intensity ratios between different spectra; black squares show plasmon enhanced SiC phonon peak towards higher frequencies; the variation D01/E3 (triangles) comes from the peak-shift of the doped SiC (E3) towards a smaller frequency of  $927\text{cm}^{-1}$

- \* Enhanced emission at SiC step edges, as well as signal fluctuations along the edges, as recently observed by Chen *et al.* [5] on quasi-free-standing monolayer graphene; in contrast, in G033/G068 the buffer layer was not decoupled;
- \* Note: over graphene regions SNOM signal remains constant for all excitation wavelengths;

- [3] A. van Bommel *et al.*, Surface Science **48**, 463 (1975)  
[4] Z. Fei *et al.*, Nano Letters **11**, 4701 (2011)  
[5] J. Chen *et al.*, Nano Letters **13**, 6210 (2013)

