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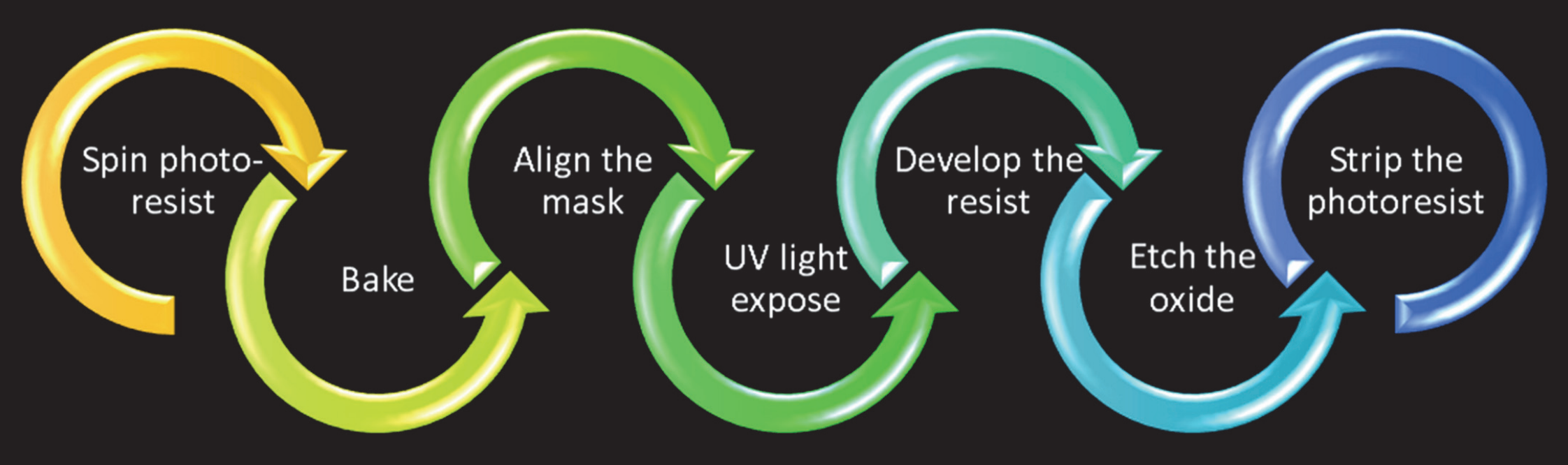
Nanoscale mapping of graphene membranes nanomechanics via Contact Resonance AFM

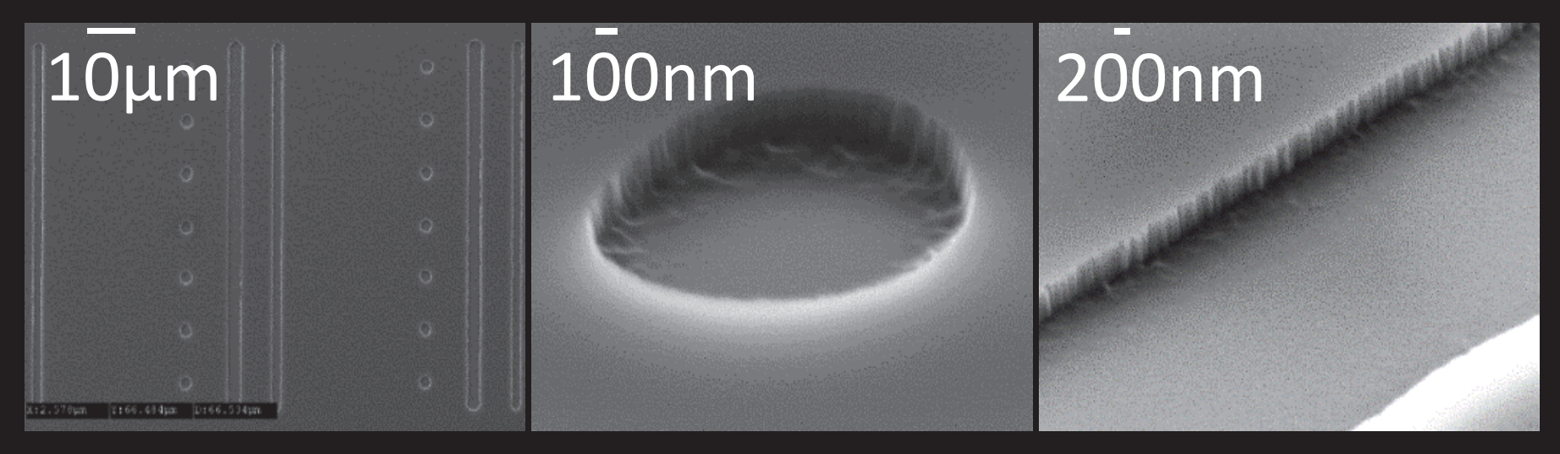
The development of new micro and nano-electromechanical systems (**MEMS** and **NEMS**), as mechanical resonators or sensing elements, requires of the implementation of new materials. **2D materials**, such as **graphene** (**GR**), have high potential in the NEMS applications owing to their unique properties – high elastic moduli and strength, low mechanical dissipation and low weight, as well as high carrier mobility and thermal conductivity [1]. This development demands matching characterization methods with nm scale resolution, sensitivity to local mechanical properties and ability to detect very low amplitude vibrations, with **scanning probe microscopies** (**SPM**) being a suitable candidate.

Here we use SPM methods based on **atomic force microscopy** (**AFM**) combined with **ultrasonic** (**US**) excitation for mapping of nanomechanical properties, dynamic response of 2D NEMS and identification of their subsurface structures.

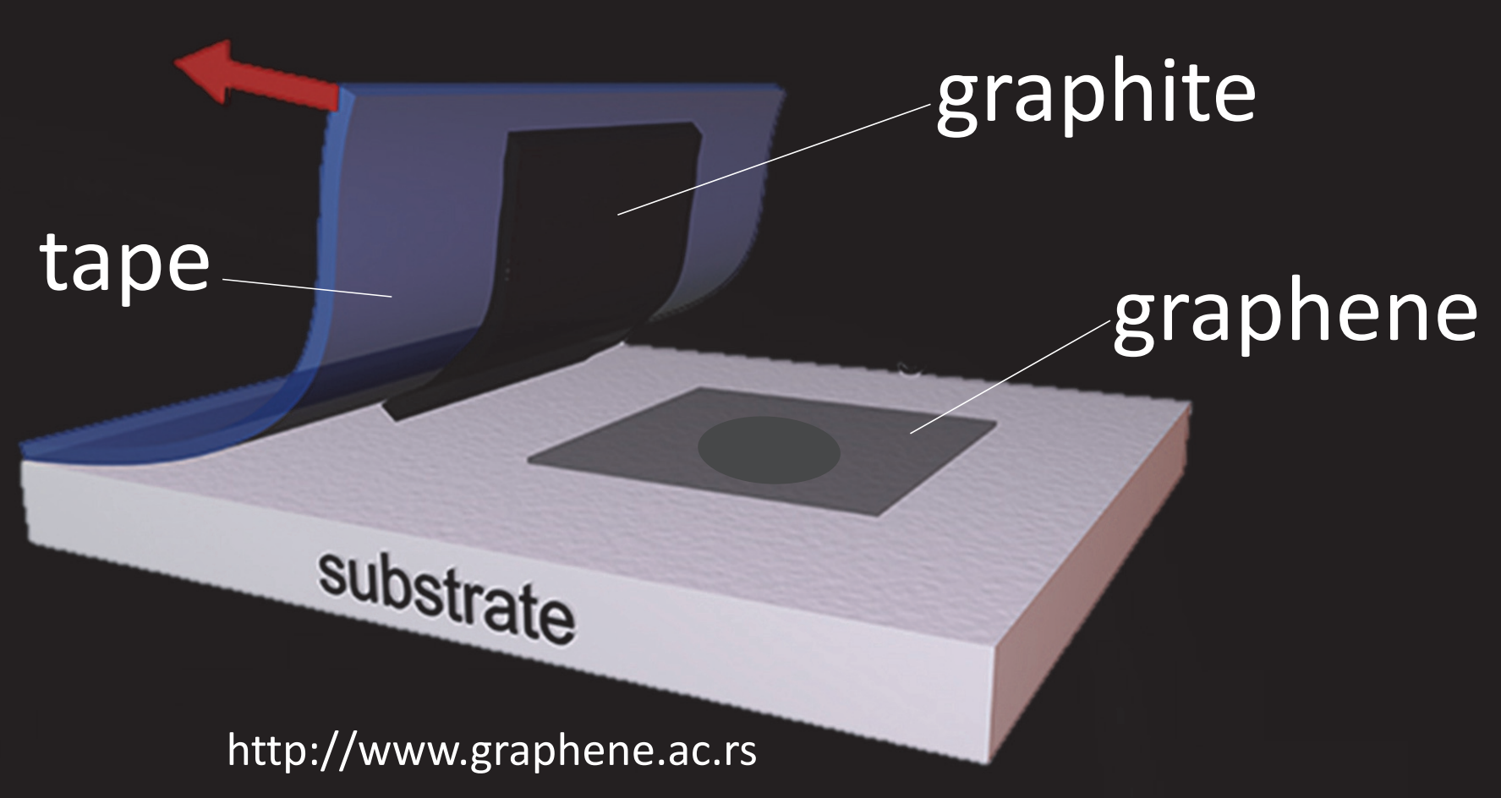
Sample preparation

Substrate processing: Photolithography





Graphene exfoliation and direct transfer



http://www.graphene.ac.rs

SPM with ultrasonic excitation

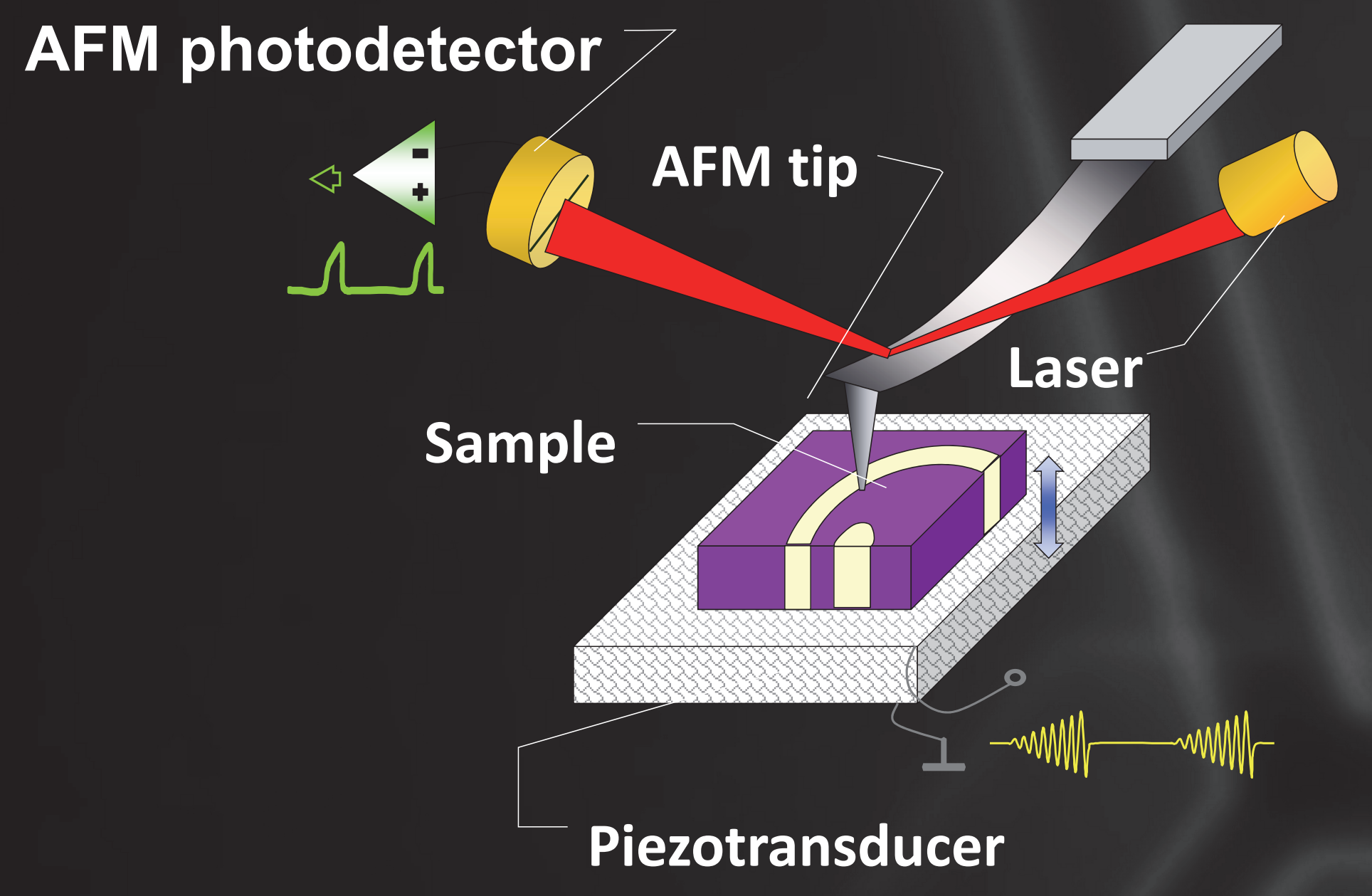
To explore the nanoscale subsurface features we use Atomic Force Microscopy (AFM) combined with ultrasonic (US) excitation. These methods allow to identify different mechanical properties of materials surfaces, as well as detect subsurface structures not seen in AFM topography [2].

The techniques has been implemented in a AFM microscope with the correspondent modifications to excite the sample via piezoelectric drive in a wide frequency range, from few kHz to MHz.

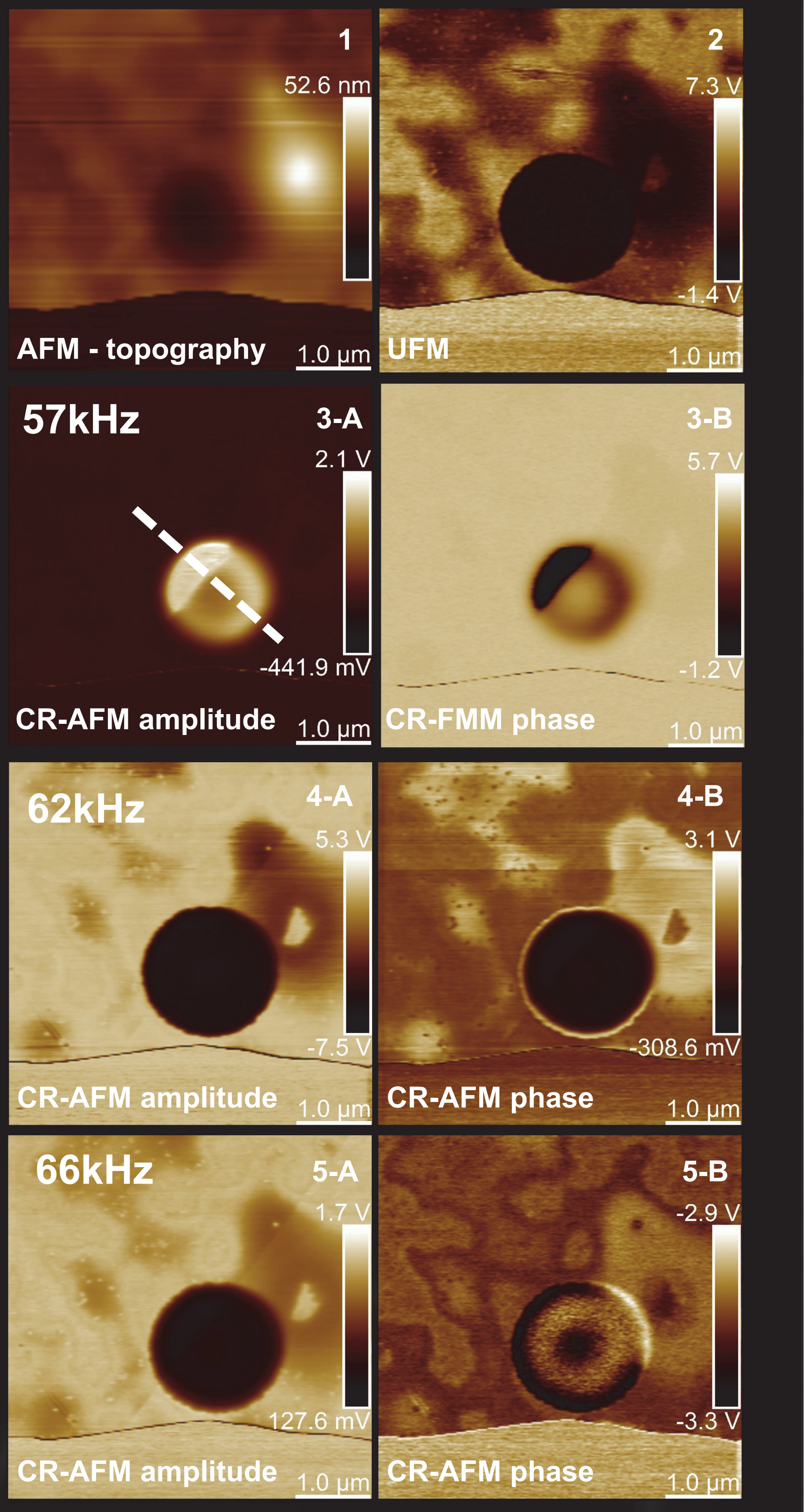
The techniques we used can be classified as:

- Low frequency - Force Modulation Microscopy (**FMM**)
  - ✓ Tip-sample ⟶ Linear regime of the contact.
- Medium frequency - contact resonance AFM (**CR-AFM**)
  - ✓ Tip-sample ⟶ Linear interaction.
- High frequency - Ultrasonic Force Microscopy (**UFM**)
  - ✓ Tip-sample ⟶ Nonlinear interaction.

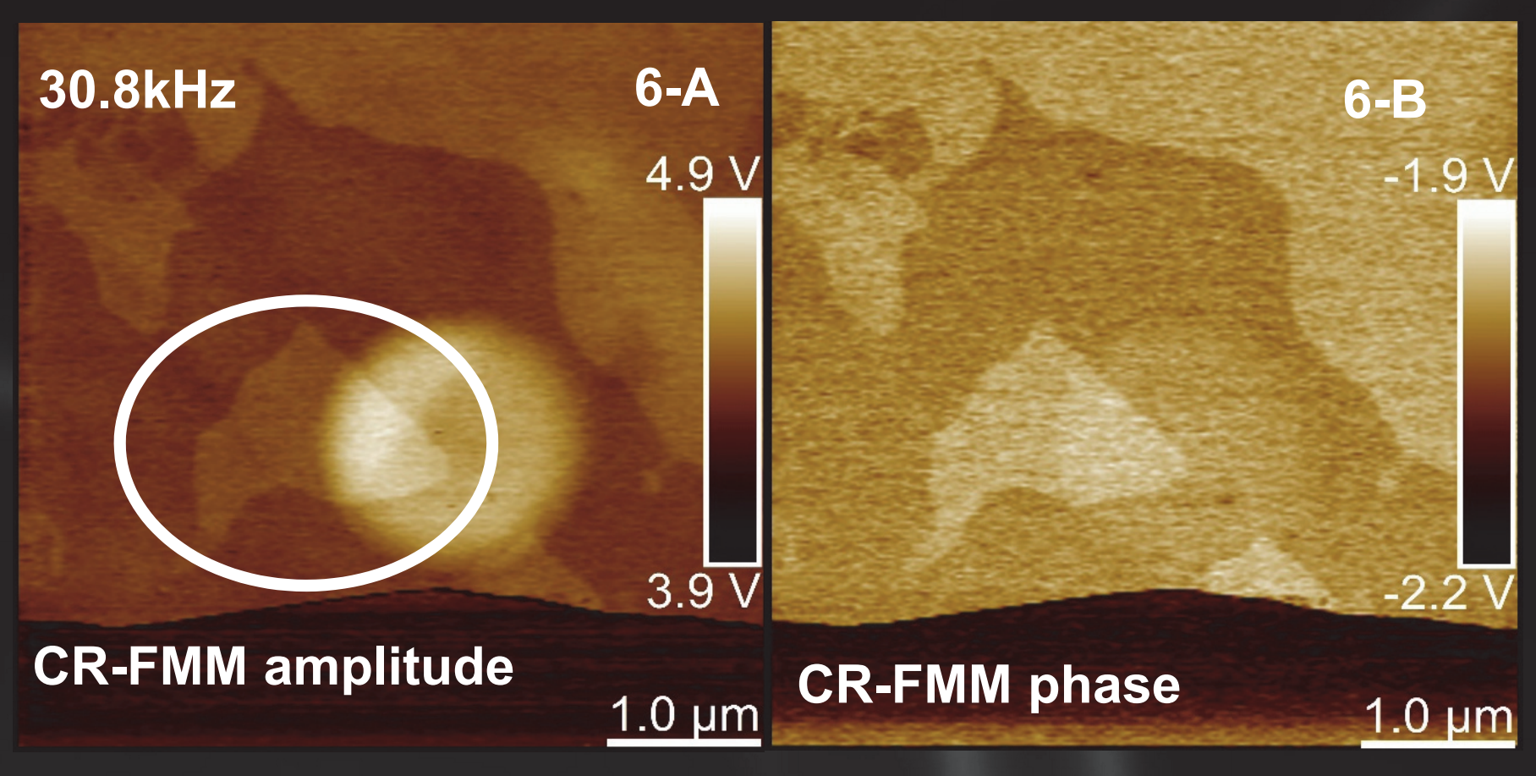
Different stiffness and elastic properties display different contrast in the images.



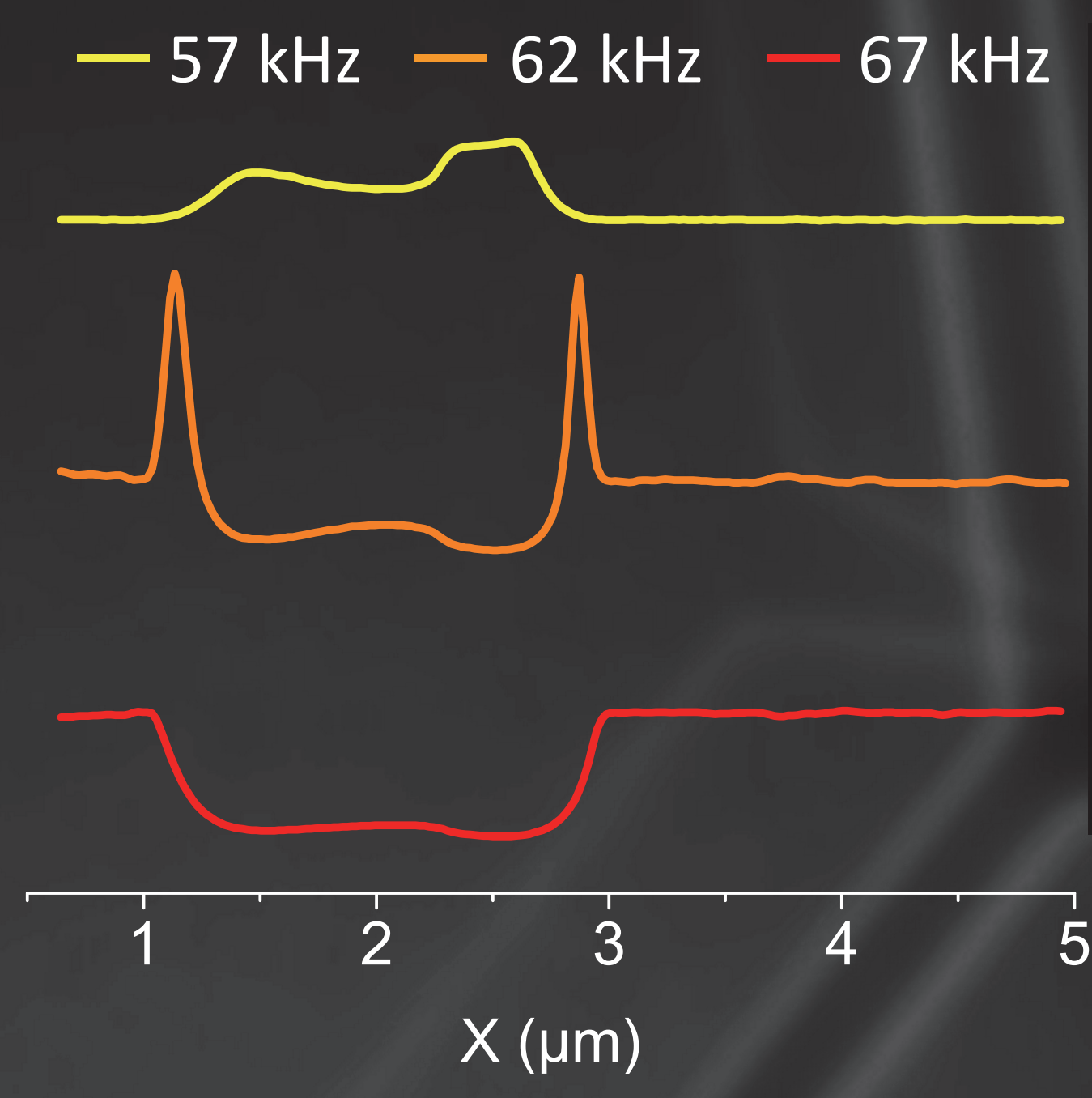
SPM Images

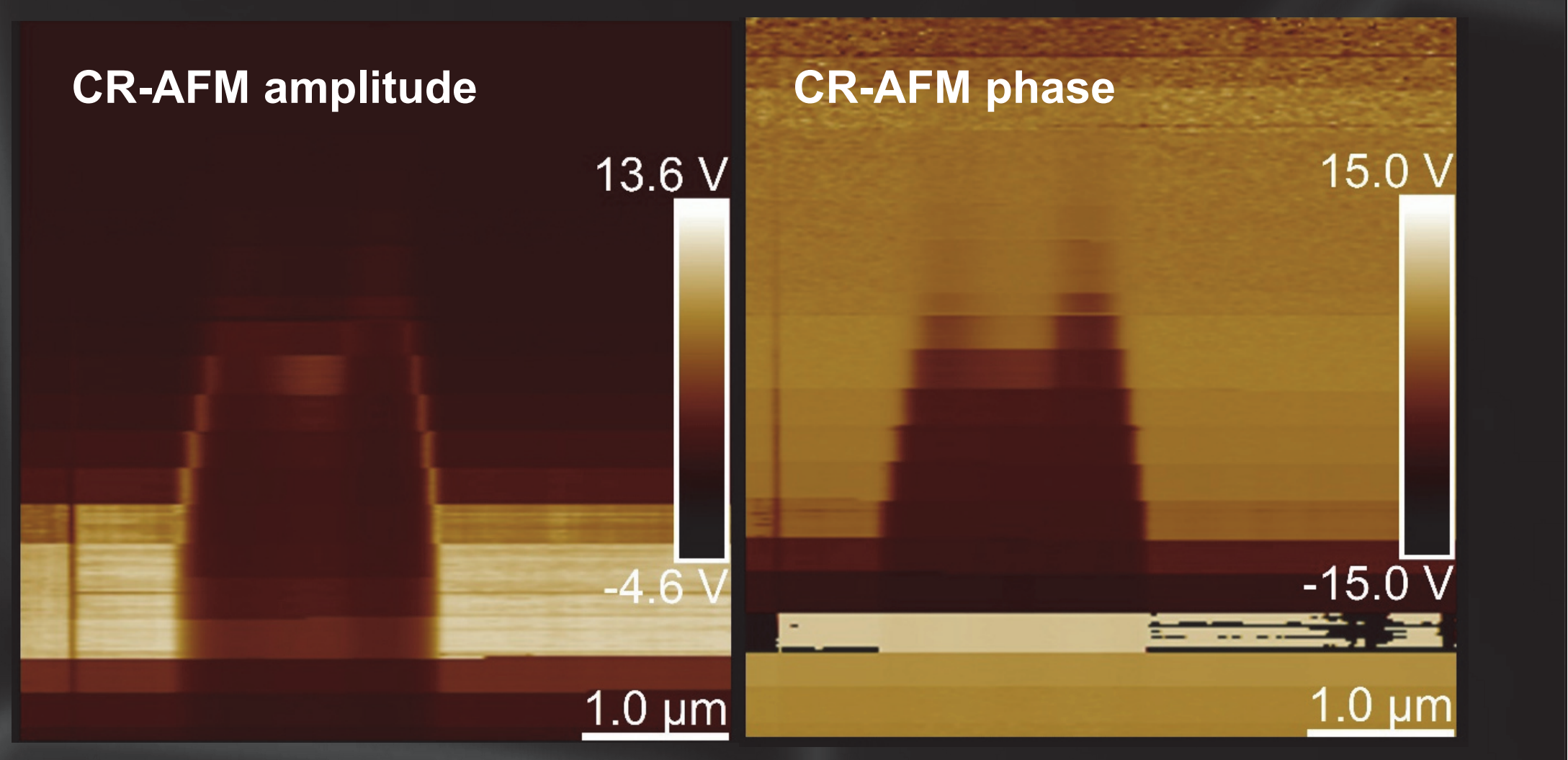


- The AFM topography images does not show well defined the shape of the hole under the GR flake (panel 1).
- UFM image displays different contrast between the substrate, the GR layer and the hole due to the differing mechanical properties of each area. (panel 2).
- CR-AFM images show different contrast in the same areas that is strongly dependent of the excitation frequency (57 – 62 – 66 kHz – panels 3-5). Excitation frequency below the cantilever CR (57kHz) provide low lateral resolution of the subsurface structures (panel 3A-B).
- Very high frequency UFM (panel 2), and other frequencies CR-AFM – panels 4,5) clearly show the shape of the features hidden under the flake. These are most likely delaminations in the areas where the flake is not well attached to the substrate surface.



- Interestingly, the CR-AFM images at 30.8 kHz show a bright area covering part of the hole which could match with a subflake.





- In order to explain the contrast in the panels 3-6 we performed the frequency sweep in the range from 50 to 69 kHz. Scanning along the single line across the centre of the flake (dotted line in panel 3A) as in the images above. It is possible to identify the resonances as a coupled resonances of the cantilever and the graphene membrane and find the flexural stiffness of the membrane from these data.

CONCLUSIONS:

- UFM images allow the highest lateral resolution of the underlying hole but can not provide membrane properties.
- CR-AFM images show the apparent geometry of the membrane depending of the driving frequency, as well as subsurface structures in the membrane and at the membrane-sample interface.
- Varying the frequency allowed a definitive interpretation of the contact resonance images and identification of the joint cantilever-membrane vibrational modes.

References:
[1] K. S. Novoselov, et al., Nature 490 (7419), 192-200 (2012)
[2] O. Kolosov, et al., NSTI-Nanotech 2012, CRC PRESS-TAYLOR & FRANCIS GROUP, Santa Clara, USA, 2012, pp. 282-285.

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