ASEC 2024 Conference
The 5th International Electronic Conference on Applied Sciences
04–06 December 2024 | Online

SEM-Tilting for the Imaging of 2D-Nanomaterials

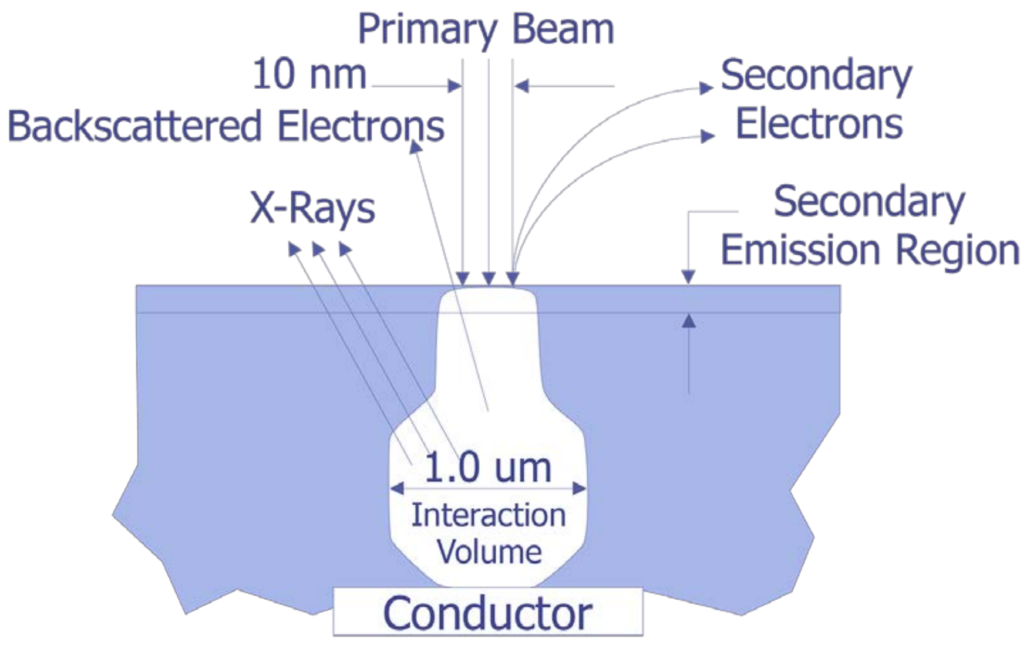
CNR IPCB
Istituto per i Polimeri Compositi e Biomateriali

Angela Longo[1], Mariano Palomba[1,], Filippo Giubileo[2], Gianfranco Carotenuto[1]*

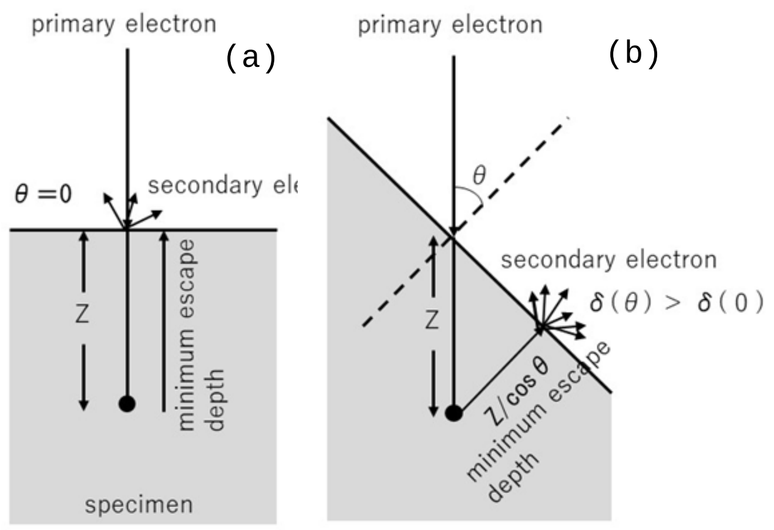
[1] Institute for Polymers, Composites and Biomaterials - National Research Council (IPCB-CNR). SS Napoli/Portici, Piazzale E. Fermi, 1-80055 Portici (NA), Italy.
[2] Institute for SuPerconductors, INnovative materials, and devices - National Research Council (SPIN-CNR). SS Salerno, Via Giovanni Paolo II, 84084 Fisciano (SA), Italy
E-mail: ***mariano.palomba@cnr.it***

Abstract: Owing to their large size and flexibility, 2D-nanostructures (e.g., graphene, graphene oxide, molybdenum bisulfide, etc.) are technologically exploited in a supported form. Glass, silicon and polymers are typical substrates. In the characterization of these 2D-nanostructures, important morphological information (e.g., size, shape factor, presence of defects, etc.) can be obtained by an investigation based on scanning electron microscopy (SEM). However, the observation of layers made by the overlapping of these extremely thin 2D-nanostructures is characterized by a poor contrast and therefore all morphological features are not clearly visible in the SEM micrograph. In this case, atomic force microscopy (AFM) could be conveniently used because of its high sensibility along the z-axis. Here, it has been shown that under a high sample tilting also SEM observations are capable to give images with very good contrast. Such high sample tilting can be obtained by positioning vertically the sample and then reducing conveniently this angle (90°) by tilting the sample up to achieve a well-focused image.

Basic principle of the tilted obsarvtion with SEM



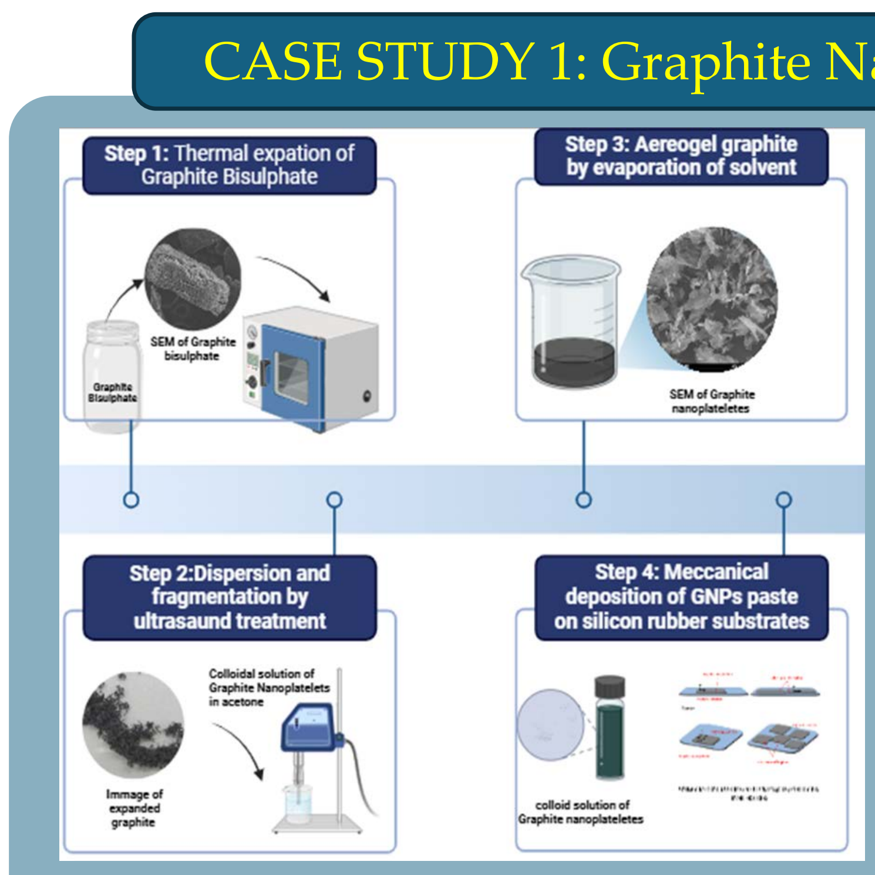
Schematic representation of the interaction between electron-beam and sample.

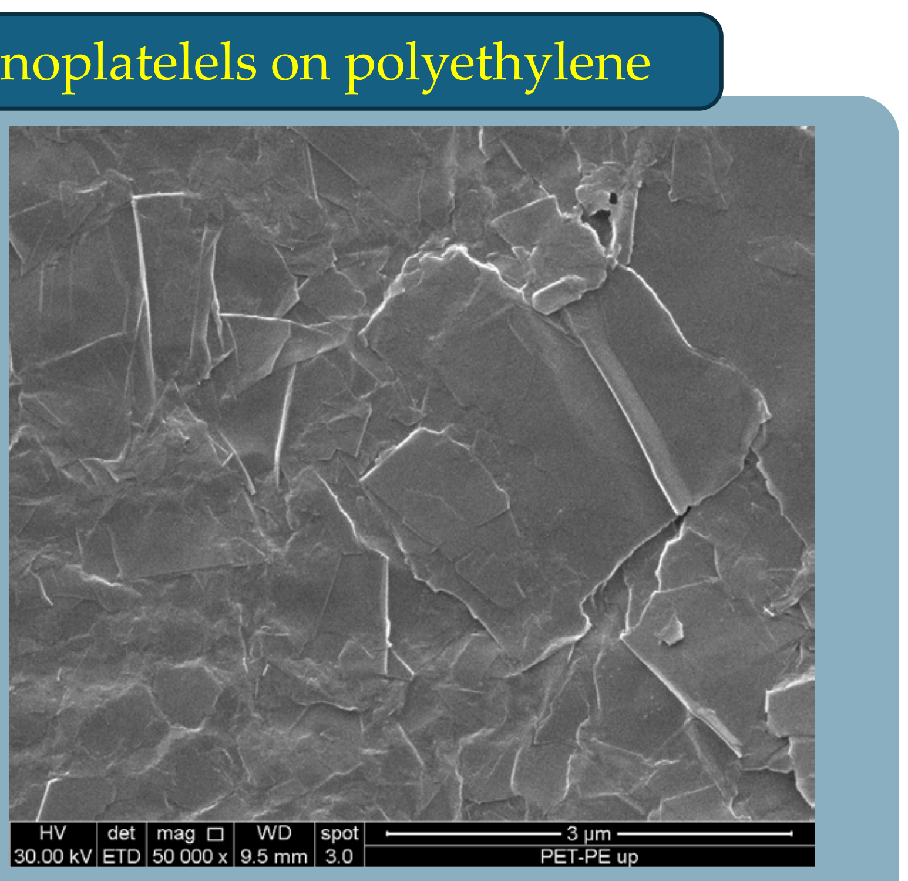


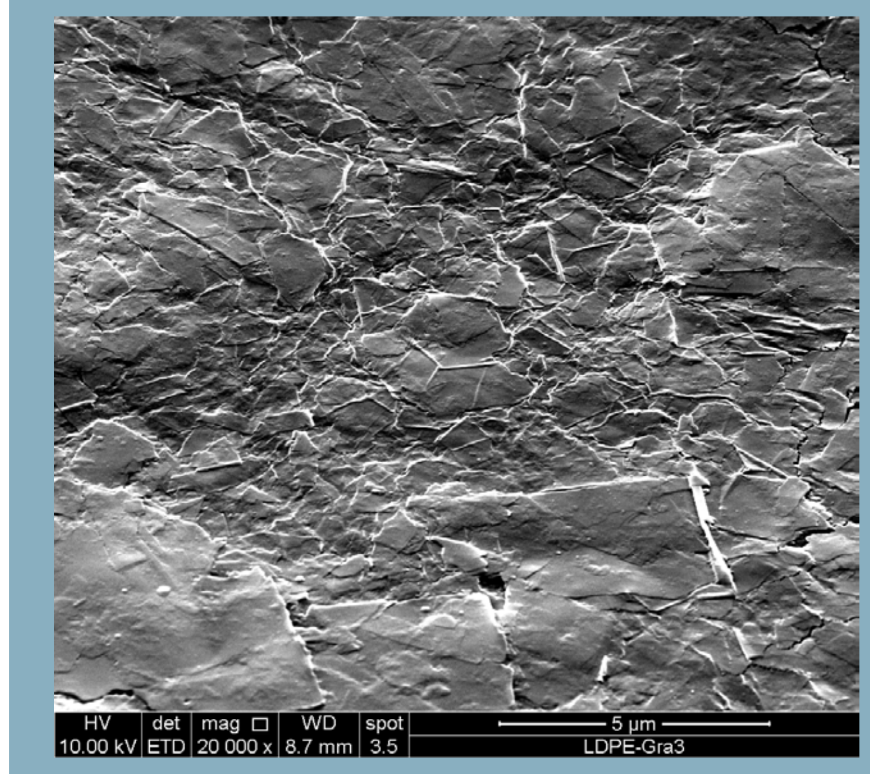
Keywords: 2D-Nanostructures; Morphology; SEM; Tilting, Oblique observation, Graphene, MoS_2.

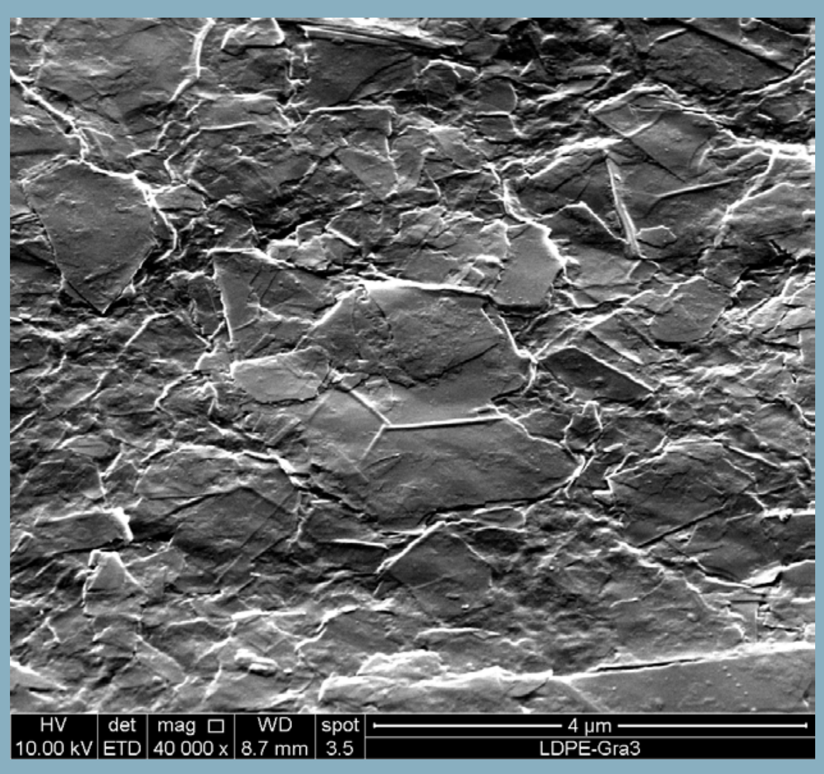
Since the secondary electron (SE) energy is very small to be a few 10eV even at maximum, the electrons excited only near the surface (approximately 10nm thick from the surface) are emitted beyond the surface. This indicates that δ increases when the tilt angle of the specimen is large against the specimen surface as it is shown in Figure a and b. Let θ be the angle between the primary electron beam and the normal to the specimen surface at the incident point of the primary electrons: $\theta=0$ for the perpendicular incidence of the primary electrons onto the specimen surface. For the perpendicular incidence, the number of secondary electrons emitted from the specimen becomes the largest at the minimum escape depth Z. When the specimen is tilted by θ, the minimum escape depth becomes small to $Z \cdot \cos\theta$. Then, the secondary electron emission increases with increasing θ, compared to the case of $\theta=0$. This means that the secondary electron image provides contrast dependent on tilt of the specimen against the incident beam.

CASE STUDY 1: Graphite Nanoplatelels on polyethylene



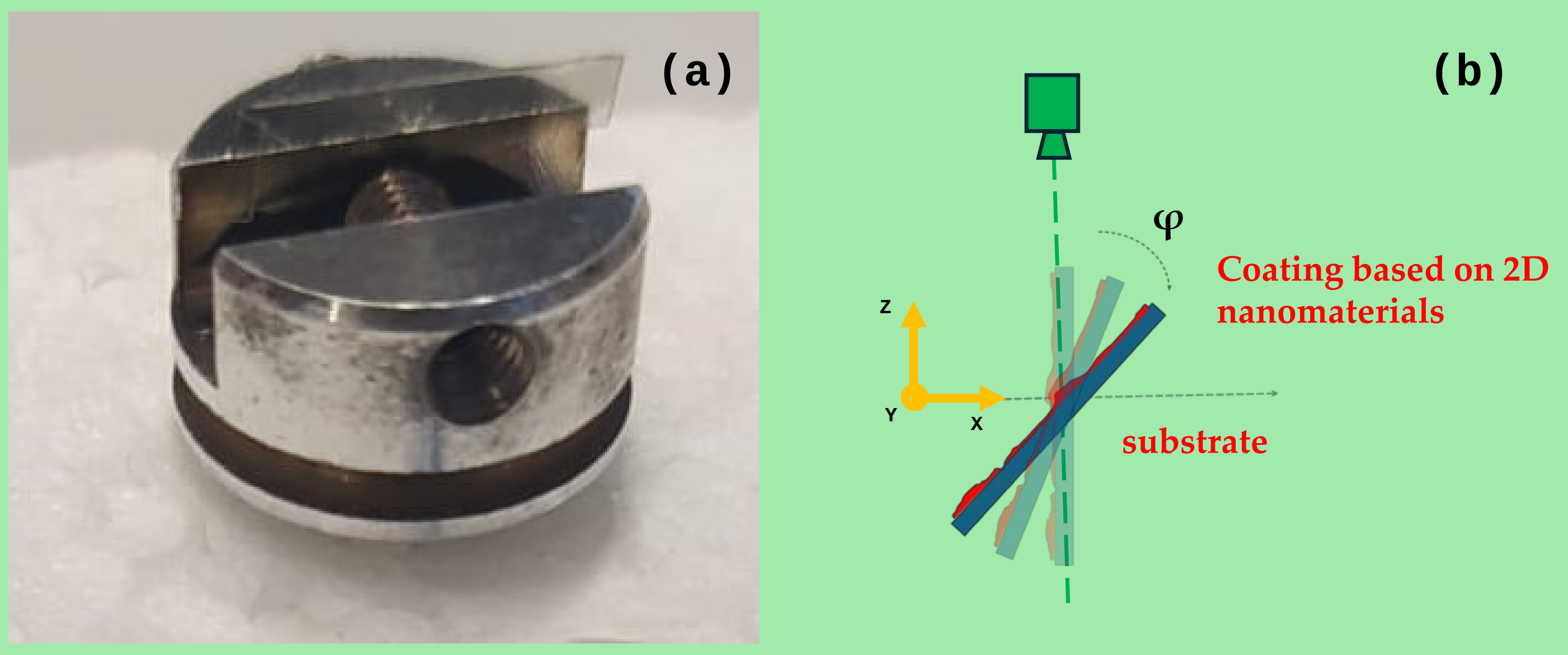




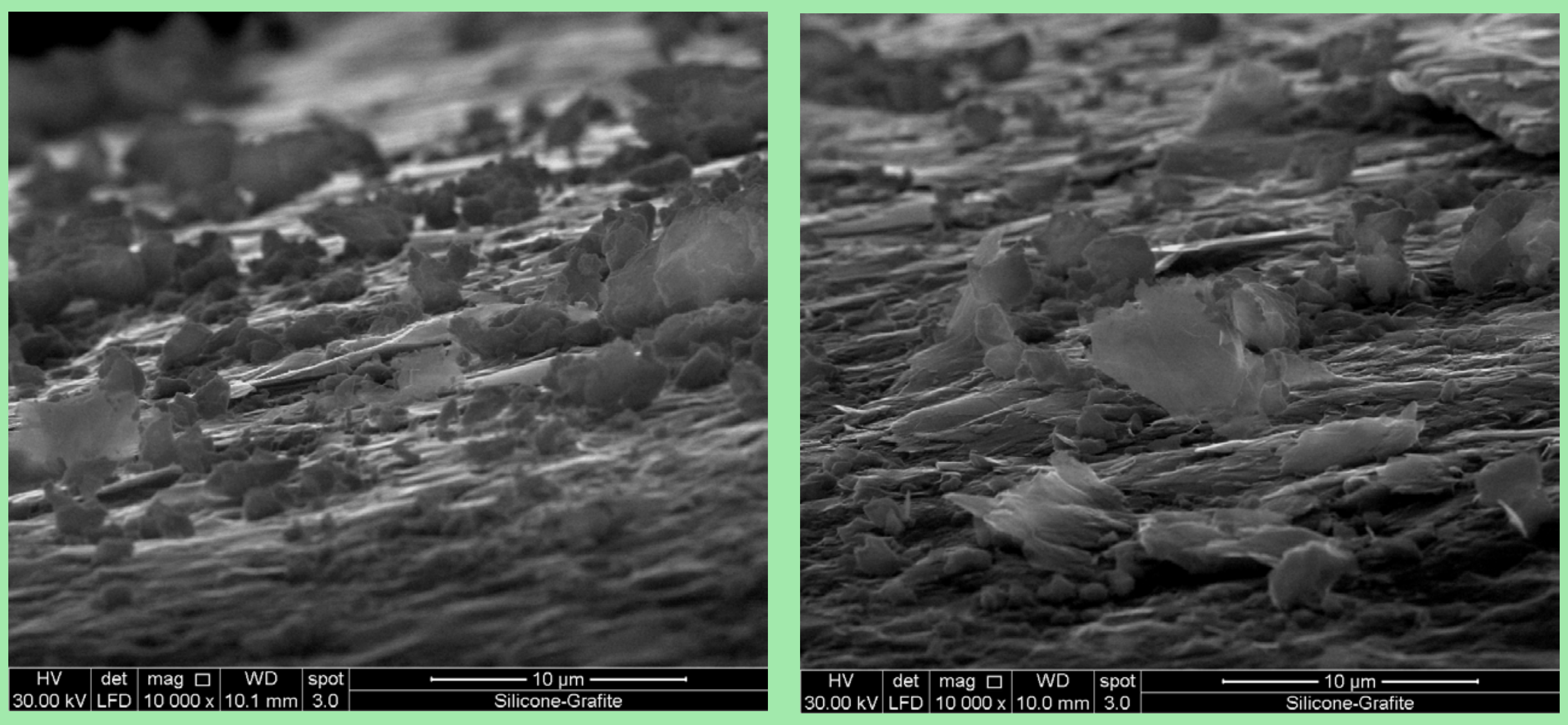


SEM-micrographs showing the top-view of a graphite coating (a) and the same sample after tilting from 90° (b).

CASE STUDY 2: Graphite Nanoplatelels on silicon rubber



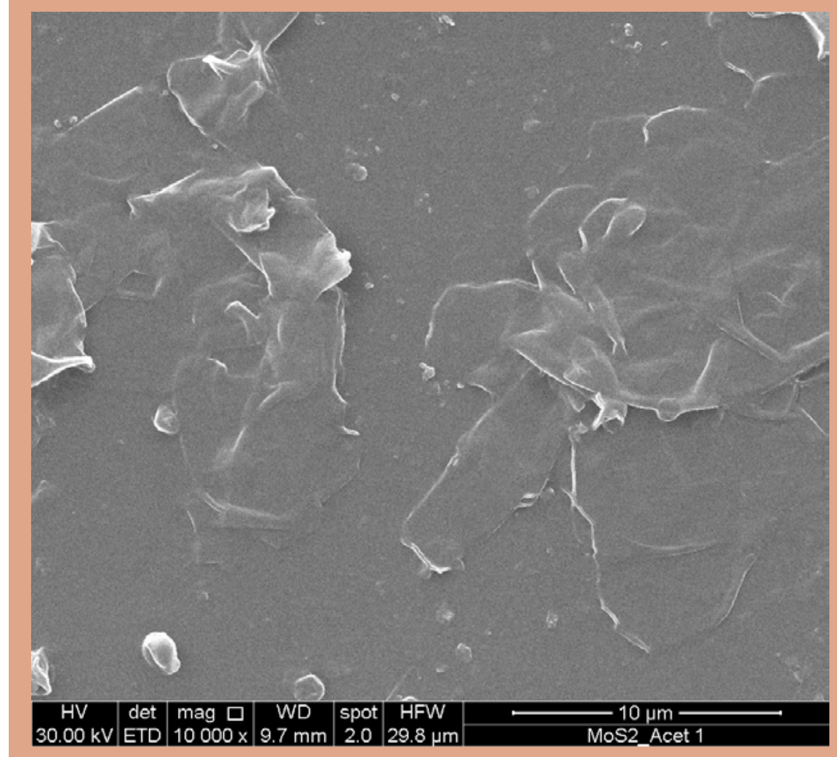
Type of stab required for a vertical arrangement of the sample (a) and schematic representation of the sample tilting from 90° positioning (b).

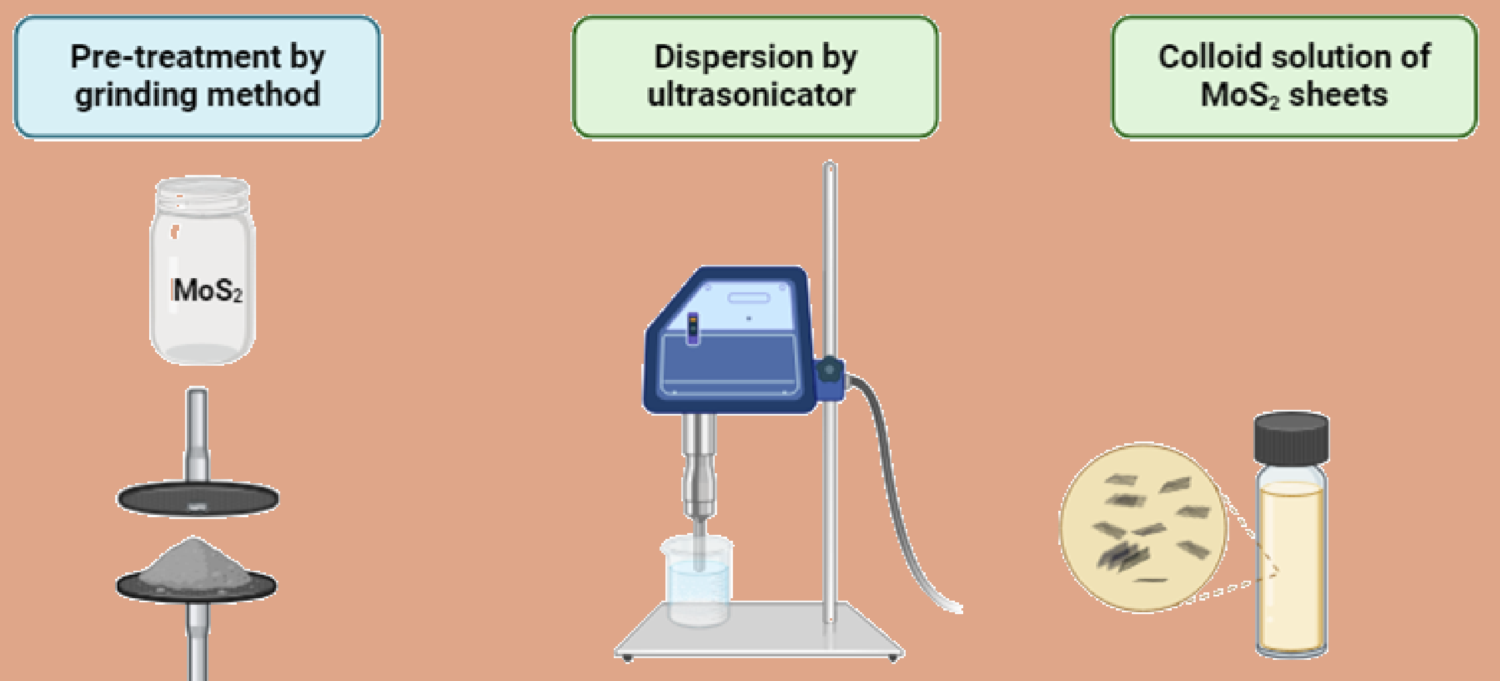


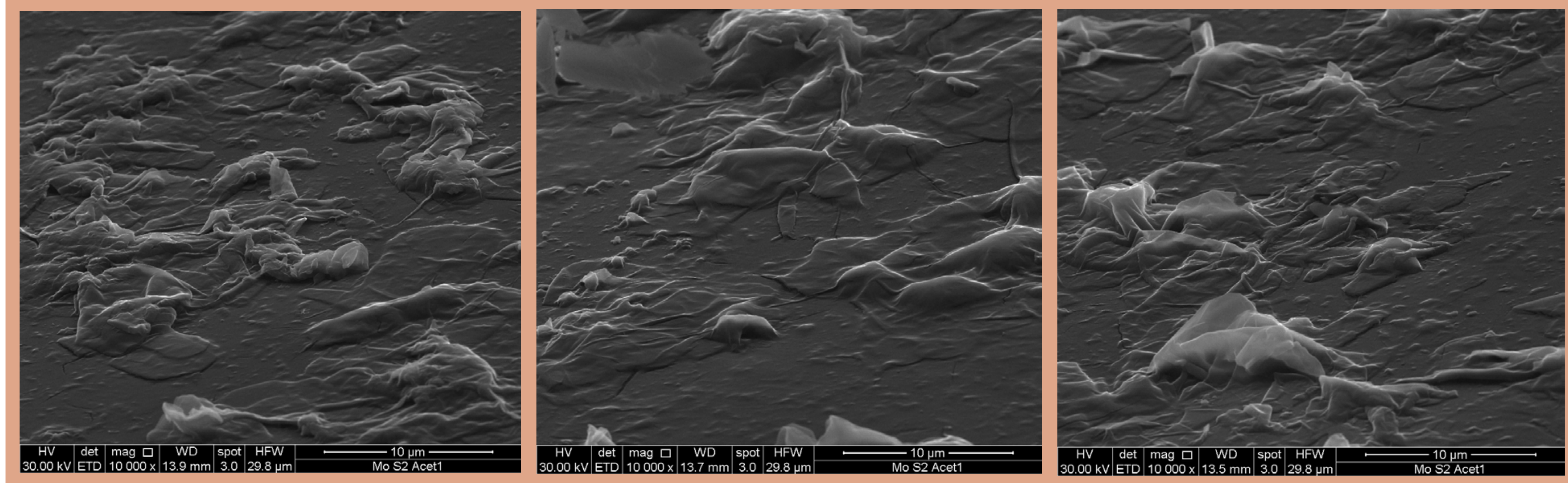
SEM-micrographs showing graphite nanoplatelets placed vertically on silicon rubber after tilting from 90°.

When a mechanical stress is applied to a silicon rubber substrate coated by graphite nanoplatelets, some of these graphite nanoplatelets are left in a vertical position. These surface features that are placed along the z direction require necessarily a tilting observation to be adequately visualized.

CASE STUDY 3: Molibdenum disulfide on glass



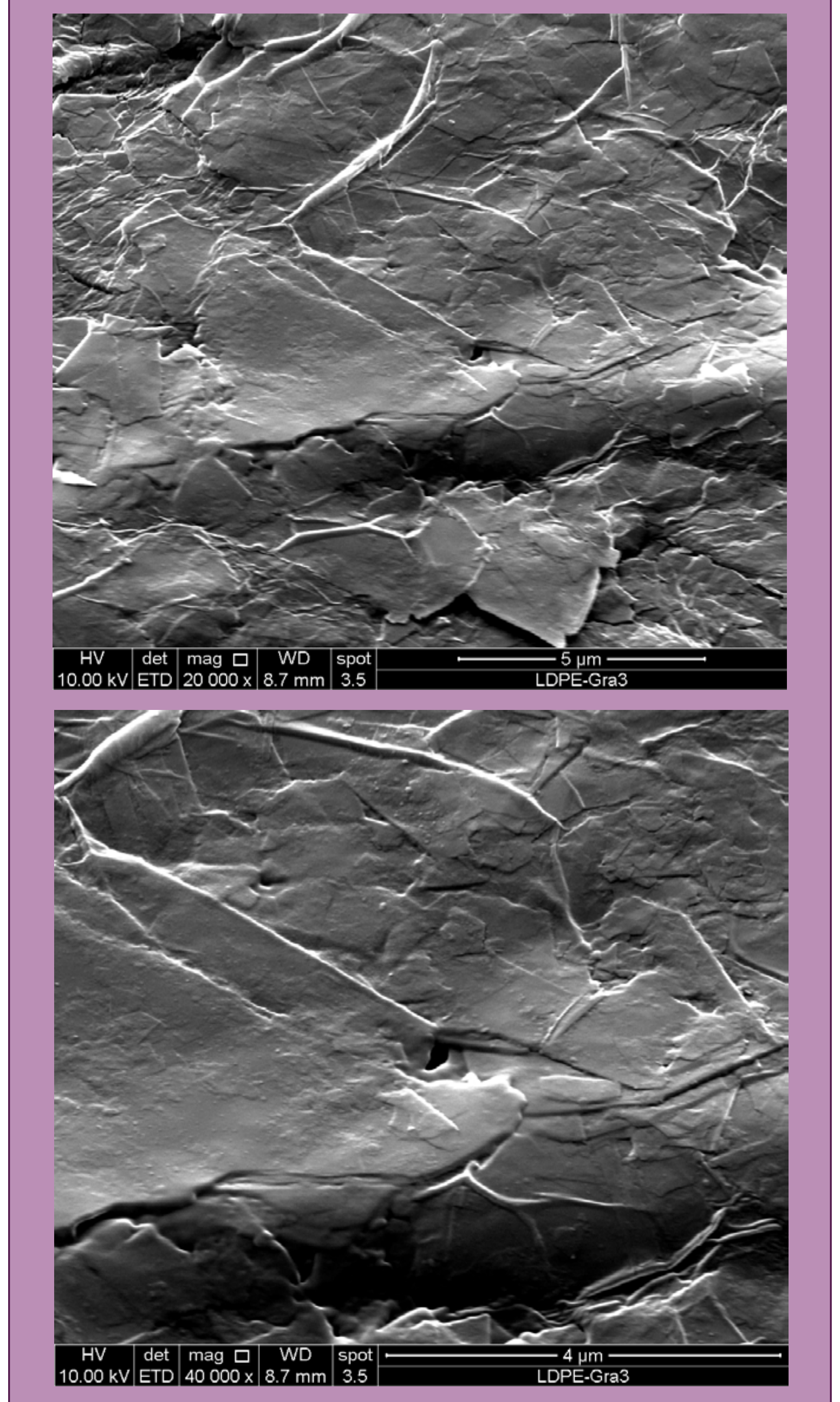




SEM-micrographs showing the top-view of few-layer MoS_2 crystals deposited on a glass substrate (a) and the same sample after tilting from 90° (b).

Few-layer crystals of MoS_2 have been prepared by applying sono-acoustic energy to a suspension of pre-treated MoS_2 powder in acetonitrile (CH_3CN). The sono-acoustic exfoliation treatment resulted effective only in the case the MoS_2 commercial sample had undergone a mechanical pre-treatment based on the application of shear stress to the pristine powder by placing it between two PTFE surfaces. In order to isolate the few-layer crystals of MoS_2, the obtained colloidal suspension was diluted and left to settle for a few weeks. MoS_2 few-layer crystals were contained in the supernatant. For optimal SEM observation, the 2D nanostructures were deposited on a glass substrate and sputtered by a Au-Pd alloy.

CASE STUDY 1



SEM-micrographs of a tilted graphite coating.

Knowledgement: The authors are grateful to Mrs. Maria Cristina Del Barone of LAMEST laboratory for the SEM characterization of 2D-nanostructures.