

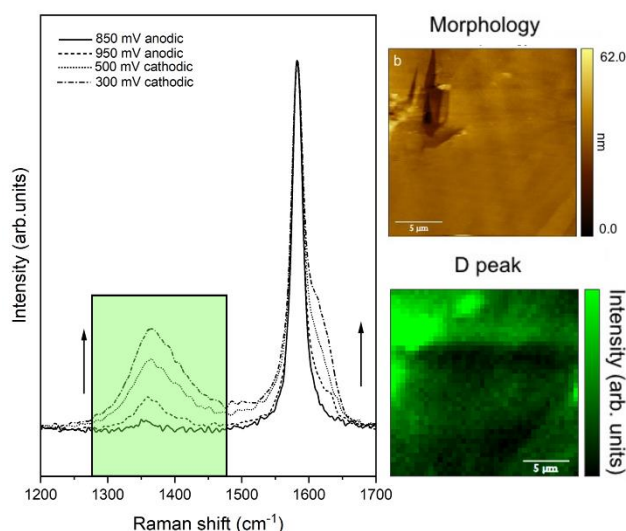


A combined Raman spectroscopy and electrochemical atomic force microscopy system for joining chemical maps to electrode surface topographies

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The intercalation of anions inside the stratified structure of highly oriented pyrolytic graphite (HOPG), which can be obtained inside an electrochemical cell and with diluted mineral acids (such as sulfuric acid), represents a model system to understand intercalation mechanisms at the molecular length-scale [1]. The complex physical-chemical processes occurring at the electrode-electrolyte interface require the use of both microscopic and spectroscopic techniques [2,3]. Generally, these studies are not directly comparable: atomic force microscopy (AFM) is coupled with a potentiostat to follow morphological evolutions of the electrode surface when different electrochemical (EC) potentials are applied to the HOPG; Raman spectroscopy is able to perform a chemical analysis during the different steps of the intercalation process. The comparison between experiments is only possible by exploiting the EC analysis that, however, does not always exactly use the same parameters in microscopic and spectroscopic experiments.



In the analysed HOPG region, the Raman spectrum was acquired as a function of the EC potential. It is also possible to select a Raman feature and visualize its intensity in different area while AFM shows the electrode morphology.

Here, we discuss the HOPG anion intercalation by exploiting an innovative experimental set-up, which is able to combine Raman spectroscopy and AFM together and inside an EC cell. The proposed set-up allows to correlate morphological images with chemical maps, as extracted from Raman data, during the oxidation process of graphite. This system thus plays a crucial role when chemical reactions, both reversible and non-reversible, are studied step by step during EC processes and/or when local chemical analysis is required.

References:

- [1] C.A. Goss *et al.* Anal. Chem. 65, 1378 (1993)
- [2] G. Bussetti *et al.* J. Phys. Chem. C 120, 6088 (2016)
- [3] R. Yivlialin *et al.* Appl. Surf. Sci. 504, 14440 (2017)