Scanning transmission electron microscopy of air-sensitive samples and under controlled low-pressure atmospheres

G. T. Leuthner[1] C. Mangler[1] T. Susi[1] J. C. Meyer[2] J. Kotakoski[1]
[1]University of Vienna, Austria
[2]University of Tübingen, Germany

Scanning transmission electron microscopy (STEM) is a powerful technique for the study of atomic structure and spectroscopic fingerprints of materials down to the level of individual atoms. However, in typical instruments the samples need to be inserted through air, which makes the study of air sensitive samples difficult with this method. In this contribution, we present first results from the Vienna setup built around a Nion UltraSTEM100 that allows the transfer of samples between external systems and the microscope in an Ar atmosphere. This enables the study of air-sensitive samples that have either been created in a protective atmosphere or made sensitive through treatment for example by energetic ion irradiation.

Additionally, in many instruments, the pressure at the sample during observation is limited to ca. 1e-7 mbar, providing a large number of gas molecules for the electron beam to crack, which can lead to structural changes in the sample. Since the exact composition of the gas atmosphere at the sample is unknown, it is difficult to study the role of different gases in the observed dynamic processes. In our setup, through the addition of an all-metal leak-valve to an instrument with a base pressure at the sample on the order of 1e-10 mbar, we can directly address this issue. In this presentation, we show the effect of leaked air as well as its constituent gases on the etching of carbon-based contamination on a mono-layer graphene sample. Air, water and oxygen all lead to etching, whereas hydrogen and nitrogen atmospheres have no influence as compared to experiments in ultra high vacuum (UHV). Oxygen provides an etching effect that is two times more efficient than that of water, p resumably due to two oxygen atoms per molecule in the oxygen gas. We also show that in UHV, graphene edges with the armchair configuration are stable even under the electron beam, whereas in an oxygen atmosphere they become unstable and only zigzag edges can be imaged. These studies show that controlled low-pressure atmospheres in transmission electron microscopes can be used to carry out chemical modification of materials at the nanometer scale.