doi:10.1017/S1431927617008480

Microsc. Microanal. 23 (Suppl 1), 2017

Ultrahigh Resolution of Electron Energy Loss Spectroscopy by a Monochromated Titan TEM: Towards Challenging Nanomaterials Characterization

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Electron Energy Loss Spectroscopy (EELS) applied in Transmission Electron Microcopy (TEM) is a powerful analytical technique for the characterization of modern nano-materials. This technique is especially useful when TEM system is equipped with a monochromator of electron source or electron gun (to minimize the energy speared inherent to electron emission process). The majority of available monochromated TEMs (e.g. FEI's Titan) has the energy resolution in the range of 150-200meV. However, for a considerable number of scientific challenges the key characteristic features of EELS measurements require the resolution better than 50meV. Among those challenges are the vibrational spectroscopy for inorganic and organic materials, including the detection of hydrogen [1], measurement of phonons distribution or bandgap states with high spatial resolution [2], and study of Van der Waals materials exhibiting intriguing structural, electronic and photonic properties [3]. Recently a significant progress is achieved in EELS-TEM systems performance. This progress is mainly associated with a monochromated scanning TEM (STEM) developed by NION Co, with a regular energy resolution of about 10meV [1]. At the same time, it has been predicted theoretically, that the original design of FEI's monochromator could deliver a comparable energy resolution (16meV) subject to the availability of a high resolution energy detection system [4].

The first successful attempt to extend the capabilities of FEI's monochromator is already described in [3]. Here we report a further development of the optimization routine to achieve the ultimate energy resolution with an FEI's EELS-TEM system (Titan 60-300) based on a combination of a monochromated gun and Gatan imaging filter (GIF 966). One of the major advantages of a Titan is the ability to operate a microscope both in conventional TEM and STEM modes. In TEM mode at 80kV acceleration voltage our finding demonstrate that by lowering the monochromator potential and compensating it with a gun adjustment, the energy resolution of 40meV can be achieved with unprecedented beam current of 400pA. While extremely usefully for high resolution EELS analysis in case high spatial resolution is not required, such a monochromated beam also significantly reduces the effects of the chromatic aberration for HR-TEM imaging with Cs correctors at low acceleration voltages. At the same time in STEM mode (also at 80kV) our optimization routine results in the energy resolution of 30meV with a probe current of about 40pA. The approach was tested on several FEI's monochromated microscopes including a low-base Titan TEM with 965 GIF at 60kV accelerating voltage, revealing nearly identical energy resolution vs. beam current.

Our approach for achieving the ultrahigh energy resolution EELS we applied for the investigation of electrical properties of ultrathin two-dimensional transition-metal dichalcogenides. These materials started to attract a significant interest due to their remarkable chemical and mechanical stability [5] and new physical properties through various quantum confinement effects [6], which make them promising candidates, for example, for nanoscale field-effect transistors and solar cell [7]. For the case of in-plane atomically sharp interfaces between a single-layer WSe_2 and MoS_2 films we demonstrate that the conduction band onsets (Fig.1a) of each of these 2D materials can be reliably traced across the WSe_2/MoS_2 interface. This essentially allows us to plot the bandgap distribution in the composite film (Fig.1b) by means of STEM spectrum imaging. On the other hand, having significant beam current in TEM mode we utilize the ultrahigh energy resolution for the momentum-transfer (q) resolved EEL spectroscopy. For a pristine single-layer WSe_2 film the analysis of the conduction band onsets variation with the momentum-transfer (Fig.1c) should, in principle, allow to verify the direct bandgap nature of that 2D material. Dealing with an order of magnitude smaller energy losses the measurement of surface plasmons distribution in various nanomaterials is another application for ultrahigh energy resolution. The plasmons even with the energy of about 100meV can be reliably detected and visualized (Fig.1d).

The estimated number of monochromated FEI's TEMs currently available at different research facilities is considerably higher than one hundred and in terms of energy resolution up until now they not are exploited to the full power. Thus, we believe that our results of the energy resolution optimization of EELS-TEM systems might be useful and interesting for the scientific community.

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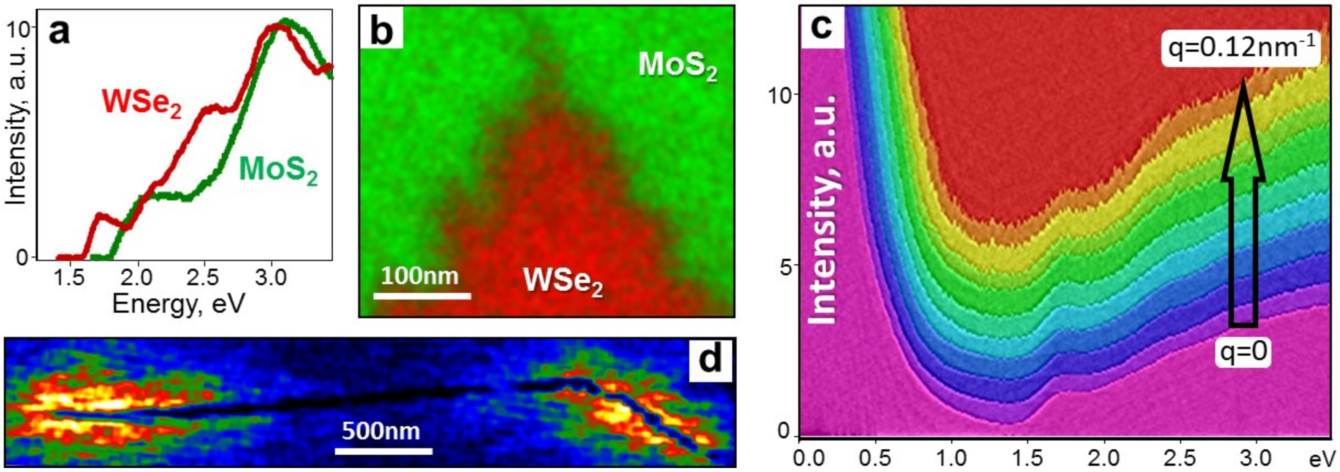

Figure 1. **a** Valence-loss spectra from a single layer WSe_2 (red dots) and MoS_2 (solid green) after ZLP subtraction. **b** EELS mapping of in-plane WSe_2/MoS_2 interface based on valence-loss features in **a**. **c** Momentum-transfer resolved EELS of the conduction band onset in a single-layer WSe_2. **d** First harmonic (110meV) plasmon map of Ag nanowire.