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White-beam electron technique for nanomaterial characterization

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White-beam technique (using energy dispersive full spectrum as a probe) such as white-beam X-ray and white-beam neutron are useful for fast and comprehensive visualization of crystal defect and distortions. They are, however, rather difficult to analyze in any qualitative way, and even a qualitative interpretation often requires considerable experience and time. Here, we develop a white-beam electron technique to characterize nanomaterials without the influence of underlying substrate signals. In this new method, we have extended such "white-beam" concept to electron beam techniques, and employ such white-beam electrons (white electrons) to extract quantitative information of target nanomaterial with extremely high efficiency. Such attempt is a novel application and extension of the conventional white-beam technique, and possibly lead to further utilization of electron beam technique.

1. Introduction

Popular surface analysis techniques and operating procedures have seen little improvement in recent decades. These techniques take measurements on the surface of a material and then data are analysed by identifying weak features in core-level signals against strong secondary electron (SE) background with the naked eye. However, such SE background signal may reflect various background processes and hold some useful information for comprehensive materials characterization. Therefore, for surface analysis techniques, there is a need for a new method that is able to obtain information about nanomaterials from SE background signals even when the nanomaterial is supported by a substrate.

Here, we propose a new train of though to characterize substrate-supported nanomaterial using SE spectrum as a white electron probe.

2. Concept of the white-beam electron technique

When we want to investigate the microscopic and

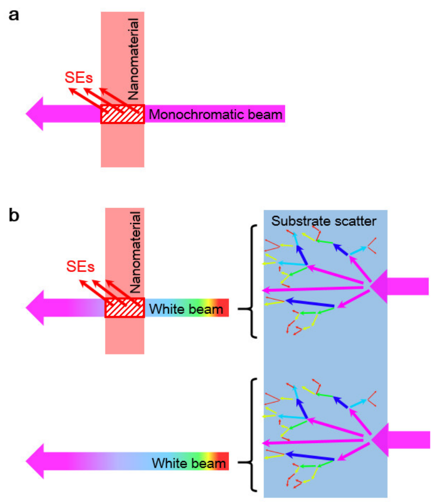

Fig. 1 Concept of the white-beam electron technique. **a**, The simplest transmission-mode experimental setup to study a nanomaterial. **b**, The two-point comparison measurement in transmission mode in which a substrate is used as a scatterer to produce a white electron beam.

electronic responses of nanomaterials such as electron–electron (e–e) interactions and electron transport, the first approach is to design an experimental setup in transmission mode with an electron probe, as shown in Fig. 1a. A high-energy monochromatic electron beam is used as an electron probe. Focusing on measuring the electron transmission of nanomaterials, only those properties at the energy of the incident beam can generally be obtained from a single measurement according to the signal attenuation of the elastic peak (zero-loss peak).

To investigate the electron transmission of a substrate-supported nanomaterial, we propose a new idea as shown in Fig. 1b. A monochromatic electron beam is injected into the back side of the substrate and corresponding secondary electrons (SEs) are produced as the incident electrons travel through the substrate. Therefore, the substrate acts as a scatterer, scattering a monochromatic electron beam to obtain dispersed electrons. The transmitted spectra emitted from the other side of substrate can be considered as a white electron beam to probe the properties of the attached nanomaterial. The initial energy distribution of these white electrons can be obtained from the control setup measuring only the substrate under the same experimental conditions. Therefore, comparing spectra obtained from these two different experimental setups allows the properties of the target nanomaterial over the whole energy range to be extracted from one pair of measurements.

This is the concept of white-beam electron technique, i.e. using a polychromatic beam composed of a mixture of energies (white electrons) to investigate the properties of a target nanomaterial, which more efficient than the basic transmission configuration.

3. Implementation of white-beam electron technique

In our previous work [1],the concept of white-beam electron technique has been applied to extract quantitative information about nanomaterials without influence from substrate signals even at low energies, leading to virtual substrate method. Here, we propose another way to implement the white-beam electron technique in SE microscopy and apply it to investigate multilayer graphene (>10 L) on a patterned SiO_2 substrate, as shown in Fig. 2.

Instead of polycrystalline Au substrate in Ref [1], a hole-patterned SiO_2 substrate was used to implement the white-beam electron technique. Four spectra measured for flat and hole regions of the SiO_2 substrate without and with a covering graphene layer are denoted as S(A), S(H), N(A) and N(H), respectively. The selected measurement points [S(A), S(H), N(A) and N(H)] are indicated in the SEM image of this graphene/SiO_2 sample in the inset of Fig. 2. The resulting transmitted spectra are shown in Fig. 2 for the energy range of 0–50 eV. A plateau structure (12–20 eV), which is related to π+σ plasmon excitation of multilayer graphene, can be clearly observed in the transmitted spectra.

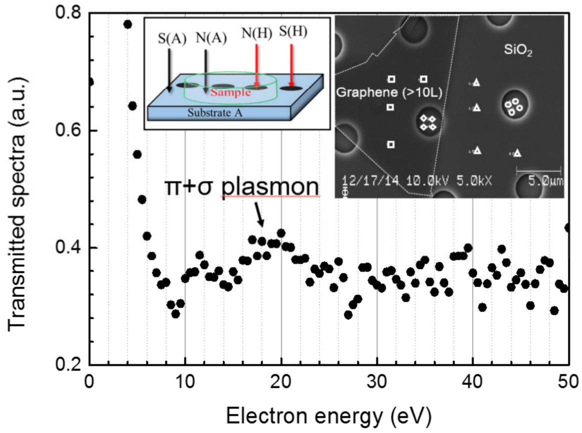

Fig. 2 Transmitted spectra of graphene obtained from the graphene/SiO_2 sample. The left inset shows the implementation of a virtual substrate measurement using a patterned SiO_2 substrate. The right inset shows the SEM image of the graphene/SiO_2 sample. The selected measurement points are marked as ○, Δ, ◊, and □ representing S(H), S(A), N(H) and N(A), respectively.

Using this white-beam electron technique in secondary electron (SE) microscopy, we successfully tracked and controlled a SE spectrum (white electrons) associated with the reflectivity difference between two different substrates to quantitatively investigate the covering nanomaterial based on subtle changes in the transmission of the nanomaterial with high efficiency rivalling that of conventional core-level electrons.

4. Conclusion

In summary, the white-beam electron technique represents a benchmark to make the use of 'white electron' in nanomaterial characterization, which in principle, can be easily implemented in many more reflection configuration techniques than surface analysis techniques and does not demand extra investment in equipment.

4. References

[1] B. Da, et al. Nature Commun. 8 (2017) 15629.