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Recent progress in scanning electron microscopy for the characterization of fine structural details of nano materials

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A B S T R A C T

Research concerning nano-materials (*metal-organic frameworks* (*MOFs*), *zeolites*, *mesoporous silicas*, *etc.*) and the nano-scale, including potential barriers for the particulates to diffusion to/from is of increasing importance to the understanding of the catalytic utility of porous materials when combined with any potential super structures (such as hierarchically porous materials). However, it is difficult to characterize the structure of for example *MOFs* via X-ray powder diffraction because of the serious overlapping of reflections caused by their large unit cells, and it is also difficult to directly observe the opening of surface pores using ordinary methods. Electron-microscopic methods including *high-resolution scanning electron microscopy* (*HRSEM*) have therefore become imperative for the above challenges. Here, we present the theory and practical application of recent advances such as through-the-lens detection systems, which permit a reduced landing energy and the selection of high-resolution, topographically specific emitted electrons, even from electrically insulating nano-materials.

Contents

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1. Introduction

For research concerning *nano-materials*, it is necessary to observe the morphology and composition of samples. At present, scanning electron microscopy (SEM) is heavily used in the pursuit of the further understanding *nano-materials,* because of the recent significant improvements in SEM for imaging, diffraction and elemental analysis in terms of spatial resolution and sensitivities [1,2]. Here, we use the word "*nano-material*" to represent a material that exhibits characteristic features in its physical and chemical properties that have origins in the material's structure at the nanoscopic level. In this review article, recent improvements in SEM and their effect on *nano-material* characterizations and their dependence upon the use of low landing energies are presented.

1.1. The basic principles of SEM

An SEM demagnifies an electron beam that is produced by a source into a probe which scans across the surface of a sample in raster fashion (Fig. 1a). The interaction between the sample and the electron probe (*impacted electrons – IE*) produces various types of *emissions,* which are captured by different detectors placed in appropriate positions.

Morphological/topological-contrast and compositional information are separately obtained by selecting specific types of emitted electrons, known as *secondary electrons* (*SEs* – with energies smaller than 50 eV) and *backscattered electrons* (*BSEs* – with energies larger than 50 eV), respectively; see Fig. 1b. Further compositional information is obtained through the detection of characteristic X-rays using an X-ray detector.

While *transmission electron microscopes* (TEMs) remain the most-used type microscopes for the characterization of crystal defects at the atomic scale, SEMs

1. do not require extensive sample preparation and yet may produce similar results through the detection of *BSEs* with selected angles;
2. retain a much large depth of field (wide observable range in *z*-axis with acceptable resolution) allowing a considerable amount