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Development of advanced electron tomography in materials science based on TEM and STEM

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Abstract: The recent developments of electron tomography (ET) based on transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) in the field of materials science were introduced. The various types of ET based on TEM as well as STEM were described in detail, which included bright-field (BF)-TEM tomography, dark-field (DF)-TEM tomography, weak-beam dark-field (WBDF)-TEM tomography, annular dark-field (ADF)-TEM tomography, energy-filtered transmission electron microscopy (EFTEM) tomography, high-angle annular dark-field (HAADF)-STEM tomography, ADF-STEM tomography, incoherent bright field (IBF)-STEM tomography, electron energy loss spectroscopy (EELS)-STEM tomography and X-ray energy dispersive spectrometry (XEDS)-STEM tomography, and so on. The optimized tilt series such as dual-axis tilt tomography, on-axis tilt tomography, conical tilt tomography and equally-sloped tomography (EST) were reported. The advanced reconstruction algorithms, such as discrete algebraic reconstruction technique (DART), compressed sensing (CS) algorithm and EST were overviewed. At last, the development tendency of ET in materials science was presented.

Key words: electron tomography; materials science; transmission electron microscopy; scanning transmission electron microscopy

1 Introduction

TEM is an important tool that can provide valuable information about the microstructure and chemistry of materials at nanoscale. However, TEM images are only two-dimensional (2D) projections of three-dimensional (3D) objects, and therefore these images provide only partial information, even erroneous information in some case because of lack of depth resolution. To understand accurately the relationships between the structures and the properties, it is essential to view directly in 3D, especially for materials with complex morphology and chemistry.

More recently, electron tomography (ET) has been successfully applied to 3D reconstruction of nanostructures with the morphologies and chemical compositions. Although the mathematical base for tomographic techniques had been established in 1917 by RADON [1], it was firstly applied to ET in biological sciences in 1968 [2,3]. With the advent of the novel tomographic imaging modes, the automation of microscope control, the optimized tilt series and the advanced reconstruction algorithms, ET has acquired revolutionary development, and has been widely used in the field of materials science in the past several decades.

According to imaging modes of ET, the types of ET based on TEM and STEM include BF-TEM tomography [4−8], DF-TEM tomography [9], WBDF-TEM tomography [10−12], ADF-TEM tomography [13], EFTEM tomography [14−19], HAADF-STEM tomography [20−28], ADF-STEM tomography [29,30], IBF-STEM tomography [31], EELS-STEM tomography [32−34], XEDS-STEM tomography [35−37], and so on. Lately, ZEWAIL [38] and KWON and ZEWAIL [39] have pioneered four-dimensional (4D) ultrafast electron microscopy (UEM) with spatial and temporal resolution, which makes dynamics ET implement.

ET mainly consists of three steps: tomography data acquisition, tomography alignment and reconstruction, and tomography visualization. In order to be suitable for tomography, all image signals along the tilt series must strictly meet the projection requirement, namely the recorded signals must be a monotonic function of some

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physical properties of the object.

The aim of this review is mainly firstly to summarize the novel tomographic imaging modes, then to introduce the optimized tilt series and the advanced reconstruction algorithms, and finally to present the further developments of ET.

2 Novel tomographic imaging modes

2.1 Based on TEM

2.1.1 BF-TEM tomography

BF-TEM tomography is suitable for amorphous materials, where mass thickness contrast is dominant. A few successful examples were reported, such as studying the location and distribution of metal (oxide) particles in zeolites and catalyst materials in 3D, and the location and the connectivity of pores in mesoporous materials [4−8]. Figure 1 shows the 3D reconstruction of an Au/SBA−15 model catalyst particle, revealing the size and location of Au particles inside the support material clearly [8]. However, for crystalline materials, BF-TEM images are mainly dominated by diffraction contrast that is highly sensitive to the direction of the incident beam. Therefore, it is very difficult to obtain BF-TEM images with clear contrast through the entire tilt range. In general, BF-TEM tomography is unsuitable for crystalline materials.

2.1.2 DF-TEM tomography

In general, DF-TEM images do not fulfill the projection requirement because the image intensity varies rapidly in a complicated manner with sample orientation. It is very difficult to acquire a tilt series of DF-TEM images of a crystalline specimen for tomography. Fortunately, KIMURA et al [9] have successfully reconstructed the $D1_a$-ordered Ni_4Mo precipitates in Ni–Mo alloy by DF-TEM tomography, and introduced how to obtain a tilt series of DF-TEM images of the Ni_4Mo precipitates. Firstly, a systematic row containing the $D1_a$ superlattice reflection at (4/5, 2/5, 0) was parallel to the tilt axis of the holder by placing carefully the specimen on the specimen holder, which made the $D1_a$ superlattice reflection exist in the tilt series from −60° to +60° (see Fig. 2(a)). Secondly, by adopting the (4/5, 2/5, 0) superlattice reflection, a tilt series of DF-TEM images of the Ni_4Mo precipitates were recorded (see Fig. 2(b)). Finally, the 3D shape and position of the Ni_4Mo precipitates were reconstructed (see Fig. 2(c)).

2.1.3 WBDF-TEM tomography

Although coherent diffraction contrast seems to violate the projection requirement, WBDF-TEM tomography has recently been proposed to investigate the 3D distribution of dislocation network in GaN film [10,11] and dislocation-precipitate interactions in an

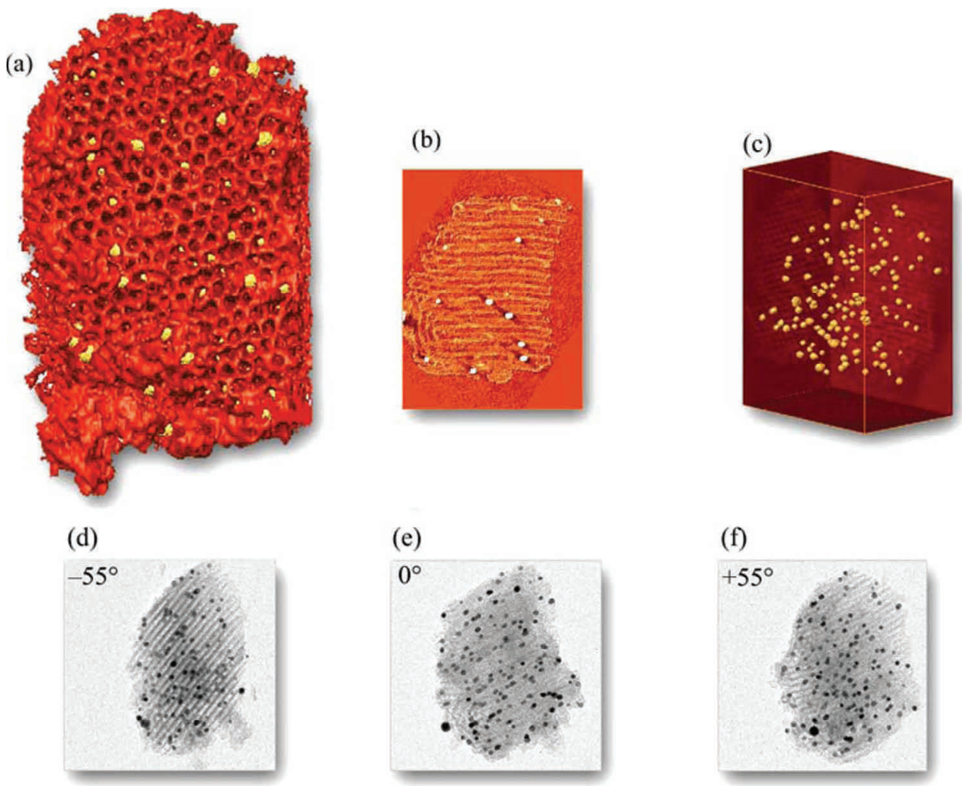

Fig. 1 Au catalyst nanoparticles inside SBA-15 mesoporous support (a), surface rendering of slice (b) from (a), surface rendering of Au nanoparticles only (c), BF images at −55° (d), 0° (e) and +55° (f) [8]