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Electron holographic tomography

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ABSTRACT

The exact knowledge about intrinsic electrostatic potentials and in particular their three-dimensional distribution at the nanometer scale is a key prerequisite for understanding the solid state properties. Electron holographic tomography (EHT), the combination of off-axis holography with tomography in the transmission electron microscope, provides a unique access to this information. We review the development and application of automated EHT to reconstruct 3D potentials in nanostructures such as the mean inner potential of a material or the diffusion potential across p–n junctions in semiconductors. We also discuss future challenges of the 3D reconstruction of electric crystal potentials at atomic resolution and magnetostatic fields as well as ways to overcome present limitations of the method.

1. Introduction

Transmission electron microscopy facilitates a two-dimensional (2D) mapping of material properties at the nanometer down to atomic scale. Therefore, this technique is an indispensable tool for the modern materials science and solid state physics. In order to obtain valuable information about the specimen, the imaging techniques used in the transmission electron microscope (TEM) should provide quantitative and reproducible results preferably in three dimensions. In particular, the three-dimensional (3D) distributions of intrinsic electric and magnetic fields within nanostructures are of high interest, because they play a crucial role for understanding the properties of emerging materials. Electron holographic tomography (EHT), i.e. electron tomography combined with off-axis electron holography, uniquely provides access to this information. In the following we mainly address the 3D reconstruction of electrostatic scalar potentials, whereas the corresponding magnetic vector field reconstruction is still in an early stage of development.

1.1. Off-axis electron holography

Off-axis electron holography (EH) quantitatively maps electrostatic and magnetostatic fields at the nanometer scale in two dimensions [1–5]. For example, it has been successfully applied for mapping diffusion potentials ("built-in" voltages) in semiconductors [6–9], for measuring mean inner potentials (MIPs) of various materials [10,11] and for characterizing charged dislocations [12]. Further it has been utilized to study the remanent magnetic field in thin films [13,14], or the collective behavior in magnetic nanoparticles [15]. The resolution of off-axis EH is ultimately given by the information limit of the TEM [16], which also enables materials analysis at atomic resolution [17,18]. In contrast to conventional imaging where only the object-modulated intensity (i.e. the absolute square of the electron wave) is detected, EH allows to reconstruct the full complex image wave in both amplitude and phase. This image wave is encoded in an electron hologram, the interference pattern formed by superposition of object wave and object-free (reference) wave. Under medium resolution imaging conditions typically used for tomographic experiments, the image wave corresponds largely to the (aberration-free) object exit wave, because lens aberrations have virtually no influence at the nanometer scale. Fig. 1a schematically illustrates the acquisition of an electron hologram in the TEM: The sample (object) is placed such that the electron wave propagates partly through the object and partly through vacuum. This leads to a modulated object wave and an unmodulated reference wave in the object exit plane. Both partial waves are imaged by the subsequent objective lens and deflected towards each other by a positively charged electrostatic biprism [19] yielding an interference pattern in the first image plane. The latter is magnified further by the projective lens system and finally recorded with the CCD camera. Fig. 1b shows a representative electron hologram of a Latex sphere shadowed with gold particles at the edge of a thin carbon support film. The fringe spac-

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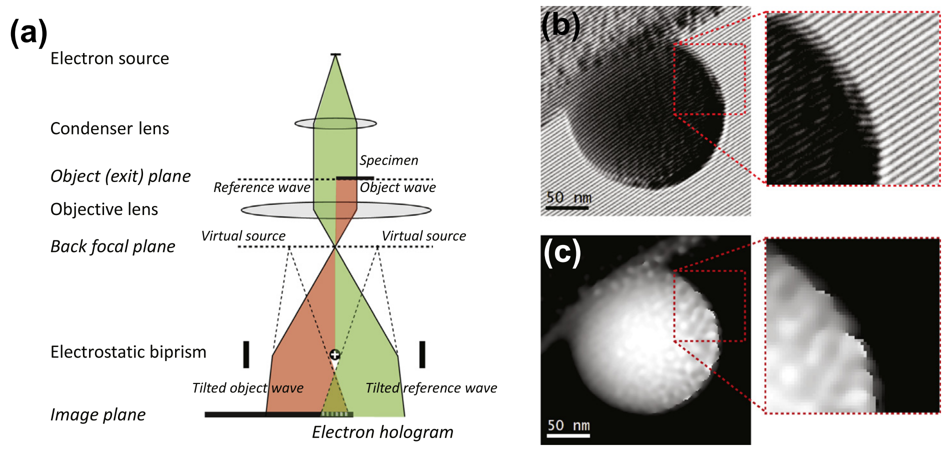

Fig. 1. Principle of off-axis electron holography. (a) TEM setup for acquisition of an off-axis electron hologram: By placing the specimen sideways from the optical axis and using a Möllenstedt biprism, object and reference wave can be brought to interference. (b) Representative electron hologram of a Latex sphere shadowed with gold particles. (c) Phase image reconstructed from (b).

ing in this hologram is 4 nm. From this it follows for the object exit wave reconstructed from the hologram by Fourier filtering a resolution of about 8 nm. The Phase image of the object exit wave is shown in Fig. 1c. More detailed information about acquisition and reconstruction of an electron object wave is described elsewhere, e.g. in Refs. [1,20].

Within the phase-grating (or phase-object) approximation (PGA) [21], field free vacuum and a particular potential gauge, the phase shift $\varphi_{obj}(x,y)$ in the object plane (x,y) with respect to the reference wave is expressed as the line integral along the electron path in z-direction by

$$\varphi_{obj}(x,y) = \int_{-\infty}^{+\infty} \left(C_E\, V(x,y,z) - \frac{e}{\hbar} A_z(x,y,z) \right) \mathrm{d}z, \tag{1}$$

with the interaction constant $C_E = \gamma m_0 e \lambda / \hbar^2$. Here $\hbar$denotes the reduced Planck constant, m_0 the electron rest mass, λ the wave length, e the elementary charge, and γ the relativistic constant. For electrons accelerated to 200 keV kinetic energy the interaction constant is $C_E = 0.0073$ rad/Vnm. This means the 2D phase distribution is proportional to the projected 3D electrostatic scalar potential $V(x,y,z)$, and, for magnetic samples, also to the projected component $A_z(x,y,z)$ of the magnetostatic vector potential $\mathbf{A}(x,y,z)$ parallel to the electron beam. It is well understood that the PGA is only valid if the influence of dynamical interaction (effects), i.e. deviations from first order Born approximation, is reduced by tilting (orienting) the specimen out of a low-index zone axis (kinematic conditions). Recently, it has been proven by multi-slice simulations that the PGA is valid for rather thick (≈100 nm) and sufficiently homogeneous specimens as investigated at medium resolution, but slightly overestimates the measured phase shift by a few percent [22] under kinematic conditions. However, in non-kinematic (dynamic) conditions, the phase shift can differ from the PGA very strongly, especially for higher atomic number materials or by the presence of sharp crystallographic interfaces. Moreover, thicker specimens produce in most cases phase shifts larger than 2π, whereas the object exit wave contains phase shifts unique only within a range of 2π. This leads to the so-called phase wrapping problem referring to the occurrence of phase jumps in the image. For this reason, the phase jumps need to be removed with specific phase unwrapping routines [23–25] which becomes complicated if the phase image is under-sampled or too noisy.

1.2. Electron holographic tomography

The drawback of a single phase image (and of single electron micrographs in general) is that the local information about the specimen along the projected dimension is lost. Thus, the mapping of electric or magnetic potentials with EH is only possible, if additional information (or assumptions) about object thickness and homogeneity can be provided. This obstacle can be overcome by combining EH with electron tomography (ET) to electron holographic tomography (EHT) in order to reconstruct 3D phase maps from a set of 2D projections. In the following we concentrate on the 3D reconstruction of electrostatic potentials and discuss the problem of magnetic field reconstruction in Section 4.

The principle EHT is displayed in Fig. 2: First, a tilt series of electron holograms is acquired in the TEM while tilting the specimen under the electron beam at commonly ±70° and increments of 1–3°. Second, the electron waves are reconstructed from all electron holograms of the tilt series in amplitude and phase. Third, the tilt series of phase images (projected potentials) is used to reconstruct the 3D potential by tomographic techniques. In

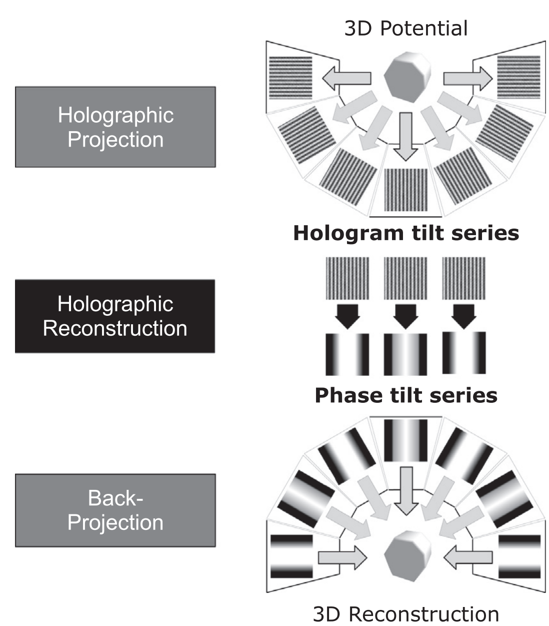

Fig. 2. Principle of holographic tomography.