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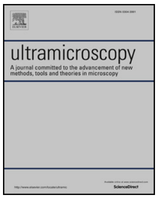

Full Length Article

Transmission imaging with a programmable detector in a scanning electron microscope[☆]

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ABSTRACT

A new type of angularly selective electron detector for use in a scanning electron microscope is presented. The detector leverages a digital micromirror device (DMD) to take advantage of the benefits of two-dimensional (2D) imaging detectors and high-bandwidth integrating detectors in a single optical system. The imaging detector provides direct access to the diffraction pattern, while the integrating detector can be synchronized to the microscope scan generator providing access to a real space image generated by integrating (pixel-by-pixel) a portion of the diffraction pattern as quantitatively defined by the DMD. The DMD, in effect, takes the place of the objective aperture in a transmission electron microscope (TEM) or an annular detector in a scanning transmission electron microscope (STEM), but has the distinct advantage that it can be programmed to take any shape in real time. Proof-of-principle data collected with the detector show diffraction contrast in samples ranging from a polycrystalline gold film to monolayer graphene.

1. Introduction

Electrons are sensitive probes of material structure due to their large scattering cross sections, short de Broglie wavelengths, and the relative ease with which they are generated and controlled with electron optics. Much of the information gleaned from an electron microscope is derived by isolating and detecting electrons that are scattered from a sample at a particular angle and/or with a particular energy. Developing detectors that selectively detect electrons based on their scattering direction and energy has been central to the evolution of electron microscopy to its present-day state as an immensely powerful materials characterization technique, capable of mapping the electronic and physical structure of materials from the atomic scale to the macroscale.

The scanning transmission electron microscope, first reported in 1938, scans a focused beam of electrons over a sample and records a portion of the electrons transmitted through the sample [1]. The first STEM image was recorded by exposing a photographic plate to the transmitted electron beam as the plate was mechanically translated beneath the sample. Since that time, STEM detectors have evolved significantly and are now widely available for the dedicated high-voltage STEM. The angular distribution of the transmitted electrons contains information about the crystallography, crystal orientation, defects, and mass-thickness, among other things. Selectively detecting portions of this angular distribution is necessary to generate images with a desired contrast mechanism. In practice, in TEM and STEM, a diffraction pattern will be observed early in an experiment in order to distinguish the various types of scattering that can be used to form a real space image.

Broadly, there are two main strategies for selecting what region of a diffraction pattern is integrated/detected to create a real space image in a TEM/STEM: (1) a physical aperture (or shaped detector) is placed in a plane approximately conjugate to the diffraction pattern to select electrons with particular scattering angles; (2) for the scanning beam technique, the full diffraction pattern is digitized for every beam position and virtual apertures can later be applied computationally. The former strategy has been employed for decades to great effect and forms the basis for nearly all TEM and STEM images reported to date [2–4]. The latter technique has become promising with recent imaging electron detector advancements [5–9]. Both strategies, however, have limitations. Physical apertures, while allowing significant control over the diffraction conditions contributing to an image, cannot take on arbitrary shapes. Imaging detectors, while allowing the user to specify arbitrary virtual masks [10,11], are relatively slow ($\approx 10^3$ frames/s), have not yet been commercialized, and/or are extremely expensive.

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STEM detection schemes have found their way into the scanning electron microscope (SEM) over the past several years, enabling what is now termed STEM-in-SEM or transmission SEM [12]. This detection mode has become increasingly popular because it enables the SEM to have many of the analytical capabilities previously available only in a TEM or STEM, for a small fraction of the financial investment. Examples include bright- and annular dark-field imaging [13] and transmission electron diffraction[14]. A further driver of their growing use lies in the fact that the relatively low-energy (typically, ≤ 30 keV) of the electron beam in the SEM carries with it some advantages for the characterization of materials. Knock-on damage in carbon substances is negligible [15], helping facilitate, for example, high-quality imaging of single graphene sheets [16]. The lower energy also leads to a significantly greater electron scattering cross-section, compared to that associated with ≥ 80 keV electrons, which means that greater information content is potentially available for analysis [16–18]. Despite the promise, however, the analytical potential of STEM-in-SEM has not yet been realized, due in part to the relative immaturity of STEM-in-SEM detector technologies. A practical problem specific to early forms of STEM-in-SEM imaging was the lack of post-specimen lenses in a SEM that would easily enable an angular selection strategy. A significant advance was recently made, however, through the introduction of inexpensive, easily-configured apertures that are placed immediately above a commercial STEM-in-SEM detector, as opposed to immediately beneath the specimen [13]. This strategy enabled angular selection over a wide range of scattering angles.

A new type of angularly selective electron detector for use in a SEM was recently described [19]. With this detector (Fig. 1), electrons forward-scattered through a sample strike a scintillator generating photons. These photons are optically imaged out of the vacuum chamber onto a digital micromirror device (DMD), a 2D array of mirrors that can be independently tilted up or down as specified by a computer [20,21]. The detector is currently operated in one of two modes. In ‘diffraction’ mode, the DMD reflects the full diffraction pattern to a CMOS camera to be digitized. In ‘imaging’ mode, the DMD is programmed to divert a precisely-defined portion of the diffraction pattern to a photodetector which integrates the incident photons. The output of the photodetector is synchronized with the microscope scan generator to create real space images. The DMD allows the detector to seamlessly take advantage of both a relatively slow imaging detector to generate a full-field diffraction pattern and a high-bandwidth integrating detector to generate real space images.[1]

In practice, the CMOS camera is used to capture a diffraction pattern from either a point or a region of the sample. Then a digital mask based on the information contained in that pattern is constructed, enabling the photodetector to generate a desired real space image. This mask is programmed to the DMD, which tilts each mirror towards the photodetector (or camera) as specified. When the electron beam is rastered across the sample the photodetector output is used to generate a real space image pixel-by-pixel. With this method, the microscope user can directly generate images identical to the virtual images generated from the 4D data sets, $I(x, y, k_x, k_y)$, of imaging detectors, but at SEM frame rates. Herein we describe an implementation of this detector, referred to as the programmable micromirror STEM (p-STEM) detector and demonstrate its use on different (poly)crystalline samples ranging ultrathin gold films to monolayer graphene.

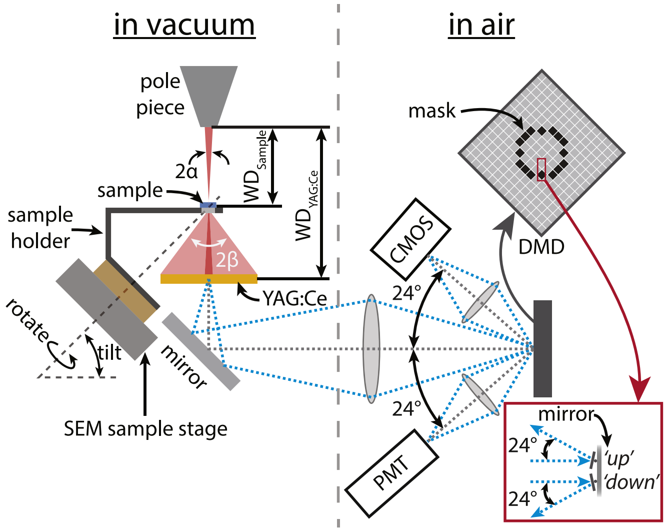

Fig. 1. Electron detector schematic. The SEM focuses a convergent beam of electrons onto a sample. Forward-scattered electrons strike a YAG:Ce scintillator which converts them into photons. The photons are imaged out of the vacuum chamber to a DMD. The DMD is then imaged to a CMOS camera or photomultiplier tube (PMT). The DMD is a (1024 × 768) array of individual mirrors that can be independently tilted to + 12° or − 12° degrees as directed by a binary mask generated via computer. When all mirrors are tilted upward in the figure, the CMOS camera digitizes the full diffraction pattern – this is referred to as ‘diffraction’ mode. When some mirrors are tilted down, the PMT integrates the photons and generates a voltage which is synchronized with the microscope scan generator and forms a real space image – this is referred to as ‘imaging’ mode.

2. Experimental

All imaging experiments were performed with a Zeiss/LEO Gemini 1525 field emission SEM operated at 30 kV accelerating voltage.[2] The user-selectable aperture (hereafter referred to as the condenser aperture) was selectable between 10, 20, 30, 60, 120, or 300 µm diameter and served as the primary control over the beam current and beam convergence angle. For reference, the typical measured beam current for each aperture was 30 pA, 120 pA, 270 pA, 1.1 nA, 4.4 nA, and 24 nA, respectively.

A five axis $(x, y, z, \theta_{tilt}, \phi_{rotate})$ stage was used for all sample positioning. A custom transmission sample holder mounted on a standard dovetail adapter allowed the sample to be positioned between the detector and the pole piece (Fig. 1). This sample holder design is unique in two ways. First, the sample holder orients the sample normal at a 45° angle with respect to the stage rotation axis coupling the stage rotation axis into a pseudo-tilt axis. Second, the center of the sample is aligned with the rotation axis ensuring that a point on the sample surface is eucentric with respect to the rotation axis. The present design roughly mimics a double tilt holder which is advantageous for diffraction imaging on crystalline materials [23,24].

The current p-STEM electron detector configuration is shown in Fig. 1[19]. A custom vacuum assembly with (x, y, z) translation capabilities positions a 100 µm thick, 12.7 mm diameter YAG:Ce scintillator crystal (Crytur) beneath the sample. A dielectric mirror located below the scintillator reflects the emitted photons through a lens system which images the scintillator crystal onto the DMD (Vialux, V-7000). The DMD is a 2D array of mirrors (1024 × 768) with 13.7 µm pitch. Each mirror can be independently tilted ± 12° with respect to the

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[1] During the course of this work, we discovered a detector described in 1979 by Cowley and Spence [22]. This detector shares many of the attributes of the detector described in the present work, but did not have the technological benefit of the DMD; instead, the authors proposed using tiny custom shaped mirrors tilted by hand to get a similar effect.

[2] Commercial instruments, equipment, or materials are identified only in order to adequately specify certain procedures. In no case does such an identification imply recommendation or endorsement by NIST, nor does it imply that the products identified are the best available for the purpose.