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Software electron counting for low-dose scanning transmission electron microscopy

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The performance of the detector is of key importance for low-dose imaging in transmission electron microscopy, and counting every single electron can be considered as the ultimate goal. In scanning transmission electron microscopy, low-dose imaging can be realized by very fast scanning, however, this also introduces artifacts and a loss of resolution in the scan direction. We have developed a software approach to correct for artifacts introduced by fast scans, making use of a scintillator and photomultiplier response that extends over several pixels. The parameters for this correction can be directly extracted from the raw image. Finally, the images can be converted into electron counts. This approach enables low-dose imaging in the scanning transmission electron microscope via high scan speeds while retaining the image quality of artifact-free slower scans.

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1. Introduction

Over the last years, aberration correction has enabled electron microscopy imaging with atomic resolution even at low acceleration voltages [1–7]. Low voltages suppress knock-on damage and allow to image e.g. low-dimensional materials with very high doses [8–15]. However, electron beam sensitive samples like organic and biological materials still suffer from radiolysis as a main damage mechanism [16–18], and even defects in low-dimensional materials still rapidly change their configuration under electron irradiation [8,9,19–22]. If the structure is not stable under irradiation, it is important to minimize the dose on the sample while extracting as much information as possible. Low-dose imaging techniques are commonly used for biological applications such as single-particle analysis, where the dose is distributed over many identical copies of an object and the desired information is obtained via a suited reconstruction algorithm [23–27]. With the same general idea (of distributing the dose over many identical objects), we have recently shown the reconstruction of defects in graphene from a set of low-dose images, recorded at two orders of magnitude lower dose than commonly used for scanning transmission electron microscopy (STEM) imaging [28,29]. Since the illumination parameters and the maximum allowed electron dose is dictated by the specimen, the detection of electrons must be optimized. For transmission electron microscope (TEM) imaging it has been shown that direct detection device (DDD) cameras can greatly improve image quality and reduce noise caused by the multiple conversions between light and electrons in regular detection systems like CCD cameras [8–11]. Also, DDD cameras can be operated in electron counting mode which further improves image quality under low-dose conditions [12,30]. So far, low-dose methods have mostly been implemented in TEMs, and therefore also the specialized detectors were all developed for TEM. However, also scanning transmission electron microscopes (STEMs) can be operated under low-dose conditions [28,31–33]. The operating conditions for low-dose STEM differ significantly from those for low-dose TEM, but the achievable doses are very similar [31,32]. For regular annular dark-field (ADF) imaging, direct electron detectors have not been used so far. The common technique for detecting electrons in ADF is a scintillator combined with a photomultiplier. Even though multiple conversion steps from electrons to photons and back are involved in the detection process, the system is still sensitive enough to detect single electron pulses [34]. This has already been shown for high-dose data, where the authors of Ref. [35] could extract the single-electron signal level from the histograms of STEM ADF images and use this information for quantitative data analysis. In contrast, in this paper we are presenting a method for low-dose conditions that is focused on fast image acquisition and processing. We also show that it is possible to correct for artifacts that occur at very high scan speeds and are caused by the finite decay time of the detection system [31].

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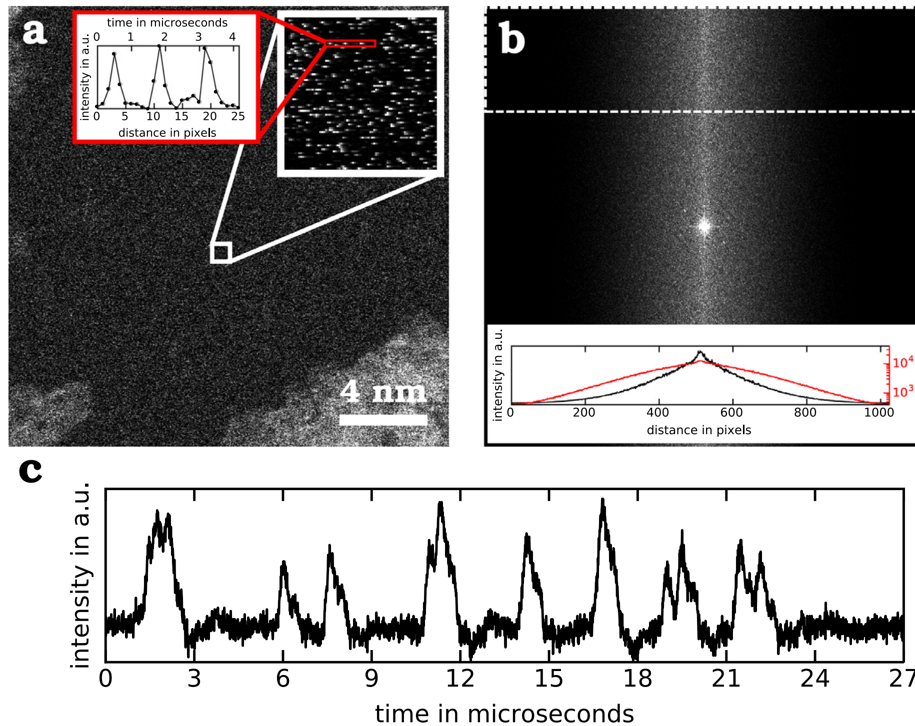

Fig. 1. (a) Low-dose image of monolayer graphene (~ 10,000 e/Å^2). Inset: Magnified part of the image and line profile across a couple of pixels. Both insets clearly show that the electron signals are spread out over several pixels. The line profile also includes a time axis to allow for an easy comparison with the oscilloscope trace in (c). (b) PS of the image in (a). The damping of high frequencies in horizontal direction is clearly visible. Inset: Line profile of the region within the white dashed rectangle. The signal shows a bilateral exponential decay towards high frequencies, which is confirmed by a plot with logarithmic y-axis (red line). (c) Oscilloscope trace of a raw ADF signal as extracted after the photomultiplier. Since the sampling rate is much higher, the electron peaks show more details, but the general shape is very similar to what is seen in the line profile of the STEM image. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

2. Low-dose STEM imaging

The simplest method for reducing the electron dose in a STEM is to reduce the pixel dwell time. The electron dose will then scale linearly with the dwell time while all other parameters that affect the achievable resolution and image quality should stay constant. In theory, only the noise level would be increased by this method. However, when going to extremely short dwell times in an experiment, a horizontal blurring appears in such images. This can also be seen in the fourier transform (FT) as a damping effect on the high frequencies in horizontal direction. The reason for this blurring is the finite decay time of the detection system consisting of a scintillator, photomultiplier and further readout electronics [34]. For our instrument, a Nion UltraSTEM 100, severe streaking and blurring start to be visible for pixel dwell times below ~ 0.5 µs, whereas above ~ 1 µs no such artifacts are visible. A solution for this problem is, as suggested in Ref. [31], to increase the pixel dwell time above the critical value of ~ 1 µs and decrease the emission current from the field-emission gun (FEG). Whilst being simple we think that this approach eliminates many of the advantages a fast scan can have. Just to name a few, a fast scan reduces distortions and jitter caused by sample drift and vibrations. Furthermore a fast scan also speeds up the whole acquisition procedure which is important especially for low-dose techniques, where the number of images acquired typically exceeds several hundreds up to several thousands. With the Nion UltraSTEM 100 for example, whose scan box is clocked with 6 MHz, the fastest possible scan speed is 167 ns/px. This is six times faster than using a dwell time of 1 µs where the artifacts of the finite decay time are not visible anymore. Besides that, changing the emission current and therefore the extraction voltage makes re-tuning of the microscope necessary when changing between low- and high-dose acquisition settings. Apart from the blurring in horizontal direction, fast scans can introduce further artifacts due to the limited response time of the scan coils. In particular, strong distortions may appear on the left-hand side of the images due to the discontinuity in the scan pattern between consecutive lines. This can be avoided by using a small delay between the lines (called ”flyback time” in the Nion software). We used a delay of 120 µs which safely avoided the distortions. The optimum delay will depend on the specific hardware, and is not further addressed here. However, one should be aware that this delay also adds additional dose to the sample, which will be localized on the left-hand side of each image.

3. Noise analysis

Fig. 1a shows a low-dose image acquired with maximum scan speed at the Nion UltraSTEM 100. The inset in Fig. 1a exemplifies how the single electron impacts are spread out over several pixels. This causes the envelope in horizontal direction of the power spectrum (PS) in Fig. 1b. A further analysis shows that the intensity is decaying, in good approximation, exponentially towards high frequencies in the horizontal direction (see inset in Fig. 1b). Since a bilateral exponential function is the Fourier transform (FT) of a Lorentzian, this suggests an approximate shape for the single electron impact peaks [36]:

$$FT\left(\frac{1}{\pi}\frac{2\gamma}{(x-x_0)^2+\gamma^2}\right)=e^{ix_0t-|\gamma|\omega} \quad (1)$$

As can be seen from the oscilloscope trace in Fig. 1c, those peaks have a slightly asymmetric shape with a longer tail on the decaying end, but due to their sparse sampling in the image this is not very pronounced and a Lorentzian seems to be good approximation. From the right side of Eq. (1) we can see that the offsets

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