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The Effect of a Pulsed Electron Beam on Damage Threshold

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The large doses delivered to specimens during high-resolution TEM work has led to renewed interest in fundamental beam-damage mechanisms, with efforts focused on overcoming associated experimental limitations. This is especially important when studying radiation-sensitive soft matter, such as organic and biological materials, where the Rose criterion stipulates that the resolution at which a material can be studied is limited by the critical dose, or the dose at which features of interest are destroyed [1]. The ionization damage mechanisms typical in these materials have aspects that proceed on a range of time scales, ranging from femtosecond-scale electronic relaxation of free radicals by crosslinking to nanosecond-scale decaying of phonons released by excited atoms returning to the ground state [2,3]. Importantly, many of these mechanisms proceed as a result of secondary electrons, and therefore the damage is not necessarily linearly proportional to the number of incident electrons [4]. As such, one of the methods for mitigating beam damage is to build up signal from a large number of independent acquisitions with a low signal-to-noise ratio. This method operates on the assumption that some of the excitations caused by such an ultra-low dose relax between each image acquisition [5,6].

Here, we present work intended to test the assumption that, given enough relaxation time and for a low enough dose rate, a statistically-significant amount of damage caused by an electron beam can be recovered or altogether avoided. If correct, one would expect to measure a higher characteristic dose (D_c) for the material under inspection. The experiments performed as part of this work aim to directly compare the D_c, defined here as the dose required to produce a ($1/e$) intensity decay of a particular Bragg reflection, from a continuous and a pulsed electron beam. The model material used in this study is the paraffin hexatriacontane ($C_{36}H_{74}$), which forms thin, orthorhombic single crystals oriented along the [001] direction upon drop-casting from solution onto a substrate [7,8]. The pulsed electron beam can be modulated over a range of repetition rates by using a femtosecond laser to generate discrete packets of photoelectrons from an unheated LaB_6 source. In this way, the duration between electron packets, the number of electrons in each packet, and, therefore, the beam current, can be delivered in a controlled-pulsed manner [9]. This additional control can be leveraged to allow significantly more time between electron delivery to the specimen compared to a continuous beam of the same current. Using the same current with two very-different dose-rate profiles allows us to test whether acquiring a series of low-dose images improves signal relative to using a more-conventional low-current continuous beam. In addition, the approach could be used to provide additional physical insight into the timescales associated with relaxation processes.

In order to account for the variety of systematic and random errors that are present in such measurements, and in order to control the large number of variables that may influence measuring D_c, a detailed statistical experimental design is employed. For example, attempt is made to account for variations in specimen preparation conditions, the crystal thickness is measured as a covariate, imaging conditions are controlled, and all other relevant factors are randomized. Practically, we measure the beam current and the illumination area, acquire a set of diffraction patterns over time from a specific crystal, and monitor the integrated intensities of the 110 Bragg spots in order to measure D_c (Figure 1).

By repeating this experiment on many individual crystals, statistical analyses can be performed and significance can be established when comparing the different dose-rate profiles [10].

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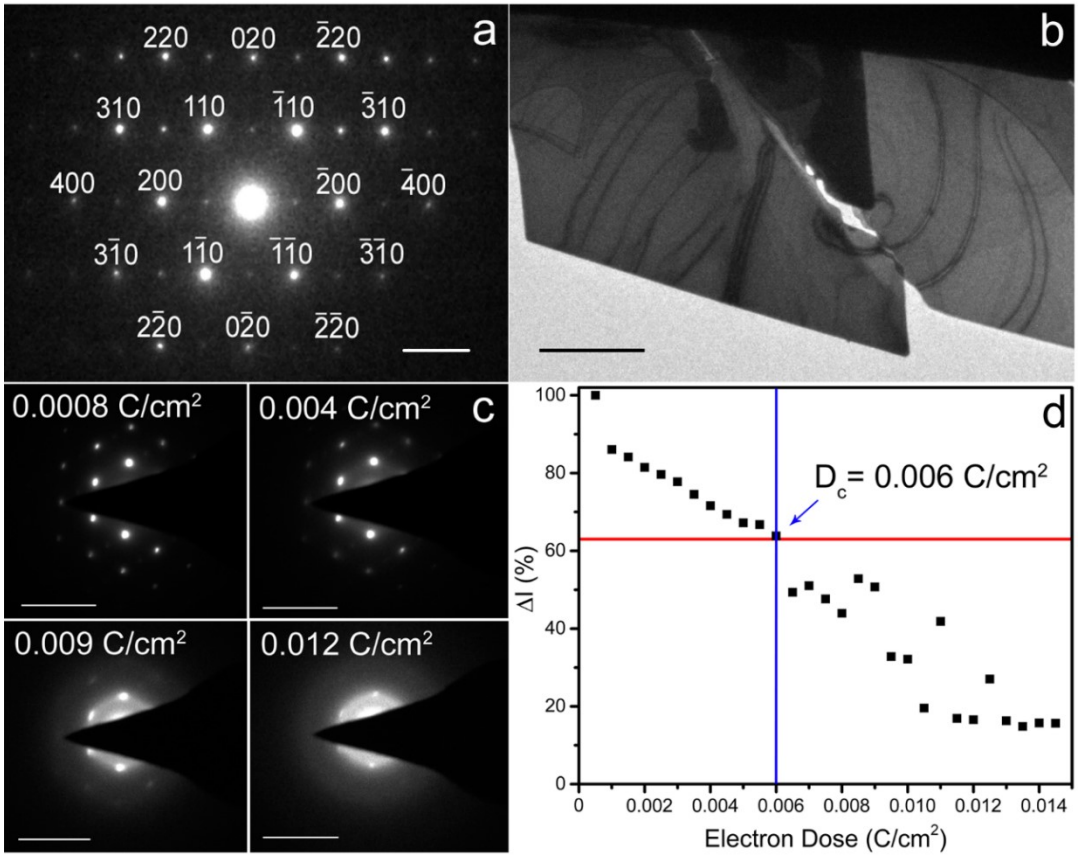

Figure 1. (a) Indexed [001] zone-axis diffraction pattern acquired with a camera length of 320 mm. Scale bar = 2 nm^{-1}. (b) Bright-field image of an orthorhombic $C_{36}H_{74}$ crystal acquired at 420x magnification. Scale bar = 5 µm. (c) Diffraction patterns from a 260 Å-thick $C_{36}H_{74}$ crystal upon exposure to a 12 pA thermionic beam current. The corresponding dose is labeled in the upper-left corner of each frame. Scale bars = 5 nm^{-1}. (d) Relative average intensity of the Bragg reflection arising from the <110> family of planes versus total accumulated electron dose, with D_c indicated. Data was calculated from the series of patterns shown in (c).

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