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Ways to Suppress Electron Beam Damage Using High-Speed Electron Beam Control by Electrostatic Shutter in Sample Observation and Analysis

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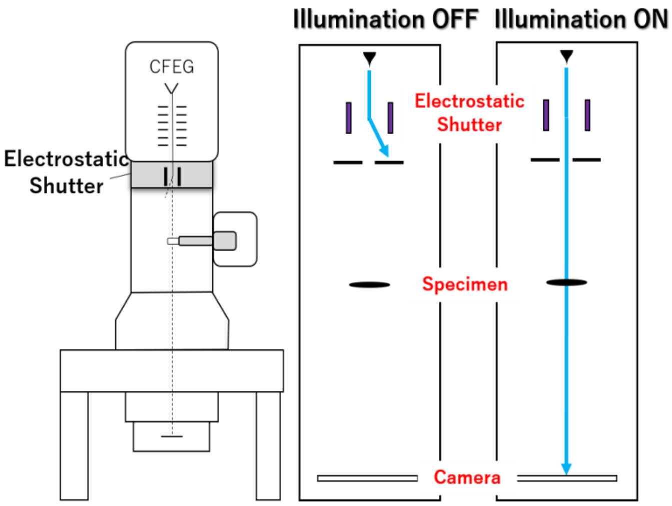
Recently, there has been increasing demand for observation and analysis of electron beam sensitive samples. Typical examples are zeolites and MOFs, which could be damaged with beam currents of only a few pA in scanning transmission electron microscopy (STEM), making the observation very challenging. The recently-introduced OBF (optimum Bright Field) STEM method is effective for high-contrast imaging at low dose condition [1], however, it can only be used for imaging and ineffective for spectral analyses with EDS and/or EELS, since the signals of these are basically proportional to the beam current. This highlights a need for general methods of reducing sample damage with smaller beam current.

To realize this challenge, we introduce a new instrumentation called EDM (Electrostatic Dose Modulation), which is a fast beam blanking system with an electrostatic shutter (Fig. 1) above sample. Since the electrostatic shutter is installed between the electron gun and the first magnetic field lens, the electron beam current can be controlled with no affections to the optical conditions. Our EDM allows a beam to have its switching speed less than 100 ns, and the rapid beam blanking can be synchronized with other systems including the microscope's scanning systems. Therefore, by rapid turning on and off of the electron beam with variable pulse duty ratio, the EDM makes it easy to adjust the time-averaged beam intensity with no need for refocus or realignment even for STEM, whose dwell time is several tens of micro second.

Next, we will introduce another method for reducing electron beam damage using EDM. Figure 2 shows the experimental results not using and using TAS, which is explained followingly. In a scanning beam microscope, every scan has a beam flyback to return the beam to the horizontal and/or vertical starting point. In the flyback term, a sample is irradiated with a dose which is no use in imaging and analysis wastefully. The TAS (True Area Scan) turns off the beam during the flyback term using the EDM system and reduces electron beam dose to the sample.

Also, depending on the dominant damage mechanism (such as knock-on or heating) for the sample in question, EDM can potentially reduce sample damage per electron beam dosage by an intermittent beam, which gives time to the sample for recovering. Figure 3 shows an experimental result using a $SrTiO_3$ sample to show how electron beam damage depends on whether the beam is continuous or intermittent. The behavior of EDS count decay on accumulated dose (frames) is taken as a measure of sample damage with mass loss. The mass-loss rate was greatly reduced from the case of (a) to case (b), where a continuous beam of 203 pA was used in (a) and an intermittent beam of 387 pA in (b), whose duty and cycle were 50 % and 80 μs. Since the dwell time per a pixel was 10 μs, the sample was scanned striped, where the beam was then turned on for 4 pixels and off for the next 4 pixels. Thus we showed, our experimental result shows several ways for suppressing sample damage utilizing the EDM in both imaging and analysis.



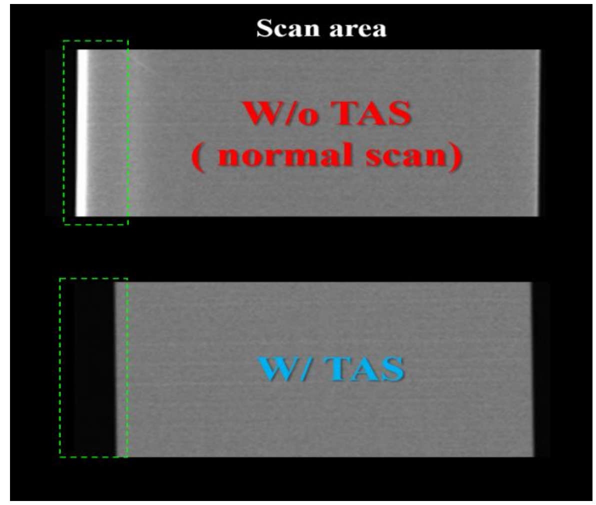


Figure 1. Schematic diagram of EDM system

Figure 2. The images taken with a camera of scanning (a) not using TAS (True Area Scan system), (b) using TAS.

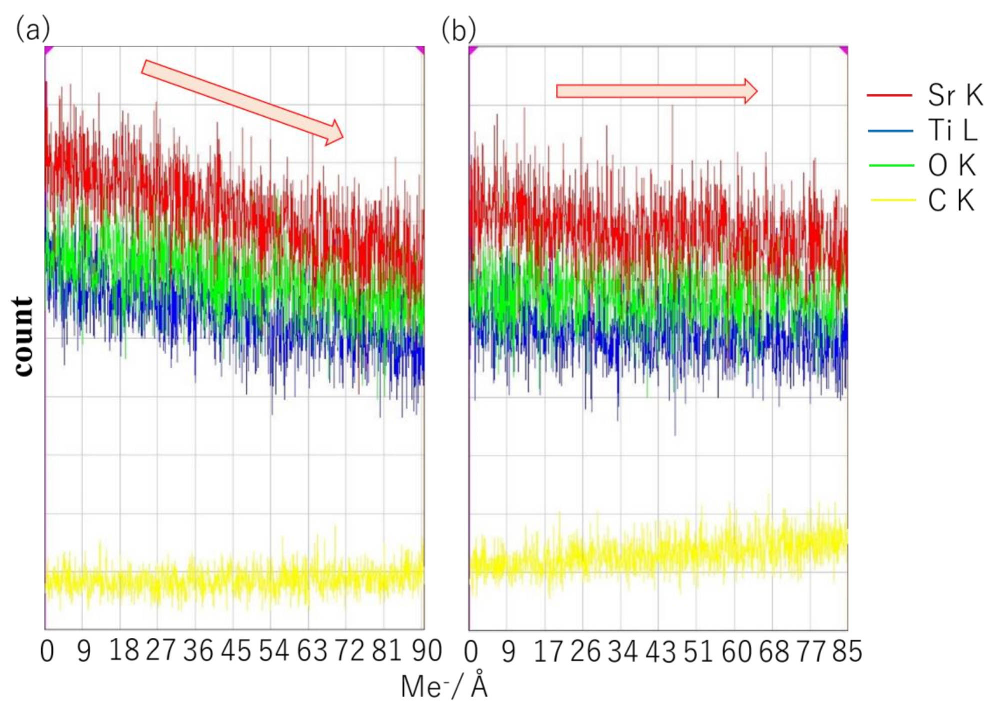


Figure 3. X-ray counts of elements, composing a sample, in a frame obtained by EDS analysis. The beam currents are 203 pA in (a) of continuous beam, and 387pA in (b) of intermittent beam.

References:

[1] K Ooe et al., Ultramicroscopy **220** (2021), p.113133.