Time-resolved TEM observation of CeO2 surface with electrostatic sub-framing system

Kanako Noguchi[1], Kazuki Yagi[2], Yuki Ninota[1], Dr. Takeshi Kaneko[3], Dr. Takeo Sasaki[1], Dr. Bryan W. Reed[2]
[1]JEOL Ltd, 3-1-2 Musashino Akishima, Japan, [2]IDES Inc., 4670 Willow Road, Suite 100, Pleasanton, USA, [3]JEOL USA Inc., 11 Dearborn Road, Peabody, USA

Time-resolved high speed TEM imaging technique is of great importance for in-situ observations such as heating/cooling, biasing, stressing, and liquid-cell experiments. Recently camera's frame rate is getting faster, but to dynamically visualize a structural change process of materials happen at a high speed, it is necessary to record TEM micrographs with a higher frame rate in the order of sub-millisecond.

Electrostatic type deflector can deflect the electron beam in tens of nanoseconds to microseconds. To using this property, there is a possibility to realize a high-speed imaging technique. In this study, we demonstrate a result of feasibility test for this system, electrostatic sub-flaming system developed by IDES Inc. This method improves temporal resolution by effectively increasing the frame rate while maintaining the same performance of the existing camera.

Fig. 1 shows schematic illustration of experimental setup. Electrostatic electron beam deflectors were installed beneath a projector lens cross-over in TEM column with a CMOS camera. The deflector itself acts as an aperture, limiting the area of the camera's sensor to which the electron beam is exposed, thus producing a small TEM image (sub-framed TEM image). By electrostatically deflecting the sub-framed TEM image at high speed in front of the camera sensor during exposure time, the images are laid out like 5x5 or 7x7 tiles. By doing this, each sub-framed image having a different time stamp. The difference between the timestamps of each sub-frame image is the time resolution.

Using this system, we observed time-series changes in (111) surface facet structure of CeO2 nanoparticles [1]. The data was recorded in 7x7 sub-frames. Since 49 sub-frames are recorded in one frame (CMOS Camera's exposure time is set to 40 ms), the exposure time per single sub-frame image is ~0.82 ms. The frame rate increased 25 fps to 1,225 fps. The 1st (0 ms) and 6th (4.08 ms) sub-frame TEM images are shown in Fig. 2. The atomic columns indicated by the arrows can be clearly seen in the 1st image, but are obscured in the 6th image. Motion of the atomic column of CeO2 (111) surface ware successfully observed in atomic level with a time scale of 4 ms by enhancing an effective frame rate of camera acquisition.

Graphic:

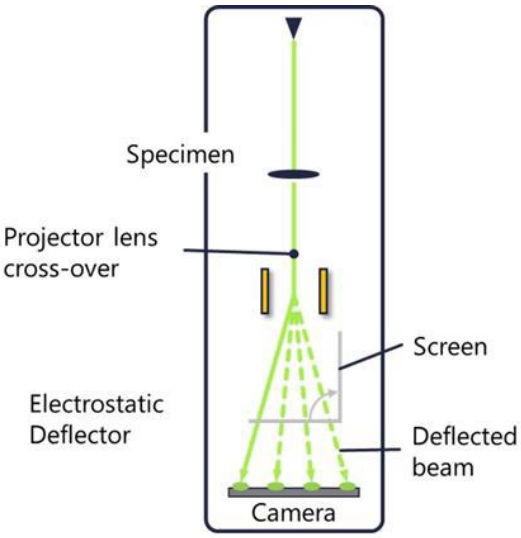

Fig. 1: Schematic illustration of electrostatic sub-flaming system installed on JEM-ARM200F

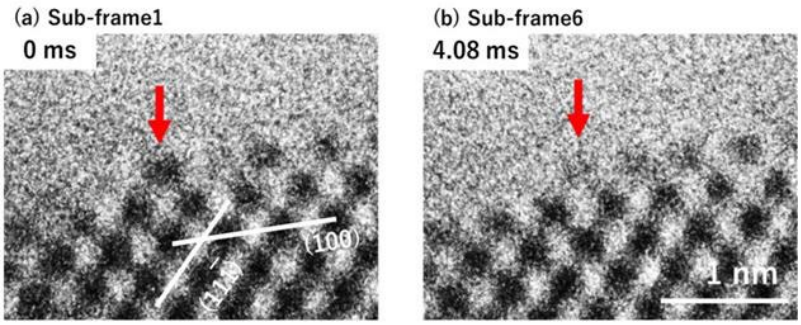

Fig. 2: Time-resolved TEM images of the surface of CeO_2 nanoparticle.
An atomic column of arrow-headed in (a) became obscured (b) after 4 ms.

Keywords:

High-speed-imaging, High frame-rate, Sub-framed imaging

Reference:

[1] CeO2 Nano particles specimen: Courtesy of Johnson Matthey