

## D-STEM: A Parallel Diffraction technique applied to Nanomaterials

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**Abstract** – A technique called D-STEM has been developed using a JEOL 2010F TEM/STEM instrument to obtain spot electron diffraction patterns in STEM configuration from nanostructures as small as ~3nm. An optimized ray path enables the formation of a 1-2 nm parallel probe scanned over the specimen to obtain a bright-field or dark-field STEM image. The beam is controlled, translated and subsequently positioned on the image at the feature of interest, while the diffraction pattern is recorded on a CCD camera.

In the D-STEM mode, the Free-Lens control feature in the instrument has been explored to optimize the ray path (Fig.1a), such that well-defined spot diffraction patterns can be obtained. In terms of electron optics, the major distinction between a conventional STEM configuration (C-STEM) (Fig.1b) and D-STEM is the use of a strongly excited condenser mini-lens (CM = 8.06 V) in the D-STEM mode; while in C-STEM, the CM is deactivated. In C-STEM mode, the parallel beam following C3 lens sets up a virtual source at an infinite distance for the objective pre-field lens, thereby resulting in a sharply converged probe at the specimen. On the other hand, in D-STEM mode, the optimized C3 and strongly excited CM create a demagnified point source of illumination at the front focal plane of the pre-field of the objective lens, resulting in a parallel beam on the specimen. The CM lens, operated at maximum excitation, together with a stronger demagnification of the electron source (C1 lens), produces a parallel beam with 1-2 nm spot size. The smallest condenser aperture of 10 $\mu$ m is employed to reduce the convergence angle and obtain sharper maxima at the back focal plane of the objective lens. Alignment in C-STEM is performed using a Ronchigram, while in D-STEM, the alignment procedure is essentially different. The low convergence angle of the beam (<1mrad) is unsuitable for observing the Ronchigram. Furthermore, the presence of an excited CM introduces distortions due to three-fold astigmatism, which are difficult to rectify using a Ronchigram [1]. Therefore, voltage-center alignment is carried out to position the electron beam along the optical axis of the objective lens. In summary, to set up a D-STEM mode capable of acquiring spot diffraction patterns from individual nanostructures, as small as ~3nm, the instrument requires a STEM bright-field or dark-field detector, FastEM<sup>TM</sup>, Free-Lens control, a small condenser aperture, and a bottom mounted CCD camera. An integration of this technique with Gatan<sup>TM</sup> software “STEM Diffraction Imaging” would automate the process and combine the rich information of electron diffraction with the spatially resolved power of spectrum-imaging, thereby enabling a fast pixel-by-pixel acquisition of diffraction patterns as a 4D data-set.

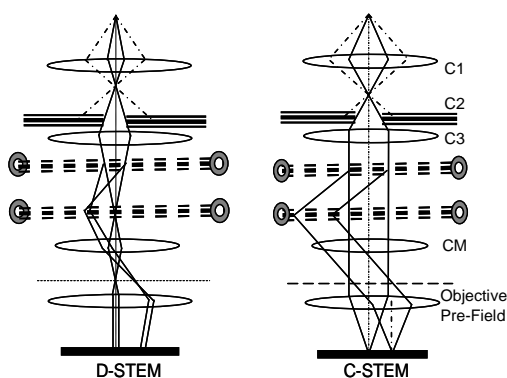


Fig 1. Schematic ray diagram showing  
(a) D-STEM (b) C-STEM

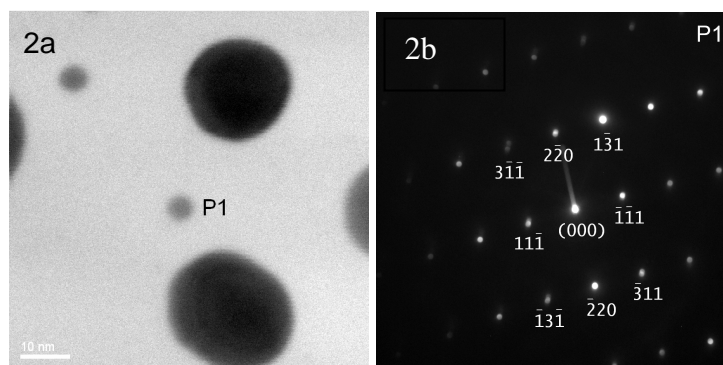


Fig 2 (a) Bright-field STEM image of Ag nanoparticles; (b) Diffraction pattern of nanoparticle P1 (~4nm in size) along the [112] beam direction

### References

[1] Voyles PM, Muller DA. Ultramicroscopy, 2002, 93, 147.