THE FIRST 300 keV DOUBLE C_S-CORRECTED AND MONOCHROMATED SUPERSTEM: APPLICATIONS IN MINERALOGY AND EXTRATERRESTRIAL MATERIALS.

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Introduction: An FEI high-base Titan 80-300 (scanning) transmission electron microscope (S/TEM) partially funded by NASA's SRLIDAP program has been installed and is now operational at Lawrence Livermore National Laboratory. It is the fist analytical SuperSTEM that has an X-twin pole piece and a Schottky FEG-gun operating at up to 300 kV, and combines double spherical aberration (Cs) correctors (one for probe and one for imaging) and monochromator in one instrument. It is also equipped with a high-resolution energy filter (GIF 866 Tridiem-ERS), a high-resolution annular dark-field detector (HAADF) and a high collection efficiency solid-state x-ray detector (EDAX/Genesis 4000). The Cs correctors (CEOS) employ pairs of hexapoles and round doublet transfer lenses, by which it is possible to correct all aberrations out to third-order. The instrument is configured to be fully remote controlled, which ensures the microscope itself is always kept in a stabile environment. Instrument performance is reviewed and examples of applications described.

Performance Evaluation: The SuperSTEM has been aligned at both 200 keV and 300 keV. All performance functions examined meet and exceed the instrument specifications. Figure 1 shows a fast Fourier transform (FFT) of a Young's fringe experiment on a cross-grating sample (gold particles are supported on amorphous carbon film) in TEM mode at 300 keV, from which the information limit of the microscope can be determined. It is obvious that the Young's fringes extend to 0.7 Å and some reflection spots can be clearly identified at 0.6 Å and even 0.56 Å. The spatial resolution of Cs-corrected STEM mode was examined by using a detector grade (Cd,Zn)Te (CZT) sample that has the zincblend structure with lattice parameter of a = 6.41 Å. The TEM sample was prepared by focused ion beam (FIB) technique using an FEI Nova 600 DualBeam FIB-FESEM instrument. Figure 2 is a low-pass filtered HAADF HR-STEM image of CZT in the [112] orientation. Characteristic (Cd,Zn)-Te "dumbbells" are clearly resolved in Fig. 2, indicating sub-Å resolution. The monochromator on the SuperSTEM is a single Wien filter design. The energy resolution of EELS achieved so far, with monochromator on, is 0.18 eV at 300 keV (Fig. 3).

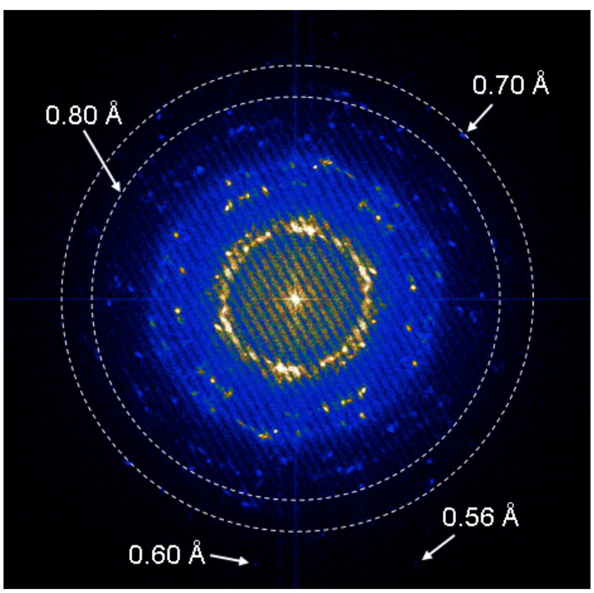

Figure 1: FFT of Young's fringe experiment from Au particles on amorphous C. Pattern shows the resolution limit after objective lens correction. The inner dash-line circle corresponds to 0.08nm, the outer circle to 0.07nm. The spots can reach to 0.056nm.

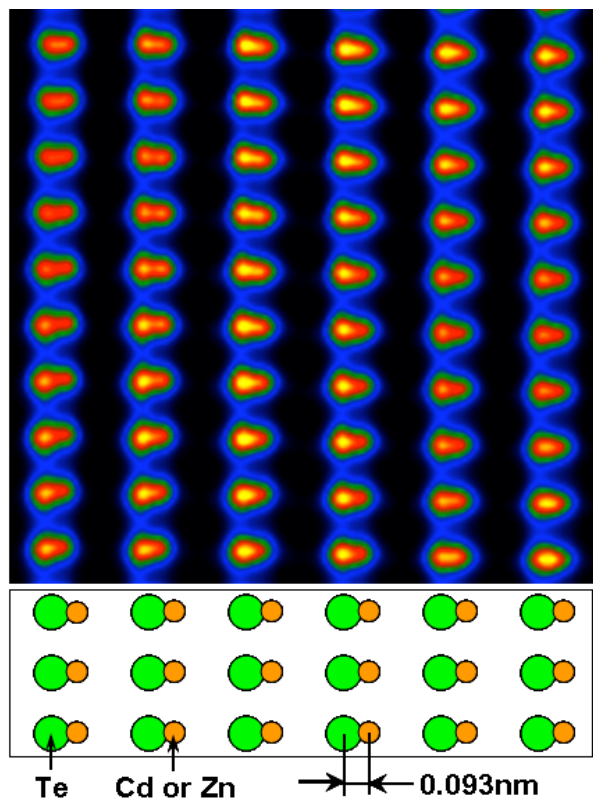

Figure 2: Cs-corrected 300 keV HAADF STEM image of CZT in the [112] orientation showing (Cd,Zn)-Te "dumbbells" (upper) and corresponding schematic of crystal projection (lower). The image demonstrates sub-Ångström resolution.

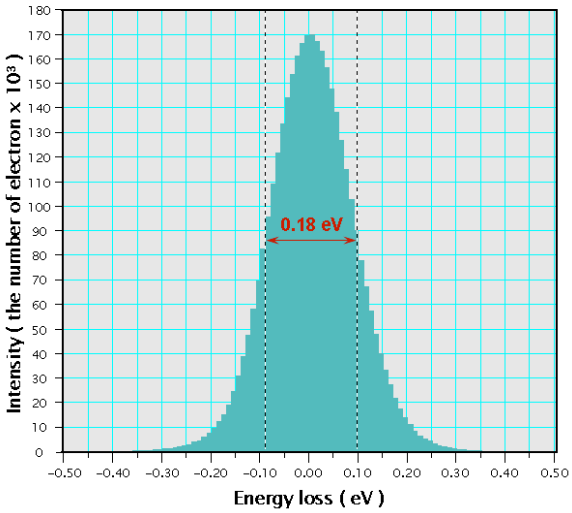

Figure 3: 300 keV zero-loss peak with monochromator on and 4200 eV extraction voltage. Energy resolution is 0.18 eV (FWHM) with an acquisition time of 1 second.

Applications: Cs-corrected STEM mode provides a sub-Ångström probe with a high-brightness. It offers the prospect of element-selective imaging of single atomic columns using the energy filter. Combined with monochromated HR-EELS, we can further investigate chemistry and electron structure-related properties by single atomic column to column, such as valence state, bonding structure, and so forth. Cs-corrected TEM mode offers a tunable spherical aberration coefficient from negative value to positive values. Properly combining a negative Cs with a positive defocus, at no cost to point resolution, a high resolution image with bright-contrast of atoms on dark background can be achieved, which can be directly interpreted without image simulation, and light elements such as oxygen atoms and even their vacancies can also be imaged[1-2]. We demonstrate this capability with pyrrhotite in the Cs-corrected HRTEM image shown in Figures 4 and 5. The corresponding FFT (inset) indicates that 2H-pyrrhotite structure (hexagonal lattice with a = 3.44 Å and c = 5.88 Å). Figure 5 shows a region of the same HRTEM image together with a schematic diagram of the structural projection of 2H-pyrrhotite. The dashed-line boxes marked on the images denote the unit cell. Obviously, Fe and S atoms can be identified from the Cs-corrected HRTEM image by comparison with the structural projection. The shortest distance between S and Fe, as marked by arrowheads, is only 1.77 Å, in which the two atoms are 100% resolved. It is now possible to use imaging to investigate mineralogy with subatomic-scale resolution.

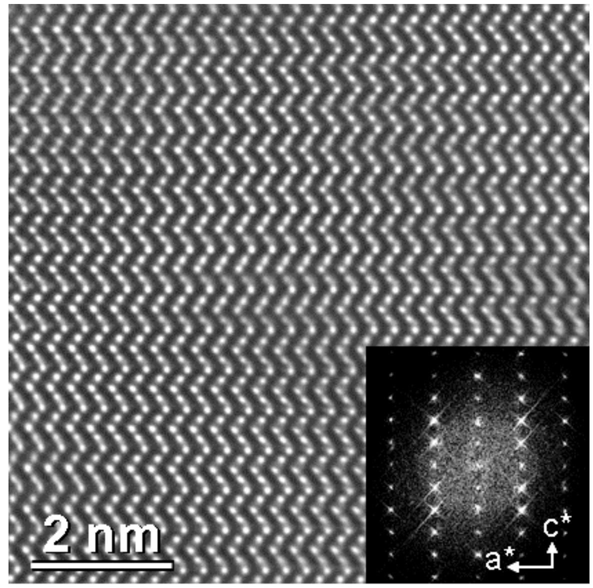

Figure 4. Cs-corrected HRTEM image of 2H-pyrrhotite and corresponding FFT (inset). Then electron beam is parallel to [-12-10] crystal direction of pyrrhotite.

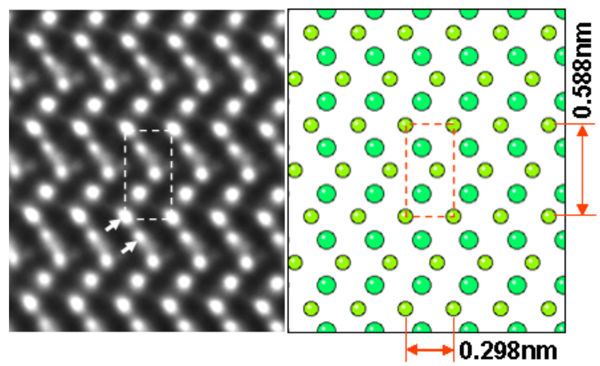

Figure 5. A part of image in Fig.4 (left) and corresponding schematic of structural projection of 2H-pyrrhotite crystal along [-12-10] direction, in which the smaller green spheres denote S atoms and bigger spheres are Fe atoms.

References: [1] Jia, C. L. and Urban, K. (2004) *Science.* 303, 2001-2004. [2] Jia, C. L. et al (2003) *Science.* 299, 870-873.

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