Development of a 200kV Atomic Resolution Analytical Electron Microscope

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Introduction

Few electron optical inventions have revolutionized the TEM/STEM as profoundly as the spherical aberration (Cs) corrector has. Characterization of technologically important materials increasingly needs to be done at the atomic or even sub-atomic level. This characterization includes determination of atomic structure as well as structural chemistry. With Cs correctors, the sub-Angstrom imaging barrier has been passed, and fast atomic scale spectroscopy is possible. In addition to improvements in resolution, Cs correctors offer a number of other significant improvements and benefits.

For a probe forming Cs corrector, more current in the same small probe can used for chemical analysis. This improves not only the resolution for STEM imaging, but also translates to higher signal-to-background and signal-to-noise ratios, reducing beam damage and enhancing detectability [1]. This allows chemical characterization to be done more rapidly, and with better spatial accuracy [2-4]. Spatial chemical characterization, such as EDS or EELS line profiling and mapping, can be performed in a fraction of the time as previously done. We have aimed to improve the EELS and EDS analytical performance of a transmission electron microscope equipped with a Schottky type electron gun and a STEM Cs corrector. We have developed a new atomic resolution analytical electron microscope, the JEM-ARM200F [Figure 1], improving the instrument's mechanical stability as well as electronic stability. Configurations include a probe forming (STEM) Cs corrector, an image forming (TEM) corrector or both. This new platform has been designed to fully take advantage of aberration correction and push the limits of atomic resolution imaging and analysis.

Figure 1. JEOL JEM-ARM200F atomic resolution analytical microscope.

Designed for the laboratory environment

Aberration correction can only improve the ultimate resolution of the microscope so far. As the resolution improves, the consideration for the environment of the room and the electronic and mechanical stability of the microscope itself should be improved. With this second generation of aberration corrected TEMs, design effort has gone into minimizing environmental effects and making these instruments less susceptible to the environment around them. The JEM-ARM200F has been designed from the beginning to incorporate aberration correction. Design improvements were made to improve the mechanical and electronic stability of the microscope, and steps have been taken to further shield the electron microscope column from thermal, magnetic, and electromagnetic influences. The overall stiffness of the electron column scales with the fourth power of the column diameter, so the thickness for the lower half of the electron column has been increased from 250 mm to 300 mm, resulting in twice the stiffness of our conventional microscopes. Computer aided design and computer aided engineering models employing the finite element method were used to optimize the microscope base design to minimize mechanical vibrations.

The electronics of the high-tension system and the electronic supplies for the lenses have been improved to minimize electronic noise and disturbances. The stability of the accelerating voltage has been improved to better than 0.5 ppm peak-to-peak, while the stability of the objective lens current has been improved to better than 1.0 ppm peak-to-peak. The stability of the deflector system has been improved about two-fold compared to our conventional microscopes, so as to maintain atomic spatial resolution for atomic resolution chemical analysis. Furthermore, in order to suppress the influences of external disturbances such as magnetic fields, temperature changes, air flow and acoustic noise, the column is equipped with a heat insulation shield, a magnetic shield, and is covered with a mechanical cover, as shown in Figure 1.

Sub-Ångstrom imaging

All of these design improvements have led to unprecedented resolution. Figure 2a is an unprocessed high angle annular dark field (HAADF) STEM image of silicon in <112> orientation. Figure 2b is an intensity profile taken across the white square in 2a, figure 2c is a histogram of the intensity in 2a and figure 2d is a Fourier transform of image 2a. The dumbbell spacing of silicon in this orientation is 0.078 nm.

This new microscope is capable of acquiring up to 4 different scanning signals simultaneously, yielding a wealth of information about the specimen. In addition to the bright field STEM and *z*-contrast HAADF images, a low angle dark field STEM image for strain contrast and a backscattered electron image of the sample surface can be acquired. Figures 3a and 3b are simultaneously acquired high angle annular dark field (HAADF) and bright field STEM images of silicon <110>. In the HAADF image, the silicon

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Ultrahigh Resolution Analytical Performance

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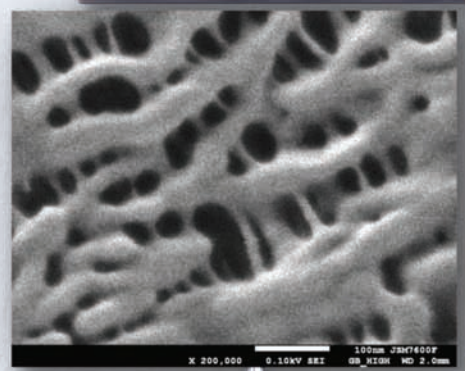
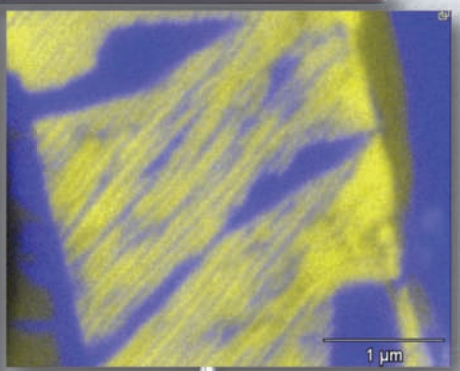
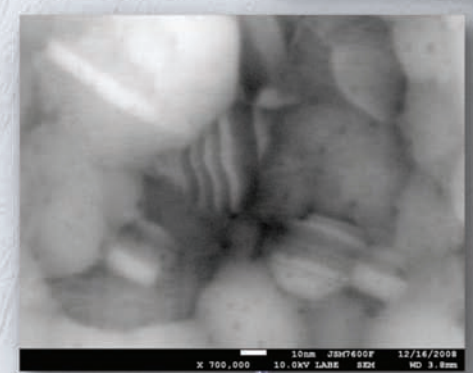
Uncoated stretched polypropylene





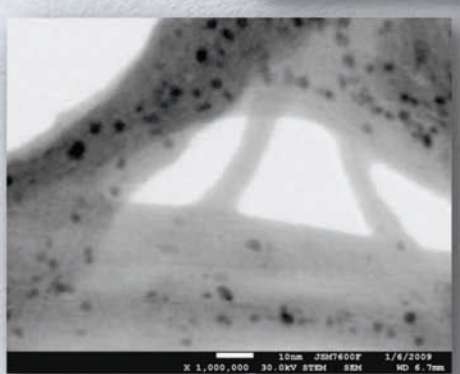
EDS/WDS
EDS map of TiO_2 in FeOx with <100nm spatial resolution 30,000x

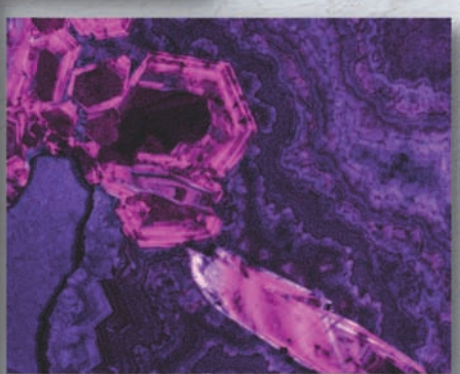
LABe
1-3 nm twinning in mineral BSE image



EBSD
Orientation map of Ni alloy

STEM
CNT with 1-3 nm Pt nanoparticles





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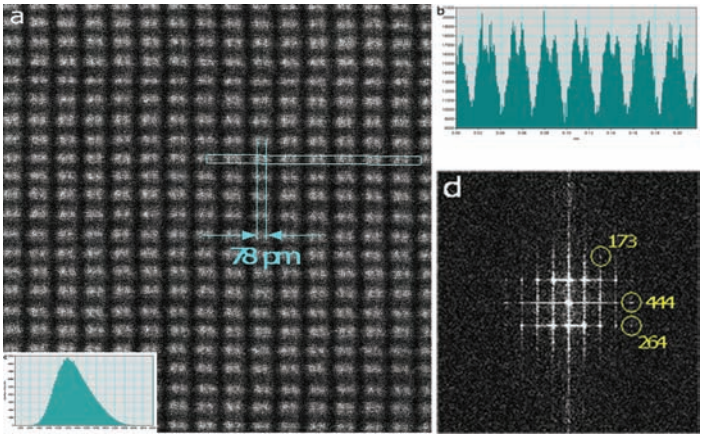

Figure 2. a) Unprocessed high angle annular dark field image of Si <112> taken by JEM-ARM200F with STEM Cs corrector. b) Line profile of the white square area from a. c) Histogram of intensity in a. d) Fourier transform of image a, showing information transfer to the 0.078 nm lattice spacing of (444).

dumbbells are bright in contrast, while in the bright field image the dumbbells appear as clearly separated black dots. These bright field images are subject to phase contrast, similar to that in aberration corrected high resolution TEM images [5].

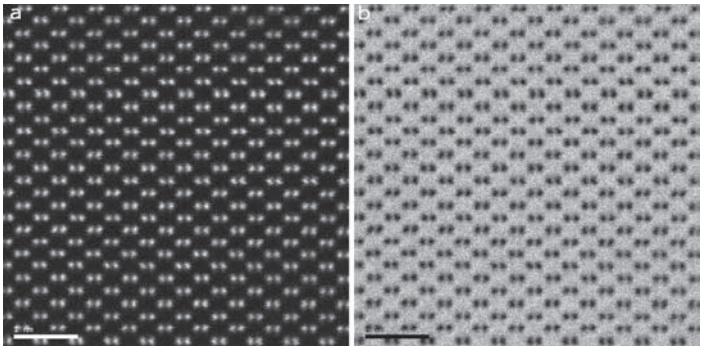

Figure 3. Simultaneous a) high angle annular dark field and b) bright field STEM images of Si <110>.

For many kinds of specimens, beam induced damage is a problem. The increased probe current from a STEM Cs corrected instrument is both helpful and detrimental to this issue. Because of the increased probe current, images and chemical maps can be acquired in less time – limiting exposure to the electron beam. However, the increased probe intensity itself can be a cause of specimen damage. One method of limiting beam damage on a specimen is to operate the instrument at a lower accelerating voltage. However, this can limit the spatial resolution of the microscope. The addition of a STEM Cs corrector helps to get some of this resolution back. Figures 4a and 4b are simultaneously acquired high angle annular dark field (HAADF) and bright field STEM images of silicon <110>, taken with the microscope operating at 120 kV. A Fourier transform of the HAADF image shows information transfer to a spacing of 0.11 nm, while a transform of the bright field image shows information transfer to a spacing of 0.136 nm.

Atomic resolution chemistry

In nanoresearch, it is important not only to know the atomic positions of the atoms in these structures, but also the chemical fingerprint of these atoms. The images previously shown have used the ultimate small probe that can be formed using the Cs corrector and condenser lens system. In a probe of this size, probe currents on the order of 10 pA are typical. As with uncorrected microscopes, higher

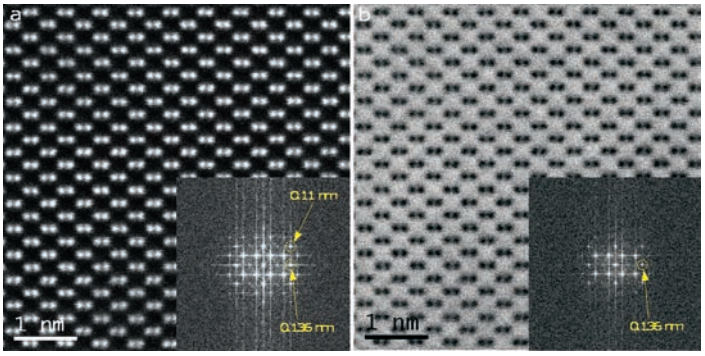

Figure 4. Simultaneous a) high angle annular dark field and b) bright field STEM images of Si <110>, taken with an accelerating voltage of 120 kV.

probe currents in a small probe are required for efficient chemical analysis. To increase the probe current, the probe size is increased, resulting in a loss of spatial resolution. With a Cs corrector, larger probe forming apertures can be used with smaller probes, allowing roughly ten times larger probe currents with good spatial resolution.

Figure 5 is a series of images of Si <110> taken under different probe conditions in STEM. Figure 5a is an image taken using a fine-imaging probe, at a probe current of approximately 30 pA. With this probe, the 0.136 nm dumbbells are clearly resolved and from the fast Fourier transform image information transfer to 0.08 nm can be seen. The images in 5b were taken with a probe current of approximately 150 pA. With this probe, the 0.136 nm silicon dumbbells are still resolved. In Figure 5c, a probe current of approximately 500 pA was used. Here, the silicon dumbbells are no longer clearly separated, but the lattice spacing of 0.192 nm can still be resolved. This indicates that it is possible to get at least 500 pA in a sub-2 Ångstrom probe.

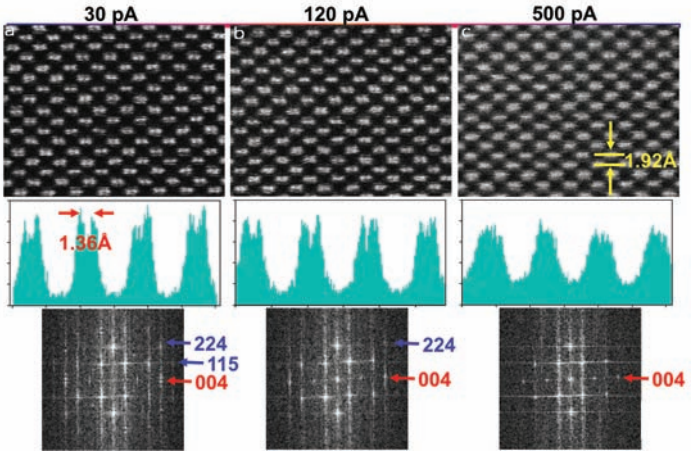

Figure 5. High angle annular dark field STEM images of Si <110> taken with electron probe currents of a) 30 pA, b) 150 pA and c) 500 pA. From c it can be seen that for a STEM probe capable of sub-2 Angstrom imaging, a probe current of 500 pA can be attained. (Images courtesy of Masashi Watanabe, Lehigh University).

This increased probe current is especially important for x-ray microanalysis in the TEM. Since a very small region of the TEM sample is exposed to the electron beam, there are much fewer x-rays generated than in a typical SEM with a bulk sample. Therefore, larger beam current in a small probe will mean the generation of enough x-rays for efficient analysis. Figure 6 shows energy dispersive spectroscopy (EDS) spectra collected from a specimen of SiAlON. This specimen contains small amounts of yttria that have

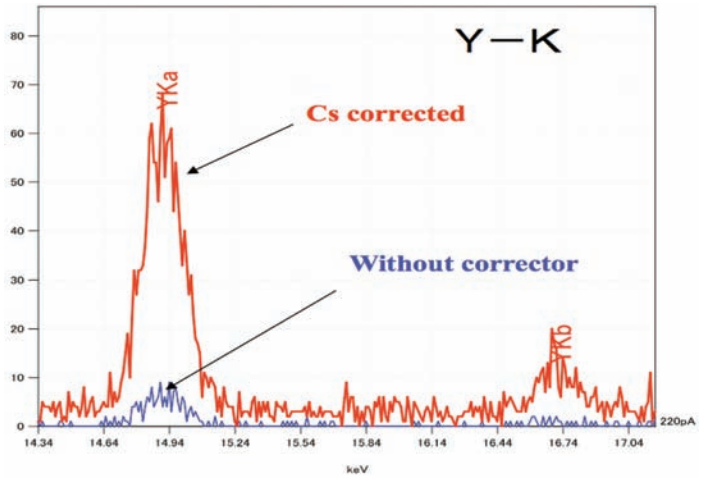

Figure 6. Comparison of EDS spectra of Y-Ka, acquired without (blue spectrum) and with (red spectrum) a STEM Cs corrector.

segregated to the grain boundaries. The spectrum in blue was acquired using a microscope without a STEM Cs corrector. The spectrum in red was acquired using a STEM Cs corrector. In both instances, a 0.5 nm electron probe was used, and the EDS spectra were acquired for 30 seconds. With the Cs corrector, about 6 times the amount of EDS signal was acquired.

The increased signal has profound impact for electron energy loss spectroscopy (EELS). In EELS, smaller probe currents may be used to reduce specimen damage because of good detection efficiency. The current in EELS experiments can be tailored to generate spectra of adequate signal-to-noise. Thus, smaller probe sizes can be used and true atomic resolution spectroscopy can be realized [4]. Figure 7 is a series of STEM EELS spectrum images of $SrTiO_3$. These spectrum images were taken using a probe size of roughly 1.5 Angstroms, with a probe current of 150pA. Under these conditions the 32 x 32 pixel chemical maps show the atomic position of each of the chemical species, Sr, Ti and O.

With the amount of probe current available due to the Cs corrector, it is possible to increase the speed of EELS acquisition without detriment to the chemical information available. Figure 8 is a series of STEM EELS spectrum images of $SrTiO_3$ taken with the same probe conditions as in Figure 7, but using a spectrum acquisition time of 3 ms. Maps of 128 x 128 pixels were acquired, still showing the atomic positions of each of the chemical species. The total acquisition time for these spectrum images was 90 seconds.

Conclusions

Aberration corrected microscopy has opened the door to unprecedented imaging and chemical analysis. Image resolution of better than one Angstrom is attainable in high resolution TEM as well as in STEM, both in high angle annular dark field and in bright field imaging. The Cs corrector allows larger probe currents to be used in these small probes, making atomic resolution spectroscopy a reality. To take full advantage of these advances, design consideration must be given to the room environment in which an aberration corrected instrument resides, as well as the electronic and mechanical stabilities of the microscope itself. To this end, a second generation of aberration corrected TEM, the JEM-ARM200F has been developed. Designed from the beginning to incorporate an aberration correction, this microscope platform will help to push the limits of atomic resolution analytical microscopy. ■

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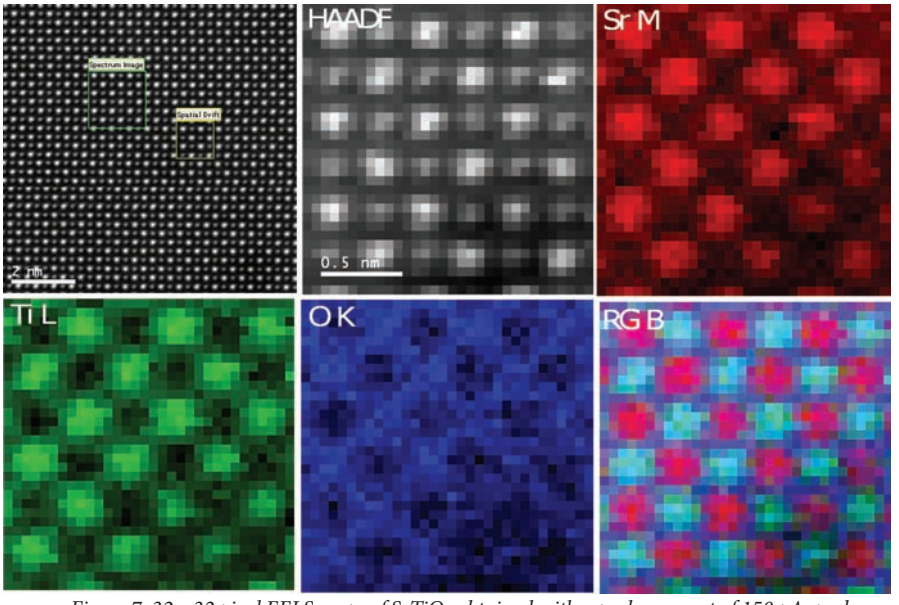

Figure 7. 32 x 32 pixel EELS maps of $SrTiO_3$ obtained with a probe current of 150 pA, probe size of 0.15 nm and an acquisition time of 0.03 seconds per pixel.

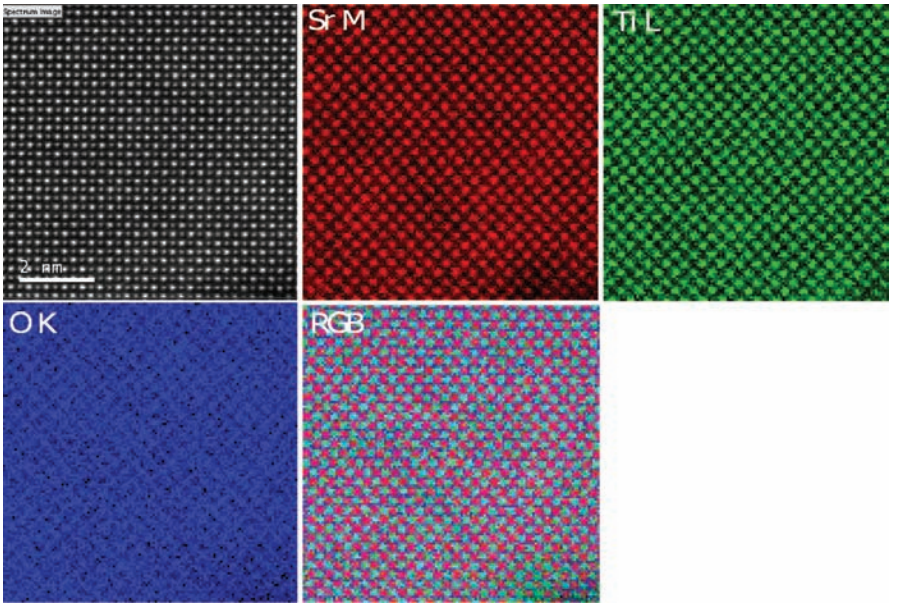

Figure 8. 128 x 128 pixel EELS maps of $SrTiO_3$ obtained with a probe current of 150 pA, probe size of 0.15 nm and an acquisition time of 0.003 seconds per pixel.