Type of presentation: Poster

IT-2-P-2425 Sensitive X-ray analysis system on an automated aberration correction FE-STEM

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In recent years the aberration-correction technique has brought a revolution in analytical microscopy by making atomic-resolution imaging and analysis routinely achievable in transmission and scanning transmission electron microscopy (TEM and STEM) [1)]. We have developed an aberration corrected STEM (Hitachi HD-2700) with an automated aberration correction function. The HD-2700 is equipped with a large solid angle Energy dispersive X-ray spectrometry (EDX) detector which enables an atomic spatial resolution and high sensitivity for EDX analysis.

In the new auto-aberration correction function, the aberration coefficients are measured from a Ronchigram image recorded on a CCD camera for an amorphous sample. Using the coefficients, the software determines the aberrations that need to be corrected and proceeds to correct them. The aberrations are measured and corrected repeatedly and automatically until they are settled under the thresholds. Experiment tests revealed that it took approximately 11 minutes to complete up to the 3rd order aberration[2)]. Figure 1 shows an example of image comparison before and after auto correction for a silicon single crystal imaged along the <110> direction. The Si dumbbell structure is clearly observed after the correction.

To improve the X-ray detection efficiency of the STEM-EDX system, we adopted a design of a windowless[3)], large area (100mm^2) silicon drift detector (SDD). The detector is located closely to the specimen to realize a large solid angle of 1.1sr. The detection sensitivity of the light element (Nitrogen) is more than 10 times higher than that of the 30 mm^2 SDD. Figure 2 shows STEM-EDX mapping results for a Pd-Pt catalyst particle specimen using a 30mm^2 Si(Li) conventional detector and the new 100mm^2 SDD, respectively. The beam energy of 200kV, probe current of 800pA, and acquisition time of 3min. were used. Clearly the maps obtained using the new SDD show much better signal to noise ratio for both nano-particles and carbon support. The high-speed X-ray analysis with the new 100mm^2 windowless SDD also largely reduces the beam irradiation damage to the specimen[4)].

1) H.Inada et al., J. Elec. Microsc., 58 (2009), 111.
2) Y. Hirayama et al., JSPS132 congress (2013).
3) S.Isakozawa et al., J. Elec. Microsc., 59 (2010), 469.
4) K. Tamura et al., Microsc. and Microanal., S2 19 (2013) 1192.

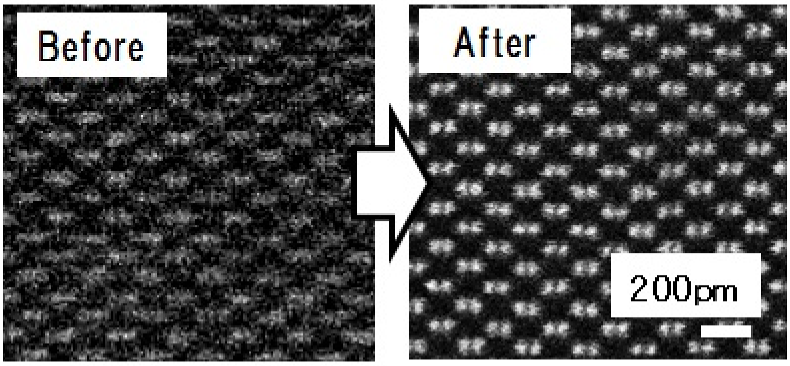

Fig. 1: Image comparison between before and after auto Cs correction of silicon dumbbells.

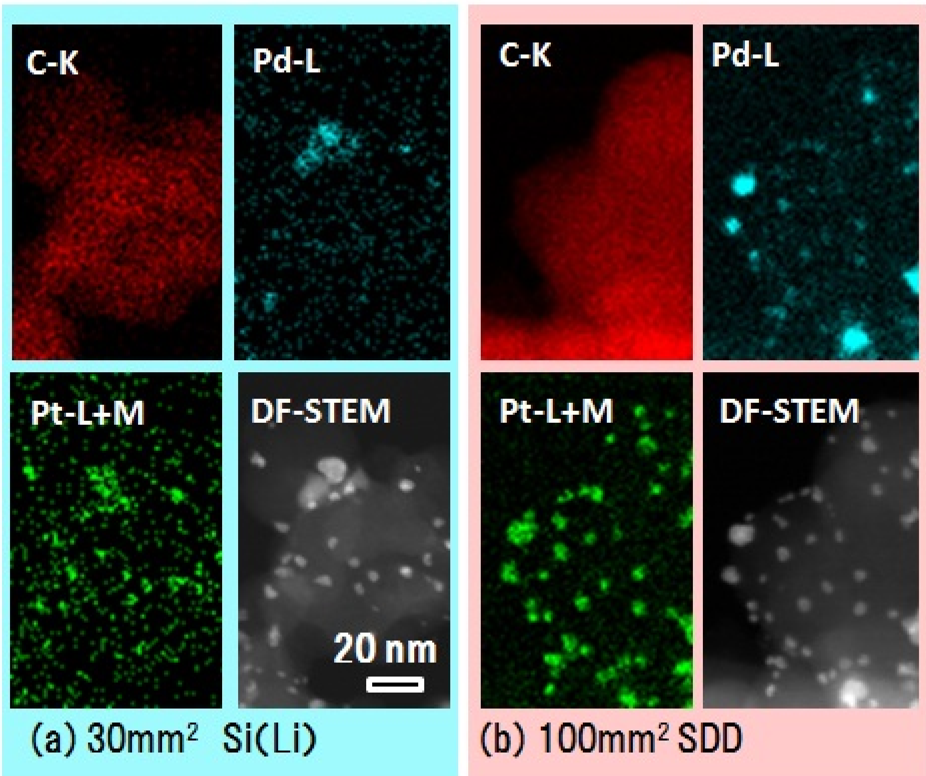

Fig. 2: EDX mapping comparison of Pt and Pd catalyst particles (a) Conventional 30mm^2 Si(Li), (b) Newly developed 100mm^2 SDD.