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Low-Voltage TEM/STEM for Atomic Resolution Imaging and Spectroscopy

Kazu Suenaga[1], Takeo Sasaki[2] and Hidetaka Sawada[2]

[1.] National Institute of Advanced Industrial Science (AIST), Tsukuba 305-8565, Japan
[2.] JEOL Ltd., Akishima, Tokyo 196-8558, Japan

Reducing the accelerating voltage of TEM/STEM is becoming essential when one aims to image any light element matters and/or beam sensitive objects. Lower accelerating voltage is preferred for observation of low-dimensional materials and small molecules made of light elements, in order not to destruct the atomic structures by the knock-on effect and, more importantly, to enhance the image/EELS contrast. In order to compensate the inferior spatial resolution due to the low acceleration voltage, more sophisticated electron optics are definitely required to reduce the residual geometric/chromatic aberrations.

Under the JST-CREST triple C project, Sawada et al. designed a new type of Cs corrector with triple dodecapole elements (the DELTA system) to reduce the six-fold astigmatism [1, 2], which is very useful for the TEM/STEM operated at low accelerating voltages around 15 to 60 kV. A JEOL 2100F equipped with this corrector shows a world best performance in terms of the spatial resolution normalized by the wave length, which was proved by the Si (224) reflection (111pm) corresponding to the 16 times of the used wave length (λ = 7 pm at 30kV) [3]. Fig. 1 shows an ADF image of a few layers h-BN which clearly shows the 108pm reflection at the 30kV operation. This is the clear proof that the atomic resolution can be achievable in STEM-ADF mode even at 30kV.

One of the major advantages of low voltage TEM/STEM has been demonstrated by chemical analysis of individual molecules without massive structural destruction [4]. However this, higher dose experiments show non-negligible irradiation damage on metallofullerene molecules proved by the escape of the encaged atoms even at 60 kV. This problem has been reasonably predicted because a simple calculation of the knock-on threshold energy based on the estimated displacement energy (E_d) [5]. The results suggests that, for the fullerene molecules with higher curvature than those of the graphene and nanotube, the accelerating voltage of 60 kV may not be low enough to completely avoid the irradiation damage. The lower accelerating voltage than ~ 35 kV seems to be preferred for observing the fullerene molecules. Here we show the single atom spectroscopy with hardly any irradiation damage realized by *further* reducing the accelerating voltage down to 30kV and then to 15kV. We have employed a spectrometer dedicated for operation at low-voltages (GIF Quantum, modified).

The high resolution image can be also achievable in TEM mode even at 15kV. Fig. 2 shows the Zemlin tableau which shows the tilting angle up to 60 mrad. It does make possible to get the high resolution image of thin specimens at 15kV. At the current stage the chromatic aberration is the major limiting factor, therefore further efforts are needed to reduce the chromatic aberration.

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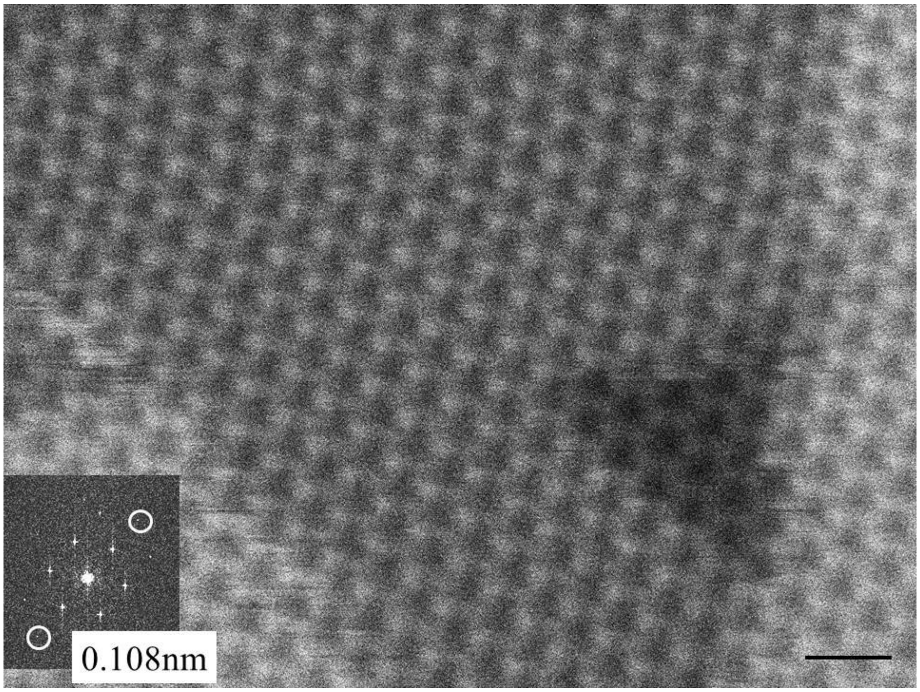
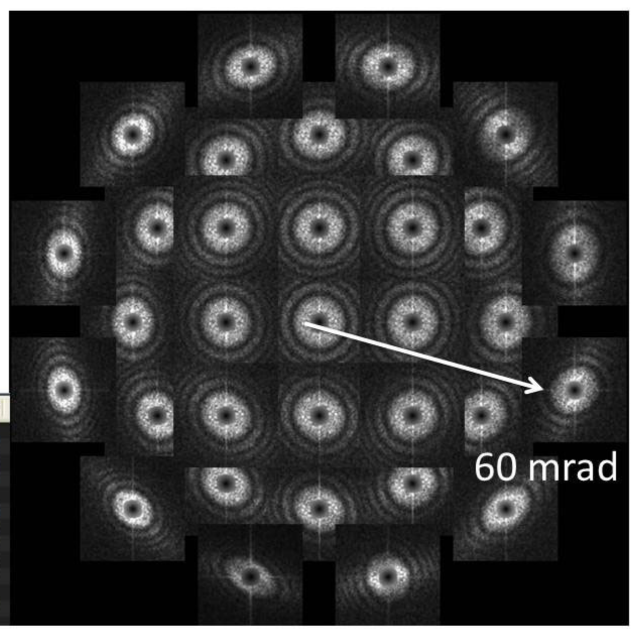


Figure 1. An ADF imag of a few layers h-BN taken at 30kV acceleration voltage. The spatial resolution can be estimated by the inset FFT pattern which clearly shows the 108 pm^{-1} reflection.



	Amplitude	Azimuth
O2	-192.366 nm	
A2	5.428 nm	-50.14°
P3	49.367 nm	109.19°
A3	72.308 nm	-55.08°
O4	0.073 um	
Q4	0.980 um	78.23°
A4	0.892 um	25.51°
P5	127.075 um	25.71°
R5	47.674 um	32.72°
A5	28.352 um	-7.05°
O6	-1.110 mm	
A6	0.366 mm	-21.29°

Figure 2. An example of Zemlin tableau recorded at TEM mode at 15 kV.

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