Configuring a 300kV cold field-emission gun for optimum analytical performance

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Abstract: The performance of a new 300kV scanning transmission electron microscope (STEM) equipped with cold field-emission gun (c-FEG) is characterised with respect to the stability for and the energy width in electron energy-loss spectroscopy (EELS) and the probe size and beam current obtainable for convergent beam and scanning probe operation. These are compared to values from a 200kV FEG-(S)TEM with standard Schottky field-emission.

1. Introduction

The recent development of stable aberration corrected electron optics has seen an increase in the commercialisation of a number of analytical transmission (TEM) and scanning transmission electron microscopes (STEM). Different analytical problems often require an appropriate electron source and adjustments to the electron gun settings are therefore needed to provide the most appropriate conditions for the required analysis, e.g. high energy resolution for energy-loss near edge fine-structure (ELNES) analysis, small stable probes for high resolution (HR-STEM) and medium size probes with high intensity for energy-dispersive X-ray (EDX) mapping [1]. For field-emission TEM/STEM instruments, these conditions are usually a function of the settings of the extraction and accelerating anodes in the electron gun combined with factors such as beam convergence and detector collection angles. Here, we investigate the operation conditions of a newly developed W<111> cold field-emission gun fitted to a double aberration corrected TEM/STEM developed by JEOL (JEM Z3100F-R005) [2]. We have studied the influence of the extraction anode (A1), accelerating anode (A2) and illumination conditions (beam convergence, spot size) to establish the optimum electron source parameters for a range of analytical conditions. A comparison of the 300kV cold-FEG data to that obtained for a 200kV thermally assisted Schottky FEG (JEM 2010F) is also made. For a 200kV Schottky FEG probe currents approaching 1nA have been reported for a probe ~1nm in diameter [3].

2. Experimental methods

Measurements from the JEOL double aberration cold-FEG TEM/STEM were obtained at an accelerating voltage of 300kV. Those obtained from the JEOL 2010F Schottky FEG were obtained at 197kV. On both instruments, the normal means of indicating the beam current is via measurement of the apparent current density on either the large or small phosphorescent screen. However, this method is restricted by the sensitivity of the built-in current density meter to a range from ~10pA/cm² to ~511pA/cm². Consequently, in this study we have measured the beam current by directly coupling the output of the phosphor screen to a high sensitivity pico-ammeter (Keithley Model 2635). To assess the likely effect of variations in the “small” 25mm diameter phosphor screen coating, measurements were
initially taken systematically at the small screen centre and at the very edge of the screen. The location at the edge was determined as the point which gave the maximum current reading. For each instrument, the small phosphor screens were calibrated against the beam current values obtained using a Faraday cage in the specimen plane (Gatan Model 641).

The nature of the 300kV c-FEG gun requires “flashing” after several hours of operation to remove gas absorbed on the tip. Beam current measurements were obtained prior to and after periodic flashing (low-flash: 1.62A for 20 seconds) and also after tip re-build (high-flash: 2.3A for 3 seconds followed by Heat-1A: A1 -2.70kV, 2.2A for 10 seconds). Values of the beam current were obtained as a function of nominal spot size in both convergent beam (CBED-α9) in which only the imaging corrector is optimized and STEM mode in which both imaging and probe correctors are optimized. In CBED mode a 150μm diameter condenser aperture was used and A2 was fixed at 4.6kV with A1 varied between 1.2 and 1.7kV for each nominal spot size. In STEM mode the value of A1 was maintained at 1.4-1.5kV while A2 was varied from 4.60kV to 4.80kV. A nominal camera length setting of 15cm was utilized with the condenser aperture setting varied from 30 and 40μm, corresponding to convergence semi-angles of 21 and 28mrad respectively. After fine adjustment of the corrector optics the probe image was focused onto the small phosphor screen edge by adjustment of the intermediate lens IL3 using free lens control. The probe diameter was estimated by consideration of the smallest resolvable lattice fringes observed in a corresponding annular dark-field (ADF) image for a given nominal spot size.

Similar investigations were performed for comparison with a Schottky FEG (JEOL 2010F at 197kV) in both CBED mode (α9) and STEM with an A1 setting of 2.75kV; A2 of 7.16kV; bias of 300V and a filament current of 2.24A, corresponding to an emission current of 147μA.

The energy spread within the primary beam was assessed in diffraction coupled mode using a 1mm GIF entrance aperture, 0.01-0.02eV/pixel dispersion and exposure times of 0.1 and 1.0s without specimen in the beam path (R005). The extraction anode (A1) of the c-FEG (R005) was adjusted from 0.9kV to 1.7kV with the accelerating anode (A2) set at 4.60kV in each case. Comparative data for the energy spread obtained for the Schottky FEG was obtained using an exposure time of 0.05-0.1s, a dispersion of 0.02eV/pixel and either 0.6mm or 3mm GIF entrance aperture. In all cases the energy resolution is stated as the full width half maximum (FWHM) of the resulting zero loss energy peak.

3. Results and Discussion

Fig. 1 shows that the zero loss peak (ZLP) in the JEOL R005 depends strongly on the setting of the extraction voltage, A1, and will typically be ~0.5eV wide (FWHM) at 300kV (~0.4eV at 80kV) for A1~1.5kV and A2 of 4.6kV. The form of the curves indicate that, for both acceleration voltages, the ZLP can be as narrow as ~0.33eV for A=1.25kV but below this the energy resolution seems to be limited by combination of the Fowler-Nordheim distribution of the field emitter and the spectrometer resolution. In the case of the 200kV Schottky emitter, however, the energy resolution is only ~0.6eV and, over the range of extraction voltages examined, depends linearly on A1 (i.e. here, A1 is limiting the performance). It is worth noting that the c-FEG produces a highly asymmetric ZLP (Fig. 2), with a tail on the low-energy side (higher energy losses) due to the tunneling of the electrons through the surface potential of the tip [4]. This will make band-gap investigations somewhat awkward, compared to the use of a monochromator [5].

The probe current depends exponentially on the settings on A2 and in particular A1, as can be seen from fig. 3. This means that for each application the A1 setting should be set to the current needed to yield a particular dose. In particular, lattice images should be recorded with the maximum current and probe that agree with the spatial resolution just necessary to resolve atomic columns in the lattice.

Fig. 4 compares probe currents recorded in CBED and STEM modes and shows, firstly, that the beam current depends strongly on the tip flashing; the tip is ~9 times brighter after a high flash at elevated temperature (which requires a subsequent “rebuild” of the tip apex and may ultimately limit its lifetime) and ~2 times brighter after a periodic low flash is performed. Secondly, our findings suggest that the setting of A2 is more important than the choice of a slightly larger condenser aperture.
Fig. 1: plots of full width at half maximum (FWHM) of the zero loss peak distribution without specimen; (a): JEOL R005 at 300kV, 0.01eV/channel dispersion, 1mm entrance aperture (EA); (b): JEOL 2010F at 0.02088eV/channel, 0.05s (0.6mm EA) and 0.1s (3mm EA).

Fig. 2: comparison of the zero electron energy-loss spectra from (a) JEOL R005 at 300kV (0.34eV FWHM) and (b) JEOL 2010F at 197kV (0.58eV FWHM) at the same energy scale.

Fig. 3: Performance of the JEOL R005 at 300kV: (a) plot of the dependence of probe current on anode voltage settings for CBED and STEM and (b) a typical lattice image of silicon [110] recorded in STEM mode with ~0.08nm spot size (probe current ~20pA).

The advance offered by Cs correction is clear if one considers the beam current versus probe size data for the 2010F, Fig 5a, where it can be seen that for the smallest probe (~0.3nm diameter) a beam...
current ~17pA is obtained resulting in very poor signal to noise in the subsequent ADF STEM image as illustrated in Fig 5b.

Fig. 4: plot of probe current for JEOL R005 in CBED and STEM mode. CBED conditions: 9, 150µm diameter condenser aperture (CA), 2µA emission, A1=1.44 kV, A2=4.6kV; STEM conditions: A1=1.5kV, 30/40µm CA. The STEM data are re-plotted on an expanded scale on the right.

Fig. 5: (a) plot of probe current vs. probe size for the JEOL 2010F (A1=2.75kV, A2=7.16kV, 147 µA emission) and (b) typical lattice image, showing noisy 0.32nm lattice fringes in an InGaAs quantum well [6].

4. Summary
The performance of a c-FEG on a newly developed 300kV FEG-STEM has been evaluated and compared to a Schottky FEG installed on a 200kV instrument. We can attain 0.3-0.5eV energy resolution and ~0.1nA current sufficient for EELS in an electron beam ~0.15nm in diameter. The authors gratefully acknowledge the assistance of Dr Mike Fay (Univ. Nottingham) for loan of the Faraday cup holder.

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