The influence of emitter conditioning on the performance of a tungsten <111> cold field emission gun operating at 300kV

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Abstract: In this contribution, we examine the influence of emitter conditioning for a <111> tungsten cold field emission gun on the emission and beam characteristics of a double aberration corrected electron microscope. By varying the post flash build-up parameters we can control the effective emitter tip radius. A sharp emitter yields an energy resolution of 0.31eV but relatively low beam current whereas an increased tip radius results in a reduction in energy resolution to 0.4eV but much higher potential beam current. Consequently, careful control of the build-up parameters can be used as a means of tailoring the emission to suit specific instrumental requirements.

1. Introduction

Recent developments in commercial field emitter design have seen a renaissance of cold-field emitters (c-FEG) in modern aberration corrected TEM and STEM instruments to optimise brightness and energy resolution [1]. The performance of these emitters is strongly dependent on their geometry and the level of vacuum around the tip. Nevertheless, even with reasonable gun vacuum levels of 10^{-9} Pa or better there is still a need to periodically recondition or "flash" the electron source to remove residual gas absorbed on the tip surface [2]. Conventional high-flash were the tip is heated to ~2400 K results in the removal of the absorbed surface gas but can effectively blunt the tip and significantly reduce the brightness of the electron source for a given extraction potential. The geometry of the emitter tip can however be restored by a build-up process using a thermal field treatment [3] in which tip is heated under the influence of a strong electric field. In the case of a tungsten <111> single crystal, the build-up process results in the diffusion of surface atoms at the apex of the tip to form {110} and {211} side facets the latter of which ultimately intersect to produce a sharp pyramid truncated by the (111) plane.

In this report, we investigate the effect of varying the post flash build-up parameters applied to a <111> tungsten c-FEG on the emission characteristics, extraction potential, probe current stability and primary energy spread.

2. Experimental methods

The W <111> c-FEG utilised in this study is fitted to a JEOL Z3100F-R005 double aberration corrected TEM/STEM normally operating at 300kV. The periodic "flashing" procedure involves an initial high-flash (HF) in which the tip is raised to a high temperature by passing a current of 2.2A for 3 seconds. The subsequent build-up (BU) treatments investigated are detailed in Table 1. In each case the emission current and corresponding probe current was measured as a function of extraction voltage (A1 kV) with a constant second anode (A2) potential of 4.6kV. The emission current was determined by indirect measurement of the potential across the appropriate test point of the emission feedback control board. This was necessary as the emission current displayed in the microscope operating software (TEM-COM) has a minimum increment of 1μA and indicates zero for emission currents below 1μA. For consistency with our previous studies the corresponding probe current was measured in CBD mode α1 with a nominal spot size of 1.2nm and a 150μm condenser aperture using a Keithley 2635 test meter via the small diameter viewing screen, previously calibrated using a Faraday cup at the specimen plane. Emission patterns were recorded using a TV rate camera positioned at the gun inspection port focused onto the surface of the perforated second anode.

Table 1: Emitter conditioning parameters investigated in this study.

	Current (A)	**A1 Bias (kV)**	**Time (Sec)**
High Flash	2.2	-	3
BU 1	2.2	-2.70	10
BU 2	2.1	-2.60	10
BU 3	2.05	-2.55	10
BU 4	2.0	-2.50	10

To assess the influence of tip conditioning on the energy spread in the primary beam an energy-loss spectrum was acquired immediately after build-up in each case for a series of equivalent probe current by recording the full width half maximum (FWHM) of the zero energy loss spectrum using a Gatan Tridium GIF with an entrance aperture of 1mm, dispersion of 0.01ev/pixel and an exposure of 0.1 second. The relative drift in beam current and gun vacuum was determined as a function of time immediately after build-up (BU-1) treatment.

3. Results and Discussion

A series of emission patterns for the different build-up conditions studied is given in Figure 1 with the respective details of the extraction potential, emission current and corresponding probe current shown in parenthesis in each case. In figure 1(a) we observe the classic three-fold symmetry of the <111> W field emission pattern.

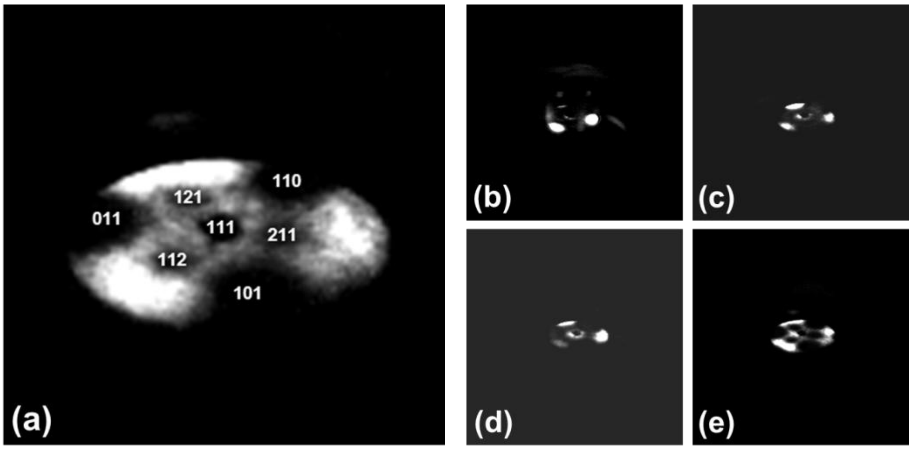

Figure 1: C-FEG emission patterns for the different conditions immediately after build-up (a) high-flash only (2.00kV, 4.08μA, 6.25nA) with the low order emitting facets indexed, (b) BU-1 (1.05kV, 5.05μA, 397pA), (c) BU-2 (1.40kV, 5.03μA, 1.35nA), (d) BU-3 (1.5kV, 4.94μA, 4.3nA) and (e) BU-4 (1.79kV, 4.26μA, 1.3nA). Note - pattern (a) is x6 magnified with respect to the subsequent patterns to show detail.

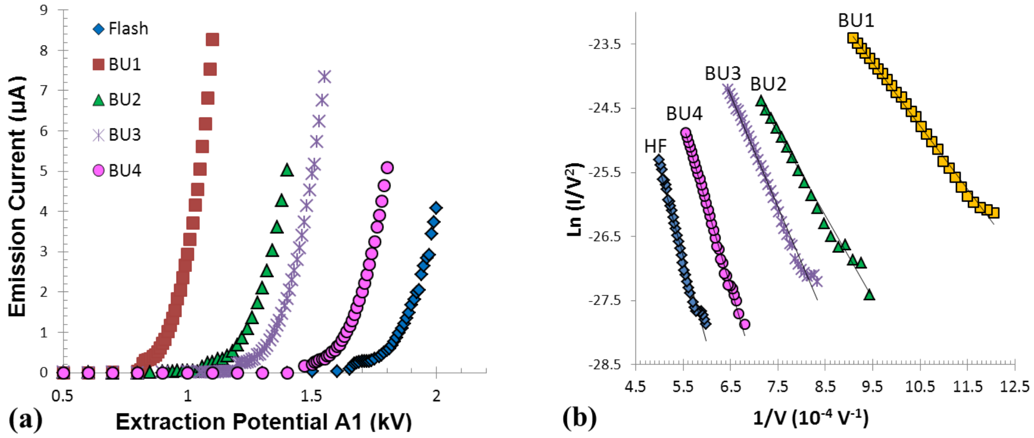

Figure 2: (a) Emission current as a function of extraction voltage, (b) the corresponding Fowler-Nordheim plots.

Plots of emission current versus extraction potential are given in Figure 2(a) with the corresponding Fowler-Nordheim (F-N) plots shown in Figure 2(b). An estimate of the tip radius can be directly extracted from the F-N field emission theory which shows the slope of the $\ln(I/V^2)$ versus $1/V$ plot to be -6.8 x10^7 $\Phi^{3/2}$ αkr [4,5] where, Φ is the work function (eV) (4.47eV for <111> tungsten), α is the Nordheim image correction term (which in this instance is assumed to be unity [4]), k is the field reduction factor and r is the tip radius (in cm). The exact value of k can vary significantly and depends on the actual tip geometry with reported vales between the limits of 3 for blunt tips to 35 for sharp tips [6]. Table 2 shows the estimated tip radii from the F-N plots in Figure 2(b). The value of kr is presented to provide a relative comparison, while the actual estimated tip radius is given for the limits k=3 and k=35. The estimates clearly illustrate the sharpening of the tip with increasing heating current and applied reverse potential.

Table 2: Estimation of the tip radii from the linear fits of the Fowler-Nordheim plots shown in Fig 2b.

	BU1	**BU2**	**BU3**	**BU4**	**Flash**
kr (nm)	160	175	278	431	505
kr (k=3) (nm)	53	58	92	144	168
kr (k=35) (nm)	4.6	5.0	8.0	12.0	14.4

The beam current at the specimen as a function of extraction potential is given in Figure 3(a). From Table 2 it can be observed that as the tip radii decreases, the required extraction potential decreases however, we find that for sharper tips the total beam current is limited by the emission current, Figure 2(a) since the latter is limited within the instrument software to 20µA and hence the extractor cannot simply be increased indefinitely. The influence on the energy spread is presented in Figure 3(b). With the exception of the high-flash only condition, the value of the FWHM of the zero energy loss peak is relatively consistent for a given value of the beam current, although the best energy resolution (0.31eV) is still achieved using the sharpest tip (BU1) while a ΔE of 0.45eV can be attained for less sharp tips (BU3, BU4) while maintaining significantly higher beam currents.

Figure 4(a) shows the gun vacuum as a function of beam-on time. While there is some slight initial degradation, likely due to out-gassing of the second anode aperture, the major component relates to the differential pressure between the gun and column. Nevertheless, the increase in gun pressure tends to plateau after 2-3 hours, remaining better than 6.5x10^{-9} Pa. Analysis of the F-N plots for the tip described in Figure 4(b) after prolonged periods without flashing reveals that the relative value of kr increases to ~180nm after 266 hours. The corresponding beam current was 290pA, approximately half the original beam current generated after initial build-up. The application of a low-flash (1.460A for 45 seconds) restored the probe current to ~750pA (for an equivalent emission current of 4μA) while the value of *kr* differed only slightly at ~190nm, indicating the successful removal of adsorbed gas but marginal change in emitter shape under low-flash conditions.

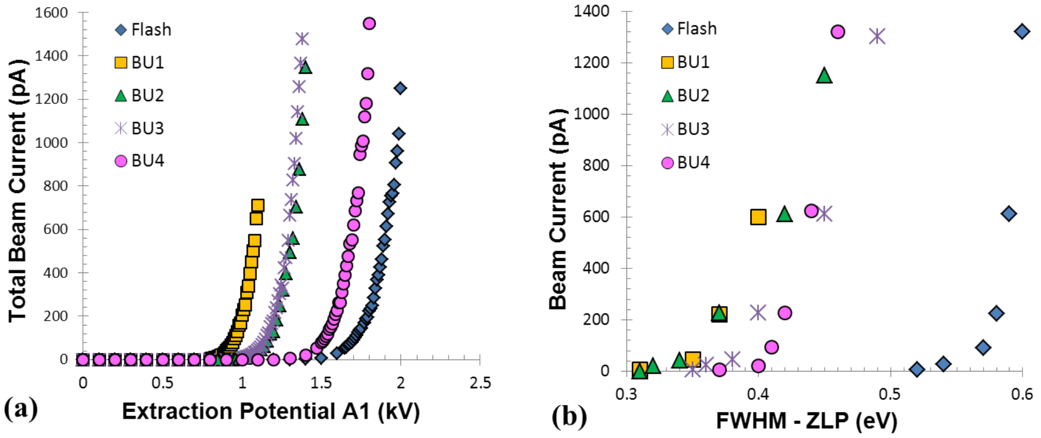

Figure 3: (a) Plot of beam current as a function of A1 with (b) the corresponding energy spread in the primary beam as a function of build-up condition for a given beam current.

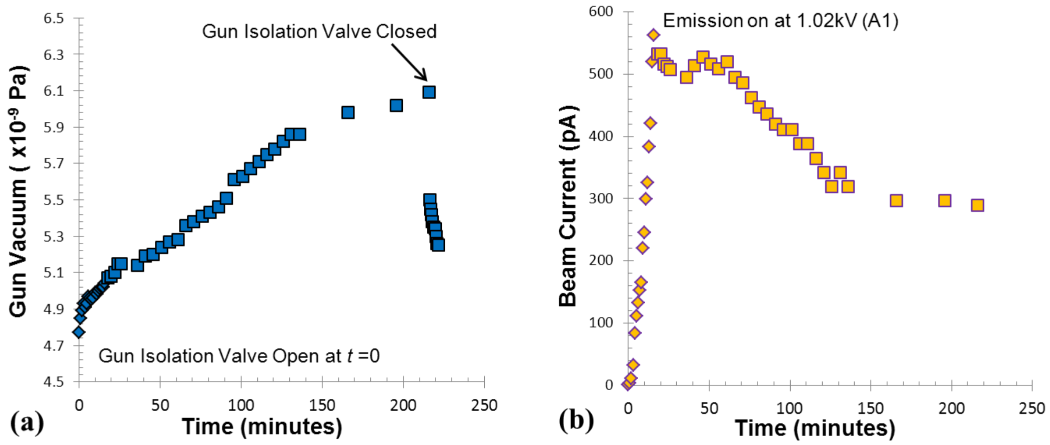

Figure 4: Plot of drift in (a) Gun vacuum and (b) beam current after high-flash and BU-1 build-up.

4. Summary

We demonstrate that the post flash build-up parameters can strongly influence the resulting emission and beam characteristics. Hence, careful choice of these parameters is required to fully optimise instrument performance for specific applications.

References

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