

Operando Observations of Zn Electrodeposition Reaction with Liquid ETEM

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Cold-field emission (CFE) electron guns have advantages of high brightness and narrow energy spreads, and therefore have been used for high resolution SEMs, STEMs, and TEMs for electron holography research. The CFE guns have two challenges: One is difficulty in stable operation for a long time. The other is under a large probe current, abrupt increase in the probe diameter in proportion to the 3/2th power of the probe current and decrease in average brightness [1, 2]. We developed a 1.2 MV-CFE - TEM [3] to observe magnetic phenomena at an atomic resolution using electron holography method. The illumination system of this microscope incorporated techniques to solve two challenges of CFE guns. First, by evacuating the vacuum around the CFE emitter with non evaporative getter pumps to the order of 10^{-10} Pa, enables stable probe current continuously for more than 8 hours, which is sufficient for TEM observations [4]. Second, a magnetic- field-superimposed CFE gun was equipped to control positions of a virtual source by the gun magnetic lens, resulting in reducing effects of spherical aberrations of the electron gun and the acceleration tube [5].

In this report we discuss results of probe evaluation of the illumination system. Figure 1 shows that the probe diameter increases gradually in proportion to the 3/8th power of the probe current, indicating a typical behavior in the axial brightness limited region [6] as is appearing in thermionic guns. This indicates suppression of spherical aberrations at the gun and acceleration tube. Figure 2 shows that observed average brightness tends to saturate with an increase in probe current. However, increase in probe current does not affect stray AC magnetic field and mechanical vibrations, therefore; these cannot cause. We conjecture that this brightness saturation is due to increases in the virtual source size caused by trajectory displacement due to Coulomb interaction. The beam energy spread including Boersh effect [7] was 0.3 to 0.5 eV, which is comparable to that of conventional CFE guns, and no extra increase due to magnetic field superposition was observed. The probe diameter measurement yielded the maximum average brightness of 1.4×10^{14} [A/m²sr]. On the other hand, 3×10^{14} [A/m²sr] was obtained by Fresnel fringes methods [8]. This is roughly due to the difference between average brightness and axial brightness.

1. Broers A. N., *Proc. Fifth Int. Conf. Electron Ion Beam Sci. Technol.* (1972) 3-25
2. Shimoyama H., *Electron Microsc.* **19** (1985) 151-164 (in Japanese)
3. Akashi T. *et al.*, *Appl. Phys. Lett.* **106** (2015) 074101
4. Kasuya K. *et al.*, *J.Vac.Sci.Technol.* **B 32** (2014) 031802
5. Veneklasen L H, *Optik* **36** (1972) 410-433
6. Fujita S. & Shimoyama H., *J. Electron Microsc.* **54**(5) (2005) 413-427
7. Boersh H., *Z.Phys* **139** (1954) 115-146
8. Akashi T. *et al.*, *Microscopy*, dfy031, <https://doi.org/10.1093/jimicro/dfy031>

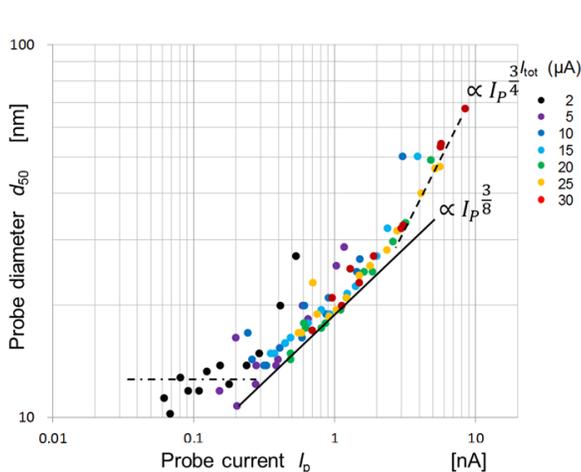


Fig.1. Probe current vs. probe diameter

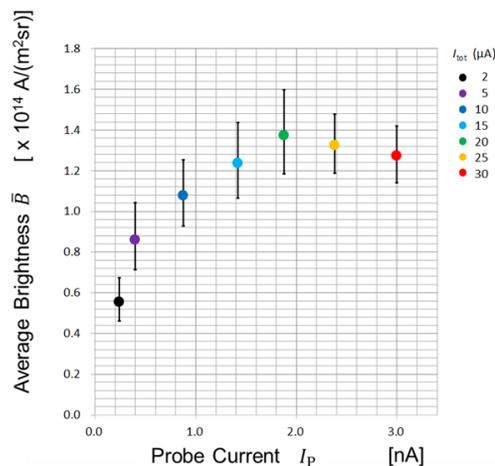


Fig.2. Probe current vs. average brightness

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