Performances of a cold FEG microscope with an objective lens aberration corrector

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The development of the aberration correctors [1] and highly coherent electron sources [2] has boosted the impact of electron microscopy on the study of materials at the atomic scale. Indeed, the last generation of instruments attains picometer-order spatial resolution [3], with an energy resolution of tenth of an eV [2]. Both, high resolution imaging and analytical microscopy have enormously benefited from the technological advances of electron microscopes, resulting in enhanced structural and chemical analysis of nanomaterials [4]. Among the electron sources with a narrow energy spread, the high brightness of Cold Field Emission Guns (CFEG) is a tremendous advantage for the study of nanomaterials, in which very weak elastic and inelastic signals emitted by small volumes of matter are key limiting factors. So far, highly coherent electron waves emitted from CFEG technology was applied to electron holography observation [5] and the analysis of EELS fine structures [6].

The present work describes the performances of an unseen 80 – 200 kV microscope, the JEOL ARM 200F, employing a newly developed CFEG and an aberration-corrected objective lens. As a first result, the capabilities of this instrument were used to determine the surprising three-dimensional morphology of CoPt bimetallic nanoparticles (NPs) and to differentiate the iron valence state in iron oxide nanostructures.

Variations of emission and probe currents after flashing the CFEG have been measured. The decay rate of the probe current was less than 5% in the first two hours after flashing making the use of this new cold-FEG much more friendly. Energy spreads of electrons emitted from CFEG have been also measured as a function of emission current. The energy spread depends on the emission current due to the statistical Coulomb effect and it varies from 0.26 eV with an emission current of 0.1 µA (which is very to the theoretical resolution value of 0.22 eV at the surface emitter) to 0.4 eV in normal emission current at 200 kV. A slightly better value of 0.23 eV was obtained at 80 kV for the lowest emission current condition. The combination of the JEOL cold FEG and the CEOS aberration corrector, associated to enhanced mechanical and electrical stabilities of the ARM 200F microscope, allows reaching a point resolution of 75 pm at 200 kV and 80 pm at 80 kV. In addition, the high brightness of the cold FEG substantially improved imaging sensitivity that is essential for quantitative high resolution imaging. This unseen point resolution at 200 kV has allowed us to study the structure of CoPt nanoparticles by observing the atomic arrangement along high indexes zone axis. A typical morphology of the as-grown NPs corresponds to triangular shape in the substrate plane (Fig. 1). These NPs show two orientations: the upper right side of the NP is oriented along the [114] direction whereas the lower left side is oriented along the [-101] direction (Fig. 1c). Due to the sub-angstrom resolution, the [114] zone axis image is a direct image of the NP structure and it allows us to prove that this configuration is formed by two twinned crystals, the twin boundary being not parallel to the electron beam as it was conventionally observed in non-corrected HRTEM imaging. This result was very well confirmed by tomography experiment (Fig. 1d).

The precise stoichiometry of two iron oxides, FeO and Fe2O3, has been determine from the analysis of iron valence state which was determined from the direct analysis of EELS fine structures spectrum of the two oxides, thanks to the high brightness and high-voltage stability of the CFEG. Figure 2 compares the ELNES of Fe L2,3 and O K edges acquired on α-Fe2O3 and FeO nanocrystals (Furuuchi Chemical Corporation™) in STEM mode with the microscope operating at 200 kV and with an emission current of 20 µA. The difference in energy and the peak shape of the Fe L3 edge as well as the difference of the pre-peak structure of the O K edge between the two spectra allow us to unambiguously determine the valence state of iron and the stoichiometry of the oxides.
References


Figure 1. (a) Bright filed image of as-grown CoPt NPs obtained on a carbon replica. (b) HRTEM image of a CoPt NP oriented along the [002] showing the truncated octahedron morphology. (c) Triangular shaped NP exhibiting the [-101] and [114] zone axes orientations. (d) 3D shape of the triangular shaped NP obtained by electron tomography showing an angel wing like morphology.

Figure 2. Comparison of (a) the Fe \textit{L}\textsubscript{2,3}-edge and (b) the O \textit{K}-edge electron energy-loss near-edge structures of \textit{\alpha}-Fe\textsubscript{2}O\textsubscript{3} (red curves) and FeO (blue curves). The pre-edge background for each edge has been subtracted using a power-law function. The contribution from transitions to the continuum states has not been removed.