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Comparison of multilayered nanowire imaging by SEM and Helium Ion Microscopy

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Abstract. The helium ion microscope (HeIM) is capable of probe sizes smaller than SEM and, with intrinsically small ion/sample interaction volumes, may therefore potentially offer higher spatial resolution secondary electron (SE) imaging of nanostructures. Here 55 nm diameter CoPt/Pt multilayered nanowires have been imaged by HeIM, SEM and TEM. It is found that there is an increased resolution of nanowire surface topography in HeIM SE images compared to SEM, however there is a reduction of materials contrast of the alternating Pt and CoPt layers. This can be attributed to the increased contribution of surface contamination layers to the ion-induced SE signal, and carbon is also observed to grow on the nanowires under prolonged HeIM scanning.

1. Introduction
Scanning electron microscopy (SEM) has long been a technique of choice to image nanostructures on substrates. The SEM is able to evaluate nanowire morphology with spatial resolutions << 5 nm, depending on factors including the probe size and accelerating voltage. The recently commercialised helium ion microscope (HeIM) is capable of probe sizes of 0.25 nm [1]; a significant improvement over the probe sizes of SEMs. The incident, highly focused He ion beam generates ion induced secondary electrons (ISE) within the sample, which can be collected from the surface to form a SE image [1-4]. In principle, the smaller probe size and the intrinsically small ion/sample interaction volumes of the HeIM compared to a typical SEM, should improve the spatial resolution when imaging the smallest nanostructures [2,3], however the mechanisms of ISE generation are quite different from ESE and the benefits of HeIM over SEM are still to be proven.

Here a Zeiss Orion Plus™ HeIM is used to obtain ISE images of 55 nm diameter multilayered CoPt/Pt nanowires consisting of alternating CoPt and Pt layers each <100 nm length. The HeIM ISE images of the CoPt/Pt nanowires are compared to SEM ESE images and the known multilayer microstructure as evaluated by TEM.

2. Experimental
CoPt/Pt multilayered nanowires were fabricated by pulsed signal electrodeposition into anodic aluminium oxide (AAO) templates which had 55 nm pore diameters [5]. The nanowires consist of

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alternating nanocrystalline layers of Pt (55 nm in diameter) and Co$_{72}$Pt$_{28}$ (variable diameter), whose microstructure has been extensively characterised elsewhere [5].

SEM and HeIM samples were prepared both by fracture of the templates, and by dissolving away the AAO templates in 0.2 M sodium hydroxide solution and rinsing with distilled water. Nanowires completely dissolved out of the templates were dispersed on SiO$_2$(100 nm)/Si substrates for HeIM analysis. The morphology of the electrodeposited multilayer nanowires were analysed using a JEOL JSM 6500F field emission gun (FEG)-SEM, a JEOL 2010F TEM, and a Zeiss Orion Plus™ HeIM. Individual nanowire samples for HeIM analysis were pre-cleaned for 5 minutes by an in-situ plasma cleaner (Evactron® 45 De-Contaminator, XEI Scientific, Inc.) inside the load lock of the HeIM to remove contamination. The plasma power was 10 watts with a radio frequency of 13.56 MHz. The samples were flushed with nitrogen before transfer into the HeIM chamber, vacuum <1.1x10$^{-4}$Pa. The helium ion beam was operated at 30-31 kV, beam current <0.3pA, and ion-induced SEs were collected using an Everhart-Thornley SE detector.

3. SEM and TEM imaging of CoPt/Pt nanowires
Fracturing the AAO templates resulted in the exposure of many aligned nanowires protruding from the AAO pores at the fracture surface. Figures 1(a,b) show typical 15kV SEM images of the CoPt/Pt multilayered nanowires, where the bright layers are Pt, and the lighter grey layers with lower secondary electron yield are Co$_{72}$Pt$_{28}$. Nanowires which retain a coating of alumina appear as a continuous bright column in SEM due to electron-induced charging (Figure 1(b)). Nanowires that have been dissolved out of the AAO templates also clearly demonstrate alternating contrast of the Pt and CoPt layers in both SEM (Figure 1(c)) and in TEM (Figure 1(d)). The as-grown nanowires typically exhibit a reduced diameter of the CoPt layers compared to the Pt layers, generating a modulated morphology along their length [5].

Figure 1. (a,b) SEM images of 55 nm diameter CoPt/Pt multilayered nanowires, in-situ, protruding from a fractured AAO template. The bright layers are Pt (marked in blue), the lighter grey layers are Co$_{72}$Pt$_{28}$ (marked in pink), and the white ‘wires’ are still embedded in a thin alumina coating. (c) SEM image of individual nanowires dissolved out of the AAO template, and lying on a gold replica of the AAO template. (d) TEM image of multilayered CoPt/Pt nanowires on a holey carbon film, showing alternating Pt (dark contrast) and Co$_{72}$Pt$_{28}$ (grey contrast) layers which grow with variable diameter.
4. HeIM imaging of CoPt/Pt nanowires

Figure 2 shows HeIM images of arrays of aligned CoPt/Pt multilayered nanowires, partially dissolved out of their AAO template. Clear modulated contrast can be observed along the lengths of some of the individual nanowires. The ion-induced secondary electron images (ISE) are similar to the electron induced secondary electron images (ESE), with the Pt layers generating more SE than the Co\textsubscript{72}Pt\textsubscript{28}. In these clumps of nanowires, which are not yet fully released from the AAO template, the surface and shape of the nanowires are not clearly defined, probably mainly due to a degree of residual undissolved alumina and contamination from the NaOH solution. For the HeIM imaging conditions used, no significant time-resolved sputtering of the nanowires was detected.

![Figure 2](image)

Figure 2. (a,b) 31 kV HeIM images of aligned 55 nm diameter CoPt/Pt multilayered nanowires, partially dissolved out of their AAO template. The bright layers are Pt, the lighter grey layers are Co\textsubscript{72}Pt\textsubscript{28}, and the white ‘wires’ are still embedded in a very thin alumina coating.

Figure 3 shows 31kV HeIM images of individual CoPt/Pt multilayered nanowires which have been fully released from their AAO template and dispersed on a SiO\textsubscript{2}(100 nm)/Si substrate. Two key features of the HeIM images of the individual nanowires are:

1. **Increase in apparent roughness of the nanowires** (Figures 3(a,b)), with few nanowires exhibiting the regular modulated morphology apparent in SEM and TEM images (Figure 1).

2. **Loss of materials contrast of the alternating Pt and CoPt layers** (Figure 3(a)). No alternating contrast from the multilayers was detected, which is clearly visible in the SEM (and TEM) images of individual nanowires prepared by the same method (Figures 1(c,d)), and also in HeIM images of partially dissolved nanowire clumps (Figure 2).

Both of these features appear to be related to:

(a) The HeIM ISE images have an increased resolution of the outer surface topography compared to the SEM ESE images. The HeIM has a much smaller spot size and smaller interaction volume than the SEM [2-4], and clearly images very fine detail in the outer contamination on the individual nanowire and substrate surface from the dissolution process (Figures 3(a,b)). Contamination from the dissolution is also present in the SEM and TEM samples, but has a very weak contrast contribution, making the nanowires appear more smooth and regular (Figures 1(c,d)).

(b) The increased sensitivity of the HeIM ISE images to surface contamination obscures the underlying materials contrast from the Pt and CoPt layers in the individual nanowires (Figure 3(a)). There appears to be much more contamination on the individual nanowires than the partially dissolved nanowire clumps (Figure 2), possibly due to attraction of contamination to the surface localised individual wires as the specimens are dried on the substrate.
(c) Notable ion beam induced deposition (IBID) of carbon also occurs during examination in the HeIM. Figure 3(c) shows the growth of contamination, and associated reduction in nanowire contrast and resolution, as a result of exposure to the He ion beam for 35 minutes. Nanowire samples exposed to an additional 30 minutes plasma cleaning to reduce carbon contamination (Figure 3(d)) did not appear significantly different.

Figure 3. Role of contamination on HeIM imaging of the CoPt/Pt multilayered nanowires. (a) 31kV HeIM image of a single multi-layered CoPt/Pt nanowire dispersed on a SiO₂ (100 nm) / Si substrate. (b) Nanowire with very irregular external morphology. (c) A nanowire scanned for 35 minutes in the marked box with the He ion beam accumulates contamination. (d) A nanowire plasma cleaned for 30 minutes within the load lock of the HeIM microscope.

5. Conclusions
It is observed that HeIM ion-induced SE (ISE) images and SEM electron-induced SE (ESE) images of the same CoPt/Pt nanowires are different in character. The ISE images reveal improved detail of the outer surface topography, but are therefore highly sensitive to surface contamination layers. For the individual nanowires examined here, the dominant contribution of surface contamination to the ISE images masks material contrast from the alternating Co and Pt layers, which can be seen in the less surface-sensitive SEM ESE images. The build-up of contamination in-situ in the HeIM during prolonged scanning is also detrimental to nanowire imaging.

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6. References
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