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DOI: 10.1017/S1431927606068450

Microsc Microanal 12(Supp 2), 2006

Three-Dimensional Nanoscale Characterization of Pt Deposition from an Organometallic Precursor Induced by a Focused Ion Beam

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In specimen preparation procedures utilizing a focused ion beam (FIB) for cutting and cleaning a specimen, often a 500-1000 nm layer of a heavy metal is deposited onto the region of interest (ROI) from an organometallic precursor to protect the ROI from Ga implantation. In our study, a layer of Pt is deposited via a gas-ion interaction process by which a Pt compound is dissociated into Pt, C, and other hydrocarbons. Previous studies have measured the composition of the deposited layer using Auger electron spectroscopy [1]. Atom probe microscopy has typically been applied to studying a variety of metals, semiconductors, and some ceramics; here, Pt deposited from an organometallic precursor was studied utilizing a laser pulsed local electrode atom probe to reveal the three-dimensional microstructure of this metallic film.

The Pt was deposited using a 30kV Ga ion beam and 300pA beam current. The sample for atom probe analysis, which requires needle geometry with an apex measuring ca. 100nm in diameter, was achieved using a focused ion beam annular milling technique [2].

Transmission electron microscope (TEM) observations of organometallic deposited Pt films reveal an amorphous structure with bright and dark regions associated with C and Pt rich regions respectively, Fig. 1. The TEM images, displaying a projection through the film reveal Pt granule sizes of 6 +/- 2 nm.

The data attained with laser pulse atom probe microscopy improves upon limitations of a TEM image of the specimen revealing a 3-dimensional view of the Pt granules, Fig. 2, their distribution, the region between them, and the anisotropic distribution of Ga around the Pt granules. The size of the Pt granules observed using atom probe microscopy is 5.5 +/- 1.2 nm in diameter with a number density of ca. 9 x 10^{23} m^{-3}. Each granule shows an increased density of Ga on the side towards the top of the specimen (the side from which both the Pt was deposited with a Ga beam and the annular FIB sharpening was applied). The overall composition (in at. %) was measured to be 56% Pt, 26% Ga, 16% C, and 2% O & hydrogen compounds, similar to the referenced Auger analysis [1]. Furthermore an analysis cylinder traversing one particle along the axis of the specimen reveals the intergranular composition is 72 4% Pt, 25 4% Ga, 2 1.5% C whereas intragranular composition is 50 4% Pt, 16 4% Ga, 32 3% C. The Ga pile-up concentration reaches 76 4% on the side exposed to the ion beam and is ca. 0.8 nm thick.

[1] Tao Tao, JaeSang Ro, John Melngailis, Ziling Xue, and Herbert D. Kaesz. J. Vac. Sci. Technol. B 8 (6) (1990).
[2] Larson, D. J., D. T. Foord, and A. K. Petford-Long. Ultramicroscopy (1999).

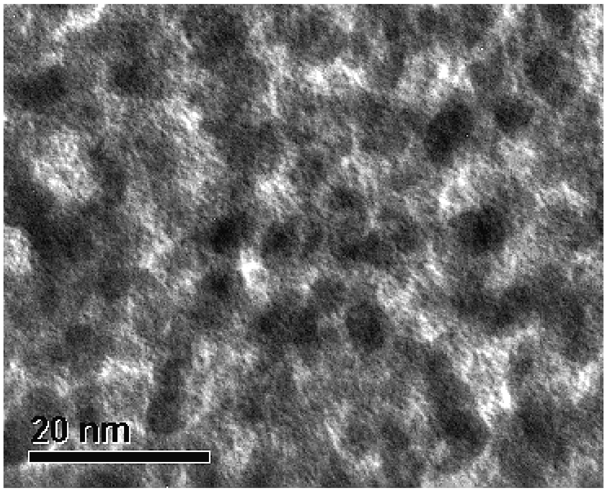

Fig. 1. TEM image of FIB deposited Pt layer. Regions of dark contrast have higher Pt concentration.

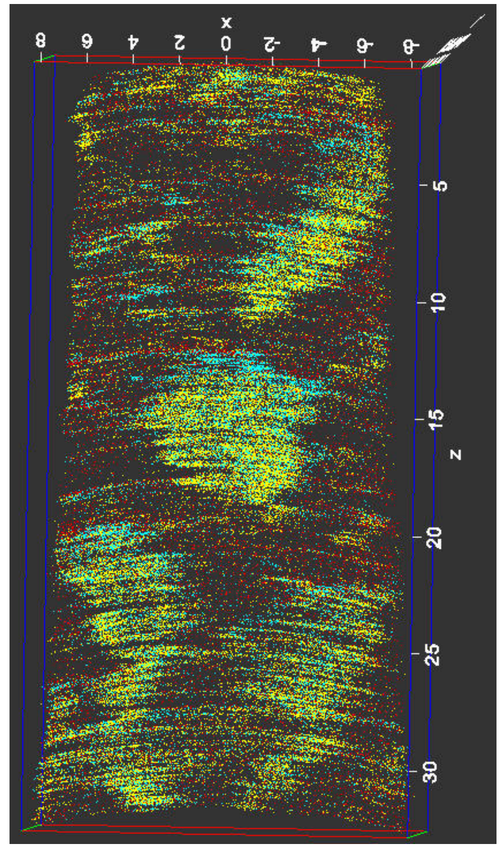

Fig. 2. 1.5 nm slice of a 3-dimensional reconstruction showing Pt (yellow), Ga (light blue), and C (red) atom locations revealing localized Pt granules and anisotropic Ga concentrations. The volume is 17 nm x 17 nm x 33 nm.

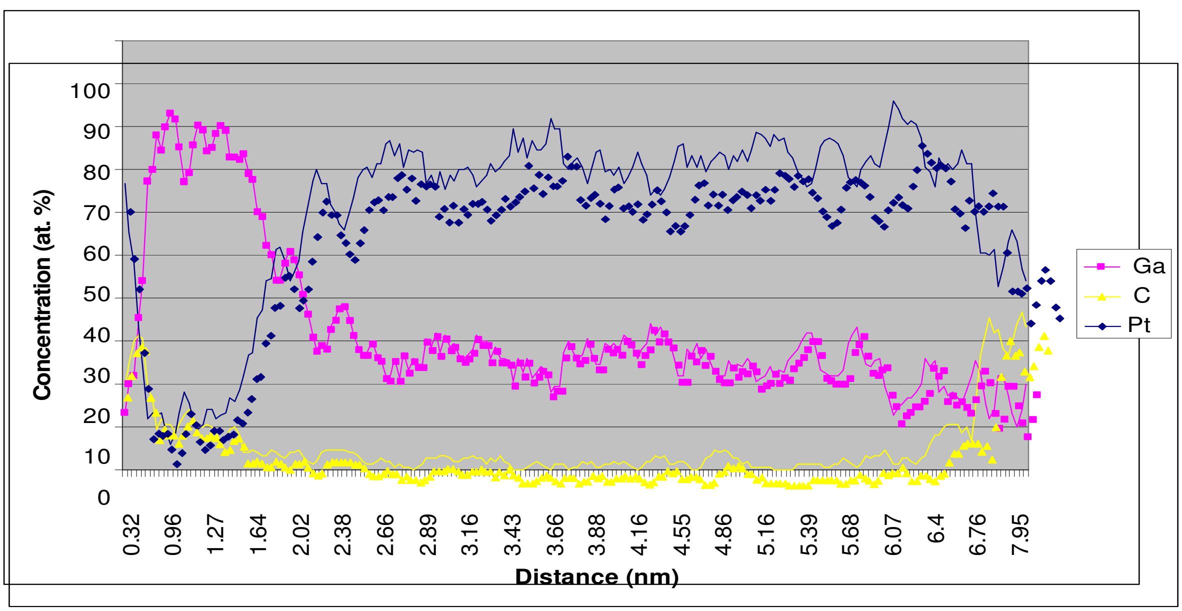

Fig. 3. Composition profile through a subsection of the 3-D reconstruction showing up to 80 at.% Ga concentration on one side of a Pt granule decreasing to below 20 at.% on the opposite side.

https://doi.org/10.1017/S1431927606068450 Published online by Cambridge University Press