

Quantitative EDS of Surface Modified Pd Powders for Hydrogen Storage.

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Palladium and its alloys are known to be useful for applications such as catalysis [1], electrocatalysis [2], and hydrogen isotope storage and separation [3, 4]. Atomic-scale surface and subsurface layers of other metals have surface hydride energetics that may enhance the kinetics of hydrogen absorption and desorption [5]. In addition, increasing the surface area/volume ratio by making Pd alloys nanoporous enhances surface-limited reaction rates. When Pd is alloyed with a higher melting temperature metal such as Rh, the temperature range over which the nanoporous structure remains morphologically stable is extended [6]. We use a variety of techniques to fabricate surface modified Pd powders at different length scales, including, the consolidation of dendrimer-encapsulated nanoparticles [7], surfactant templates [8], and atomic layer electroless deposition (ALED) [9]. It is crucial to understand the spatial distribution of alloying elements in these structures because of the large effect it has on thermal stability, hydrogen storage properties, and the kinetics of hydrogen uptake and release.

Quantitative EDS was performed on Rh and Pt coatings applied to Pd powders at several length scales. Surface-modified powder particles with 5 nm diameter were fabricated using dendrimer encapsulation [10], 100-nm diameter particles were fabricated with surfactant templates [11], and ALED was used on powders >1 μm in diameter [9]. In all cases, the same analysis routines that utilize a combination of multivariate statistical analysis and multiple-least squares fitting was used to quantify the composition and thickness of the surface modified layer(s). We will discuss the importance of large-area SDD EDS detectors for acquiring data on these surface modified layers that range from tens of nm to sub-nm dimensions.

References:

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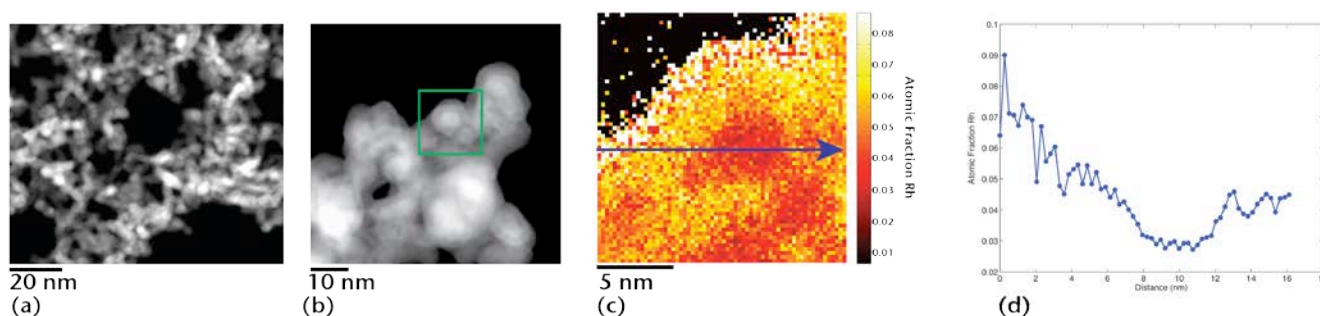


Figure 1: (a) Lower magnification HAADF STEM image of 5 nm-diameter Pd particles made with dendrimer encapsulation. The green-boxed region in (b) is selected for EDS analysis. The Rh concentration is mapped in (c), and a line profile for the region marked with a blue arrow is shown in (d). The core-shell compositional structure of the particles is visible in the Rh concentration map.

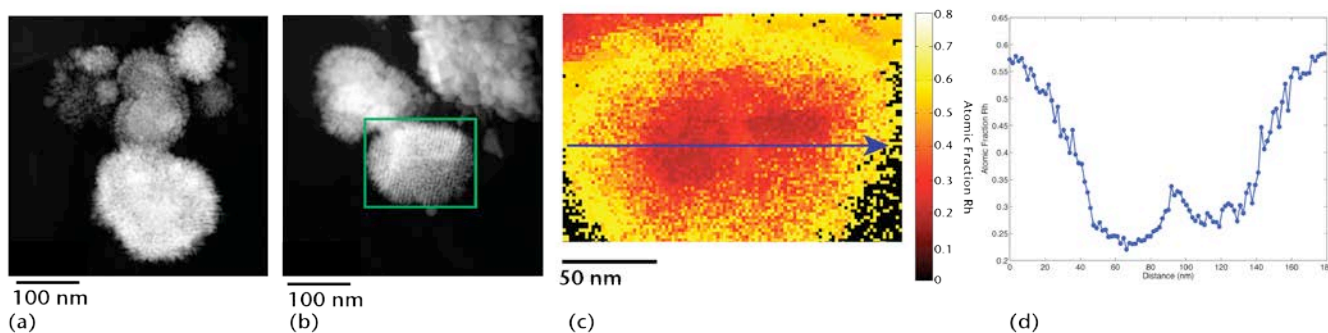


Figure 2: (a) A HAADF STEM image shows several nanoporous, 100 nm-diameter Pd particles made with surfactant templates. The green-boxed region in (b) is selected for EDS analysis. The Rh concentration is mapped in (c), and a line profile for the blue arrow is shown in (d). Again, the core-shell compositional structure of the particles is visible.

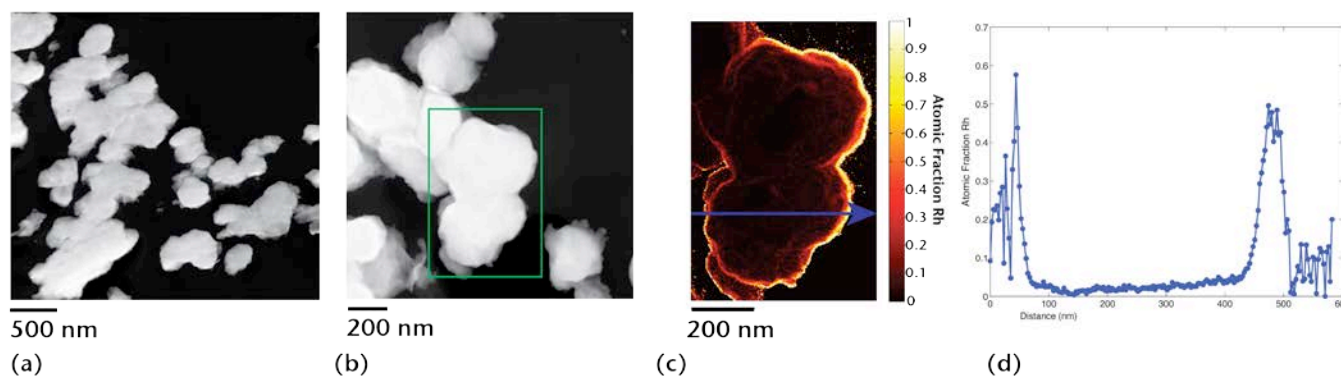


Figure 3: (a) HAADF STEM image of ~ 1 μm -diameter Pd particles that have been surface modified with ALED. These samples were prepared using ultramicrotomy. The green-boxed region in (b) is selected for EDS analysis. The Rh concentration is mapped in (c), and a line profile for the blue arrow is shown in (d). The surface enrichment in Rh is visible.