

Low-Energy EELS of Au Nanoparticles

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Gold nanoparticles possessing a variety of geometries (spheres, rods, triangles, etc.) have been characterized using transmission and reflection mode electron energy-loss spectroscopy (EELS) [1]. In a transmission electron microscope (TEM) with a highly focused high-energy (> 100 keV) electron beam, bulk atomic composition, bulk electronic structure, and chemical state can be determined at the nanoscale; however, there is limited surface sensitivity and energy resolution [2]. In high-resolution (HR) reflection EELS experiments, a low-energy (< 70 eV) broad-spot electron beam incident on a specimen can provide surface-sensitive information, including phonons, surface plasmons, and vibrational spectra of adsorbates with thicknesses less than a molecular layer on the surface and energy resolutions below 10 meV (roughly 80 cm^{-1}) [3]. However, the macroscopic beam spot precludes analysis of individual nanoparticles. The present study demonstrates how an analyst can perform reflection EELS at low energy in an Auger electron microscope, thereby achieving some of the surface sensitivity of an HREELS instrument at resolutions approaching that of a TEM or other high-energy EELS instrument. Increasing surface sensitivity with decreasing beam energy is also demonstrated.

Scanning electron microscopy (SEM) and low-energy EELS were carried out on a JEOL JAMP 7830F Scanning Auger Microprobe equipped with a concentric hemispherical analyzer [4] to study both the graphite substrate and gold nanoparticles [SPI SEM gold-on-carbon test spec small 1504 lot#1100216 (nominally 10 nm in diameter)], as well as a gold foil for comparison. Individual gold nanoparticles were identified with the SEM (Fig. 1), after which EELS spectra were collected at beam energies from 800 eV to 2000 eV. At lower beam energies, the surface spectral features were more pronounced than at higher energies (Fig. 2). Principal component analysis and linear discriminant analysis were used to post-process the data and identify trends in the data with changing beam energy (Fig. 3).

References

- [1] B. Schaffer, K. Riegler, G. Kothleitner, W. Grogger, and F. Hofer (2009). Monochromated, spatially resolved electron energy-loss spectroscopic measurements of gold nanoparticles in the plasmon range. *Micron* **40**, 269-273.
- [2] R. F. Egerton (1996). *Electron Energy-Loss Spectroscopy in the Electron Microscope – Second Edition*. New York, NY: Plenum Press.
- [3] D. P. Woodruff and T. A. Delchar (1994). *Modern Techniques of Surface Science – Second Edition*. Cambridge, UK: Cambridge University Press.
- [4] The full description of the procedures used in this abstract requires the identification of certain commercial products and their suppliers. The inclusion of such information should in no way be

construed as indicating that such products or suppliers are endorsed by NIST or are recommended by NIST or that they are necessarily the best materials, instruments, software or suppliers for the purposes described.



Fig. 1. SEM of Au nanoparticles on graphite (20 keV, 50 kX).

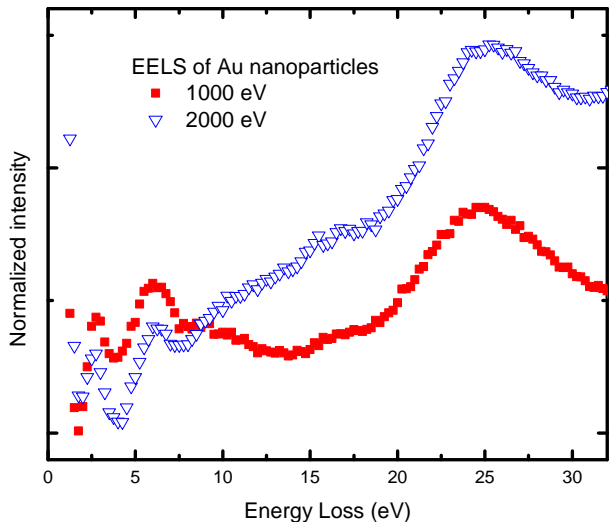


Fig. 2. Mean of the zero-centered and unit-scaled EELS spectra of gold nanoparticles at two different accelerating voltages. The low-energy surface sensitive features (<10 eV) are more prominent in the spectra collected using a 1000 eV incident beam.

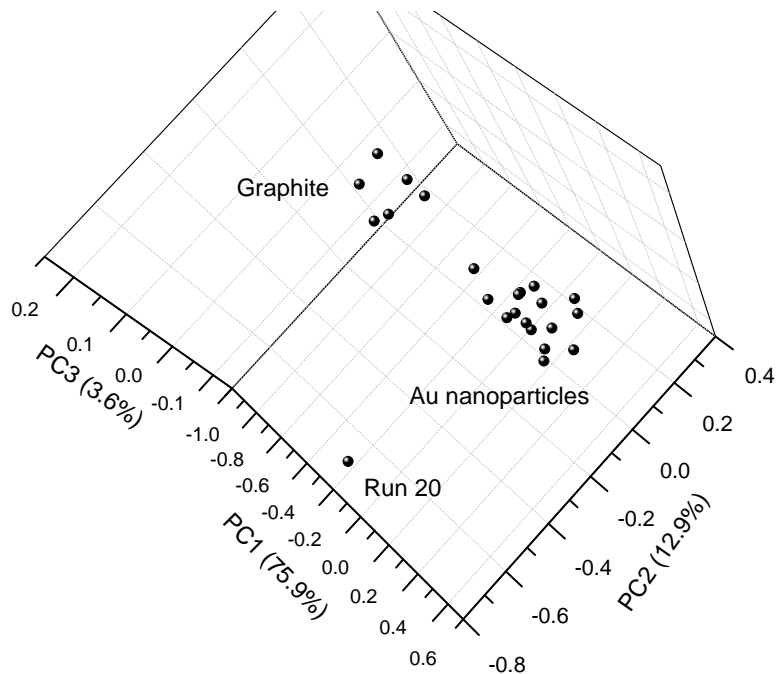


Fig. 3. Principal component analysis of graphite and gold nanoparticles EELS data collected at 2000 eV beam energy. The first three principal components of the set are shown, and the graph is rotated for best visualization of the two main clusters. A deviation from the two main clusters (Run 20) could indicate cross-particle interactions, contamination, or other effects.