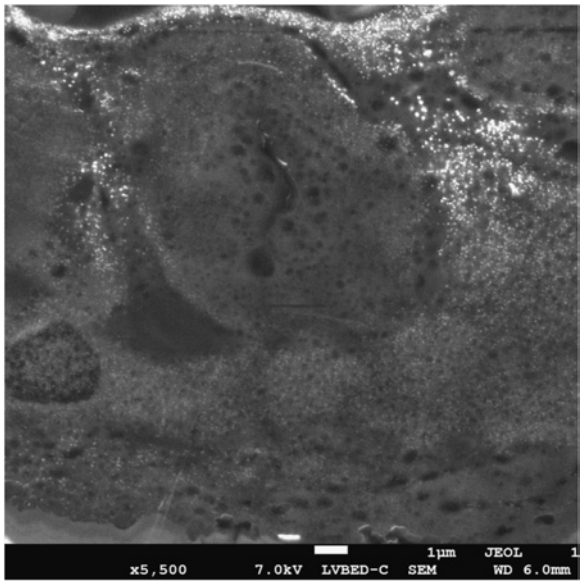
Highlights from Microscopy AND Microanalysis

Materials Applications

An Examination of the Surface and Sub-Surface of Modern and Historical Platinum Photographic Prints Using Low Vacuum High-Resolution Scanning Electron Microscopy by Patrick Ravines, N Erdman, and R McElroy, *Microsc Microanal* 22 (2016) 857–64

Photographic prints of platinum metal on paper supports, produced from about 1890 to the 1920s, are some of the most exquisite and expressive in fine art photography, and platinum/palladium printing remains as one of the most expressive media in use today. The chemical and material nature of these valuable prints is of great interest to those interested in their long-term preservation, in the intersection of science and art, and in the scientific/technical study of cultural heritage. A nineteenth-century print by the Smith Curry Studio and a 2005 palladium/platinum print prepared using Bostick and Sullivan chemical solutions were compared to determine platinum nanoparticle distribution. Low vacuum scanning electron microscopy and energy-dispersive spectroscopy (EDS) of the surface and sub-surface of argon plasma prepared cross sections showed that the nineteenth-century print is composed of platinum nanoparticles embedded in the upper layers of the paper's cellulosic fibers. The greatest number of particles was immediately beneath the surface in the primary wall and deep within paper fibers up to a depth of 10 µm. The bulk of image particles deposit directly beneath the fiber's surface in the primary wall, between the primary wall and outer secondary wall, and between other more internal walls.

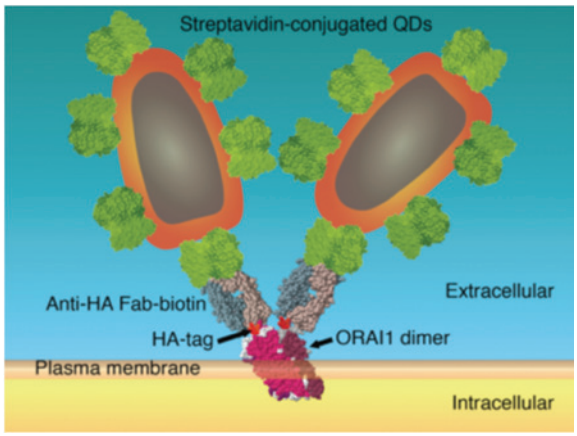


Backscattered electron image of a photographic print cross section prepared using argon ion plasma showing the internal distribution of the Pt image nano-particles. In this sample Pt particle size is below 50 nm.

Biological Applications

Visualizing Quantum Dot Labeled ORAI1 Proteins in Intact Cells via Correlative Light and Electron Microscopy by DB Peckys, D Alansary, BA Niemeyer, and N de Jonge, *Microsc Microanal* 22 (2016) 902–12

ORAI1 proteins are ion channel subunits that are the essential pore-forming units of the calcium release activated calcium (CRAC) channel complex essential for T cell activation and other cellular processes. While these proteins are synthesized as monomers, they assemble into multimeric complexes to form active hexameric channels. But these structures have never been observed directly in the intact plasma membrane of a whole cell. Environmental scanning electron microscopy (ESEM) was used in combination with scanning transmission electron microscopy (STEM) to image plasma-membrane-expressed ORAI1 proteins in whole Jurkat T cells in the liquid state. For visualization of individual protein positions, streptavidin coated quantum dots (QDs) were coupled to the proteins via an extracellular human influenza hemagglutinin (HA)-tag cloned into the protein. The schematic model shows the individual QD label positions, from which the cluster formation was analyzed. From data analysis of cells that also contained unlabeled endogenous protein, it followed that labeled ORAI1 was present in hexamers in a small fraction only and resided mostly in monomers and dimers.



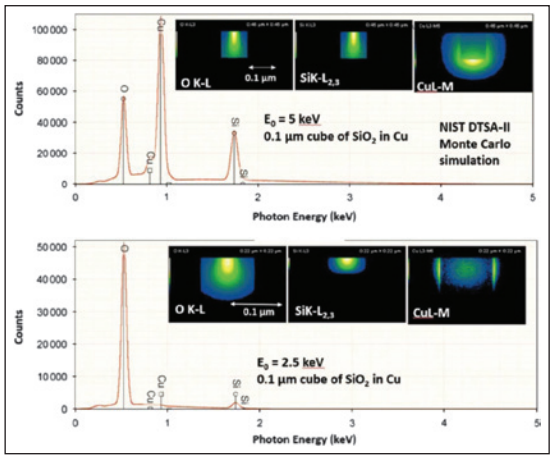
Schematic model of the coupling of two QDs to an ORAI1 dimer. Individual ORAI1 proteins in the intact plasma membrane of whole cells were imaged in the liquid-phase, which provided information on the location of the QD labels attached to ORAI1 proteins.

Microscopy AND Microanalysis

Techniques and Instrumentation Development

Electron-Excited X-ray Microanalysis at Low Beam Energy: Almost Always an Adventure! by DE Newbury and NWM Ritchie, *Micros Microanal* 22 (2016) 735–53

"Low beam energy" x-ray microanalysis performed in the scanning electron microscope at landing energies $E_0 \leq 5$ keV offers improved lateral and depth resolution, typically sampling volumes with linear dimensions of a few hundred nanometers or less. Special analytical challenges arise because the reduced beam energy can only stimulate x rays from low-ionization-energy atomic shells. Thus, transition metals usually analyzed with K-shell x rays must instead be analyzed with L-shell x rays, while for intermediate and high atomic number elements the M-shell must be used. Low photon energy x-ray families have low yield and complex, variable shapes from multiple, closely spaced peaks. Nevertheless, accurate quantitative analysis (relative to elemental and simple compound standards) can often be achieved by employing the peak fitting and matrix correction procedures embedded in the NIST DTSA-II EDS analysis and modeling software engine. Other challenges to analytical interpretation arise because surface layers such as natural oxides, which are so thin as to be insignificant at high beam energy ($E_0 \geq 10$ keV), account for a much larger fraction of the material sampled at low beam energy. Iterative DTSA-II analysis must be performed wherein the residual spectrum after peak fitting is inspected for peaks of unanticipated constituents.



Monte Carlo simulation (NIST DTSA-II) of electrons bombarding a 100 nm cube of SiO_2 in a Cu matrix. At 5 keV, electrons penetrate the SiO_2 cube and excite Cu x rays, but at 2.5 keV few electrons reach the Cu, so the Cu peak is barely detectable above background.