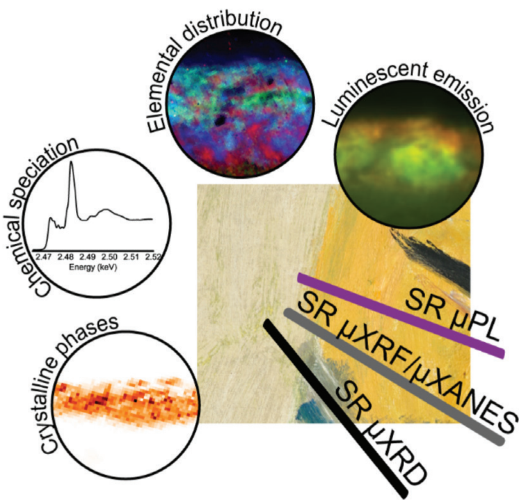
Highlights from Microscopy AND Microanalysis

Materials Applications

Application of Synchrotron Radiation-Based Micro-Analysis on Cadmium Yellows in Pablo Picasso's *Femme* by M Ghirardello, V Gonzalez, L Monico, A Nevin, D MacLennan, C Schmidt Patterson, M Burghammer, M Réfrégiers, D Comelli, and M Cotte, *Microsc Microanal* | https://doi.org/10.1017/S1431927622000873.

The combination of synchrotron radiation (SR) X-ray micro-techniques is often exploited by the cultural heritage community to investigate microsamples taken from artworks. Less frequently, these techniques are combined with other complementary measurements exploiting SR sources. Although it is not straightforward to correlate maps obtained by probing the samples with different radiation energies related to different physical phenomena, the combination of information provides a unique insight into the paint chemistry. In this work, we present the combination of SR X-ray and SR photoluminescence techniques to study two paint microsamples taken from Pablo Picasso's painting *Femme* (1907) (Figure). The painting contains two cadmium yellow paints: one relatively intact and one visibly degraded. SR micro-analyses demonstrated that the two cadmium yellow paints differ in terms of structure, chemical composition, and crystal defects. Based on the combination of different SR measurements, we hypothesize that the degraded yellow consists of nanocrystalline CdS with a high amount of Cd(OH)Cl. These two characteristics have enhanced the reactivity of this paint, affecting its stability and leading to its degradation.

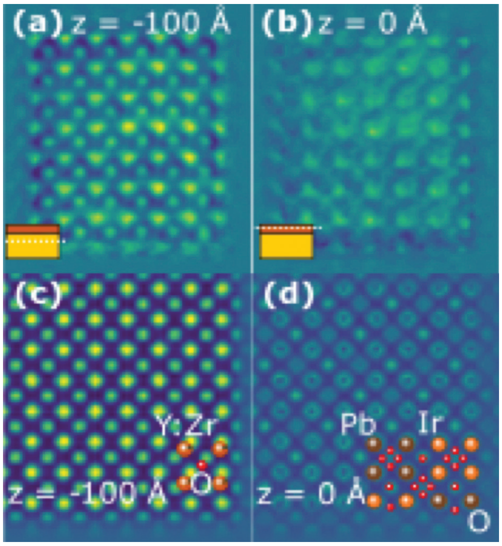


Overview of the different information obtained on the microsamples taken from Picasso's painting *Femme* using synchrotron radiation (SR) X-ray-based techniques (micro-X-ray fluorescence (XRF), X-ray absorption near edge structure (XANES), and X-ray diffraction) and SR deep-UV micro photoluminescence (PL).

Techniques

A Three-Dimensional Reconstruction Algorithm for Scanning Transmission Electron Microscopy Data from a Single Sample Orientation by HG Brown, PM Pelz, S-L Hsu, Z Zhang, R Ramesh, K Inzani, E Sheridan, SM Griffin, M Schloz, TC Pekin, CT Koch, SD Findlay, LJ Allen, MC Scott, C Ophus, and J Ciston, *Microsc Microanal* | https://doi.org/10.1017/S1431927622012090.

While a single transmission electron microscopy (TEM) image contains only two-dimensional information about one's sample, often three-dimensional information is of interest. This proof-of-principle experiment demonstrated three-dimensional imaging of a $Pb_2Ir_2O_7$-$Y_{0.05}Zr_{0.95}O_2$ heterostructure by a depth-sectioning approach from the scattering (S) matrix. The S matrix formulation is widely used to represent multiple scattering in quantum mechanics, which we reconstruct here from 4D-STEM data. The result is shown in the figure with distinct (a) $Y_{0.05}Zr_{0.95}O_2$ and (b) $Pb_2Ir_2O_7$ layers visible. The Pb-Ir columns scatter electrons so strongly that phase-wrapping — dips in the measured phase of the columns — become apparent. Reconstructions from equivalent simulated data are shown for the same layers in (c) and (d) for comparison. This research demonstrates how three-dimensional information is encoded in the rich datasets recorded by the 4D-STEM technique, even for a single-specimen orientation.



Different depth sections reconstructed from 4D-STEM experimental data of a $Pb_2Ir_2O_7$-$Y_{0.05}Zr_{0.95}O_2$ heterostructure. (a) $Y_{0.05}Zr_{0.95}O_2$ and (b) $Pb_2Ir_2O_7$ layers are compared with reconstructions from simulated data in (c) and (d) respectively.

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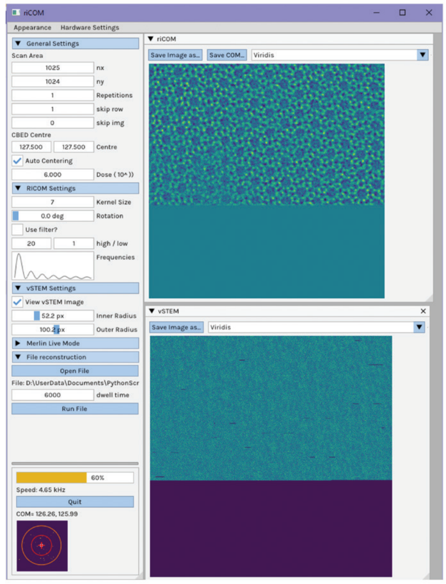
Techniques

Real-Time Integration Center of Mass (riCOM) Reconstruction for 4D STEM by CP Yu, T Friedrich, D Jannis, S Van Aert, and J Verbeeck, *Microsc Microanal* | https://doi.org/10.1017/S1431927622000617.

Scanning transmission electron microscopy (STEM) utilizes the intensity distribution of the convergent beam electron diffraction patterns (CBEDs) at each probe position to build a 2-dimensional representation of the sample, for example, by the number of electrons falling into the dark field region. The integrated signal of the center of mass (iCOM) shift of the CBED is another popular method. It has desired properties such as low-dose robustness and the capability of imaging atomic columns of large differences in projected potential. In this work, the iCOM algorithm is altered such that the integration range is tunable and individual CBEDs from single probe positions can be independently processed. This enables simultaneous data collection and real-time update of the reconstructed image. The tunable integration range shows interesting high-pass filtering effects, which strongly alters the contrast of the reconstructed image. This enables users to emphasize long-range, across-sample contrast, or localized, atomic scale contrast. Data, codes, and the program for reconstruction are shared in online repositories https://doi.org/10.5281/zenodo.5572123 and github.com/ThFriedrich/riCOM_cpp.



Screen shot of real-time iCOM reconstruction (top) and virtual annular detector reconstruction (bottom) on silicalite-1 zeolite. Parameters can be adjusted in the left panel, including the number of pixels in each direction, kernel size, virtual detector range, and dwell time.