

Development of a Wide-Range X-ray Emission Spectroscopy Measurement System with Transition Edge Sensors and Microwave Multiplexed Readout

Matthew H. Carpenter¹, Kathryn G. McIntosh¹, Joel N. Ullom^{2,3}, Douglas A. Bennett², Daniel S. Swetz², Carl D. Reintsema², Johnathon D. Gard³, Daniel T. Becker³, John A.B. Mates³, Kelsey M. Morgan³, Jozsef Imrek³, Abigail L. Wessels³, Mark Croce¹ 1) Los Alamos National Laboratory; 2) National Institute of Standards and Technology; 3) University of Colorado

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NIST

Motivation

Goal: Measure high-resolution X-ray emission spectra (XES) of model compounds to calibrate atomic models and build library of chemical spectral information.

LANL Hyperspectral X-ray Emission (HXI) **Project application: Uranium compounds** Existing U M emission spectra typically only address metallic U. We need comprehensive measurements of U M spectra of different chemical compounds to demonstrate chemical differences and to build accurate theoretical models. (See Croce et. al HXI poster LA-UR-19-26600)

Example published metallic U M spectra showing minor peaks (satellites) and spectral structure:

XES System: Design

Adapt existing ADR cryostat instrumented for microwave TES readout with hybrid X-ray TES array and X-ray analysis chamber of bulk samples. X-ray excitation gives low-background (Bremsstrahlung-free) emission spectrum.

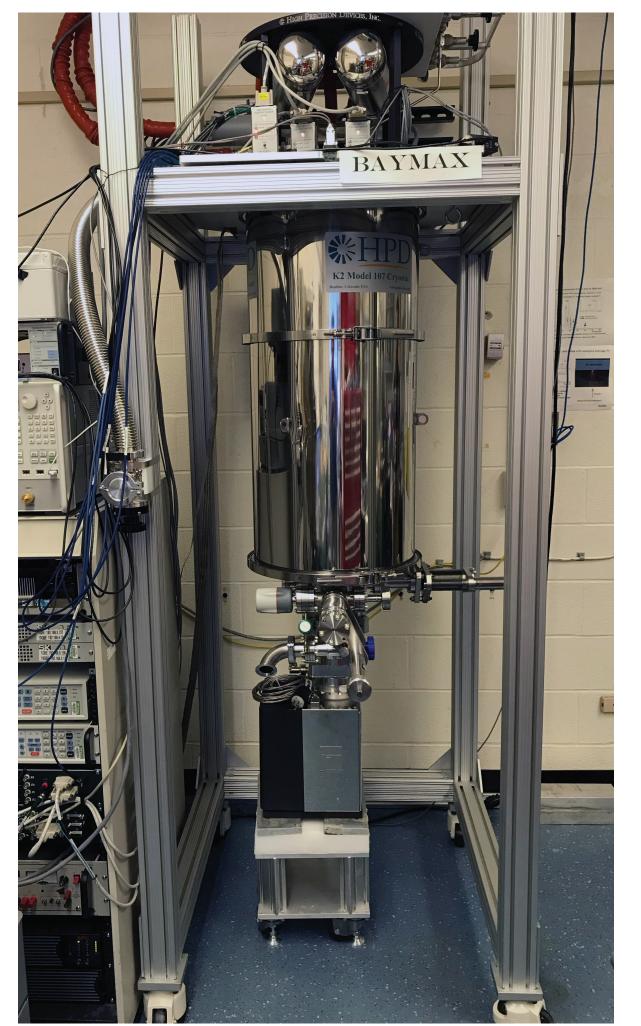
- Separate vacuum between cryostat and chamber
- lon pump for min. vibration
- 5 sample positions using SEM pin stubs
- Sample-detector distance approx. 140 mm

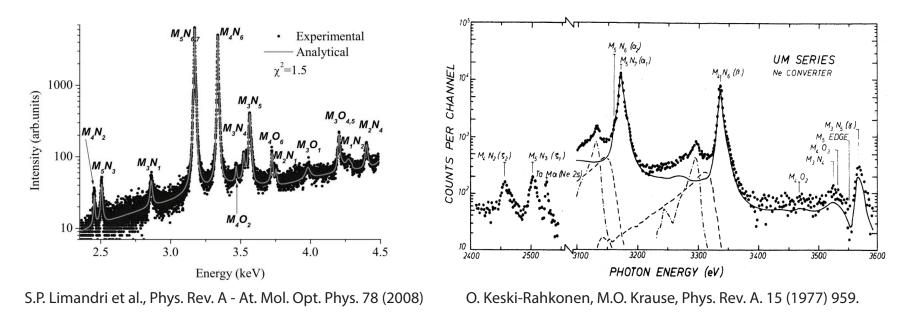
System Commissioning

Current status:

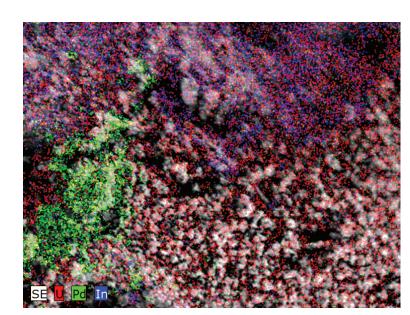
- Detector package installed in cryostat, microwave readout tested
- X-ray analysis chamber assembled and awaiting X-ray generator installation

X-ray sample chamber installed on cryostat

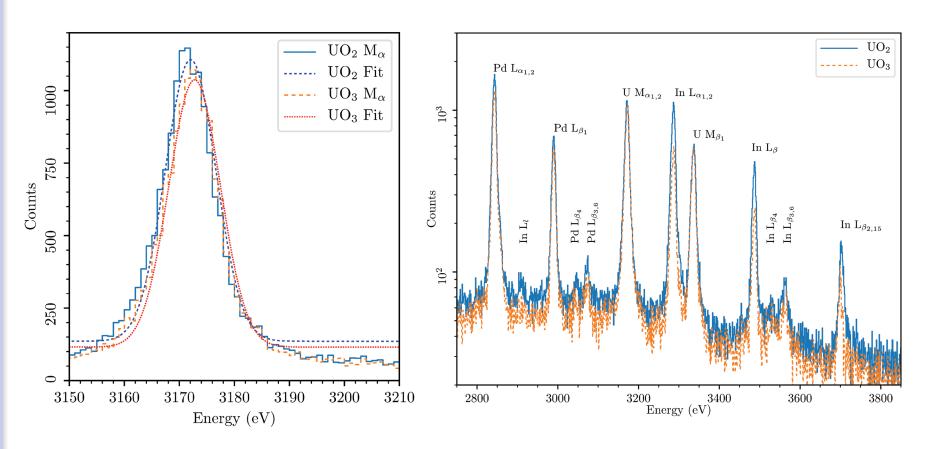


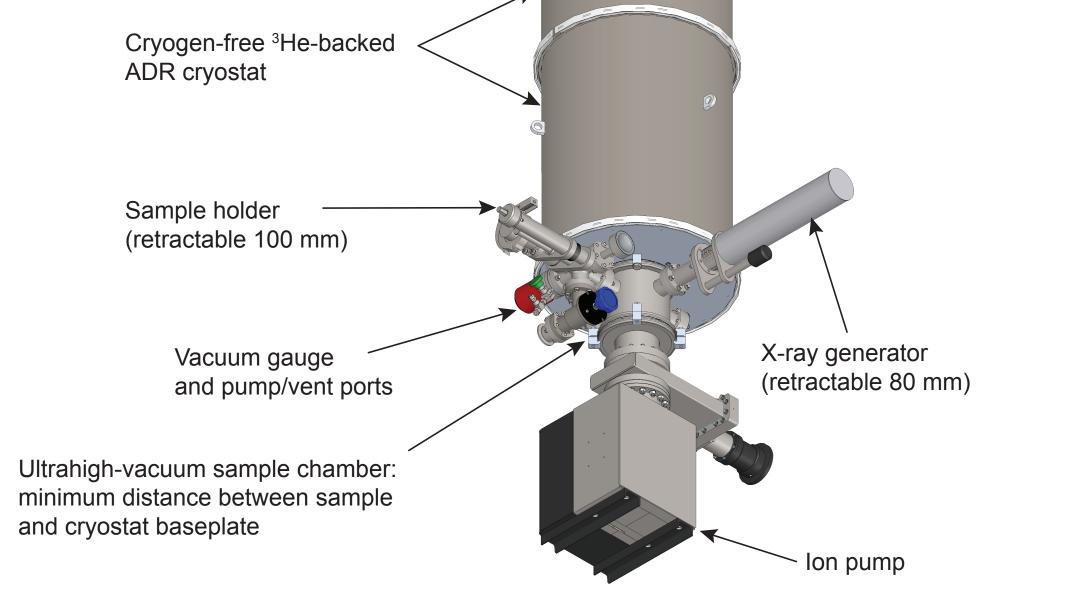


HXI Project application: U M spectroscopy with in-situ calibration in SEM. Upper Left: SEM image of UO₂ mixed with Pd and In. Lower Right: U M region acquired with STAR Cryoelectronics MICA-1600. *Lower Left*: U M_a showing shift of ~700 meV. Precise in-situ calibration is needed to constrain

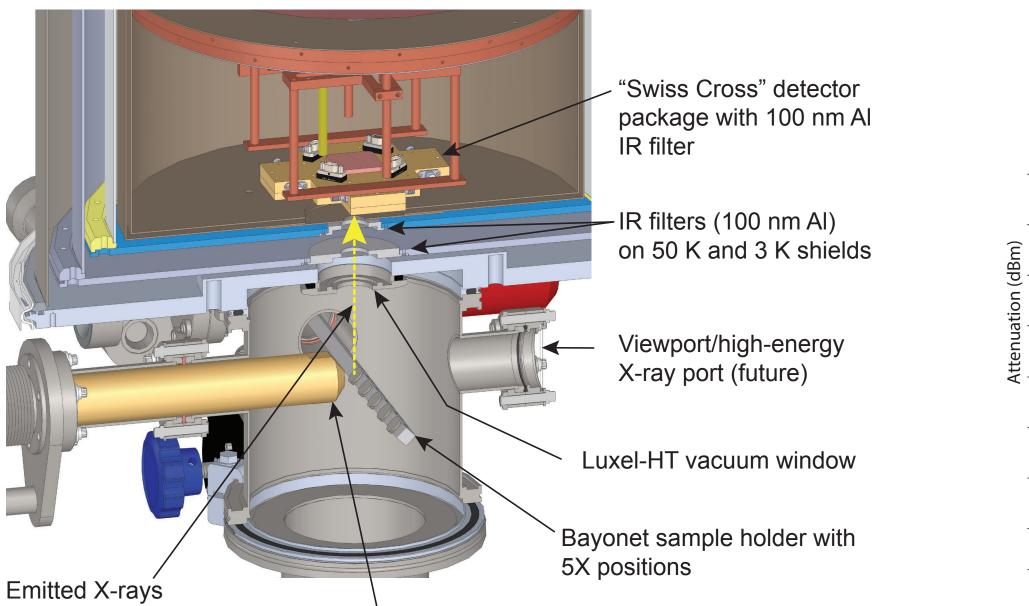


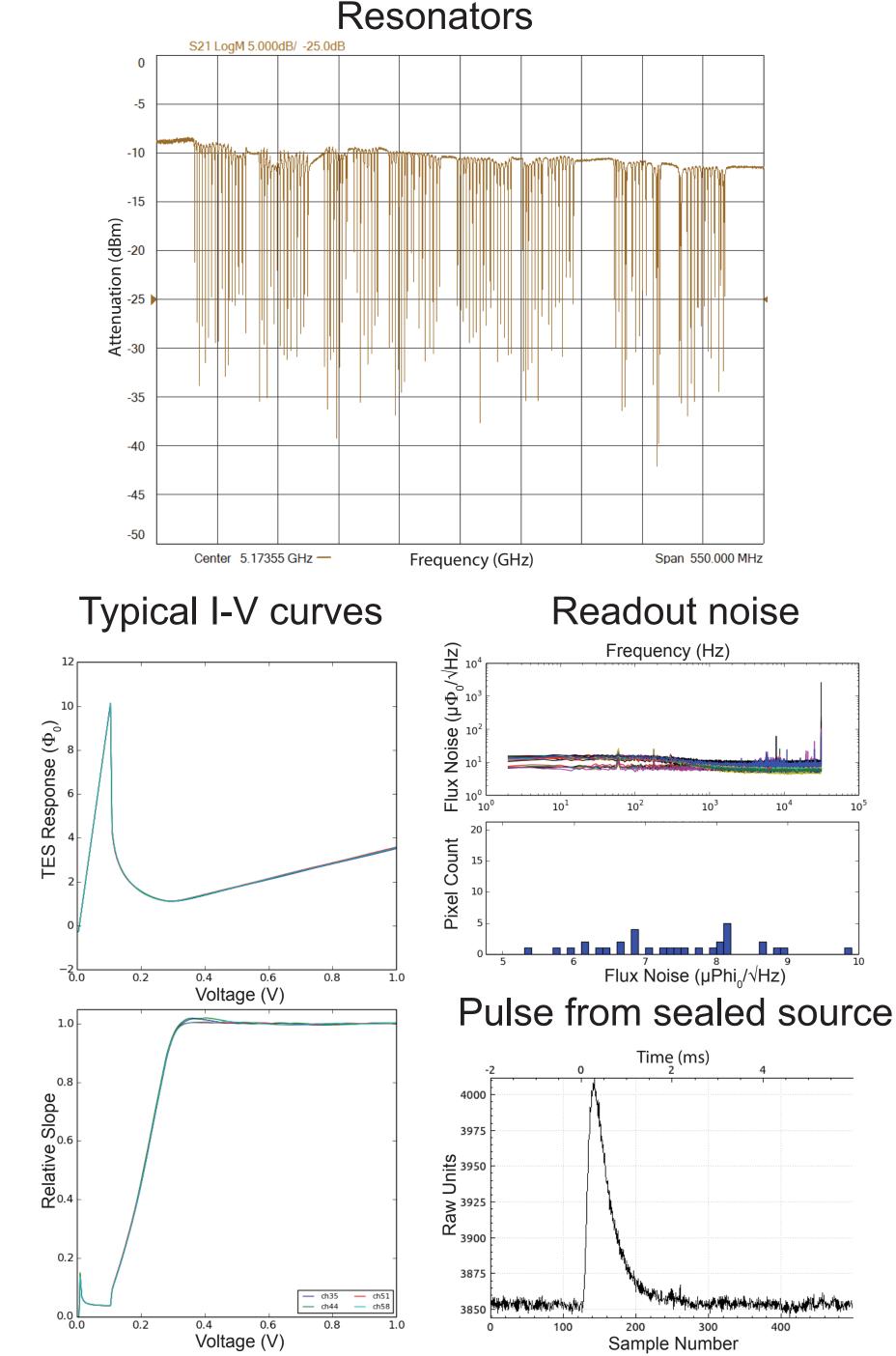
the U M peak position within 100 meV. High-resolution X-ray emission spectroscopy will validate theoretical models and provide a database of chemical shifts and positions/intensities of minor peaks (satellites).





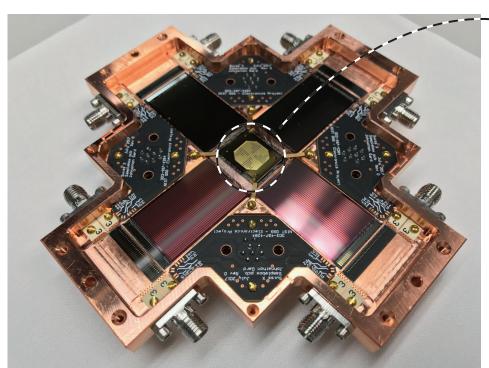
Cutaway view of sample chamber and cryostat



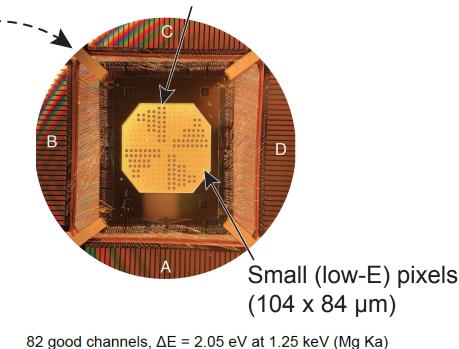


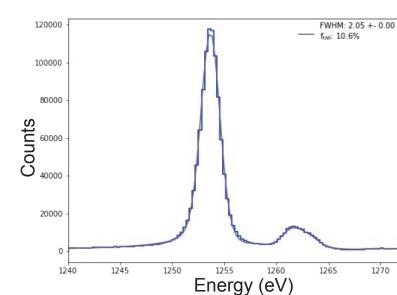
NIST TES Detector Arrays

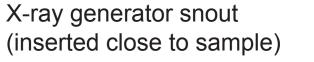
New 128-pixel TES detector arrays from NIST incorporate two pixel sizes for lowand high-energy-range performance, with RF-SQUID microwave multiplexed readout from the University of Colorado. Large (high-E) pixels (320 x 305 µm)



"Swiss Cross" detector package: 128 pixels bonded at once (choose large or small). Integrated **RF-SQUIDS** and resonators allow readout of all 128 pixels on single feedline @ ~5 GHz.



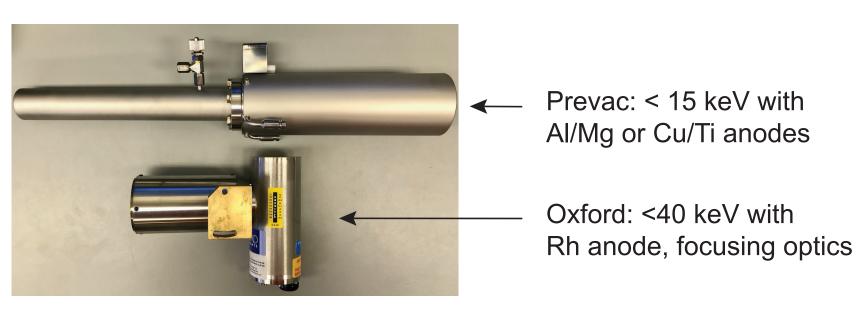




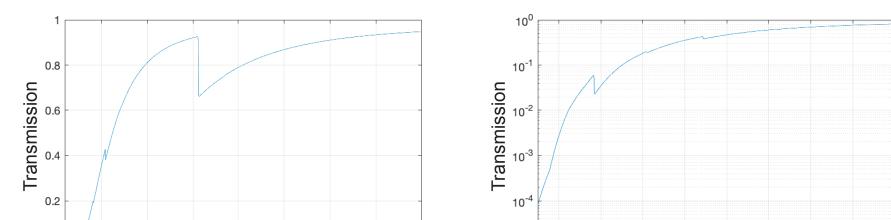
Choice of X-ray generators:

(to detector)

- Prevac RS40B1 for low energy (U M/N emission, low-Z elements); Al/Mg or Cu/Ti anode
- Oxford XTF5011A for high energy (U L emission), Rh anode with focusing optic



X-ray transmission of vacuum window + IR filters: suitable for light element analysis (K emission from C, N, O, F) simultaneous with heavy elements



Summary: X-ray spectroscopy system based on TES detectors will allow high resolution over a large energy range (200 eV - 15 keV). This will enable simultaneous high-precision measurement of X-rays from heavy and light elements in a variety of compounds to validate

