doi:10.1017/S1431927614007466
Microsc. Microanal. 20 (Suppl 3), 2014

Ultrahigh-Resolution X-ray Microanalysis with a Cryogen-Free Microcalorimeter Spectrometer

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X-ray detectors based on superconducting microcalorimeters currently represent the most attractive technology capable of <5 eV resolution for energy dispersive spectroscopy and microanalysis applications. The superior energy resolution as compared with conventional solid state X-ray detectors enables the analysis of nanometer-scale device features, as well as quantitative analysis for the inspection, characterization, and compositional analysis of nanometer-scale contaminant particles and defects. In order to analyze features at this length scale, the X-ray generation volume in the sample must be nanometer scale as well. With current-generation field emission scanning electron microscopes it is possible to reduce the X-ray generation volume to a depth under 100 nanometers for high spatial resolution imaging by operating at reduced electron beam energies. At these low energies, however, the electron beam excites only low-energy elemental X-ray lines, which conventional solid state detectors are unable to resolve owing to severe peak overlaps at these low energies.

Cryogenic microcalorimeter X-ray detectors based on superconducting transition edge sensors (TES) offer up to a roughly 60-fold improvement in energy resolution as compared with conventional detectors for energy-dispersive spectrometry. The best energy resolutions demonstrated to date are 2.0 eV full width at half maximum (FWHM) at 1.5 keV (Al-K α) and 2.4 eV FWHM at 5.9 keV (Mn-Kα). The energy resolution of state-of-the-art microcalorimeter detectors rivals the resolution of spectrometers for wavelength-dispersive spectrometry (WDS), yet microcalorimeters offer all the advantages of EDS detectors - ease of use, long-term stability, and the ability to quickly provide qualitative as well as quantitative chemical analysis.

An X-ray microcalorimeter consists of three parts: an absorber that captures the energy of the incident X-ray, an extremely sensitive thermometric element to measure the temperature rise of the absorber following an X-ray absorption event, and a support and thermo-isolation structure that determines the rate of heat loss from the microcalorimeter. The thermometric element for a TES microcalorimeter consists of a superconducting thin film operated at its superconducting transition. A TES is particularly attractive for these applications owing to its high sensitivity $\alpha = d(\log R)/d(\log T)$ and fast response time achievable using a novel electrothermal feedback technique. Normal metal/superconductor bilayers are typically used to fabricate transition edge sensors, since the superconducting critical temperature can easily be tuned to the desired operating temperature (around 0.1 K) by adjusting the strength of the proximity-effect coupling, and they can be made with low resistivity to ensure fast thermal response times. Following a photoabsorption event, the sudden temperature rise of the microcalorimeter results in a current pulse through the TES (the height of the pulse being proportional to the incident X-ray energy), which is measured using a low input-impedance superconducting quantum interference device (SQUID) amplifier. The third part of a TES microcalorimeter is the thermal isolation structure, usually a thin membrane upon which the TES bilayer and absorber are fabricated. Typical TES microcalorimeters are fabricated on a micromachined silicon nitride membrane formed by depositing a low-stress,

silicon-rich silicon nitride film on a Si wafer and then etching the backside of the wafer down to the membrane, or using a surface micromachining technique to preferentially etch away a sacrificial lay er underneath the silicon nitride membrane.

Microcalorimeter detectors have been used to develop our MICA-1600 EDS spectrometer based on a pulse tube cryocooler and adiabatic demagnetization refrigerator (ADR) that does not require any liquid cryogens. An example of the ultrahigh-resolution capability is shown in Figure 1, where we show spectra for a 20 nm thick W over 20 nm thick Ta bilayer on a Si substrate at using 3, 5 and 7 keV electron beam energies. At all three electron beam energies, the W, Ta and Si lines are clearly resolved. At 3 keV, the X-ray excitation volum e is almost entirely limited to the top of 20 nm W layer and only the W M α line and a weak T a M α line are observable. As the beam energy is increased, the emergence of the Ta Mα and Si Kα lines can clearly be seen.

In this presentation we describe the cryogenic system and automated controls that simplify the operation of the spectrometer, the installations on Hitachi S-4800 and S-6600 SEMs, and recent application data including qualitative/quantitative analysis and X-ray mapping/line analysis data.

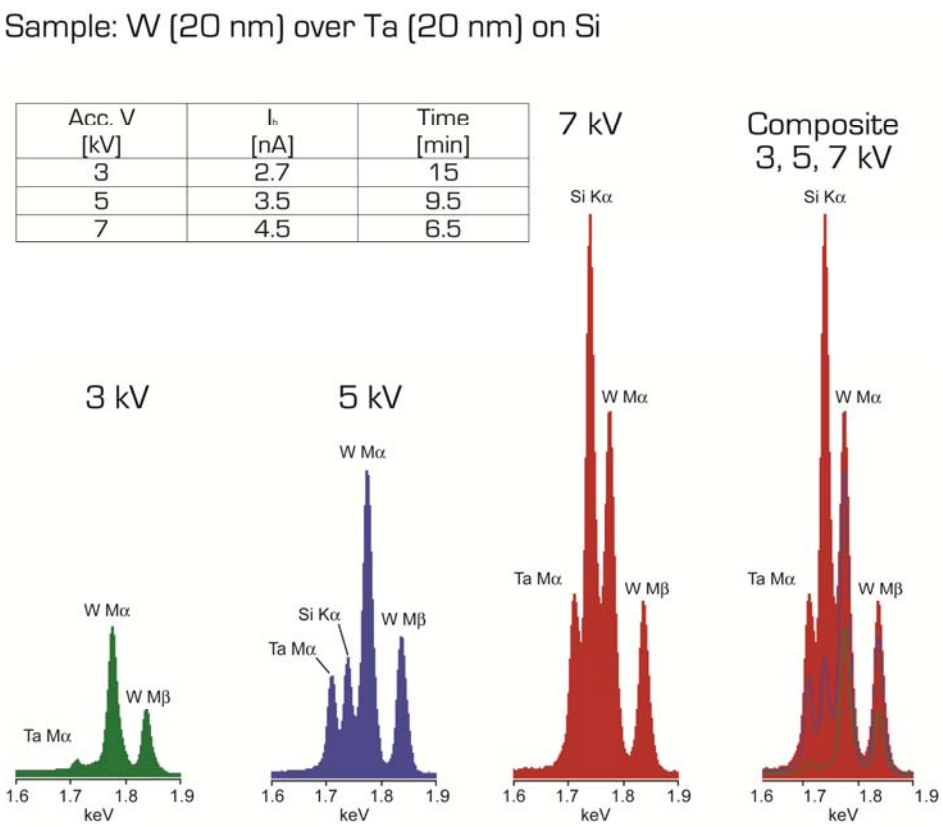

Figure 1. The MICA-1600 clearly resolves Ta, Si, and W peak overlaps around 1.75 keV, enabling surface analysis of nanometer-scale films. The effect of the X-ray excitation volume dependence on beam voltage can easily be seen (5,200 counts/eV full scale, all three spectra have 500,000 counts (range 0 - 4 keV)).

https://doi.org/10.1017/S1431927614007466 Published online by Cambridge University Press