A Microcalorimeter Spectrometer for High-Resolution X-ray Microanalysis

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X-ray detectors based on superconducting microcalorimeters currently represent the most attractive technology capable of <10 eV resolution for energy dispersive spectroscopy and microanalysis applications in the semiconductor industry. This energy resolution is required for the inspection, characterization, and compositional analysis of contaminant particles and defects as well as for the analysis of nanometer-scale device structures. In order to carry out the compositional analysis of nanometer-scale features, the X-ray generation volume in the sample must be nanometer length 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 semiconductor detectors are unable to resolve owing to peak overlaps at these low energies. Cryogenic microcalorimeter X-ray detectors based on superconducting transition edge (TES) sensors offer up to a roughly 60-fold improvement in energy resolution as compared with conventional semiconductor 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 and 2.4 eV FWHM at 5.9 keV. 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 layer underneath the
silicon nitride membrane. These detectors have been used to develop a compact, next-generation EDS spectrometer based on a cryogen-free adiabatic demagnetization refrigerator (ADR).