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Nanocalorimetry: Using Si-micromachined Devices for Thermodynamic Measurements of Thin Films and Tiny Crystals¹

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We have used Si micromachining to fabricate membrane-based calorimeters for measuring thermodynamic properties of microgram-quantity samples over a temperature range from 1.7 to 550K in magnetic fields to 8T. Prototype scaled down devices have been made which allow precise measurements of nanogram quantities. Different types of thermometers are used for different purposes and in different temperature ranges. Current development efforts are extending the temperature range to 0.3 - 800K, and we are collaborating with the national high magnetic field lab to extend the field range to 65T in pulsed magnets. These devices are particularly useful for specific heat measurements of thin film samples (100-400 nm thick) deposited directly onto the membrane through a Si micromachined evaporation mask. They have also been used for small bulk samples attached by conducting paint or In, and for powder samples dissolved in a solvent and dropped onto devices. The measurement technique used (relaxation method) is particularly suited to high fields because thermal conductance is measured in zero field and is field independent, while the relaxation time constant does not depend on thermometer calibration. The devices have been used with little modification for thermal conductivity and thermopower measurements, and are well suited¹ to measurements of calorimetric signals such as those occurring at phase transitions or under irreversible thermal behavior. I will discuss device fabrication and thermal analysis which allow us to precisely identify heat flow in the devices and consequent limits on the absolute accuracy, as well as possible future directions for device development. I will also briefly discuss examples of measurements on several materials of current interest: 1) amorphous Si and its alloys, 2) high precision critical temperature studies of $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$ and $\text{La}_{1-x}\text{Ca}_x\text{MnO}_3$, 3) antiferromagnetic CoO nanoparticles and thin layers, 4) Fe/Cr giant magnetoresistance multilayers.

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