MICRON-SCALE DIFFERENTIAL SCANNING CALORIMETER ON A CHIP

This application claims the benefit of U.S. Provisional Patent Application Ser. No. 60/063,192, filed Oct. 20, 1997.

TECHNICAL FIELD

The invention relates to calorimetry, particularly to differential scanning microcalorimeters.

BACKGROUND OF THE INVENTION

Calorimetry is a measurement technique used to measure the changes in heat of an isolated system. Differential scanning calorimetry applies an approximately linear temperature profile to an isolated system while a reaction occurs in one part of the system. Differences in temperature across the temperature scan provide information about the thermodynamics of the reaction. Microcalorimetry is a measurement technique based upon a small sized calorimeter and as a result is applicable to analyzing the reactions of small samples, for example, thin films. A microcalorimeter is able to measure the heat of reactions of thin films because of the small size of the instrument; in ordinary calorimeter ovens, the thin film is too insignificant in the device to gain any information about its chemical reaction.

A non-scanning, that is, constant temperature microcalorimeter device is described in U.S. Pat. No. 5,451,371. The device is built on a silicon base and the base is etched, leaving a frame of silicon supporting two suspended polysilicon platforms. A catalyst is used on one platform to sense the presence of hydrocarbons. Platinum resistors on each platform serve as heaters and thermometers.

An article entitled "Thin Film Microcalorimeter for Heat Capacity Measurements from 1.5 to 800 K", Denlinger et al., Review of Scientific Instruments, American Institute of Physics, 1994, describes a microcalorimeter fabricated from a silicon nitride membrane mounted in a silicon frame. The membrane provides a platform that contains a platinum heater, a thin film platinum thermometer for high temperatures, and a Nb—Si low-temperature thermometer. The device does not have separate sample and reference zones for accurate scanning measurements.

The prior art fails to adequately resolve issues of thermal isolation and measurement accuracy in scanning microcalorimeters. It is an object of this invention to provide a scanning microcalorimeter on a chip with good thermal isolation between sample and reference zones to enable the measurement of small samples and thin films or monolayer films over a large range of temperatures.

SUMMARY OF THE INVENTION

The invention is a micron-scale differential scanning calorimeter produced on a silicon or gallium arsenide chip that allows for microscopic differential scanning calorimetry measurements of small samples. In several embodiments, the microcalorimeter includes a reference zone and a sample zone, each with an integrated polysilicon heater and a thermopile. In one embodiment, the reference and sample zones are on separate suspended platforms. In other embodiments, the reference and sample zones are at opposite ends of a single suspended platform. With a chip produced from silicon substrate, the thermopile consists of multiple polysilicon/aluminum junctions that are connected in series and that alternate between the reference and sample zones. The thermopile voltage provides a measure of the

temperature difference between the two zones and helps cancel the effects of common-mode thermal variations in the surrounding environment. In one embodiment, a single heater provides heat to the sample and reference zones. In another embodiment, the heater comprises an oven that contains the reference and sample zones, in which case the microcalorimeter may be designed without an integrated heater.

In performing a differential scanning calorimetry measurement according to the invention, the reference and sample zones are heated simultaneously with a ramped temperature profile. The electrical power profiles to the heaters may be calibrated such that the output voltage of the thermopile is zero in the absence of any differences in the thermal processes occurring in the two zones. A sample material or a sensing material may then be applied to the sample zone. As the temperature is scanned, a loss or gain of heat associated with a reaction or phase transition in the sample zone results in the production of a difference signal by the thermopile. Mapping the difference over a range of temperature provides information about the reaction. Types of sensing materials include a catalyst for chemical sensing, a material that exhibits a phase transition, and a chemically selective reactive material.

BRIEF DESCRIPTION OF THE DRAWING

FIG. **1** is a schematic block diagram of a first embodiment of the microcalorimeter of the invention.

FIG. **2** is a cross-sectional elevation view of the microcalorimeter taken along line **2**—**2** of FIG. **1**.

FIG. **3** is a schematic block diagram of a second embodiment of the microcalorimeter of the invention in which a single suspended platform has reference and sample zones.

FIG. **4** is a schematic block diagram of a third embodiment of the microcalorimeter of the invention that includes a dual concentric suspended structure.

FIG. **5** is a schematic block diagram of a fourth embodiment of the microcalorimeter of the invention that shows a single heater.

DETAILED DESCRIPTION

FIG. **1** is a plan view of a microcalorimeter **10** of the present invention comprising a dielectric such as silicon oxide layer **20** attached to one side of a substrate such as silicon substrate **12** thereby forming a single chip. Silicon substrate **12** is not visible in FIG. **1** but is shown in FIG. **2**. A suspended reference platform **14** and a suspended sample platform **16** are located within the boundaries of the silicon oxide layer **20**. The reference platform **14** and the sample platform **16** are silicon oxide platforms with layers of polysilicon and aluminum embedded therein. The platforms **14** and **16** are suspended over pits **34** and **32**, respectively, which are etched into the silicon substrate. The pits **34** and **32** in the bulk silicon are visible in FIG. **1** through openings in the silicon oxide layer **20** that forms the platforms **14** and **16**. The platforms **14** and **16** are held in place by four silicon oxide arms **47**, **49**, **51** and **55** extending from the perimeter of the silicon oxide layer **20**. Arms **51** and **55** form bridges over the pits from the platforms to the chip periphery where connecting wirebond pads are located for heaters **22** and **24**. A thermopile **15** is buried within layer **20** and crosses between the platforms **14** and **16**. A ridge **9** of bulk silicon is present under silicon oxide arms **47** and **49** thereby separating the pits **34** and **32**. Ridge **9** is shown in FIG. **2**.

To illustrate the size of the instrument in an actual prototype, platforms **14** and **16** are each about 50 microns in the vertical dimension and about 35 microns in the horizontal dimension.