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Abstract

Microhotplate as a Platform for Calorimetry †

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Abstract: Calorimetry is a powerful method for characterising materials. The heat of a reaction can be directly measured with good accuracy. The established method usually requires large amounts of materials, which is a major drawback when studying thin film reactions. A MEMS microhotplate-based microcalorimeter is developed in this study; it allows us to investigate thin film reactions by using a very small amount of materials. The temperature scale is calibrated by a well-known heat method of melting of two metal films. Energy calibration is also solved, and thus real information can be extracted for various solid-phase thin film reactions. In order to study reactions taking place close to room temperature, a cooled sample holder is developed, and the measurements can be started well below 0 °C.

Keywords: microcalorimetry; MEMS membrane structure; microheater chip; signal processing; temperature and energy calibration; cooled sample holder

1. Introduction

Thin film reactions play an important role in many fields of materials science and technology. There are many structural characterisation methods that are used to investigate thin films before and after each technological step (SEM, TEM, SIMS, etc.). However, the reaction heat balance of the studied system is not simple due to the very small amounts of material available [1,2]. Membrane-type twin microhotplates with a diameter of about 150 µm each are applied in the microcalorimeter [3]; it allows us to investigate solid-state reactions of a very low volume of materials in various fields of interest. The electrical control system of two independent channels is developed in order to record differential calorimetry signals.

A fundamental point of calorimetry is to determine the temperature scale with high precision. By using ≈200 nm thick In and Sn films with a melting point of 156.6 °C and 231.9 °C, respectively, temperature calibration can be performed. In the present work, the new microheater design with better temperature uniformity and an upgraded measurement system is presented. The energy calibration of the system is also essential for the exact determination of the reaction heat and description of the studied reaction. The first attempts at using the energy calibration method are also presented.

2. Design of Microcalorimeter System

The layout of the Pt heater element is shown in Figure 1a. The temperature uniformity over the heated surface with a diameter of 150 µm is better than ±1.5% up to the investigated 750 °C, as measured by calibrated optical pyrometry. It is worth noting that up to 550 °C, this method can be used for several thousand hours of stable operation, whereas filament degradation becomes significant and leads to an exponential reduction in lifetime

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over this temperature. The Pt microheaters are formed and embedded in independent membranes of SiO_2/Si_3N_4 (Figure 1b).

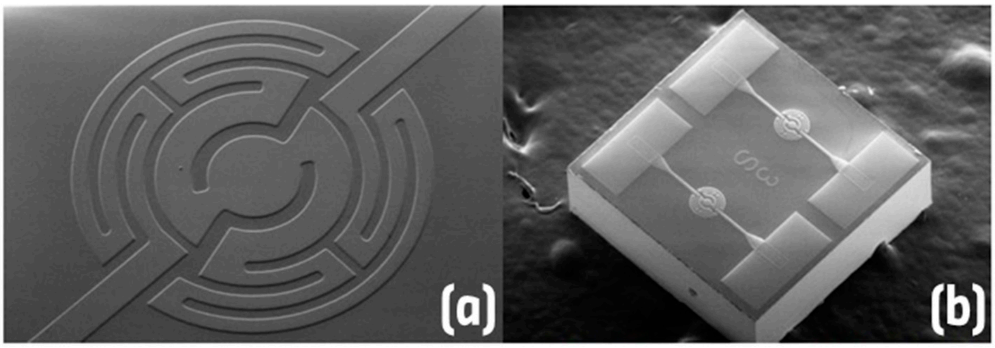

Figure 1. Layout of the Pt heater element with a diameter of 150 mm with enhanced temperature homogeneity over the surface (**a**); Microcalorimeter chip with two independent heater elements embedded in separated dielectric membranes (**b**).

The two Pt heater elements are driven by two independently controlled current sources, and the voltage drop on the heaters is measured. The analogue control loops keep the temperature of the heaters always at the same level. The differences in the necessary powers for maintaining identical temperatures contain the DSC signal characteristic of the reaction.

If the thin film reaction takes place at a temperature range of ≈100–500 °C, the measurements can be started from room temperature. However, in many cases, the reaction takes place at lower temperatures, close to or below room temperature. In this case, the measurement has to be started at a much lower temperature, e.g., −100 °C. A special sample holder was built in order to cool down the microheater chip to a well-controlled temperature; therefore, the heating process can be started well below 0 °C.

Besides the temperature calibration of the measurement, the exact calibration of the power difference between the two heaters may lead to the exact determination of the reaction energy. By using thin metal films with well-known melting energy, the energy calibration was performed.

3. Summary

In this work, we present a microcalorimeter system which has twin, membrane-based microheaters with enhanced temperature uniformity. The analogue control system drives the heaters independently and as an output, the power difference is provided. After the demonstrated temperature and energy calibration, this signal provides the reaction energy of the investigated thin film reaction.

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References

1. Lopeandia, A.F.; Valenzuela, J.; Rodríguez-Viejo, J. Power compensated thin film calorimetry at fast heating rates. *Sens. Actuators A Phys.* **2008**, *143*, 256–264. [CrossRef]
2. Wang, Y.; Zhu, H.; Feng, J.; Neuzil, P. Pavel Neuzil, Recent advances of microcalorimetry for studying cellular metabolic heat. *Trends Anal. Chem.* **2021**, *143*, 116353. [CrossRef]
3. Harasztosi, L.; Szabó, I.A.; Biró, F.; Kiss, R.G.; Battistig, G. Microcalorimeter development and calibration based on a twin micro-heater platform. In Proceedings of the 8th International Conference on Sensors Engineering and Electronics Instrumentation Advances (SEIA' 2022), Corfu, Greece, 21–23 September 2022.