TEMPERATURE DEPENDENCE OF THE RESONANCE FREQUENCY OF THERMOGRAVIMETRIC DEVICES

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Abstract

This paper investigates the temperature dependence of the resonance frequency of thermogravimetric (TG) devices for tip heating over the temperature range of 25-600°C. The resonance frequency of a fabricated TG device shows to be temperature independent for tip heating up to about 600°C. This allows a direct TG measurement without any temperature calibration of the resonance frequency.

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1. Introduction

Thermogravimetric analysis (TGA) is a thermal analysis technique used to determine changes in weight of a sample in relation to temperature changes. A TG device with a resonating sensing cantilever and two thermal actuators has been presented at MEMS'10 [1]. The shift of the cantilever resonance frequency gives information on the mass change of the sample while heated by the integrated heater. The temperature increase of the heater not only changes the sample mass but can also directly influence the resonance frequency of the cantilever and a frequency shift might occur without a mass change of the sample [2]. In this case a calibration of the resonance frequency versus temperature has to be carried out before a TGA measurement. It has been shown that for Si cantilever the distance of the hot spot from the edge might gives less or more dependence of the resonance frequency on the temperature [3]. This paper presents an experimental study on the influence of thermal gradient on the resonance frequency of inhomogeneously heated TG devices. The use of a cantilever paddle and the choice of SiN as structural material instead of Si, that has a thermal conductivity 50 time higher than SiN, resulted in temperature independence of the resonance frequency for tip heating up to about 600°C. Direct TG measurements can be performed up to 250°C with all the measured devices.
Fig. 1. Image of one of the investigated TG devices (device A). The cantilever size is 200×66×1.1 μm³.

1. Theory

The TGA device is based on the concept that changes in the sample mass with increasing temperature (due to phenomena such as oxidation and evaporation), will correspond to a shift in the resonance frequency of the cantilever. The resonance frequency of a non-damped cantilever with a rectangular cross section and with a sample attached to the free end of the cantilever can be calculated using the formula:

\[
f = \frac{1}{2\pi} \sqrt{\frac{k}{0.25m_c + m_s}}
\]  

where \( k \) and \( m_c \) are the spring constant and the mass of the cantilever respectively; \( m_s \) is the mass of the sample. In eq. (1) has been assumed that the sample mass does not contribute to the stiffness of the cantilever. If heated to a certain temperature the mass sample changes and the resonance frequency will also change in accordance with eq. (1). The temperature increase at the tip of the cantilever not only affects the sample mass but can also alter mechanical properties of the cantilever such as Young’s modulus, geometry, and stress. In fact, if the TG device is uniformly heated, the change of the ambient temperature strongly affects the resonance frequency of the device, see Fig. 2. In Fig. 2 the temperature dependence of the resonance frequency has been approximated to be linear.

\[
y = 2.15E-04x
\]

During a TG measurement the ambient temperature is kept constant and only the temperature of the integrated heater increases. This corresponds to a non uniform distribution of the temperature along the cantilever, see Fig. 3. Fig. 3 shows a thermal map obtained for one of the investigated device (device B) when actuator and heater are biased respectively at 1.5 V and 8 V. This measure has been made with an infrared thermo-camera equipped with a focal plane array (FPA) of 320×256 InSb sensors sensitive in the 3-5 μm wavelength range. The spatial resolution is about 6μm while noise equivalent temperature difference (NETD) is estimated to be 0.5 K. The hot spot is located at the free end of the cantilever, which is the lowest stressed region.
The most stressed region is next to the fixed end, where only a negligible temperature increase respect to the temperature of the frame exists. Indeed looking to Fig. 3 it is possible to see that in the first 1/3 of the cantilever length the temperature is already decreased of more than 70% of its highest value.

2. Device

The main characteristics of the measured devices are listed in Table 1. The devices are made using the same fabrication process. The first device, device A, has been designed with a length of 200 \( \mu m \); device B has approximately the same length of device A but a paddle has been added at the free end.

Table 1: Important parameters of the TG devices

| Parameter                  | Value  | Value  |
|----------------------------|--------|--------|
| Device                     | A      | B      |
| Cantilever size [\( \mu m^3 \)] | 200x66x1.1 (device Fig.1) | 214x61x0.9 |
| Paddle size [\( \mu m^3 \)]  | Not present | 70x30x0.9 |
| Resonance frequency [kHz]   | 40.7   | 12.8   |
| Actuator resistance [\( \Omega \)] | 900   | 300   |
| Heater resistance [k\( \Omega \)] | 4.5   | 7     |
| Heater interconnection      | PolySi and Au | Au     |

3. Fabrication

The chips are made using a thin-film bulk-micro-machining process. The starting material is a 300-\( \mu m \) thick \(<100>\) silicon wafer of 100 mm diameter, on which 600 nm LPCVD low-stress SiN and 300 nm LPCVD low-stress poly-silicon (poly-Si) are deposited. After implantation of the poly-Si to make low-resistive n-type and p-type regions (50\( \Omega \)/sq and 75 \( \Omega \)/sq, respectively) the poly is patterned and covered by another 300 nm SiN. The contact openings to the poly-Si are made and an anti-diffusion barrier (40 nm titanium and 80 nm titanium nitride) is deposited. The Ti/TiN layer is then patterned and Rapid Thermal Annealing (RTA) is performed to get titanium silicide (TiSi2). After the silicidation step 10 nm Cr and 400 nm Au are deposited and patterned. Then SiN membranes are etched by anisotropic etching of silicon from the back side in a KOH solution. To release the final structures, plasma etching from the front side is used. After dicing the chips are ready for assembly.

4. Experimental results

Experiments started with the measurements of the resonance frequency of the devices without tip heating. The devices were mounted on a dedicated holder and a lock-in amplifier (SR830) was used to excite the device and to read out the output of the Wheatstone bridge [1]. The measurements were remote controlled with a LabView program. The resonance frequencies of the devices without tip heating are reported in Table 1.
The resonance frequency was then measured applying increasing voltages on the heater resistor. In Fig. 4 the resonance frequency as function of the heater temperature is shown. Device A shows a temperature independence of the resonance frequency up to 250°C. For device B the resonance frequency is temperature independent till about 600°C.

For both device A and B the amplitude of the output signal at resonance frequency resulted also to be sensitive to temperature, decreasing with the increase of the tip temperature.

5. Conclusions

The influence of tip heating on the resonance frequency of TG devices has been investigated. When uniformly heated the resonance frequency of the TG device shows a temperature coefficient $\Delta f/f_0$ of about 200 ppm/K. When heated at the tip the resonance frequency results to be less or more sensitive to the tip temperature in dependence of the specific design of the device. In particular it has been found that for cantilever pad with a total length of 280 μm the resonance frequency resulted to be temperature independent till 600°C for tip heating. This is attributed to the good thermal isolation of the device, making that even at a high sample temperature, the areas with high stress levels remain basically at ambient temperature. Direct TG measurements without any temperature calibration of the resonance frequency can be performed up to 250°C with both the measured devices.

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