

Temperature calibration and electrical characterization of the differential scanning calorimeter chip UFS1 for the Mettler-Toledo Flash DSC 1

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Abstract

This paper reports on the temperature calibration and electrical characterization of the calorimeter chip UFS1 (internal design XI-400) developed for the new commercially available differential scanning calorimeter (DSC), the Flash DSC 1 of Mettler-Toledo. The chip consists of 2 identical membranes both with a p-type polysilicon microheater in the center of the membrane and a p/n-type polysilicon thermopile for measuring the sample temperature. The temperature calibration of the XI-400 is performed in the temperature range from 208 K to 723 K. An isothermal calibration is first performed to calibrate the heater resistance and the obtained curve is used to calibrate the integrated thermopile. The accuracy of the calibration is then determined by measuring the extrapolated onset temperature (T_e) of primary standards. A detailed electrical characterization of the device is also reported. The calibration method implemented and the good temperature reproducibility of the device allow to use devices with uncalibrated heater resistance in the temperature range from 208 to 723 K with a typical maximum error of ± 5 K.

Keywords: calorimeter chip; microheater; thermopile; temperature calibration.

1. Introduction

Calorimeter chips have become widely used to study thermal properties of sub-microgram samples as function of temperature. They are powerful tools as the small addenda of the calorimetric cell enable to study small amounts of material [1-2]. Ultra-fast-scanning calorimeters are commercially available and extensive

characterizations are present in literature [3-5]. These chips give new possibilities especially for studying the phase transitions of polymers. Scan rates as fast as 1 kK/sec prevent recrystallization of the sample during the scan and thermal properties of polymers can be studied under the same conditions as during injection moulding [6-8]. Different designs of calorimeter chips have been proposed with the intent to come to a device capable of fast temperature scan rates [3,9-10]. A common way to achieve this purpose is to reduce the thermal resistance of the calorimeter chip. In this paper an electrical characterization of device XI-400 is carried out to determine its capability in terms of heating and cooling scan rate. Not only the scan rate but also the accuracy of the temperature read out at which a thermodynamic phenomenon occurs is of importance during a calorimetric measurement. The calorimeter chips have to be calibrated in order to establish the relation between the measured signals and the sample temperatures. A common method to calibrate calorimeter chips is with primary standards of which the phase transitions are often fixed or defined ITS-90 points. The output of the chip temperature sensor (i.e. the thermopile output voltage for XI-400) measured for the phase transition of the primary standard is associated directly with the corresponding temperature value present in literature. Different primary standards can be chosen to cover the entire temperature range of interest. The drawback is that for each primary standard, so for each measured temperature, a different chip has to be used [12] since the sample has to be deposited directly on the chip. Moreover, since the chips used for the calibration are contaminated, calorimetric measurements can be performed only with not calibrated devices. Techniques based on the use of IR camera are not accurate because it is possible to detect only the incoming radiation power intensity and not the temperature itself. The temperature can be calculated if the emissivity ϵ of the specimen is known, which is source of large uncertainty [19-20]. In [13] a calibration method that allows the use of calibrated devices for calorimetric measurements is proposed. First the chip is inserted into an oven and the heater integrated on the calorimeter chip is calibrated in the temperature range of interest. Then the obtained calibration curve is used for the calibration of the temperature sensor, i.e. the thermopile. In this paper the chip XI-400 has been calibrated according to the method presented in [13] for temperatures ranging from 208 K to 608 K. In order to determine the accuracy of the calibration the T_e of primary standards has been measured with chips having uncalibrated heater resistance. The primary standards have been

chosen so that their phase transition is inside the temperature range from 208 K to 723 K. In the first part of the paper the main characteristics of the chip XI-400 are briefly outlined. A detailed electrical characterization of the chip follows. In the last part of the paper the temperature calibration of the device is presented. At the end conclusions on the calibration accuracy are outlined.

2. Device description

A photo of the chip is shown in Fig.1 and its main properties are reported in Table 1.

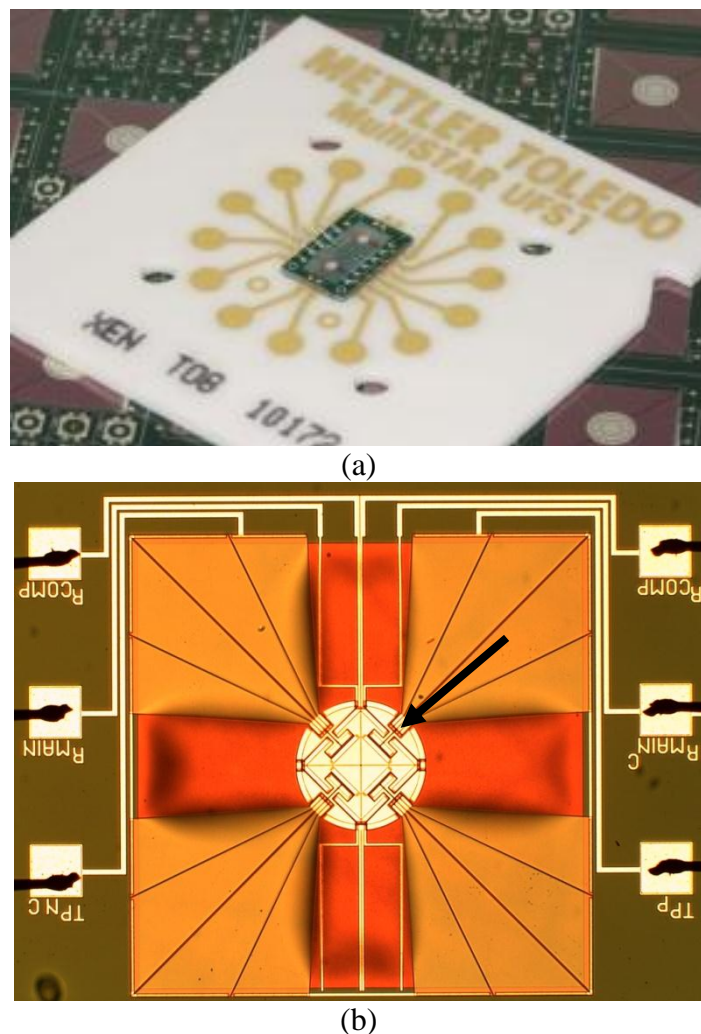


Fig. 1 Photograph of device UFS 1, internal design XI400: (a) Device on ceramic package (b) Close up of one cell: the heater covered with Aluminium layer in the center of the membrane makes up the sample area; the hot junctions of the 8 thermocouples (2 are pointed by the arrow) are inside the sample area.

The chip is made of two identical cells, the reference and sample cell, respectively. Both cells consist of a $1.6 \times 1.6 \text{ mm}^2$ large and about $2 \text{ }\mu\text{m}$ thick membrane.

Table 1 Main characteristics of Device XI400

Symbol	Quantity	Value @ room Temperature	Unit
R_{main}	Main heater resistance	5	k Ω
R_{comp}	Compensation heater resistance	4	k Ω
R_{tp}	Thermopile resistance	13	k Ω
$N\alpha_s$	Thermopile sensitivity	4	mV/K
S_a	Sample area	0.2	mm ²
L^*W	Membrane area	2.5	mm ²
T_c	Time constant for cooling of empty sensor	12	ms
R_{amb}	Thermal resistance sample to ambient	5	kK/W
S	Transfer of chip	20.4	V/W

The membranes of the two cells are thermally separated by a thick Si frame of 300 μm that acts as heat sink. Two heaters are integrated in the center of the membrane. The main heater, with a resistance of about 5 k Ω , is used for the general temperature scan program. The other heater, of about 4 k Ω , is active only in power compensation mode and has to compensate for temperature differences between the reference and the sample cell. The two heaters, covered by an Aluminium (Al) layer, make up a circular area with a diameter of 0.5 mm, where the sample can be loaded. The Al layer is used to get temperature uniformity in the sample area. The temperature of the heated area is measured with an integrated thermopile. The thermal resistance (R_{th}) between the sample area and the surrounding is given by:

$$R_{th} \left[\frac{K}{W} \right] = \frac{S}{N\alpha_s} \quad (1)$$

where S is the device transfer defined as the ratio between the output voltage of the thermopile and the input power in the main heater resistance; N is the number of thermocouples forming the thermopile and α_s is the Seebeck coefficient of the thermopile. The calculated value of R_{th} is 5 kK/W at room temperature. The chip is mounted on a custom-designed ceramic base plate MultiSTAR UFS1 (24×24×0.6 mm³) with 14 connections pads, and wire bonded using Al bonding wires.

3. Electrical characterization

Main heater

The electrical resistance of the main heater (R_H) is about 5 k Ω at room temperature, with a relative standard deviation (σ) of 0.8%, calculated over 270 chips belonging to two different wafers. With a temperature coefficient of the resistance (TCR) of about 0.1 %/K, the calculated σ corresponds to a deviation of about 8 K.

Since the calibration method proposed in this paper is independent of the specific chip, the resistance deviation between the chips does not constitute a problem for calorimetric measurements with devices having uncalibrated heater resistance. This issue is discussed more extensively in section 4. The time stability of the heater resistance has also been investigated. At room temperature R_H shows a variation of only 30 ppm/hr. The time stability decreases with temperature, showing a drift of the main heater resistance of about 300 ppm/hr at 623 K, see Fig. 2. This drift corresponds to a drift in temperature of about 0.3 K/hr.

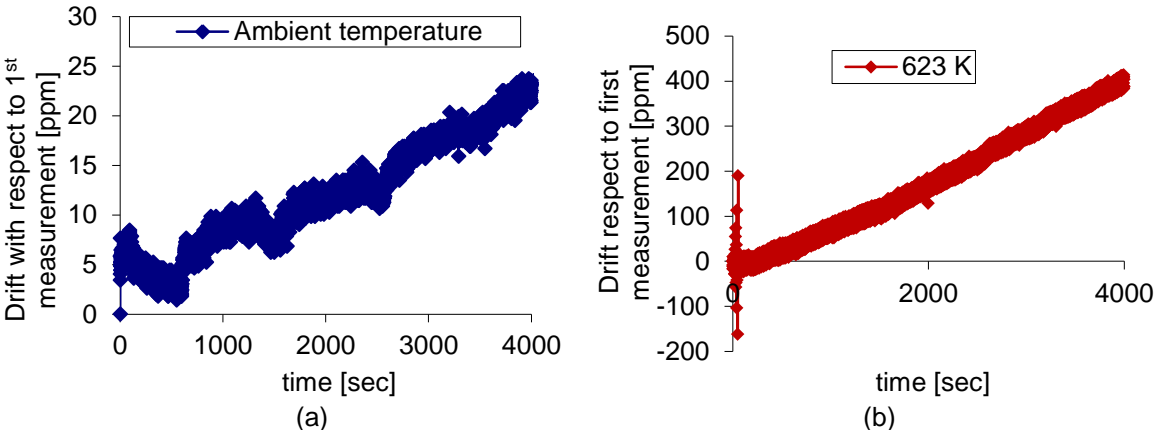


Fig. 2 Main heater resistance stability at (a) 300 K and (b) 623 K. Although the stability decreases with the temperature, the drift of the heater resistance is only 300ppm/hr at 623 K, that corresponds to about 0.3 K/hr.

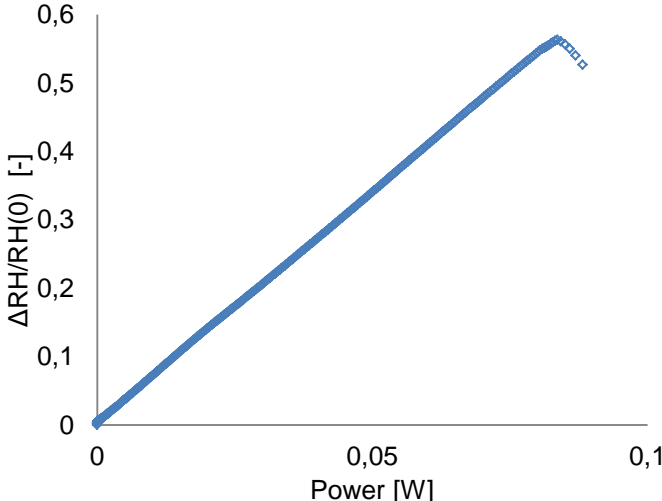


Fig. 3 Variation of the heater resistance with input power. The linear increase of the heater resistance with temperature reverses above about 800 K.

In Fig. 3 the heater resistance is shown as function of the input power. The resistance of the main heater increases with increasing heating power and thus with

increasing temperature. At sample-area temperatures above 800 K the trend reverses and the resistance starts to decrease with increasing heating power. Similar behavior of the heater resistance with heating power has been found in literature for a membrane device [14] with an n-type polysilicon heater sandwiched between a SiN and SiO₂ layer. At about 850 K the membrane cracks. The failure of the device is probably due mainly to the high stress present on the membrane caused by the different expansion coefficients of the SiN and Al layers, which are about 3.3 and 24 ppm/K, respectively. Another possible reason is the electrical breakdown of the SiN [16].

Although the device has been proved capable to sustain heater temperature up to about 800 K, a maximum sample-area temperature of 723 K is advised for reliable and repeatable measurements.

Thermopile

An integrated thermopile, which consists of 8 p/n-type polysilicon thermocouples, is used to measure the temperature of the heated area. The electrical resistance of the thermopile (R_{tp}) is about 13 kOhm at room temperature. Measurements on 270 devices showed a relative standard deviation of about 0.8% of its value at room temperature. The variation of the output of the thermopile (U_{tp}) for 86 devices is about 0.6 K at about 453 K. Although the variation of the thermopile output is much smaller than the method inaccuracy the U_{tp} it is measured for all the devices before use since the built in software of the Flash DSC 1 allows to measured the U_{tp} as function of the heater temperature for each loaded device. The hot junction of the thermocouples is located inside the Al circular plate delimiting the sample area while the cold junction is on the Si frame that acts as heat sink. The thermopile measures directly the temperature difference between the heated area and the Si frame. The temperature resolution of the measurement is limited by the thermocouple Johnson noise:

$$u_n \left[\frac{V}{\sqrt{Hz}} \right] = \sqrt{4k_B T R_{tp}} \quad (2)$$

where R_{tp} is the thermopile resistance, T the absolute temperature in Kelvin and k_B the Boltzmann's constant. With $R_{tp} = 13$ kOhm, $T = 300$ K, the noise voltage is about $u_n = 15$ nV/ \sqrt{Hz} . For a bandwidth of 100 Hz this will correspond to about 150 nV that is equivalent to a temperature noise of about 40 μ K.

Device Time constant

An impulse of 170 msec is applied to the heater resistance and the output voltage of the thermopile is measured. The heating and cooling time constant of the chip XI-400 are measured to be about 10-12 ms at 423 K. With a temperature elevation of the sample area of 400 K and a thermal time constant of 10 ms the maximum cooling rate is about 40 kK/sec.

4. **Temperature calibration**

The temperature calibration of the chip XI-400 consists of two steps: 1) Isothermal calibration of the main heater resistance and calibration of the thermopile sensitivity; 2) Calibration accuracy determination with primary standards.

Isothermal calibration and Calibration of the thermopile sensitivity

The isothermal calibration is one of the existing methods for the temperature calibration of heater resistances. Other possibilities are the calibration with reference materials [12] or with Raman spectroscopy [16], the last one suited for non metallic resistors and especially for those made in Silicon. During an isothermal calibration the chip is loaded into an oven or placed on a hot plate and the heater resistance is measured at a fixed number of set temperatures. The method is straightforward and it can be applied to heater resistors made in any material. The disadvantage is that for some particular heater designs the heater resistance values are under/overestimated. This is the case for heaters where the electrical resistance of the heater legs, which are connections to the heater made of the same material of the heater, constitute a considerable percentage of the total heater resistance [16]. In fact, while in normal operation only the heater reaches high temperatures and the heater legs have a temperature gradient, during the calibration the whole chip is brought at high temperature and the heater legs are heated isothermally. The heater designed for the chip XI-400 does not have heater legs as Al connections are available just outside the heated area. Moreover, as already mentioned above, the heater resistance value for the chip XI-400 is about 5 kOhm at room temperature which is much higher than the Al connections of only a few tens of Ohm. The isothermal calibration is therefore suited for the chip XI-400, and a description of the used measurement set up is outlined in the next section.

During the isothermal calibration of the heater resistance, if different bias voltages are applied to the heater, the output of the thermopile can be measured. In this way a graph of the output voltage of the thermopile as function of heater resistance is

obtained at each fixed oven temperatures. If a correct set of bias voltages is chosen these curves overlap in temperature. This makes possible to obtain just one curve [13]. Using the calibration the curve of the heater resistance the Seebeck coefficient can be then extract as a function of temperature.

Measurement set-up

A picture of the measurement set-up is shown in Fig.4.



Fig. 4 Measurement set-up for isothermal calibration. (a) Probe for measurement in the Metrology Well. From the right to the left: the TO-5 package; the Teflon socket and the Inconel tube; (b) Metrology well; (c) close up of the CTS T-65/50 oven inside. The top part of the TO5 packages, where the chips are glued, are inserted in an Al block and the temperature is measured with a PRT thermometer.

A Metrology Well (FLUKE 9173) is used to calibrate the chips in the temperature range from 308 K to 608 K. An external secondary 25.5 Ohm-PRT (FLUKE 5628) is used to monitor the Metrology Well temperatures, for a more accurate temperature reading, and because it is not practical to align the chips with the built-in reference thermometer of the Metrology Well [17]. The chips are glued on a TO-5 package and connected through a Teflon TO-5 socket to a custom made probe. The probe consists of shielded wires for the electrical connections between the chip and the instruments and an Inconel tube for mechanical support. The electrical signals are measured with

a 6-digit Digital MultiMeter from Agilent (34970A). All the parts of the set-up are suited for temperature calibration up to 773 K but the TO-5 socket limits the highest temperature to be 608 K. To extend the temperature calibration towards lower temperatures (208-358 K) the chips are inserted in a CTS T-65/50 oven. Also in this case the chips are glued on a TO-5 package and connected, through a wired PCB board, to the Agilent 34970A. The chips are mounted on an Aluminum custom-made block in which a hole is drilled to accept a 100-Ohm PRT thermometer (FLUKE 5618B) that measures the temperature of the Aluminum block. The temperature ranges chosen for the two ovens overlap in the range from 300 K to 360 K in order to verify the continuity of the measurements. A custom-made LabView program is realized to remotely control the measurements.

Extraction method

The heater resistance R_H is connected in series with an external precision series resistor (R_S) (Econisto 8E16). The voltages across the heater resistor (U_H) and the series resistor (U_S) are measured in order to calculate R_H :

$$R_H = R_S \frac{U_H}{U_S} \quad (3)$$

A maximum dissipated power of 0.05 mW has been chosen as compromise between low noise measurements and low self heating of the heater resistors. The R_H is measured for a fixed number of temperatures in the range from 208 K to 608 K with a 25 K of interval and for each calibrated chip the curve of the dependency of the heater resistance with the temperature can be drawn. Since it is not practical to calibrate every chip before a calorimetric measurement, the goal of the temperature calibration presented in this paper is to build a calibration curve independent of the specific chip so that chips with uncalibrated heater resistance can be used for calorimetric measurements using the calibration curve determined with the calibrated chips. For this reason, for all the wafers of the fabricated batch, only a few chips randomly chosen per wafer are calibrated. For each of these chips the R_{Hratio} , defined as the ratio between R_H and its value at 273.15 K, is calculated. The R_{Hratio} is not related to a specific chip, and its variation with the temperature is taken as calibration curve. The temperature calibration curve for the chips XI-400 is shown in Fig.5.

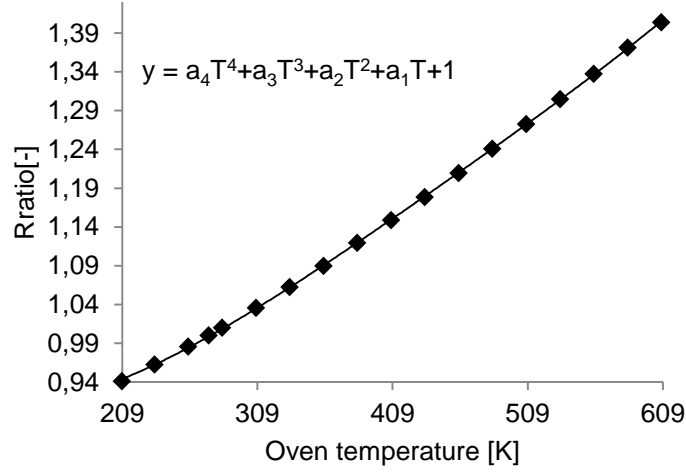


Fig. 5 Heater resistance versus temperature normalized to its value at 273.15 K. The measurement points are fitted with a fourth order polynomial.

The best fitting of the R_{Hratio} points is found to be a fourth order polynomial. The error made in approximating the measurement points with the polynomial is less than 0.6 K. Repeated measurements of R_H show an uncertainty of about ± 1.5 K in the temperature range considered. The reproducibility of 20 different chips has an uncertainty of less than ± 2 K. Once the heater resistance values are measured for all the temperatures of interest, the temperature coefficient of the resistance (TCR) between two adjacent temperatures, j and $j+1$, can also be calculated as:

$$TCR_{(j,j+1)} \left[\frac{ppm}{K} \right] = 10^6 \frac{R_{H(j+1)} - R_{H(j)}}{(T_{(j+1)} - T_{(j)}) \frac{1}{2} (R_{H(j+1)} + R_{H(j)})} \quad (4)$$

The calculated TCR as function of temperature is reported in Fig. 6. Once the relation between temperature and main heater resistance is known the thermopile can also be calibrated. The output voltages of the thermopile (U_{tp}) are recorded for different input power, that is for different heater resistance values. Using the measured heater resistance values at different temperatures it is possible to draw the dependencies of the U_{tp} with the temperature, see Fig. 7. This curve can be obtained for each chip before a calorimetric measurement using the built-in software of the Flash DSC 1. In this case a fourth order polynomial is used to fit the measurement points. The U_{TP} can be expressed as:

$$U_{TP} = (\alpha_{s,p} - \alpha_{s,n}) N \Delta T \quad (5)$$

where ΔT is the temperature difference across the thermopile and $\alpha_{s,p} - \alpha_{s,n}$ is the relative Seebeck coefficient of the p-type and n-type polysilicon thermopile, which can be extracted as a function of the temperature. The calibration curve obtained with

this method can then be used for DSC measurements with chips having uncalibrated heater resistance.

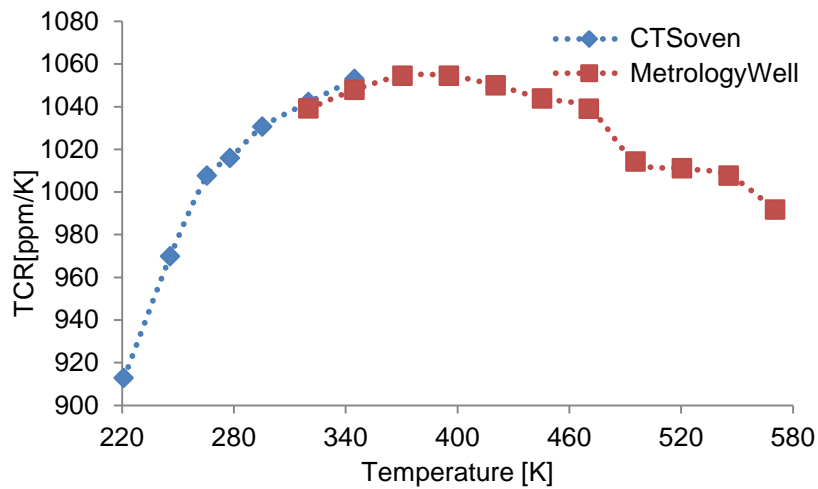


Fig. 6 TCR of the main heater resistance as function of the temperature. The two curves correspond to the measurements performed with the two ovens (CTS and Metrology Well).

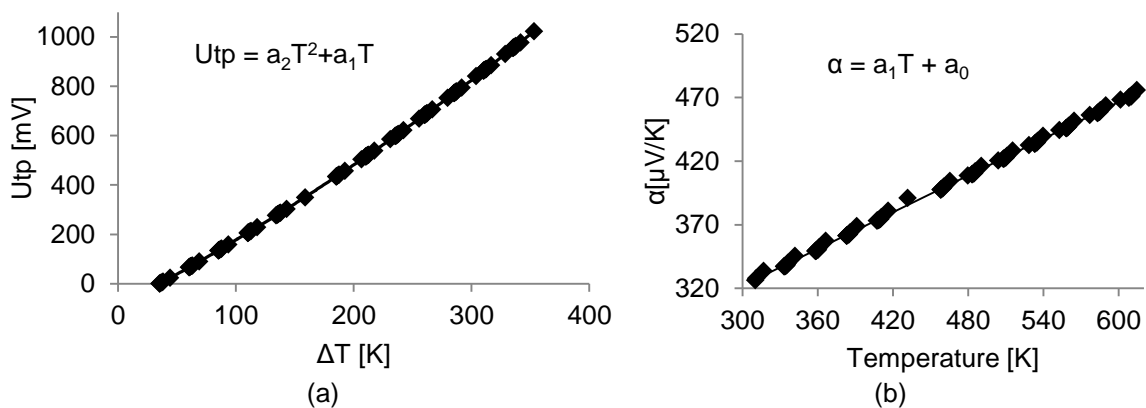


Fig. 7 (a) Output voltage of the thermopile (U_{tp}) as function of the temperature difference between hot and cold junctions of the thermopile. The Seebeck coefficient of the thermopile (b) is the derivative of the U_{tp} with respect to the temperature.

Calibration accuracy determination with primary standards

The accuracy of the temperature calibration using the heater resistors is determined by measuring the extrapolated onset temperatures (T_e) of primary standards. The T_e is defined as the temperature of the intersection between the base line and the line through the leading edge of the peak [18]. The primary standards have been selected on the basis of their phase transition temperatures situated in the temperature range of interest and they are: Adamantane (not a primary standard), Indium (In), Tin (Sn) and Zinc (Zn). For the measurements a chip with an uncalibrated heater resistance is positioned in the Flash DSC 1.

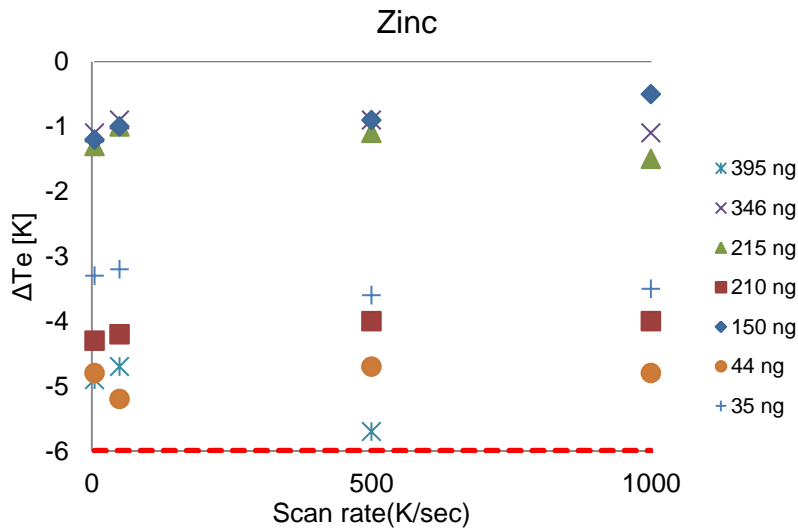
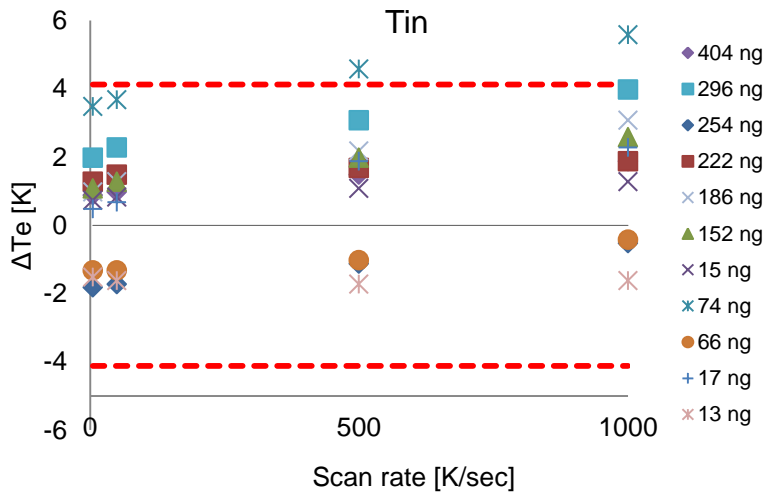
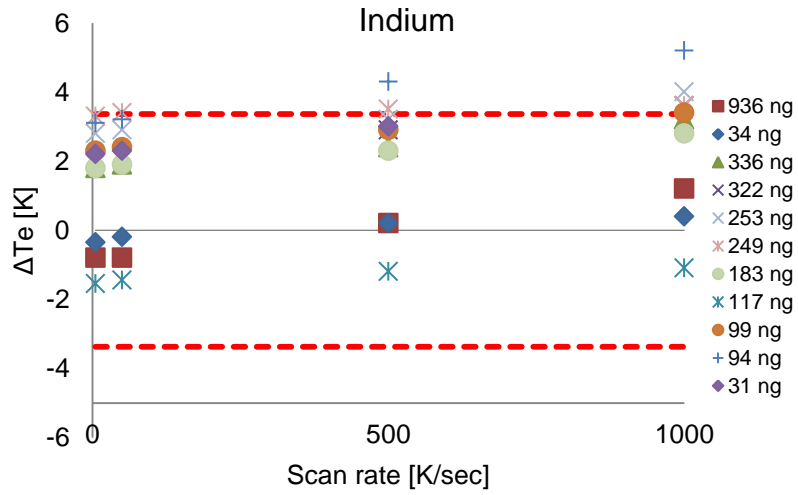


Fig. 8 Difference between the extrapolated onset temperature and the theoretical phase transition temperature for various scan rates (K/sec) and for different sample masses. The red lines show the tolerance interval of the instrument ($(T_{trs} - T_{ambient}) * 0.01 + 2^\circ\text{C}$).

The heater calibration curve is loaded in the software available on the Flash DSC 1 and a software calibration is performed to obtain the U_{TP} as function of the temperature, specific for the used chip. After the annealing of the chip at 723 K, the sample is loaded on the chip and the T_e is measured at various scan rates. Different devices have been tested with samples having masses ranging from about 30 ng to 940 ng. In Fig. 8 the measurements performed with In, Sn and Zn are reported. The measurements have been performed in air with an ambient temperature of about 296 K. The graphs show the difference (ΔT_e) between the T_e and the theoretical phase transition temperature (T_{trs}), for different sample heating rates (K/sec) and different sample masses. For each new measurement a new device is used. The two red lines in Fig.8 show the tolerance interval of the instrument ($(T_{trs} - T_{ambient}) * 0.01 + 2 K$). The values of the sample masses, calculated from the measured enthalpy of fusion, are also reported in Fig. 8. To measure the solid-solid phase transition of Adamantane a two-stage Intracooler is connected to the Flash DSC 1.

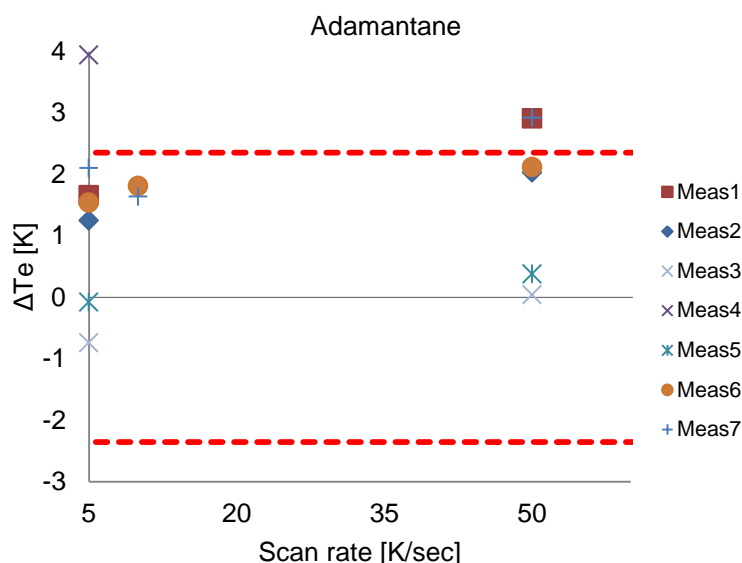


Fig. 9 Differences between the extrapolated onset temperature and the theoretical phase transition temperature of Adamantane for different scan rates (measurements performed at 173 K as ambient temperature). The maximum scan rate is 50 K/sec as the onset temperature could not easily be extrapolated at higher scan rates. The red lines show the tolerance interval of the instrument.

The measurements, shown in Fig. 9, were performed with an ambient temperature of about 173 K, also in this case in air. For the lowest scan rate used, all the measurements are within the tolerance interval, except for one measurement with Adamantane, deviating from the other measurements probably for a bad thermal contact between the sample and the chip membrane. However, from Figs. 8 and 9 it

can be concluded that the calculated ΔT_e is less than ± 5 K for all the tested chips. The average calculated ΔT_e can be used to correct the calibration curve for a better accuracy, which is especially clear in the case of Zinc, see Fig. 8.

5. **Conclusions**

This paper presents the electrical and thermal characterization of the calorimeter chip UFS1 (internal design XI-400) developed for the new commercially available differential scanning calorimeter (DSC) instrument, the Flash DSC 1 of Mettler-Toledo. The heater resistance characterization shows that for reliable measurements the maximum advisable temperature for the sample area is 723 K. Using an isothermal calibration the dependence of the heater resistance with temperature is measured for a few chips per batch. The obtained heater calibration curve is used for the thermopile calibration of all the chips of the batch. The calibration method shows an uncertainty of less than ± 2 K for measurements on different devices. The onset temperature of *Adamantane*, *Indium*, *Tin* and *Zinc* is measured to determine the accuracy of the temperature calibration. The combination of an electrical calibration, and the use of primary standards together with the good chip reproducibility makes it possible to use chips with uncalibrated heater resistance in the temperature range of 208-723 K with a typical maximum error of ± 5 K.

6. **Acknowledgements**

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7. **References**

- [1] K. Allen, F. Hellman "Specific heat of C60 and K3C60 thin films for T=6–400 K" Phys. Rev. B 60, 11765–11772 (1999)
- [2] K. Allen, F. Hellman "Specific heat of endohedral and higher fullerene thin films" J. Chem. Phys. 111, 5291 (1999)
- [3] A. Minakov, J. Morikawa, T. Hashimoto, H. Huth, C. Schick "Temperature distribution in a thin-film chip utilized for advanced nanocalorimetry" Meas. Sci. Technol. 17 199–207 (2006)

- [4] A.A. Minakov, S.A. Adamovsky, C. Schick "Non-adiabatic thin-film (chip) nanocalorimetry" *Thermochimica Acta*, 432, 177–185 (2005)
- [5] M.Yu. Efremov, E.A. Olson, M. Zhang, S.L. Lai, F. Schiettekatte, Z.S. Zhang, L.H. Allen "Thin-film differential scanning nanocalorimetry: heat capacity analysis" *Thermochimica Acta*, 412, 13–23 (2004)
- [6] V. Brucato, F.G. Crippa, S. Piccarolo, G. Titomanlio "Crystallization of Polymer Melts Under Fast Cooling I: Nucleated Polyamide 6" *Polymer engineering and science*, vol. 31, no. 19, 1411-1416, (1991).
- [7] Z. Ding, J.E. Spruiell "An Experimental Method for Studying Nonisothermal Crystallization of Polymers at Very High Cooling Rates" *Journal of Polymer Science*, vol. 34, 2783-2804, (1996)
- [8] V. Brucato, F. De Santis, A. Giannattasio, G. Lamberti, G. Titomanlio "Crystallization during fast cooling experiments, a novel apparatus for real time monitoring" *Macromol. Symp*, 185, 181-196, (2002).
- [9] S.L. Lai, G. Ramanath, L.H. Allen, P. Infante, and Z. Ma, "High-speed (104 OC/S) scanning microcalorimetry with monolayer sensitivity (J/m²)" *Appl. Phys. Lett.*, vol. 67, pp. 1229-1231, (1995)
- [10] S. Zhang, Y. Rabin, Y. Yang, and M. Asheghi, "Nanoscale calorimetry using a suspended bridge configuration" *J. Microelectromech. Syst.*, vol. 16, pp. 861-871, (2007)
- [11] R. E. Cavicchi, G. E. Poirier, N. H. Tea, M. Afridi, D. Beming, A. Hefner, I. Suehle, M. Gaitan, S. Semancik, and C. Montgomery, "Micro-differential scanning calorimeter for combustible gas sensing" *Sens. Actuators B, Chem.*, vol. 97, pp. 22-30, (2004)
- [12] W. Winter, G.W.H. Höhne "Chip-calorimeter for small samples" *Thermochimica Acta*, 403, pp 43-53 (2003).
- [13] E. Iervolino, A.W. van Herwaarden, P.M. Sarro "Temperature calibration of fast scan calorimeter chips" *Proc. EUROSENSORS*, pp. 773-776, (2008)
- [14] M.Ehmann, P.Ruther, M. von Arx, H.Baltes, O.Paul "Ageing behavior of polysilicon heater for CMOS microstructures operated at temperature up to 1200 K", *JMEMS*, pp. 147-150, (2001)
- [15] M Graf, R Jurischka, D Barrettino, A Hierlemann "3D nonlinear modeling of microhotplates in CMOS technology for use as metal-oxide-based gas sensors" *J. Micromech. Microeng.*, 15, pp. 190–200, (2005)

- [16] B.A. Nelson, W.P. King "Temperature calibration of heated silicon atomic force microscope cantilevers" *Sensors and Actuators A* 140, pp. 51–59, (2007)
- [17] <ftp://ftp.hartscientific.com/publications/2510068.pdf>
- [18] G. Vanden Poel, V.B.F.Mathot "High-speed/high performance differential scanning calorimetry (HPer DSC): Temperature calibration in the heating and cooling mode and minimization of thermal lag" *Thermochimica Acta*, 446, pp. 41–54, (2006)
- [19] L. La Spina, A. W. van Herwaarden, H. Schellevis, W. H. A. Wien, N. Nenadović, L. K. Nanver, "Bulk-micromachined test structure for fast and reliable determination of the lateral thermal conductivity of thin films" *J MEMS*, 16, pp. 675-683, (2007)
- [20] A. Jacquot, W. L. Liu, G. Chen, J.-P. Fleurial, A. Dauscher, and B. Lenoir, "Improvements of on-membrane method for thin-film thermal conductivity and emissivity measurements," in *Proc. IEEE Int. Conf. Thermoelectronics*, pp. 353–361, (2002)