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Temperature distribution in ultrafast chip-nanocalorimeters measured by micro-scale infrared thermography

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Abstract

To study the kinetics of phase transitions and to obtain artificial materials with improved physical properties, a set of thin-film high-sensitivity sensors for ultra-fast scanning nanocalorimetry has been constructed. To investigate the dynamics of the temperature distribution in thin-film calorimetric sensors, high-resolution high-speed infrared thermography has been applied as a tool of non-contact thermal imaging in combination with ultra-fast scanning calorimetry.

1. Introduction (Arial, 9pt, bold)

Advances in ultrafast chip-calorimetry provide possibility to generate non-equilibrium states and to study phase-transition kinetics at microsecond and even faster time scales. Ultrafast chip calorimetry can be applied towards micro- and nanoscale objects to study kinetics of thermodynamic processes. Heating and cooling rates of several mega K/s became accessible with the sensors similar to that available from Xensor Integration, NL, [1], see Fig. 1a.

We consider the experiments on the dynamics of the temperature distributions in the XEN-39472 calorimetric sensor studied by high-speed high-resolution infrared thermography. The origin of limitations imposed on the maximum attainable controlled cooling rate is investigated, and the possibility of improving the calorimetric sensor is proposed.

2. IR thermography of the sensor used for ultrafast calorimetry

To investigate the dynamics of temperature distribution in sensors used for ultrafast calorimetry, the IR thermography as a tool of non-contact thermal imaging is applied in combination with ultrafast scanning calorimetry. A high-speed high-resolution IR camera (640 x 512 matrix of InSb photodetectors) with the original-designed microscopic lens (magnification x 7) is used. The images of the infrared radiation intensity (*IRI*) from the top view of the sensor membrane were studied. The images were taken at 0.1 ms exposure time, acquisition rate of 400 Hz (1 shoot every 2.5 ms), and the spatial resolution was 4.29 μm per pixel.

3. Results and analysis

The distribution of the infrared radiation intensity $IRI(t,x,y)$ from the central heated part of the membrane of the sensor was measured simultaneously with the temperature $T(t,0,0)$ obtained from the thermopile at the central point at $x=0$ and $y = 0$. Thus, the dynamics of the distribution $IRI(t,x,y)$ measured in relative units can be compared with the dynamics of the actual temperature $T(t,0,0)$ at the central point of the membrane. The temperature in the center of the heating region was scanned linearly with time t from the initial value T_{in} = 320 K to the maximum T_{max} 550 K at heating and back to 320 K at cooling. Thus, the interval of the temperature scanning equals ΔT= 230 K. The scanning rate is $R = \Delta T/t_0$, where t_0 is the time interval from the beginning to the end of the heating scan. Accordingly, the temperature and IRI attain the maximum values $T(t_0, 0,0) = T_{max}$ and $IRI(t_0,0,0)=IRI_{max}$ at $t = t_0$.

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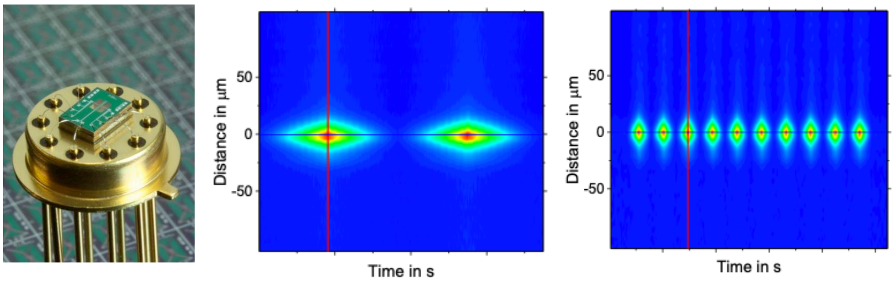
The first result is that the temperature changes only near the central heated region of the membrane during heating-cooling scans. (Fig. 1 b, c) The main change in the membrane temperature occurs in the region ca. 10 µm around the central point $(x, y) = (0,0)$. This means that the heat generated by the heater is removed by the ambient gas before the heat reaches the membrane periphery in accordance with the model proposed previously [2].



a b c

Fig. 1: Photograph of the thin-film sensor, XEN-39472, used as a calorimetric measuring cell (a) Infrared radiation intensity** $IRI(t,0,y)$ **in relative units vs. time and distance along** Oy **axis measured on the membrane of XEN-39472 during two temperature scans at** R = **500 K/s (b). during ten temperature scans at** R = **10^4 K/s.(c)

3.1 Analysis

The temperature measured by the thermocouple in the center of the membrane can be used for the calibration of the infrared radiation intensity, which is measured in relative units. The profiles that are presented in Fig.1 can be transformed into temperature. Indeed, $T(t,0,0)^4$ is proportional to $(IRI(t, 0, 0) - IRI_{bg})$, where IRI_{bg} is some background level that is associated with thermal noise and dark current of the photodetectors. Thus, the profile of the infrared radiation intensity $IRI(t, 0, y)$ can be transformed into the temperature profile $T(t, 0, y)$ by the following relationship:

$$T(t,0,y) = T(t_0,0,0) \cdot \left(IRI(t,0,y) - IRI_{bg}\right)^{1/4} / \left(IRI_{max} - IRI_{bg}\right)^{1/4} \qquad (1)$$

It is noteworthy that $IRI(t, 0, 0)$ can be transformed to the *linear dependence* $T(t, 0, 0)$ with only one fitting parameter $k = 0.935$ for several scanning rates in the range $5{\cdot}10^2$ – 10^6 K/s. Similar results were obtained for different rates (Fig. 2).

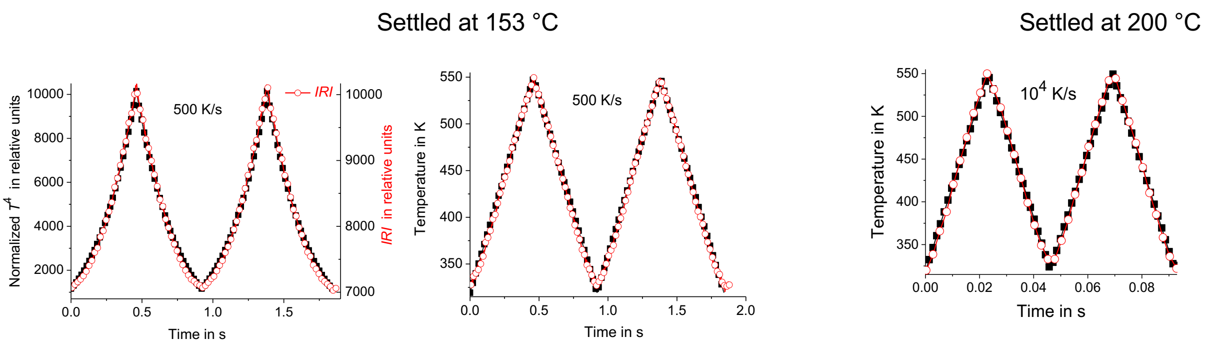


***Fig. 2:** Time dependencies of normalized $(T(t, 0,0))^4$ (squares) and IRI(t, 0, 0) (circles) **(a)**, as well as T(t, 0, 0) (squares) and normalized to temperature $(IRI(t, 0, 0)-IRI_{bg})^{1/4}$ (circles) **(b)** at R= 500 K/s and IRI_{bg} = 6620 r.u. as well as 10^4 K/s and IRI_{bg} = 6564 r.u. (**c**).*

REFERENCES

[1] van Herwaarden A. W., Overview of calorimeter chips for various applications, Thermochim. Acta 2005;432: 192-201.

[2] Minakov A. A., Morikawa J., Hashimoto T., Huth H., Schick C., Temperature distribution in a thin-film chip utilized for advanced nanocalorimetry, Measurement Science and Technology 2006; 17: 199-207.

[3] Minakov A., Morikawa J, Zhuravlev E, Ryu M., van Herwaarden A. W., and Schick C., High-speed dynamics of temperature distribution in ultrafast (up to 10^8 K/s) chip-nanocalorimeters, measured by infrared thermography of high resolution, Journal of Applied Physics 125, 054501 (2019).