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Study and characterization of a microcalorimeter thermostat

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Abstract- A high-accuracy adiabatic microcalorimeter has been developed at Istituto Nazionale di Ricerca Metrologica (INRIM, Torino, Italy) for the realization of the radio frequency and microwave power primary power standard. The apparatus is intended to evaluate the efficiency of proper power sensors of thermoelectric or bolometric type by measuring their losses in terms of heat produced. These kind of measurements require a good thermal stabilization of the measurement chamber, therefore a dedicated thermostat has been designed, assembled and characterized at INRIM in order to obtain a thermal stability of ±1 mK on a daily basis.

Keywords: Microcalorimeter, thermostat, microwave power standard.

I. Introduction

In the Radio Frequency (RF) and Microwave (MW) fields, power is a key quantity. The measurement of the electrical losses of power sensors is, therefore, of the utmost importance because it allows the determination of the effective efficiency of the sensors themselves.
Primary RF and MW power standards are realized by National Metrology Institutes (NMIs) mainly by means of microcalorimeters that allow the measurement of the electrical losses of proper sensors through the heat produced. In the past, microcalorimeters were thermally stabilized by using water or oil baths [1, 2]. The drawback of this method is that seepages can destroy the sensors and corrosion ruins contacts and metal components. Therefore a frequent and careful maintenance of o-rings and electrical connectors is mandatory but cumbersome. Recently, INRIM developed a dry microcalorimeter in which the thermal stabilization was realized by means of Peltier elements [3]. This approach has been also followed by others [4]. This allowed the implementation of a system that was easier to operate and whose measurement chamber the thermal stability was of some 20 mK. We recently evidenced that this thermal behaviour is one of the main error sources in measurements [5], therefore a new thermostat has been designed, manufactured and tested at INRIM in order to achieve a better thermal stability in the mK range.

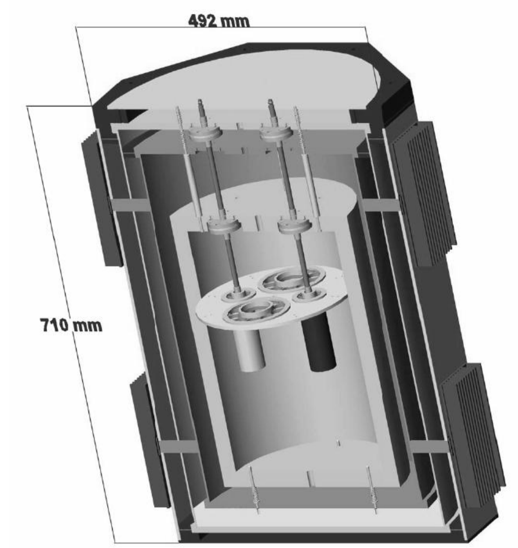

Figure 1. Schematic view of the microcalorimeter thermostat

II. System description

Thermostats for microcalorimeters have quite unique characteristics. The one developed at INRIM is a double inset coaxial system. It means that two coaxial lines enter the thermostat. One is used to supply RF and MW power to the sensor under test placed into the measurement chamber, while the other is used as thermal reference. Through these lines heat can flow in and out the thermostat deeply worsening the thermostat performance. For this reason insulating coaxial sections have been developed that employs thin wall tubes, gold plated brass, steel, plastic and ceramic elements to obtain a high thermal impedance and, consequently, a good filtering of the room thermal disturbances [6].
The thermostat is composed of three concentric cylindrical shields. The external and inner ones made of Aluminium. The intermediate shield is a Copper artefact. The space between the external and the intermediate shields is filled with polystyrene spheres. The inner cylinder delimits the measurement chamber. The overall system (depicted in

Figure 1) is enclosed into a transparent plastic box that was metalized to reflect radiative contributions. Glass wool fills the space between the box and the external Aluminium cylinder. In the picture, the two coaxial insulating sections installed on each feeding line are visible. At the end of each feeding line a power sensor is connected. One is the sensor under test that will be supplied with RF and MW power, while the other acts as thermal reference. A thermopile plane, connected in differential configuration, senses the temperature difference between the base of the connectors of the two sensors. From the behaviour of the thermopile voltage, the effective efficiency of the sensor under test can be calculated [3],[7],[8]. On the measurement plane, two thermopile system are presents in order to fit for sensors of different dimensions. Other electrical connectors placed on the top cover (not visible in Fig. 1) allow the access to the thermopile voltage, the sensor output and the thermometers that sense the system thermal stability.

III. Temperature stabilization

Two independent control stages are employed to obtain the required stability as described in Section II. The coarse stabilization system acts on the intermediate copper shield by means of 8 Peltier elements (four of them are visible in Figure 1) which drive current is proportionally and integratively (PI) controlled. The Peltier system is divided in two sub-systems; one is responsible for the upper Peltier ring and the other for the lower Peltier ring. Each ring has its own PI controller. Their maximum power is about 60 W. The PI loops are closed with two AD590 sensors, one per ring, placed just behind the copper thermal fingers of the related Peltier elements.
The fine stabilization system is composed of a Constantan wire heater that is rolled with equally spaced spires on the top cover of the measurement chamber and provides up to 12 W. Its PI controller is based on an AD590 sensor placed on the internal surface of the measurement chamber top cover. Due to the high thickness of the internal Aluminium shield walls (2.5 cm) it allows a fast and fine control of the residual thermal fluctuations that still bypass the intermediate shield.
The microcalorimeter temperature is monitored, through the contact thermometry technique, by means of two Pt-100 resistance thermometers. The first one is placed on the internal surface of the measurement chamber and the second one on the thermopile plate.
The microcalorimeter has been designed to operate inside a shielded room which environment is stabilized at a temperature of (23.0 ± 0.5) °C and a relative humidity of (45 ± 5) %. The design aims to stabilize the temperature of the thermopile plane so that it presents a stability of the order of ±1 mK for a measurement period of one day. In this application, the absolute temperature do not play a key role. Anyway, it is conventionally set around 297 K.
The PT100 resistance thermometer and the amplified thermopile voltage are read with commercial multimeters. All the system is under PC control by means of IEEE-488.2 bus and a measurement software developed at INRIM in Python language [9]. Measurements are taken every 0.5 s and averaged every minute to minimize the random noise. This measurement cadence has been chosen taking into account the timing of the microwave measurements, in particular the thermal time constant of the DUT when the microwave is applied that is about 40 minutes.

IV. Results

The thermostat has been characterized with respect to the isolation from the external perturbations and to the stability in operative condition.
Isolation is the parameter that quantifies the ability of the thermostat to attenuate the external disturbances. It can be defined as the ratio of the temperature variation of the system as a consequence of a given temperature variation of the environment when the stationary condition has been reached. High isolation let the microcalorimeter to operate in worse temperature conditions, allowing to relax the laboratory environmental requirements. It permits, also, to foresee the behavior of the system for a given laboratory temperature stability.
The stability in operative condition has been evaluated in terms of peak-to-peak amplitude of the thermopile plate thermal fluctuation with and without the presence of the wire heater fine controller.

A. Isolation from external perturbations

The two main external perturbation sources are: a heat flow entering the measurement chamber through the coaxial cables (being the section of the other cables so thin that their contribution can be neglected), and the fluctuation of the environmental temperature.
For minimising the contribution due to the coaxial feeding lines, proper insulating sections have been added as described in Section II. Their characterization has been presented in [6]. When inserted into the microcalorimeter feeding lines system, they are connected to the shields in such a way that the residual thermal disturbances

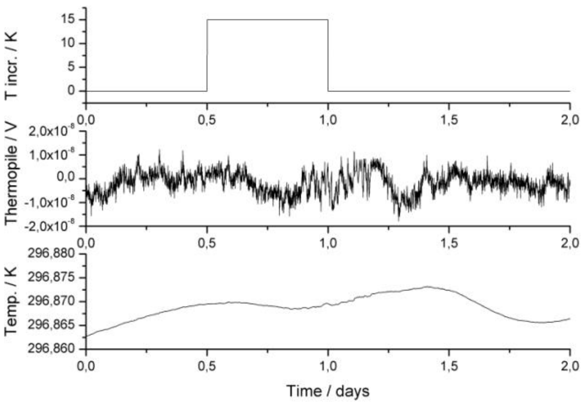

Figure 2. Thermopile voltage and thermopile plane temperature when a heat flow is forced into a coaxial input of the microcalorimeter

coming from the outside through the coaxial lines are discharged onto the shields (see Fig. 1) so that we do not expect any relevant heat flow to the thermopile plane. To test their effectiveness, we connected to one of the two coaxial inputs of the microcalorimeter a coaxial cable rolled up with a Constantan wire heathed to about 312 K. This means a temperature difference of about 15 K with respect to the thermopile plane temperature. If some heat flow is present, the coaxial line should became hotter than the other coaxial line that is used as a reference (see Fig. 1) causing a variation of the output voltage of the differential thermopile system. For these measurements, the Constantan wire fine controller has been kept inactive. As it can be seen from Fig. 2, the effects of such a temperature step are lower than 10 mK, leading to an isolation greater than 1000. Furthermore, Fig. 2 also shows that the peak-to-peak oscillation of the thermopile system voltage is about 20 nV and no correlation with the stimulus appears.

Likewise the isolation from the environment has been evaluated by stepping down and, then, up the laboratory temperature. The results of the test are shown in Fig. 3. Figure 3a, presents the thermopile plane temperature variation when the Constantan wire fine controller is switched off. It highlights that a step variation of about -0.75 K of the laboratory temperature leads to a decrease of about 40 mK of the thermopile plate that, in turn, means an isolation of about 20. The time constant is of the order of 1 day and it is due to the inertia between the intermediate shield (where the loop sensors are placed) and the plane where the thermopiles and the relative monitor termometer are located. Data of Figure 3b refer to measurements performed with the fine controller active. As it can be seen, the performance are much improved, because the response to a temperature variation of 1 K, leads, after a transient of about 8 hours, to a variation below 1 mK. The corrisponding isolation is, therefore, greater than 1000.

B. Stability in operative condition

The thermostat capability in stabilizing the thermopile plane temperature is shown in Figure 4. The left panel (Fig. 4a), presents the coarse stabilization made by the Peltier system that keeps the thermopile plane temperature stable to the order of ten mK. Figure 4b, instead, shows the fine stabilization made with the complete stabilization system active. Now the temperature oscillates in a range of ±1 mK on a daily basis.

Figure 4a puts in evidence daily oscillations with an amplitude of 20 mK peak-to-peak due to the low isolation of the thermostat when only the Peltier controllers are active (see also Fig. 3a). Conversely, when the fine controller is active (Fig. 4b), these fluctuations are no longer visible thanks to the high isolation that, in this case, exceeds 1000 (see also Fig. 3b). Nevertheless, some noise still remains that is not correlated with the environmental

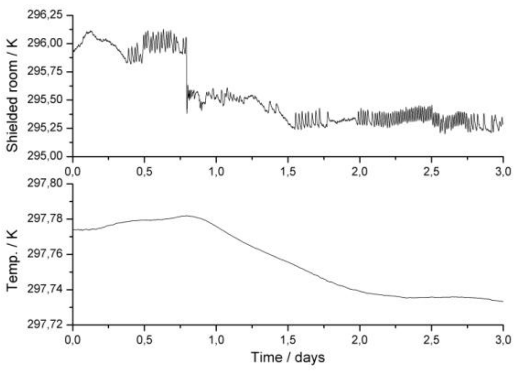

Figure 3a. Laboratory temperature fall and relative thermopile plane temperature when the Constantan fine controller is off

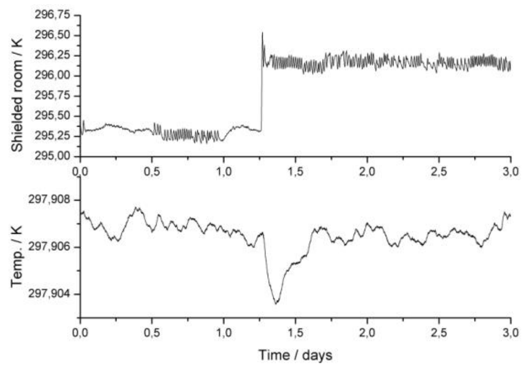

Figure 3b. Laboratory temperature rise and relative thermopile plane temperature when the Constantan fine controller is on

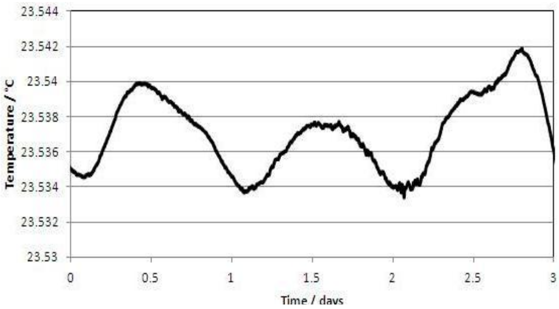

Figure 4a. Temperature stability of the thermopile plane with only the Peltier controllers on

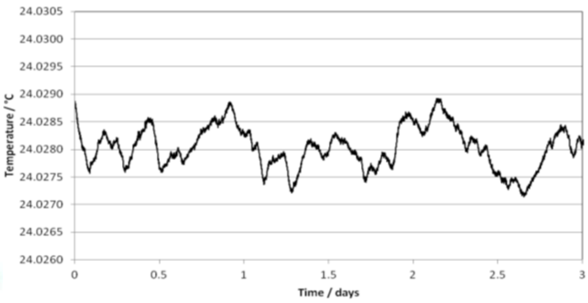

Figure 4b. Temperature stability of the thermopile plane with all the controllers active.

temperature. We believe that this small but faster random fluctuations are due to the noise of the fine controller that, at this level, is no longer negligible. As a consequence, a further enhancement of the temperature stability is still possible, if required, by replacing the fine control system with a less noisy one.

IV. Conclusions

The construction of a microcalorimeter for the realization of the RF and MW primary power standard requires the design of a thermostat that has been here presented. It has been completely designed, assembled and tested at INRIM. The thermostat is based on three cylindrical concentric chambers. The middle one is thermally stabilized by means of a Peltier system. The inner one is equipped with a Constantan-wire heater for fine control. Both stabilization systems are PI controlled. Thanks to custom made special coaxial insulating sections of INRIM design and to the fine controller, the isolation of the thermostat from external environmental temperature exceeds 1000. Due to this high isolation, the thermostat performances in terms of temperature fluctuation of the thermopile plane have been demonstrated to reach the target specifications of ±1 mK during a measurement period of one day.

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