Calibration of a Single-Cell Calorimeter in a New Transient-state Test Bench

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Abstract—The nuclear radiation energy deposition rate is a key value for the thermal design of experiments, on materials and nuclear fuels, carried out in experimental channels of nuclear research reactors. Studies are led for two kinds of sensor currently dedicated to quantifying this value and corresponding to calorimeter. Development of new sensors but also improvement of their calibration and their associated interpretation methods are necessary. These aims are possible by many ways such as numerical simulations of sensor, characterizations under laboratory conditions and experimental campaign under irradiation conditions. The calibration step under non-irradiation conditions represents a crucial phase. This phase requires the development of specific benches. The present paper focuses on a new thermal-transient bench and its use to perform calibration of a polish single-cell calorimeter. The new bench is detailed. First studies of the influence of external conditions (temperature, velocity) on the calorimeter sensitivity are presented and discussed.

Keywords—Calorimetry, MTR, Nuclear Heating Rate, Heat Exchanges, Calibration, Transient State

I. INTRODUCTION

In the field of nuclear energy, the control and measurement of the energy deposition rate per unit mass induced by the interactions of radiation with matter (usually expressed in W·g⁻¹) are crucial. This parameter, also called nuclear heating rate, is used in order to carry out accurate studies on the ageing of materials and the behaviour of nuclear fuels under irradiation inside research reactors. In particular, several experiments require this key parameter in order to design thermal device allowing the achievement of specific and stable thermal conditions.

In this context, the IN-CORE program [1-3], run jointly by the CEA and Aix-Marseille University since 2009, was created. Currently, two kinds of in-pile sensors are used to measure nuclear heating rate inside Material Testing Reactors (MTRs): the single-cell calorimeter [4,5], and the differential calorimeter [5-10]. Thus, for each kind of sensor, the IN-CORE program is involved in a scientific axis focusing on the enhancement of sensor design, their out-pile calibration means, methods (protocols) and their in-pile quantification methods, and interpretation analysis. For that, various experimental actions are conducted such as fabrication of specific experimental test benches [10,11] suited to the sensor studied, measurements of thermal properties of materials and developments of multi-sensor mock-ups (cf. Fig.1).

Fig. 1. Description of the IN-CORE research axis dedicated to the improvement of the in-pile calorimeter

In this paper, the studied in-pile calorimeter is a single-cell calorimeter which corresponds to the KAROLINA calorimeter (cf. Fig.2) [3, 5]. This calorimeter was designed and developed by the Polish National Center of Nuclear Research (NCBJ) to carry out experiments in the MARI A Reactor in 2014 and 2015. The aim of these two irradiation campaigns was the comparison of different calorimeters: KAROLINA calorimeter and gamma thermometer then KAROLINA calorimeter and two French differential calorimeters (CARMEN [5] and CALORRE [12,13]).

This single-cell calorimeter designed for a nuclear heating rate range up to 5W·g⁻¹, is composed of a graphite sample surrounded by a thin layer of Helium gas and a stainless steel jacket. Moreover, this sample is located in the center of the jacket by means of two dedicated sample holders made of porous polymer foam chosen in order to reduce nuclear heating

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deposition on these two pieces. The gas chosen to fill space between the jacket and the sample is helium due to its high thermal conductivity. Finally, this calorimeter is instrumented by two K-type thermocouples, called Ts and Tf, located at the center of the sample and at the middle height of the external surface of the jacket respectively as shown in Figure 2.

![Thermocouples](image)

**Fig. 2.** Scheme of the KAROLINA single-cell calorimeter (in the left–hand section) and its picture integrated inside the INTERCAL probe for the irradiation campaign performed in 2015 (in the right–hand section).

The calibration method of this type of sensor is very different from that of differential calorimeters. In fact, the differential calorimeters integrate heating element in each calorimetric cell, which allows the experimental simulation of an amount of energy, by means of the Joule effect, equivalent to the nuclear heating rate expected in the research reactor and lead to calibration curves by analysing thermal steady states. Therefore, single-cell calorimeters, that do not include heating elements, require other calibration methods. In this case, the calibration methods are based on the analysis of transient states. The processing of the temporal response curve allows the determination of the sensitivity of the sensor from a characteristic value corresponding to thermal time constant. This paper focuses on the experimental calibration of the KAROLINA calorimeter with a new transient-state test bench. More precisely, this paper begins by the analytical determination of the thermal time constant and the sensitivity of a single-cell calorimeter. Then a detailed presentation of the new transient-state test bench and its experimental protocol are performed. Finally, the experimental temporal response of KAROLINA calorimeter, obtained with this bench, is shown and preliminary studies of the influence of thermal and hydraulic external conditions on this response r and the calorimeter sensitivity are given.

II. THEORETICAL DETERMINATION OF THE SENSITIVITY OF A SINGLE-CELL CALORIMETER

The determination of the sensitivity of a single-cell calorimeter is realized thanks to a transient calibration in two steps. In the first step, the calorimeter is put inside a hot surrounding in order to reach a first thermal steady state at a hot temperature. In the second step, the calorimeter is as fast as possible inserted into a cold surrounding for instance, in order to cool the sensor up to reach a second thermal equilibrium inside it (cf. Fig.3).

![Diagram](image)

**Fig. 3.** Diagram of the transient-calibration protocol of a single-cell calorimeter

Thanks to this out-of-pile cooling curve representing temperatures versus time, the thermal time constant $(\kappa/s)$ and the sensitivity of the sensor can be obtained. Indeed, in the case of laboratory conditions by considering conductive transfer only, the heat equation is inside the calorimeter:

\[
\frac{\partial T}{\partial t} = \lambda \nabla^2 T
\]

where $\rho$ is the density (kg.m$^{-3}$), $c_p$ is the specific heat (J.kg$^{-1}$.K$^{-1}$), $\lambda$ is the thermal conductivity (W.m$^{-1}$.K$^{-1}$).

By applying a heat balance on the calorimeter, the heat accumulation is equal to the heat transfers towards the cooling flow, that leads to this formula:

\[
\frac{\partial T}{\partial t} = \frac{1}{\rho c_p V R_a} \left( T_s - T_f \right)
\]

where $T_s$ is the sample temperature (K), $T_f$ is the fluid temperature (K), $V$ is the volume of the area where heat transfers are considered (m$^3$), and $R_a$ is the thermal resistance from the sample to the external surface of the calorimeter jacket.

Thus, with a variable change $(\theta = T_s - T_f)$ and a time integration, the equation (2) becomes:

\[
\theta = A e^{-\kappa t}
\]

with

\[
\kappa = \frac{1}{\rho V c_p R_a}
\]

Consequently $\kappa$ can be determined experimentally by analyzing the transient-state part of the calibration curve and the sensitivity $S$ can be deduced by applying the formula:

\[
S = \frac{1}{\kappa c_p}
\]

Finally, from this key value, the nuclear heating rate $E_{n\gamma}$ (in W.g$^{-1}$) can be also calculated with this formula:

\[
E_{n\gamma} = \Delta T_0 C_p \kappa
\]

To realize this transient-calibration method based on the two successive step, an automatized test bench was developed.
III. PRESENTATION OF THE BENCH FOR CALORIMETER CALIBRATION UNDER TRANSIENT CONDITIONS (BERTRAN)

To determine accurately the time constant of single-cell calorimeters and their sensitivity, the BERTRAN bench was designed and developed. The main metrological goals for its fabrication were the control and minimization of the transfer time between the hot and the cold surroundings, the capability to perform reproducibility and repeatability and parametrical studies.

A. Detailed characteristics of this new transient-state test bench

This new experimental setup is composed of:
- Two separate tanks filled with silicon oil in which the single-cell calorimeter is alternatively inserted. These two tanks are used to impose various external thermal and flow boundary conditions to the instrumented cell (a flow in each tank with controlled temperature and velocity) (cf. Fig. 5). The tank located on the right is designed to have a cold fluid temperature from 20°C to 80°C thanks to a heating cartridge of 750W coupled with a cooling device of 300W. The second tank located on the left concerns the hot fluid temperature. A setup from 120°C to 250°C can be imposed by means of three heating cartridges of 750W each. In each tank, a fluid circulation system is used for the control of the velocity of the flow from 500 rpm to 1250 rpm, which corresponds to a fluid velocity lower than 1.5 m/s.

- A mechanical system composed of two drive chains with two idler sprockets, which move a cart where the calorimeter is fixed thanks to two V-flanges (cf. Fig. 6). This system allows the displacement of the single-cell calorimeter from one tank to the other automatically.

The automation of the transfer system of the calorimeter leads to a constant transfer time equal to about 1.3 s.

- A data acquisition system connected to a computer. The device consists of an Agilent data acquisition unit (34970A) containing a 34901A multiplexer of 20 channels used for temperature measurements with the calorimeter K-type thermocouples.

- An extraction system to remove silicon oil vapours.

B. Experimental protocol and temporal response

In order to realize KAROLINA calibration, the temperature of the cold surrounding was fixed to 30°C (a value corresponding to that in the pool of MARI reactor). The temperature of the warm tank was fixed to 200°C in order to have a significant difference and to reach temperature obtained by the calorimeter sample during irradiation campaigns. An identical velocity in the two baths is also chosen and imposed.

The protocol explained in section II is applied. First of all, the calorimeter is inserted in the hot bath. The temperatures inside the calorimeter increase. When the steady state is reached, the calorimeter is moved and put in the cold bath. The temperatures decrease up to the occurrence of a new steady state as shown on Figure 7. The step 2 is analyzed to determine the time constant (cf. Fig. 9). Figure 7 gives results obtained for two experiments (Day 1 and Day 2). A good reproducibility is observed.
IV. PRELIMINARY STUDIES WITH THIS NEW TRANSIENT-STATE TEST BENCH

Preliminary experiments were performed in order to study the influence of the velocity of the fluid flow of the two tanks and the influence of the cold temperature on the sensor sensitivity.

A. Influence of fluid velocity conditions

Various velocities of fluid flow have been tested from 500 to 1250 rpm.

Curves of the sample temperature (Ts) versus time are represented for four velocities in Figure 8. The curve reaches the final cold temperature more quickly when the fluid flow velocity increases. This expected behaviour is due to the intensification of the convective heat exchanges. Nevertheless, a slight influence of the fluid flow velocity can be observed.

Figure 9 shows the cooling part of the temporal curves after a simple mathematical transformation (linearization by considering logarithm functions). Consequently, a direct determination of the constant time $\tau$ can be realized as it corresponds to the slope of the linear curve. The four tested velocities lead to this range of time constant: $0.0290 \text{ s}^{-1} > \tau > 0.0283 \text{ s}^{-1}$. This range induces a low variation of the sensitivity (sensitivity calculated by using equation 5) versus the fluid velocity for a same considered temperature of the sample: diminution of about 1°C.g/W from 500 to 1250 rpm (cf. Figure 10).

B. Influence of fluid temperature conditions

As the fluid temperature inside an experimental channel of a reactor changes versus the axial position, different temperatures of the cold tank were tested.

Figure 9. Temporal curves representing the linearization of the transient state during the cooling step for four fluid velocities from 500 to 1250 rpm and with a hot temperature of 200°C and a cold one of 30°C

But by taking account the variation of the temperature sample leading to a $c_p$ change, a significant variation of the sensitivity versus the sample temperature can be observed (cf. Figure 10). This kind of variation has been also obtained in the case of the differential calorimeters having a high sensitivity in particular.

The variation in the case of differential calorimeters is linked to the modification of the thermal conductivity of structural materials of the calorimeter and its gas but also to the change of the nature and intensity of heat transfers (increasing of the radiative thermal transfer inside the cell for instance).

B. Influence of fluid temperature conditions

As the fluid temperature inside an experimental channel of a reactor changes versus the axial position, different temperatures of the cold tank were tested.
Figure 11 presents the temperature of the sample of the calorimeter versus time for three cold temperatures. It can be also noted a low variation of the temporal response curve. Consequently a low variation of the constant time is observed too.

V. CONCLUSIONS AND OUTLOOKS

First experiments were performed with a new automated transient-state test bench on a Polish single-cell calorimeter (KAROLINA calorimeter) in order to determine the constant time and the sensitivity of this sensor. A low influence of the velocity of the fluid flow was shown on the response of the calorimeter, on its time constant and on its sensitivity for a same sample temperature. By against, a significant influence of the sample temperature on the sensitivity of this sensor was noted. A sensitivity diminution of more than 15°C g/W has been observed for a temperature increasing from 30°C to 140°C. First experiments of the influence of the cold temperature were carried out.

New parametrical studies by changing hot and cold temperatures and other fluid velocities will be performed to complete these first results of the calibration of single-cell calorimeter. Moreover, a new experimental campaign will be performed for a French compact differential calorimeter called CALORRE. These results will be compared with those obtained by means of a common calibration using heaters (steady state calibration).

ACKNOWLEDGMENT

We acknowledge the IN-CORE program to financially support these works.

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