DIRECT MEASUREMENTS OF THE MAGNETOCALORIC EFFECT OF GADOLINIUM SAMPLES AT NEAR ROOM TEMPERATURE

Paulo V. Trevizoli, trevizoli@polo.ufsc.br
Jader R. Barbosa Jr, jrb@polo.ufsc.br
Pablo A. Oliveira, pablo_oliveira@polo.ufsc.br
Alvaro T. Prata, prata@polo.ufsc.br
Rogério T. S. Ferreira, rogeriotsf@gmail.com
POLO - Laboratórios de Pesquisa em Refrigeração e Termofísica, Departamento de Engenharia Mecânica, Universidade Federal de Santa Catarina, Florianópolis, SC, 88040-900, Brasil.

Abstract. *Direct measurements of the adiabatic temperature change are an attractive alternative for characterizing the magnetocaloric effect of promissing materials for application in magnetic cooling systems. In the present work, a simple experimental apparatus was designed and constructed specially for performing the direct measurements. The main purpose of the paper is to characterize the magnetocaloric temperature change of gadolinium (Gd) samples as a function of temperature. A satisfactory agreement was observed between our direct measurement results and those obtained via an indirect approach reported in the open literature (Dan'kov et al., 1997). The differences between the adiabatic temperature change can be attibuted to thermal losses in the sample and to an intrinsic departure from the equilibrium condition which is characteristic of the direct measurement approach.*

Keywords*: magnetocaloric effect, adiabatic temperature change, direct measurement, gadolinium, active magnetic regenerator.*

1. INTRODUCTION

Magnetic refrigeration is a cooling technology based on the magnetocaloric effect (MCE). The MCE is characterized by a reversible temperature variation of the material when subjected to a changing magnetic field. Some classes of magnetic materials exhibit a significant MCE (of the order of 2 K/T) at room temperature, which makes them potential candidates for utilization as refrigerants in active magnetic regeneration refrigeration (AMRR) cycles. To date, the most widely examined of such materials is gadolinium (Gd), a rare-earth metal which presents a MCE of typically 2.3 K/T at a Curie temperature (T_C) of 293 K when subjected to a magnetic field variation of up to 2 T (Tishin and Spichkin, 2003; Dan'kov *et al.*, 1998).

In principle, the MCE can be measured directly or indirectly. The so-called direct measurement procedure presents some advantages in comparison with the indirect approach, which requires calorimetric and magnetic measurements in addition to the utilization of a theoretical Maxwell relation to obtain the adiabatic temperature change. The direct approach, on the other hand, can be undertaken with conventional instruments for temperature measurement, such as thermocouples or resistance temperature detectors (Gschneidner and Pecharsky, 2000; Pecharsky *et al.*, 2001). However, in order to obtain meaningful and reliable results with the direct method, several experiment design requirements have to be met, such as a rapid change of magnetic field, a uniform magnetic field volume (if the sample is moved in and out of it), an effective thermal insulation of the sample, a satisfactory compensation technique to eliminate the effect of the magnetic field change on the temperature sensor reading, and a sample volume (mass) much greater than that of the temperature sensor (> 1 g) (Gschneidner and Pecharsky, 2000; Dan'kov *et al.*, 1997).

The direct approach has been explored to some extent in the open literature. Benford and Brown (1981) described direct measurements of the MCE in polycrystalline Gd in applied fields of 1, 3, 5 and 7 T, with maximum temperature variations of 3.6 K (1 T), 7.8 K (3 T), 11.0 K (5 T) and 13.8 K (7 T) at T_C ~ 292.5 K. The magnetocaloric temperature change decreased to half of the peak value at both higher and lower temperatures within ± 40 K of T_C. They attributed the main sources of error to the temperature drift due to non-adiabatic conditions (with drift rates less than 0.3 K/min) and slow thermocouple response time. Dan'kov *et al.* (1997) discussed in detail the principal requirements for good direct measurements with thermal contact sensors, the main sources of error, the error compensation techniques, and the comparison procedures between direct and indirect calorimetric measurements. Gopal *et al.* (1997) described the development of an automated sample translation-type apparatus for measuring directly the MCE of magnetic refrigerants between 10 to 325 K in applied fields of up to 9 T. They reported measurement repeatability of ± 30 mK in the determination of the adiabatic temperature change of gadolinium (Gd), dysprosium (Dy), holmium (Ho), and a gadolinium-yttrium alloy ($Gd_{52}Y_{48}$). Giguère *et al.* (1999) conducted direct measurements of the giant MCE in $Gd_5Ge_2Si_2$ samples, and revealed peak temperature changes of approximately 4 K (at 272 K), 8.5 K (between 270 and 280 K) and 10 K (at 280 K) for 2, 5 and 7 T, respectively. Fujieda *et al.* (2004) evaluated the MCE in $La(Fe_xSi_{1-x})_{13}$ samples and observed discrepancies between results obtained with direct and indirect measurements of the order of 2 K at 188 K, which were attributed to thermal losses due to an imperfect insulation of the sample in the direct measurements. More recently, Wada *et al.* (2007) performed direct measurements of giant MCE in $Mn_{1+\delta}As_{1-x}Sb_x$

samples and reported maximum adiabatic temperature changes of the order of 10 K and 9 K for x = 0.1 and 0.3, respectively, in a field change of 5 T.

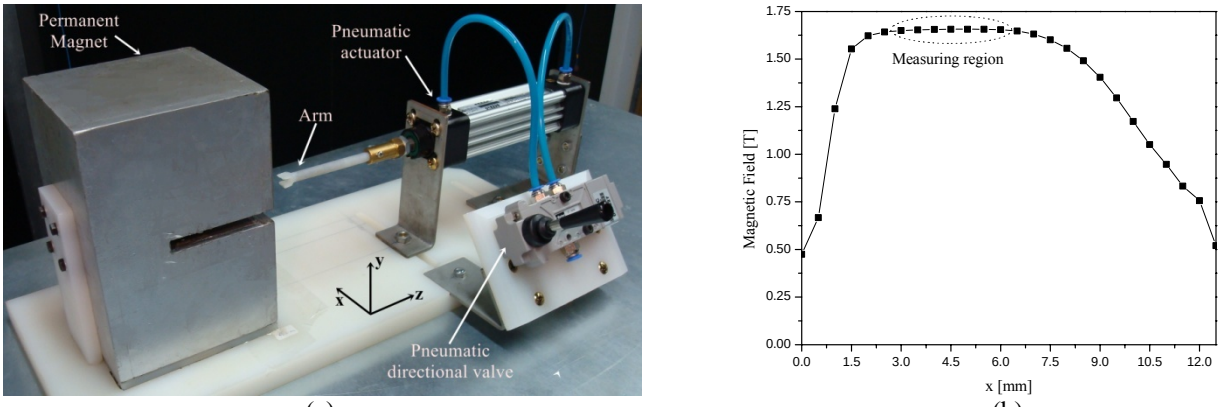
The present paper advances an experimental procedure for evaluating the MCE at near room temperature via the direct approach. The measurements of *magnetocaloric temperature change* (this terminology seems more appropriate than *adiabatic temperature change* since the experimental conditions are not perfectly adiabatic) were carried out in a sample prepared from Chinese commercial Gd. The tests were conducted under a field change from 0 to 1.65 T, between 283 and 303 K. The main objectives of the study are as follows:

(i) To quantify the magnetocaloric temperature change of Gd samples as a function of temperature;
(ii) By using the same magnet employed in our AMRR cycle demonstrator, to provide a more realistic assessment of the actual temperature change achieved under real (not perfectly adiabatic) conditions;
(iii) To calculate the corresponding decrease in the instantaneous cooling capacity of the magnetic refrigeration cycle through a comparison of the cycle performance under an ideal adiabatic temperature change with that observed using the actual temperature change (direct approach). This is achieved by incorporating the magnetocaloric temperature change of Gd (ideal and real) into a mathematical model for a Brayton-based parallel plate AMRR cycle (Oliveira *et al.*, 2009a, 2009b).

As will be discussed in the paper, the results are in satisfactory agreement with the indirect measurements published in literature (typically 0.5 K at T_C). Also, the non-ideal conditions under which the magnetization occurs in the regenerator act toward a reduction of the instantaneous cooling capacity, which influences negatively the performance of the cooling system.

2. EXPERIMENTAL WORK

The direct measurement apparatus (DMA) is composed of a NeFeB permanent magnet arranged in a so-called Hallbach array and a pneumatic circuit to move the sample into and out of the magnetic field, as shown in Fig. 1(a). The pneumatic actuator provides a rapid magnetic field change in order to improve the measurement precision (Gopal *et al.*, 1997; Gschneidner and Pecharsky, 2000). Figure 1(b) shows the variation of the average magnetic field in the transverse direction, x (normal to the actuator arm, which moves along z), and the position where the sample enters the magnetic field (measuring region). The magnetic field was measured by a Bell model 640 gaussmeter and a transverse gaussmeter probe model HTB4-0608. The experimental uncertainty was calculated at ± 2% of experimental value.



(a) (b)

Figure 1. (a) Direct measurement apparatus; (b) average magnetic field in the transverse direction.

The measurements were carried out in an environmental test chamber capable of controlling the average ambient air temperature to within ± 0.3 K. During the tests, the ambient temperature was varied between 283 K and 303 K.

The samples were prepared from a single piece of Chinese commercial Gd and composed by two sheets with dimensions of 13.00 × 7.20 × 1.9 ± 0.05 mm (2.45 ± 0.03 g). Between the sheets, a T-type thermocouple was installed. Thermal contact between the Gd plates and the thermocouple was improved by placing a thin layer of thermal grease during the preparation of the samples. The samples have been calibrated for temperature measurements according to the following procedure: 1) the samples were connected to the same data acquisition system used in the direct measurement; 2) the samples were inserted in a thermostatic water bath and maintained in thermal equilibrium; 3) the temperature data were acquired for 300 s at the same acquisition frequency of the direct measurement; 4) the temperature averaged over the 300 s was compared with a standard thermometer; 5) a calibration curve between 278 K

to 298 K was produced. After calibration, the samples were thermally insulated with a 9 mm layer of polystyrene. The step-by-step preparation of the samples is illustrated in Fig. 2.

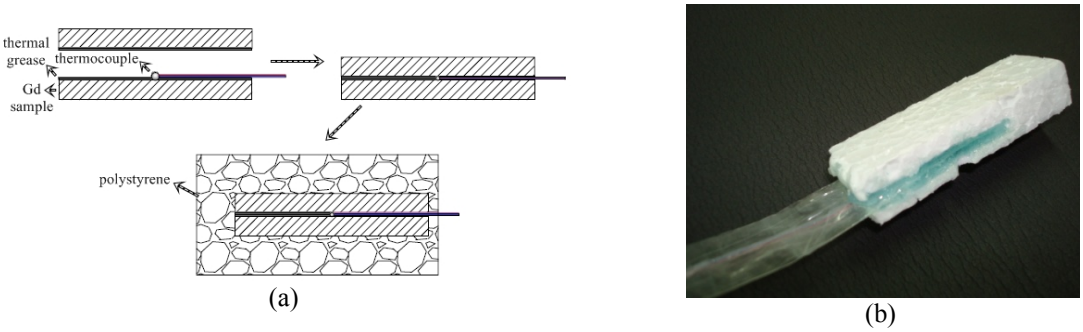


Figure 2. (a) Preparation of the Gd samples for the DMA; (b) a typical sample.

The thermally insulated sample was attached to the tip of the nylon arm in the DMA and the thermocouple was connected to the data acquisition system. A sampling rate of 67 Hz was used in the experiments. Once the desired ambient temperature was set, some time is necessary for the sample to reach thermal equilibrium with the surroundings. When the difference between the sample and the ambient air becomes less than 0.1 K, the pneumatic valve is manually operated and the sample is displaced by 100 mm (actuator stroke) into the magnetic field. The time response of the thermocouple is recorded and, after a few seconds (typically five), the sample is removed from magnetic field. Again, some time is required for the sample to regain thermal equilibrium with the surroundings. This procedure was repeated 5 times for each ambient temperature in order to guarantee the reproducibility of the measurements and to decrease the experimental uncertainty. Based on the calibration procedure and on the error propagation analysis, the experimental uncertainties were calculated at ± 0.20 K.

3. RESULTS AND DISCUSSIONS

The time-dependent temperature signal for each sample can be divided into 3 periods, as shown in Fig. 3(a): (1) before magnetization, (2) during magnetization and (3) MCE measurement.

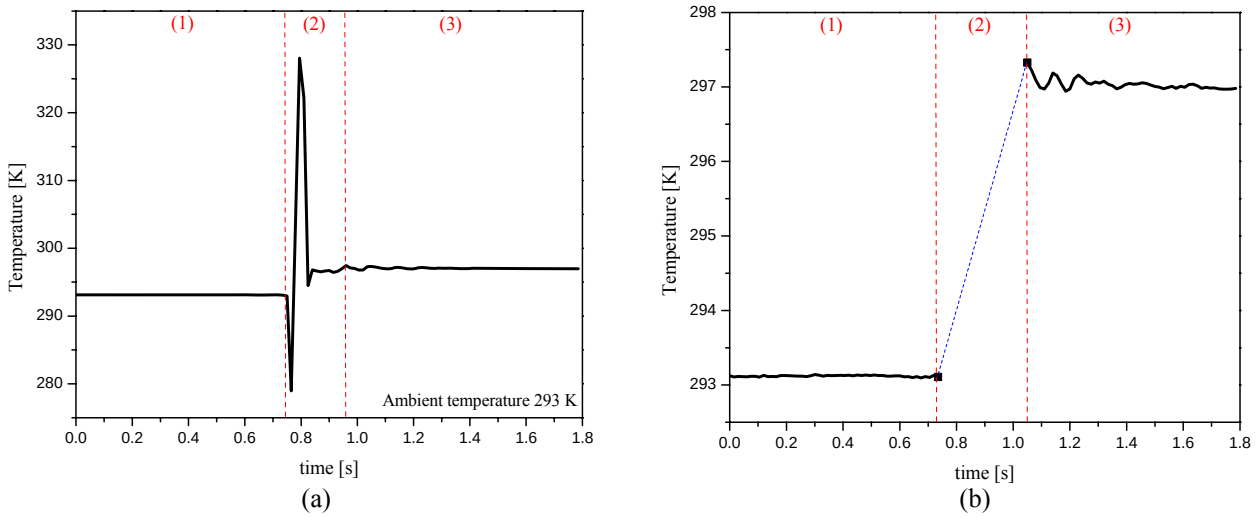


Figure 3. (a) The 3 periods of the temperature signal; (b) temperature signal after treatment.

The second period is characterized by a strong oscillation of the temperature signal due to the variation of the magnetic field and the motion of the sample, both of which influence the thermocouple response. Based on observations of our own database and on similar behavior reported by Dan'kov *et al* (1997), the second period was seen to last for approximately 0.3 s in all measurements. This period is initially disregarded in the experimental analysis, as seen in Fig. 3(b) (dashed line). However, a procedure is proposed to compensate the thermal losses and the effect of the magnetic field change on the temperature sensor reading (Gschneidner and Pecharsky, 2000) taking place during the second period. By applying this correction, it is possible to infer the actual magnetic temperature change from the temperature measurements obtained in the third period.

For statistical significance, a total sampling time of 0.75 s (50 measurements) was adopted as of the beginning of the third period. The experimental magnetocaloric temperature change is calculated as the arithmetic average of the temperature measurements over the total sampling time. As can be seen from Fig. 3(b), the temperature shows a slight decreasing trend as a function of time in the third period due to heat transfer between the sample and the surroundings (Canepa *et al*, 2005).

The MCE is assumed to take place instantaneously (Brück, 2005) and, therefore, its calculation based only on the temperature signal during the third period may represent an underestimation of the true adiabatic temperature change. In order to account for the MCE in the second period and for the thermal losses during both second and third periods, a mathematical model is proposed in which the transient heat conduction equation is solved in the magnetocaloric material and polystyrene insulation. The 1D model geometry is shown in Fig. 4, which assumes symmetry with respect to the plane x = 0 (where the thermocouple is located). The thickness of the Gd layer (equivalent to one sheet) is L and the polystyrene layer is assumed to behave as a semi-infinite medium. The physical properties of the materials are shown in Table 1. At t = 0, the magnetic field is applied and the temperature of Gd layer increases instantaneously by an amount equal to the actual magnetic temperature change corresponding to the equilibrium temperature, ΔT_m.

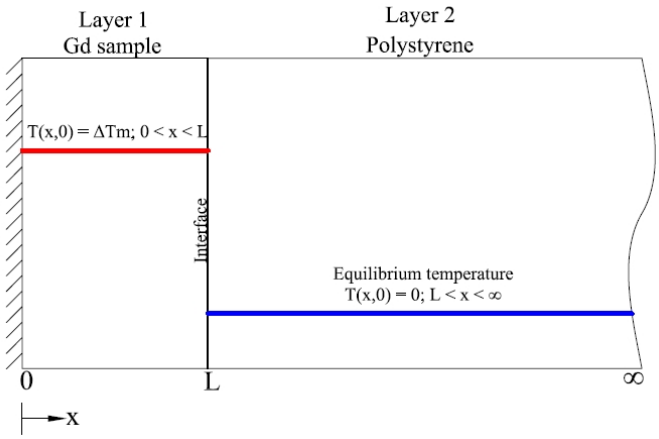


Figure 4. Model geometry and initial conditions.

The dimensionless temperature is defined as:

$$\theta = \frac{T(x,t)}{\Delta T_m} \tag{1}$$

where T(x,t) is the excess temperature with respect to the equilibrium value ($0 \leq T \leq \Delta T_m$). The governing equations for heat conduction in the Gd and insulation are given by:

$$\frac{\partial^2 \theta_{Gd}}{\partial x^2} = \frac{1}{\alpha_{Gd}} \frac{\partial \theta_{Gd}}{\partial t}, \text{ for } 0 < x < L \tag{2}$$

$$\frac{\partial^2 \theta_{PS}}{\partial x^2} = \frac{1}{\alpha_{PS}} \frac{\partial \theta_{PS}}{\partial t}, \text{ for } L < x < \infty \tag{3}$$

where α is the thermal diffusivity. The boundary and initial conditions are as follows:

$$\frac{\partial \theta_{Gd}}{\partial x}\Big|_{x=0} = 0 \tag{4}$$

$$\theta_{Gd}(L,t) = \theta_{PS}(L,t) \tag{5}$$

$$k_{Gd} \frac{\partial \theta_{Gd}}{\partial x}\Big|_{x=L} = k_{PS} \frac{\partial \theta_{PS}}{\partial x}\Big|_{x=L} \tag{6}$$

$$\theta_{PS}(\infty,t) = 0 \tag{7}$$

$$\theta_{Gd}(x,0) = 1 \tag{8}$$

$$\theta_{PS}(x,0) = 0 \tag{9}$$

The analytical solution of the system of equations was proposed by Ozisik (1993):

$$\mu = \sqrt{\frac{\alpha_{Gd}}{\alpha_{PS}}};\ \beta = \frac{1}{\mu}\frac{k_{Gd}}{k_{PS}};\ \gamma = \frac{\beta-1}{\beta+1};\ \alpha = \frac{k}{\rho c_p} \tag{10}$$

$$\theta_{Gd} = \frac{T_{Gd}}{\Delta Tm} = 1 - \frac{1-\gamma}{2}\sum_{n=0}^{\infty}\gamma^n\left\{\operatorname{erfc}\left[\frac{(n+1)L-x}{2\sqrt{\alpha_{Gd}t}}\right] + \operatorname{erfc}\left[\frac{(2n+1)L+x}{2\sqrt{\alpha_{Gd}t}}\right]\right\},\ \text{for } 0 < x < L \tag{11}$$

$$\theta_{PS} = \frac{T_{PS}}{\Delta Tm} = \frac{1+\gamma}{2}\sum_{n=0}^{\infty}\gamma^n\left\{\operatorname{erfc}\left[\frac{2nL+\mu(x-L)}{2\sqrt{\alpha_{Gd}t}}\right] - \operatorname{erfc}\left[\frac{(2n+2)L+\mu(x-L)}{2\sqrt{\alpha_{Gd}t}}\right]\right\},\ \text{for } L < x < \infty \tag{12}$$

Table 1. Summary of physical properties.

Properties	**Gd sample** Petersen *et al. (2008)*	**Polystyrene** Incropera and DeWitt, (2002)
Density $-\ \rho$	$7900.0\ {}^{kg}/_{m^3}$	$55.0\ {}^{kg}/_{m^3}$
Thermal conductivity $-\ k$	$10.50\ {}^{W}/_{mK}$	$0.027\ {}^{W}/_{mK}$
Specific heat capacity $-\ c_p$	Siddkov, *et al.* (2005)	$1210.0\ {}^{J}/_{kgK}$

The procedure for quantifying the MCE consists of finding by successive iterations the value of the actual magnetocaloric temperature change, ΔT_m, which minimizes the following objective function:

$$\omega = \left|\overline{T}^{C}_{x=0} - \overline{T}^{E}_{x=0}\right| \tag{13}$$

where $\overline{T}^{C}_{x=0}$ and $\overline{T}^{E}_{x=0}$ are the calculated and experimental time-averaged values of the temperature at x = 0 (i.e., at the sample symmetry plane) between 0.3 s and 1 s. Figure 5(a) illustrates the calculated temperature distribution in the Gd layer and in the insulating material layer as a function of time for an equilibrium temperature of 293 K and a magnetic field of 1.65 T. The temperature decrease in the plane x=0 can be observed in Fig. 5(b).

By applying the proposed procedure, an empirical relationship was derived for compensating the effects of changing magnetic field and thermal losses during the second and third periods, and the compensation relationship is given by:

$$\Delta T_m = 1.009(\Delta T^{E}_{m}) \tag{14}$$

where ΔT^{E}_{m} is the magnetocaloric temperature change measured experimentally using the time average of the temperature signal in the third period. Figure 5 (c) presents the theoretical compensation of the magnetocaloric temperature change at x=0 (i.e., the position of the thermocouple in the sample). As can be seen, the temperature signal is well picked up by the model, indicating that the effect of heat loss in the insulating material is being dealt with appropriately. The influence of the sample geometry and orientation on the magnetization process, as reported by Bahl and Nielsen (2009), was disregarded in our work. However, these factors are known to exert some influence on the magnetocaloric temperature change and shall be evaluated more systematically in our research in the future.

Figure 6(a) shows the magnetocaloric temperature change results (after compensation) as a function of the ambient equilibrium temperature. For comparison purposes, results of an indirect approach measurement of the MCE in a Chinese polycrystal Gd (corrected for H = 1.65T) reported by Dan'kov *et al*. (1998) are also shown in Fig. 6(a). A satisfactory agreement is observed between the curves, given the differences associated with the purity of the samples and the experimental conditions under which both experiments were performed; while the indirect approach requires quasi-static and carefully controlled conditions (so as to minimize the departure from thermodynamic equilibrium), the direct approach renders a more suitable evaluation of the MCE taking place in real applications such as in the regenerator of an AMRR apparatus (Pecharsky and Gschneidner, 1999).

Figure 6(b) shows a comparison with the direct measurements reported by Canepa *et al.* (2005) (also corrected for H=1.65T). The magnitudes of the magnetocaloric temperature change are in agreement with each other, but a difference in the value of T_C was observed. This difference is not significant since values for T_C are reported in the range of 290 K – 297 K for low magnetic fields. (Bahl and Nielsen, 2009).

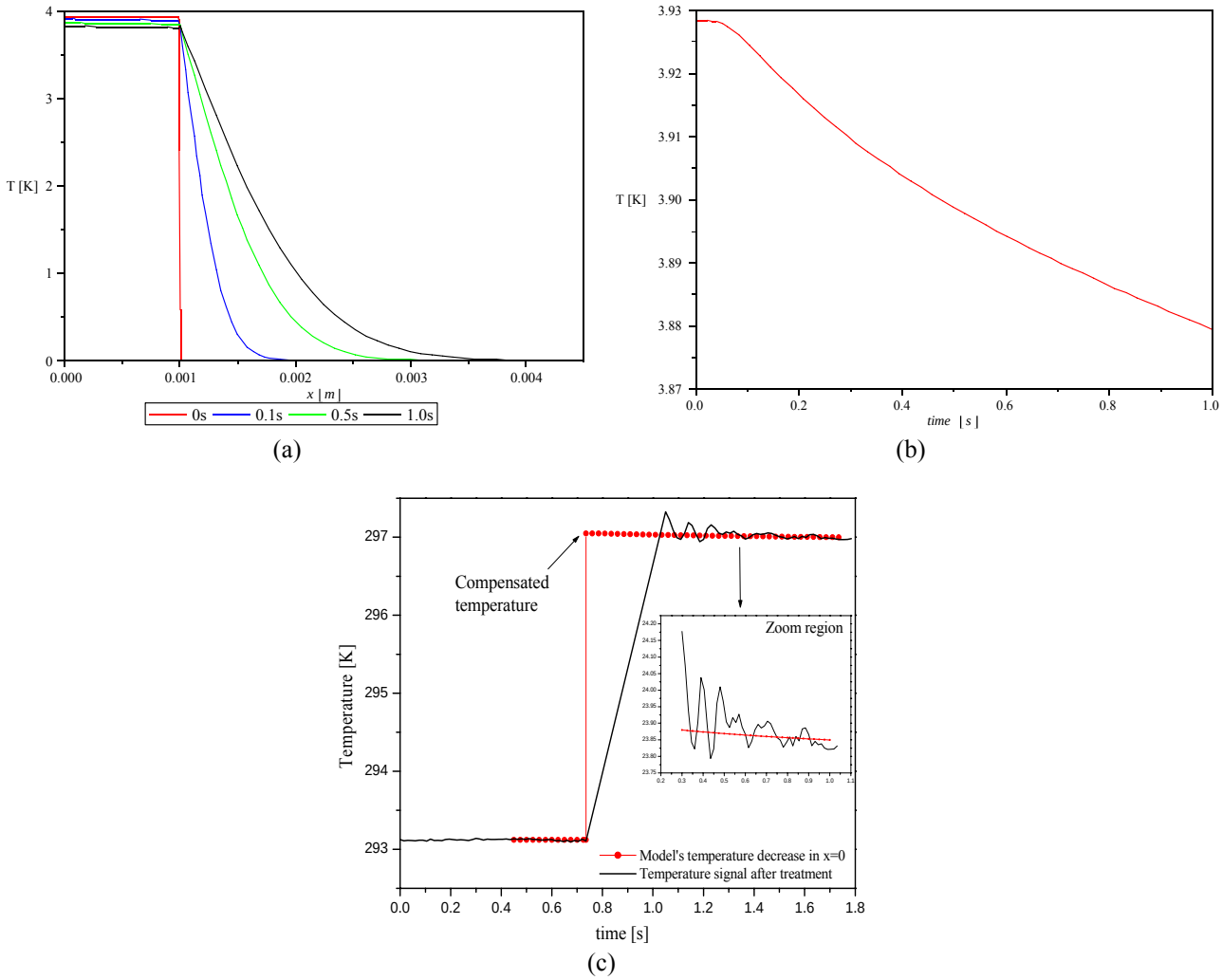


Figure 5. (a) Calculated temperature distribution in the Gd and polystyrene layers at different times; (b) Temperature decrease in the Gd layer at x=0; (c) temperature compensation at x=0.

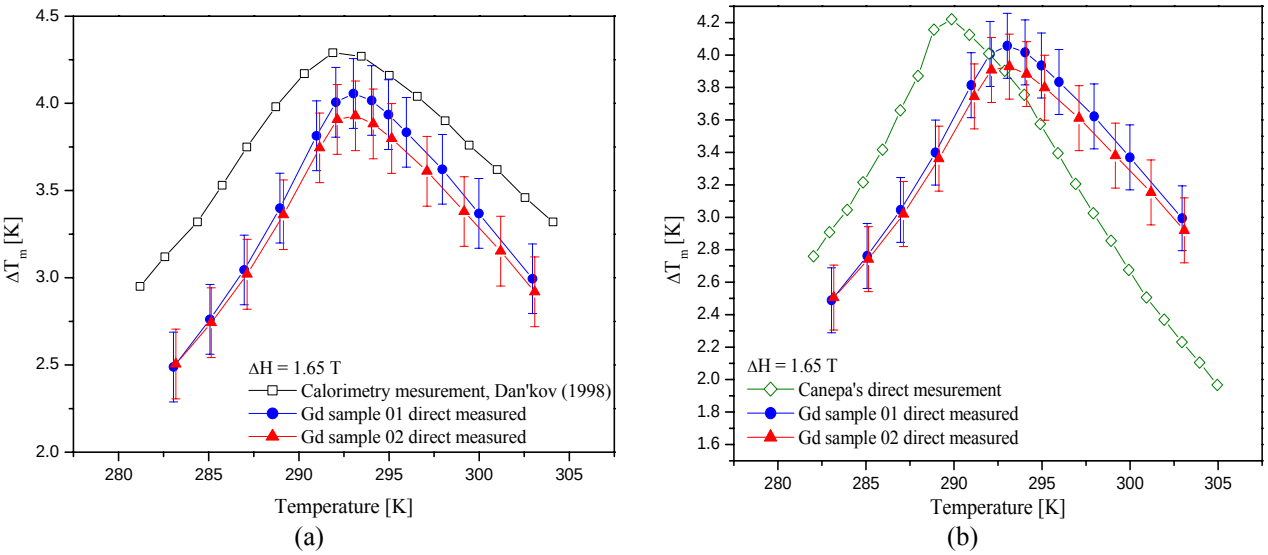


Figure 6. Magnetocaloric temperature change for Gd sample
(a) Comparison with indirect method (Dan'kov et al., 1998); (b) Comparison with direct method (Canepa et al., 2005).

In order to advance alternatives for improving the thermal efficiency of magnetocaloric refrigeration devices, it is necessary to understand the nature of the energy losses taking place in the cycle and to quantify the contribution of each type of loss (e.g., thermodynamic cycle, magnetic material, frictional etc.) to the departure from the ideal cycle efficiency.

A computational model developed by our group (Oliveira *et al.*, 2009a, 2009b) enabled the simulation of the four thermodynamic processes in a Brayton-based reciprocative AMRR cycle using the magnetocaloric temperature change curves obtained experimentally. The computer code simulates the flow of the working fluid (water) in the parallel channels and the coupled heat transfer between the magnetocaloric solid and the working fluid according to a 2D transient hybrid model. The conditions of the simulation are presented in Table 2. In the present analysis, the specific heat of Gd was obtained from Siddkov, *et al*. (2005) and the applied magnetic field was assumed constant and equal to 1.65 T to simplify the simulation. The properties of the water were obtained from Petersen *et al*. (2008).

Table 2. Parameters utilized in the AMRR cycle simulation.

AMRR cycle variable	Value
frequency – f	1 Hz
Mass flow rate – $\dot{m}$	12 $^{kg}/_{h}$
Gd mass	0.228 kg
Magnetic field variation – ΔH	1.65 T
Temperature span – ΔT	10 K
Curie temperature – Tc	293 K
Hot resevoir temperature – T_{Hot}	298 K
Cold resevoir temperature – T_{Cold}	288 K
fluid	water

Figure 7 shows a comparison of the instantaneous cooling capacity calculated with the hybrid model using the magnetocaloric temperature change for the Gd and using the MCE obtained via an indirect approach for polycrystal Gd (Dan'kov *et al.*, 1998). The abscissas represent the elapsed time during the *hot-to-cold blow*, i.e., the portion of the cycle immediately after the removal of the magnetic field (which decreases the Gd temperature). As can be seen, the curves present a similar behavior, i.e., the cooling capacity increases during the first portion of the cycle due to the high water velocity and low Gd temperature, but decrease toward the end of the cycle because of the decreasing water velocity and due to the reduction of the temperature difference between the fluid and the solid (Oliveira *et al.*, 2009b). The cycle average cooling capacity obtained with the computational code is shown in Table 3.

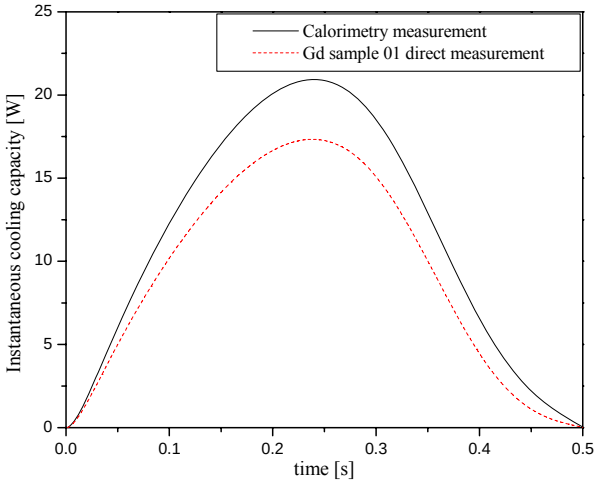


Figure 7. Instantaneous cooling capacity.

The cycle-average cooling capacity (calculated based on the time integral of the curves in Fig. 7) shows a clear decrease. When comparing the average cooling capacity the reduction is close to 20%. Although this is certainly an impressive figure, a more extensive analysis is needed in order to evaluate the effect of the operating conditions on the relative cooling capacity decrease. Additionally, an experimental confirmation is needed to validate the theoretical results.

Table 3. Cycle-average cooling capacity.

Sample	Average cooling capacity
Gd sample	4.71 W
Calorimetry measurement	5.84 W

4. CONCLUSIONS

An experimental procedure for evaluating the MCE at near room temperature via the direct approach was proposed in this paper. The results of our direct measurements using samples made with commercial Gd showed a satisfactory agreement with both indirect and direct approaches encountered in the literature (Dan'kov et al., 1998; Canepa et al., 2005). The associated decrease of the instantaneous and average cooling capacities of a Brayton-based parallel plate AMRR cycle was estimated via an existing mathematical model (Oliveira *et al*., 2009a, 2009b) which, for a typical set of operating conditions, indicated a decrease in the average cooling capacity of around 20%. These results represent a loose on AMRR cycle performance.

5. ACKNOWLEDGEMENTS

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