Experimental on-site measurement of thermal conductance by means of heat flow meter applied to nanocomposite thermal insulating mortar coating

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Abstract. In the energy refurbishment of buildings, the declared thermal conductivity is the key to meet the minimum requirements concerning energy efficiency and tax incentives. Regardless of the assessment method for thermal conductivity, in situ measurements using the heat flow meter technique can be always performed, especially in case of very low and self-declared thermal conductivities. In the present work, the reliability of the heat flow meter approach is validated considering a multilayer wall, with all thermal properties declared by means of technical regulations or CE marking. The measured total conductance of the wall is in very good accordance with the calculated one. Then, the heat flow meter has been applied to a plastered stone masonry wall before and after the application of a nanocomposite, thermal insulating, mortar coating 8 mm thick, with a self-declared thermal conductivity of 0.0019 W/(m K) without CE marking or appropriate laboratory tests. The thermal conductance obtained from the onsite measurements is about five times greater than the expected one. The increase in thermal resistance due to the mortar is comparable to a material with the same thickness, but with the thermal conductivity one order of magnitude greater than the self-declared value.

1 Introduction

Within the EU, buildings account for 40% of final primary energy consumption and 36% of energy-related greenhouse gas emissions [1]. The "green homes" EPBD IV directive [2], just approved by the European Parliament, sets challenging objectives to reduce energy consumption and greenhouse gas emissions of the building stock within 2050. In this context, the availability of high efficiency insulators is of paramount importance to reach better performances with reduced thicknesses. Indeed, thick insulating coatings may have both low compatibility with technical and aesthetic aspects of the facades and the cost effectiveness [3, 4]. The declared thermal conductivity is the key parameter to meet the minimum requirements concerned with both energy efficiency and benefit from tax incentives. The manufacturers of insulating materials can declare the performance of their marketed products by means of two different approaches.

• CE marking based on harmonised standard: it can be either mandatory or voluntary and it is adopted in case of traditional insulators for which standardized and harmonised tests to assess thermal conductivity are available.

• Laboratory tests: they must be carried out by a third-party laboratory accredited to one of the EU member countries. The declared thermal conductivity must be statistically representative to ensure the consistency of the material performance, according to the UNI EN ISO 10456 standard [5]. This approach has also been implemented by the recent Italian standards UNI 10351 [6] and UNI/TR 11936 [7]. Namely the tests have to be performed over a sufficient number of samples (at least three) to consider the obtained result reliable. Laboratory tests are typically used for recent insulators, still lacking a CE marking approach. This issue is due to two main reasons: firstly, these products are protected by trade secret and secondly their recent intensive development has not allowed the definition of a standard assessment approach.

As concerns the carried-out studies about insulating materials based on nanotechnology, [8] provides basic information about some products available on the market (i.e. EPS including graphite powder additive, aerogel, nanoparticle-based vacuum insulation panels, nanoceramic thermal insulation coatings). The thermal properties of most of the above-mentioned materials are assessed according to the previously illustrated approaches (i.e., CE marking or statistically representative tests carried out by accredited laboratories). On the other hand, other materials, such as nanoceramic coatings, present contradictory information either self-declared or obtained by incomplete tests, which are usually non-compliant with the standard approaches. These painton insulation products contain ceramic microspheres with a diameter in the range of 20-120 µm with a cellular wall thickness of 50-200 nm. The microspheres are supposed to be vacuum-hollow balls made of melted glass or ceramic on high gas-pressure and high temperature (1500 °C). After cooling down, the decrease in pressure should leave vacuum inside the microspheres which are then inserted in a rubber/polymer-based matrix.

Literature concerning liquid nano-ceramic thermal insulation coatings gives different and contradictory values of the thermal conductivity with respect to the Manufacturers' declarations. In fact, most of technical sheets of nano-ceramic products self-declare a thermal conductivity of about 0.001-0003 W/(m K), but the few, available scientific papers estimate a far higher range of thermal conductivity, from 0.01 W/(m K) to 0.7 W/(m K), as discussed below. In addition, the recent standard UNI/TR 11936:2024 [7] states that "*at the date of publication of the present standard, no marketed thermal mortar or paint can be associated to conductivities certified by tests in accredited laboratories lower than 0.025 W/(m K) (i.e., conductivity of still air)*".

Bozsaki experimentally investigated the thermal properties of a liquid nano-ceramic coating [9] according to EN 12667 standard [10], both indirectly by spraying 1-2 mm thick liquid nanoceramic layer on three different types of conventional thermal insulation materials, (i.e., expanded polystyrene (EPS), extruded polystyrene (XPS) and fiberwood), and directly on pure nanoceramic solidified samples. The two applied methods led to much greater values of conductivity with no evidence of the claimed thermal effect. Measured thermal conductivity of pure material was about 0.11 W/(m K) for wet samples and 0.069 W/(m K) at the end of the drying stage.

Other studies involved laboratory measurements carried out on a 25 cm perforated brick wall plastered on both sides were carried out under two scenarios: firstly, with an aerogel panel 1 cm thick, then with a nanotechnological mortar 8 mm thick [11]. By inserting the specimens between two thermostated chambers and using the heat flow meter technique according to UNI ISO 9869- 1 [12], it is possible to indirectly trace the thermal conductivities of both coatings. The thermal conductivity of the aerogel was found to be equal to $0.0145 W/(m K)$, in agreement with the data declared for the products on the market. The conductivity of the nanotechnological mortar was

about 0.7 W/(m K), incredibly far from the very low values declared by the producers (i.e., 0.001- 0.0025 W/(m K)).

Laboratory tests (hot box method and holometrix apparatus) [13] on a brick wall coated by a 2 mm thick insulating paint composed by vacuum hollow spheres lead to a thermal conductivity of about 0.02 W/(m K), a value which is still at least one order of magnitude greater than the optimistic values declared by the producers.

Bozsaki, basing on the former studies, assumed that the thermal resistance provided by such nanotechnological coatings was not due to conductance. For this reason, he conducted specific laboratory experiments [14] to verify the effect of surface heat transfer resistances. Tests were carried out according with EN 12667 by inserting the nanoceramic paint layer in air gaps and measuring the equivalent thermal conductivity of inhomogeneous multilayer structures. The paper does not explain how the considered air gaps were coupled to the surface temperature sensors of the HFM when they were located at the end or beginning of the sample. In addition, the nanoceramic paint was always coupled to insulated samples, without enquiring the case of materials with higher conductivity (e.g., bricks, wood). Moreover, the size of the air gap (either 100x100 mm or 200x200 mm) is very likely to be subjected to edge effects, since the whole sample has a size of 300x300 mm. Besides of the illustrated issues, the paper confirms that the extremely low thermal conductivity claimed by the producers is not endorsed by laboratory tests, even enquiring in detail the contribution of surface heat transfer resistance. The paper tries to relate the modest insulation effect of the nanoceramic to the thickness of the air gap and the convective heat transfer coefficient. In fact, a low number of tested samples seems to show a reduction in conductivity up to 10-12% with nanoceramic and a large air gap. However, the phenomenon is not present in all the tests and most of samples lead to variations (positive and negative) of about 5-6%. For this reason, the highlighted fluctuations are not due to the added thermal resistance, but to uncertainties associated to the instrumentation accuracy.

Another relevant study [15] enquires various types of thermal paint coating applied to skimmed 12 mm plasterboard samples, involving both the measurement of the thermophysical properties (i.e., emissivity and thermal resistance in particular), and the use of the electron microscope to see the actual internal structure of the material. A mixture of spherical particles of ceramic insulating additive embedded within a continuous structure of fine, irregular shaped paint matrix material is observed. Scanning Electron Microscopy indicates that the additives are pre-dominantly micro-porous with pore sizes between 0.1 and 100 μm. Microscopy also suggests that the particles of additive are not sufficiently robust to maintain a vacuum and are probably air filled, although they appear to be closed cell. According to the work, the added thermal resistance can be compared to the one provided by a conventional lining paper due to this aspect. The tests on emissivity show that the thermal resistance in nanotechnological coatings cannot be due to low surface emissivity. In fact, the study concludes that they are "*poor inhibitors of infrared radiant heat loss, no better than conventional building surfaces*". Moreover, density and thermal conductivity of the powdered insulating paint additive are too high to provide a significant contribution to the thermal insulation of structures.

From the analysis of the available literature, based mainly on laboratory measurements, a great uncertainty emerges regarding the real insulation properties of nanocomposite mortar coatings. Furthermore, there are no studies aimed at characterizing the real in-situ behaviour of these materials. The aim of this work is to make a contribution through in-situ experimental investigation to verify the effectiveness of this technology. This contribution is particularly important in the current historical phase, where the achievement of energy saving objectives is

one of the most important aspects of European policy. On-site measurements are always allowed and strongly advisable especially when the conductivity reported in the technical sheet of the insulator is just self-declared, without CE marking or it is not based on laboratory tests, compliant with the standards. These self-declarations are often associated to very low conductivities, implying that the minimum energy requirements can be met with very low thicknesses of material.

The on-site measurements can be performed by means of the Heat Flow Meter technique (HFM), as indicated in the standard UNI ISO 9869-1.

The present paper deals with HFM on-site measurements to assess the contribution of nanotechnological mortars to the total thermal conductance of a wall.

2 The heat flow meter approach

2.1 The average method

On site measurements regarding conductance can be performed according to the standard UNI ISO 9869-1 [12], concerned with the heat flow meter technique. This kind of measurements always occurs under transient regime since the steady state conditions can only be granted in controlled environments such as laboratories. For instance, considering a vertical, perimetral wall, the internal temperature can be easily thermostated, while the external one still varies during the day, even in harsh climates. For this reason, the standard adopts the progressive average method: namely, the conductance is obtained by dividing the mean density of heat flux rate by the mean temperature difference between internal and external surfaces. The approach requires a relatively long testing period, to ensure the convergence of the value of conductance (i.e., about 3 days) and to cope with transient effects. The thermal conductance Λ [W/(m²K)] can be then obtained as follows:

$$
\Lambda = \frac{\sum_{j=1}^{M} q_j}{\sum_{j=1}^{M} (T_{si,j} - T_{se,j})}
$$
(1)

Where:

 q_i is the density of heat flux rate referred to the j-th measurement [W/m²];

 $T_{si,j}$, $T_{se,j}$ are respectively the internal and external surface temperatures referred to the j-th measurement [K];

M represents the total number of measurements collected during the test.

Then the thermal transmittance U [W/(m²K)] can be computed with the addition of the liminary resistances as illustrated in (2):

$$
U = (R_{si} + \Lambda^{-1} + R_{se})^{-1}
$$
 (2)

Where:

 R_{si} , R_{se} respectively internal and external liminary resistances [m²K/W].

The illustrated approach is based on the HFM, which consists of two measurement nodes as illustrated in Figure 1:

- internal node: equipped with both the heat flux (q) and two surface temperature (T_{si}) sensors;
- external node: equipped with two surface temperature (T_{se}) sensors.

The redundancy of the temperature sensors is meant to check the uniformity in temperature of the enquired element.

2.2 Assumptions of the HFM method

According to UNI ISO 9869-1, the convergence value of conductance is representative of the real thermal performance of the element if the following assumptions are met:

- the HFM is not exposed to solar radiation;
- the thermal conductance of the enquired element does not vary during the test;
- the heat content of the element is the same at the beginning and the end of the measurement.

Furthermore, the standard adds the following conditions for elements with a specific heat per unit area higher than 20 kJ/(m^2K):

- minimum duration of the test 72 h;
- the conductance obtained at the end of the test does not deviate by more than $\pm 5\%$ from the value obtained 24 h before;
- two values of conductance are calculated: the former considering an interval starting from the beginning of the measurement, with a duration $D = 2/3$ of the total duration of the test. The latter is obtained by an interval with the same duration D, ending at the end of the measurement. These two values must not deviate by more than ±5%.

The condition of "heavy" elements (i.e., thermal capacity greater than 20 kJ/((m^2K)) is met for almost all traditional walls since even a wall made of perforated bricks 8 cm thick plastered on both sides reaches a thermal capacity of about 40 kJ/(m^2K).

Moreover, two last conditions are needed:

- high differences in temperature between external and internal are strongly advised (at about 5-10 K) to grant a relevant heat flow to be correctly measured by the instruments;
- the investigated element has to be free from thermal bridges or other similar perturbations. For this reason, a previous analysis of the elements by means of thermal camera is always advised.

2.3 Accuracy of the HFM method and used instrumentation

According to UNI ISO 9869-1, section 9, the uncertainty on the thermal conductance of the proposed approach is between 14% and 28% if the following assumptions are met:

- the temperatures don't show large fluctuations compared to the temperature difference between both sides of the element;
- the test is long enough (i.e., 72 hours of minimum duration);
- no solar radiation or strong thermal influences affect the wall;
- the HFM has a negligible thermal resistance if compared to the enquired element.

Considering the case studies presented below, all the measurements have been performed adopting the Thermozig Ble heat flow meter – Optivelox with the following data:

- heat flow meter sensor: 0.01 W/m^2 resolution, $\pm 5\%$ accuracy,
- temperature sensor: 0.01 °C resolution, $(0.15+0.001 \text{ |t|})$, $[t]=[^{\circ}C]$ accuracy.

As shown by the plots of the results for each measurement, all the above conditions are met and therefore the measured conductances shall be associated to an uncertainty between 14% and 28%.

3 Validation of the heat flow meter approach

Before considering the effects of nanotechnological insulators, the HFM was applied to a multilayer, perimetral wall with the insulated stratigraphy reported in Table 1. The wall has no windows, pillars or other elements that can be associated to thermal bridges. Moreover, the sensors were installed in the middle of the wall, far from the edges. All thermal properties are a priori known by means of technical regulations or CE marking and therefore the comparison between theoretical and measured conductance provides a field test of both the instrumentation and the approach itself.

Table 1. Stratigraphy and thermal properties of the multilayer wall for validation.

3.1 Theoretical expected conductance

The theoretical expected conductance Λ_{theo} [W/(m²K)] can be calculated as follows:

$$
\Lambda_{\text{theo}} = \left[\sum_{i} (\mathbf{s}_{i} / \lambda_{i}) \right]^{-1}
$$
 (3)

Where:

 λ_i is the thermal conductivity of the i-th layer [W/(m K)];

 s_i is the thickness of the i-th layer [m].

Basing on the information in Table 1, $\Lambda_{\text{theo}} = 0.768 \text{ W/(m}^2\text{K)}$.

3.2 HFM positioning and testing

The HFM has been installed in the middle of a wall in Genoa, North-East facing, after checking the absence of thermal bridges by means of a thermal camera. The external temperature sensors were installed aligned with the internal ones, providing a reflective protection against unwanted solar radiation. Figure 2 shows the installed instrumentation on both external and internal sides. The test lasted 72 hours and it occurred during winter season, to ensure a relevant difference in temperature between internal and external.

Figure 2. HFM installation on the external (on the left) and internal (on the right) sides.

3.3 HFM results

According to Figure 3, the difference in temperature between internal and external surfaces remained greater than 10 °C (about 13 °C) with an almost cyclic oscillation between day and night.

Figure 3. Measured internal (solid, blue line) and external (dashed, orange line) surface temperatures.

Figure 4. Measured heat flux.

Figure 4 shows the heat flux exchanged through the wall during the test.

Then, the conductance has been computed following the approach illustrated in section 2, using Eq. 1. The convergence of conductance over the duration of the test is shown in Figure 5.

Figure 5. Conductance computed with the average method, plotted over the duration of the test.

In particular, the measured value of conductance is $0.734 W/(m^2K)$, in very good accordance with the expected result (i.e., $0.768 W/(m^2K)$), with an uncertainty of about 4%. The result meets all the assumptions reported in subsection 2.2.

The obtained result validates the reliability of the approach and the correct working of the used instrumentation.

4 Measurement of conductance with and without nanotechnological mortar

Accomplished the validation stage, the HFM was applied to a stone wall, plastered on both sides before and after the application of a nanotechnological mortar.

4.1 Choice of the wall

The stone wall is located in Genoa, and it has a total thickness of 70 cm plus 2 cm plaster on each side; it is north-facing and it has been examined by means of thermal camera to check both its uniformity and the absence of thermal bridges. Figure 6 shows the view of the internal and external surfaces of the wall, while Figure 7 reports the thermal images of the areas in Figure 6 showing good thermal uniformity. The wall divides a heated room from the outside.

Figure 6. External (on the left) and internal (on the right) surfaces of the stone wall.

Figure 7. External (on the left) and internal (on the right) view with the thermal camera of the wall in Figure 6.

With reference to Figure 7, the thermal camera is used in a qualitative and not quantitative approach to check the thermal uniformity of the wall. Therefore, the results are not influenced by the choice of emissivity and the surface temperatures reported are not representative of the real surface temperature.

4.2 HFM measure without nanotechnological mortar

Following the same approach adopted for the validation stage, the sensors of the HFM were installed (Figure 8), granting the alignment between internal and external sides, protecting the sensors outside with a reflective shield against unwanted radiation or rain.

Figure 8. HFM installation on the external (on the left) and internal (on the right) sides.

Figures from 9 to 11 resume the main results. Namely the internal and external surface temperature, the heat flux and the conductance.

Figure 9. Measured internal (solid, blue line) and external (dashed, orange line) surface temperatures.

Figure 10. Measured heat flux.

Figure 11. Conductance computed with the average method, plotted over the duration of the test.

Figure 11 shows the convergence of the conductance at about 2.536 $W/(m^2K)$. The result meets all the assumptions reported in subsection 2.2. Assuming standard liminary resistances for horizontal heat flow $(R_{se} = 0.04 \text{ m}^2\text{K/W}, R_{se} = 0.13 \text{ m}^2\text{K/W}$, the transmittance of the wall is $1.77 W/(m^2K)$. Besides of the former validation stage, the obtained result is aligned with the abacus of transmittances provided by the Italian standard UNI/TR 11552 [16] that reports a transmittance of 1.95 W/(m²K) for a stone wall of 70 cm plus 2 cm of plaster on each side (wall code MPl02). Clearly no destructive investigation was performed on the wall, so the difference between the measured value and the one in the abacus (about 9%) is very likely to be due to slight variations in the thicknesses and in the kind of materials actually adopted to build the wall. Anyway, the comparison provides further evidence of the reliability of the HFM method.

4.3 Supply and installation of the nanotechnological mortar

Subsequently, the nanotechnological mortar was installed on the external surface of the wall, according to the Manufacturer's instructions, applying three different layers, each one after the drying of the former. The application of each layer has been performed by a third-party mason who has successfully attended the Manufacturer's course to correctly lay down the mortar. Figure 12 resumes the main stages during the laying of the mortar. As typically occurs for similar products, no specific information about the mortar composition is available and the so called "technical sheet" lacks complete tests, compliant with the standard approaches. In particular, an equivalent conductivity of $0.0019 \, W/(m K)$ is self-declared. In addition the considered mortar has a density of about 700 kg/m³ and a specific heat of 1290 J/(kg K).

The total thickness is of about 8-10 mm as measured (Figure 13).

Figure 12. Laying of the three different layers of the nanotechnological mortar (first layer on the left, last on the right).

Figure 13. Total thickness of the nanotechnological mortar at the end of the laying.

4.4 HFM measure with nanotechnological mortar

Following the same approach previously adopted, the sensors of the HFM were installed (Figure 14), granting the alignment between internal and external sides, protecting the sensors outside with a reflective shield against unwanted radiation or rain.

Figure 14. HFM installation to measure the contribution of the nanotechnological mortar. Particular of the external side.

Figures from 15 to 17 resume the main results. Namely the internal and external surface temperature, the heat flux and the conductance.

Figure 17 shows the convergence of the conductance at about 1.051 W/(m^2K) with an average difference between the two surface temperatures of about 9 °C. The result meets all the assumptions reported in subsection 2.2. Assuming standard liminary resistances for horizontal heat flow $(R_{se} = 0.04 \text{ m}^2 \text{K/W}, R_{se} = 0.13 \text{ m}^2 \text{K/W}$, the transmittance of the wall is 0.891 W/(m²K).

Figure 15. Measured internal (solid, blue line) and external (dashed, orange line) surface temperatures.

Figure 16. Measured heat flux.

Figure 17. Conductance computed with the average method, plotted over the duration of the test.

4.5 Comparison of the results

Table 2 allows a comparison about the measurements of the wall conductance without and with nanotechnological mortar.

Table 2. Stratigraphy and thermal properties of the multilayer wall.

^a Considering standard liminary resistances for horizontal heat flow (R_{se} = 0.04 m²K/W, R_{se} = 0.13 m²K/W).

The expected transmittance and conductance have been computed adding the thermal resistance provided by the mortar, according to the Producer's self-declared technical sheet $(\lambda_{\text{mortar}} = 0.0019 \text{ W/(m K)}, \text{S}_{\text{mortar}} = 0.01 \text{ m})$, to the measured conductance of the bare stone wall.

According to section 9 of UNI ISO 9869-1, the measured conductances have an uncertainty between 14% and 28% (subsection 2.3 for more information), even if the validation stage showed a better accuracy, about 4%.

Two main conclusions can be drawn.

Direct conclusions: the measured value of thermal conductance is about five times greater than the expected one, showing that the self-declared conductivity is not reliable. This means that the only application of the nanotechnological mortar would not be enough to meet the minimum requirements concerned with both energy efficiency and benefit from tax incentives. In other words, the application of the nanotechnological mortar 8-10 mm thick is not an equivalent insulating solution, if compared to traditional ones (e.g., conductivity 0.028-0.030 W/(m K), 10- 12 cm thick). Even accounting for the accuracy of both the instrumentation and the approach, the measured value is dramatically different from the declared one. In fact, the UNI ISO 9869-1 identifies a range between 14% and 28%, while the result is 594% of the expected value.

Indirect conclusions: considering the initial conductance of $2.536 W/(m2K)$, the added thermal resistance compared to the applied thickness of 8-10 mm allows an indirect estimation of the mortar conductivity. In particular, a similar conductance is analytically obtained considering a fictitious material 1 cm thick, with a conductivity of about 0.015-0.02 $W/(m K)$.

5 Conclusions and future developments

The present work enquires the effect of a nanotechnological mortar on the total thermal conductance of a stone wall using the heat flow meter approach reported in UNI ISO 9869-1 for on-site measurements.

A former validation stage has been performed on a multilayer wall with standard materials and insulators, showing very good accuracy (4%).

Then, the HFM has been applied to measure the thermal conductance of a stone wall with and without a nanotechnological mortar. The measured conductance of the wall with the mortar is five times greater than the theoretical, expected value, obtained adding the declared thermal resistance of the mortar to the initial measured conductance of the bare wall. In addition, the increase in thermal resistance is comparable to the one of a fictitious material of the same thickness and with a conductivity between 0.015-0.020 W/(m K).

According to these results, the nanotechnological mortar cannot be used as a substitute of standard insulators. Indeed, the measured conductance leads to a transmittance which is very far from the limits which are compulsory concerned with both energy efficiency and benefit from tax incentives in most of Countries.

As concerns the future developments, the performance of other nanotechnological mortars should be tested, by means of both on-site measurements and laboratory tests, compliant with the standards. Moreover, a theoretical model accounting for the heat transfer in nanocomposite material should be carried out and then tested by means of laboratory measurements.

Another interesting issue is concerned with the durability of the material, regardless of the reliability about the declared conductivity. Indeed, the very thin, insulating mortar is likely to be subjected to damage, exfoliation or impacts since it is exposed to an aggressive environment which might reduce the thermal performance, long before the end of the useful life of the envelope on which the mortar is laid.

Nomenclature

- q Heat flux per unit area W/m²
- R Thermal resistance m2K/W
- s Thickness m T Temperature K
-
- U Thermal transmittance W/(m2K) Λ Thermal conductance $W/(m^2K)$
- λ Thermal conductivity W/(m K)

Subscripts

- se referred to external surface
- si referred to internal surface

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