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Determination of uncertainties for emissivity measurements in the temperature range [200°C - 900°C]

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Abstract

A new test apparatus for the measurement of spectral emissivity of good conductive opaque materials is described. Spectral emissivity is measured in the infrared spectral range [2.5 µm - 14 µm] and in the temperature range [200 °C - 900 °C]. A comprehensive study of uncertainties has been made. Uncertainties of each parameter and influence of each assumption are quantified. The final *combined standard uncertainty* is calculated by combination of all the uncertainties. The first results of a comparison validate the calculated level of uncertainty.

1. Introduction

Knowledge of spectral emissivity of materials is necessary for quantitative interpretation in Infrared thermography. In many cases literature values of emissivity are not reliable enough for precise quantitative interpretations. Therefore, measuring the emissivity in a specialized laboratory is the only way to get reliable values for a specific material. Emissivity measurements are known to be very difficult, and comparisons carried out during the last years showed high level of discrepancies between results of different laboratories. Therefore many laboratories are asking for national reference laboratories. Laboratoire National d'Essais (LNE) in France is working in this way with the Bureau National de Metrologie (B.N.M.).

An apparatus for the measurement of spectral emissivity of opaque materials in the temperature range [200°C, 900°C] is presented. The measurement principle used is a direct method based on the comparison of the spectral radiance of the sample with the spectral radiance of a black body. A comprehensive work has been made for the determination of uncertainties, the main points of this work are described. The first results of an comparison are presented for validation of the final uncertainties.

Determination of uncertainties is made using the method recommended by the Comité International des Poids et des Mesures (CIPM) for calculation and expression of uncertainties.

2. Emissivity measurement technique

2.1. Description of the apparatus

The radiation comparison method is used because, for high temperatures, the relative variation of spectral radiance with temperature decreases when temperature increases. Therefore uncertainties on temperature measurements involve lower uncertainties on emissivity for high temperatures. *Figure 1* shows a schematic view of the apparatus. The specimen and the reference black body are located in a vacuum chamber which allows to work in an inert atmosphere. The walls of the chamber are coated with a black paint and cooled at a stable temperature. The reference black body is a stainless-steel cylindrical cavity heated in an electrical furnace. The specimen (diameter 30mm, thickness 10mm) is heated with an electrical resistor. A black-body at ambient temperature (*cold black body*) is used for the correction of incident radiance on the specimen. A rotating system carrying an elliptical and a plane mirror collects successively radiation from the specimen, from the reference black body and from the cold black body. The radiation is chopped before passing through the window and filtered by a band-pass interference filter. The radiation is then focused on a M.C.T. detector and the signal is measured using a lock-in amplifier. Temperatures of the reference black body and the specimen are measured using type S (Pt/PtRd) thermocouples. Surface temperature of the specimen is determined using the *gradient method*. Two thermocouples are

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buried at known depths in the specimen. The gradient in the specimen is assumed to be constant and the surface temperature is linearly extrapolated from the two measured temperatures, the temperature on the surface is assumed to be uniform.

2.2. Measurement principle

Radiative signals are measured successively on the reference black body, on the cold black body and on the specimen. The temperatures are measured simultaneously. The spectral emissivity is then calculated using the following model :

$$\varepsilon_e(\lambda,T_s)=\frac{V_s - V_o}{V_r - V_o}.\varepsilon_r.\frac{\int_{\lambda_{\inf}}^{\lambda_{sup}} S(\lambda).\tau_{fil}(\lambda).\tau_{win}(\lambda).\tau_{atm}(\lambda).\tau_{mir}(\lambda).\left[\left(\overset{\circ}{L}_\lambda(T_r)-\overset{\circ}{L}_\lambda(T_o)\right)\right].d\lambda}{\int_{\lambda_{\inf}}^{\lambda_{sup}} S(\lambda).\tau_{fil}(\lambda).\tau_{win}(\lambda).\tau_{atm}(\lambda).\tau_{mir}(\lambda).\left[\left(\overset{\circ}{L}_\lambda(T_s)-\overset{\circ}{L}_\lambda(T_o)\right)\right].d\lambda} \qquad (1)$$

$\lambda_{\inf}, \lambda_{sup}$	spectral range of the filter	λ	central wave-length of the filter
V_s	specimen signal	T_s	specimen surface temperature
V_r	reference black body signal	T_r	reference black body temperature
V_o	cold black body signal	T_o	cold black body temperature
ε_r	reference black body emissivity	$\tau_{fil}(\lambda)$	filter spectral transmittance
$\overset{\circ}{L}_\lambda(T_s)$	specimen spectral radiance	$\tau_{mir}(\lambda)$	mirrors spectral reflectance
$\overset{\circ}{L}_\lambda(T_r)$	reference black body spectral radiance	$\tau_{win}(\lambda)$	window spectral transmittance
$\overset{\circ}{L}_\lambda(T_o)$	cold black body spectral radiance	$\tau_{atm}(\lambda)$	atmospheric transmittance
$S(\lambda)$	detector relative spectral sensitivity		

Interference filters have a rather broad bandwidth (about 0.6µm), therefore the variations of spectral parameters (spectral sensitivity, spectral transmittances) are considered. Otherwise errors can occur if the surface temperature of the specimen and the temperature of the reference black body are significantly different.

The main assumptions made in setting up the model are :

- the response of the spectro radiometer is linear,
- the temperature gradient in the thickness of the specimen is constant,
- the surface temperature of the specimen is uniform,
- the vacuum chamber is a black body at ambient temperature,
- the radiance of the cold black body is equal to the radiance in the chamber.

3. Determination of uncertainties

3.1. Method for the determination of uncertainties

The method used by L.N.E. is the one recommended by the CIPM in the Guide to the Expression of Uncertainty in Measurement [1]. This guide establishes general rules for evaluating and expressing uncertainty in measurement.

If a measurand Y is determined from N other quantities $x_1, x_2, \ldots, x_N$, assumed to be independent, through a functional relationship *f* : $Y = f(x_1,x_2,\ldots,x_N)$, the *combined standard uncertainty U(Y)* is given by :

$$U^2(Y)=\sum_{i=1}^{n}\left(\frac{\partial f}{\partial x_i}\right)^2.U^2(x_i) \qquad (2)$$

where $U(x_i)$ is the *standard uncertainty* evaluated for each input parameter.

$U(x_i)$ is a standard uncertainty evaluated either from series of repeated observations (standard deviation) or estimated by scientific analysis based on all information available (manufacturer's specifications, calibration certificates, experience, model, ..., etc).

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3.2. Practical determination of uncertainty for emissivity measurements

Uncertainties of all the parameters and the influence of the assumptions have been quantified. The main sources of uncertainties and their contribution to the final uncertainty are given below.

3.2.1. Temperatures

The thermocouples and the temperature measuring chain are calibrated. The uncertainty of the black body temperature is estimated twice as big as the uncertainty of calibration of the thermocouple. The uncertainty on the specimen surface temperature is calculated using the linear extrapolation law. Uncertainty on surface temperature against conductivity of the specimen is plotted in *figure 2*.

3.2.2. Radiative signals

Noises on signals are very small, thus the main source of uncertainty to be considered is the non-linearity of the spectroradiometer which gives a global uncertainty on the ratio of signals. The linearity of the spectroradiometer has been experimentally controlled using the *flux addition method* [2] . The spectroradiometer can be considered linear with a relative uncertainty lower than ± 0.4% for signals increasing in the ratio of 1 to 30.

3.2.3. Emissivity of the reference black body

The temperature distribution along the cavity has been measured and the emissivity of the black body is calculated using the spectral radiosity method. The spectral emissivity of the reference black body can be assumed to be equal to unity with an uncertainty lower than ± 0.002 .

3.2.4. Spectral sensitivity of the detector and spectral transmittance of optics

The spectral sensitivity of the detector versus wave-length is given graphically by the manufacturer and spectral transmittances of the optics have been measured using a FTIR spectrometer. The uncertainties of these parameters are unknown. Variations have been numerically simulated and the influence of these parameters on the final emissivity has been shown to be negligible.

3.2.5. Atmospheric spectral transmittance

The atmospheric spectral transmittance is calculated from data given in [3]. These data are given for distances much larger than the actual distance in the apparatus (0.5 m outside the chamber), therefore the atmospheric transmittance has been extrapolated assuming an extinction coefficient of atmosphere constant regardless of distance. The stability of the atmospheric transmittance during a comparison is the main point to be considered. A variation of the atmospheric spectral transmittance, due to a variation of temperature and humidity, has been simulated. For temperatures and relative humidity varying respectively in the range 23°C ± 1°C and 50 % ± 5% and CO_2 concentrations varying in the range 0 ppm to 1500 ppm, the relative errors on emissivity can be ± 1% for wave-lengths in the absorption bands of atmosphere (6.4µm).

3.2.6. Reflections in the chamber

The vacuum chamber is assumed to be a black body at ambient temperature but in fact, the emissivity of the coating (black paint) is not equal to unity. Therefore a part of the radiation produced by the sample and the heating system is reflected by the chamber, increasing the radiance of the specimen and consequently the measured emissivity. *Table 1* gives the relative error on emissivity, considering an emissivity of the black coating equal to 0.85 .

3.3. Final uncertainty

The final uncertainty is calculated using model (2), by combination of the uncertainties of parameters used for the calculation and the uncertainties on assumptions. The maximum final uncertainties for a thermal conductivity of specimen above 20 W/m.K are tabulated in *table 2*. It can be noticed that the relative uncertainty is higher for low temperatures than for medium temperatures. That arises from the fact that the temperatures uncertainties are almost

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independent of temperature in the range [200 °C - 600 °C] and that an error on temperatures have more influence for a low temperature than for a high temperature.

3.4. Validation of the uncertainty estimated level

An comparison between seven French laboratories is in progress. The comparison is made on a bulky platinum sample with a sandblasted surface. All the participants use the same two calibrated thermocouples for the measurement of surface temperature thus the error on temperature is the same for every laboratory. *Figure 3* shows some results of measurements on platinum for the temperature 350°C. For the long wavelengths a drift of spectral emissivity can be noticed. That arises probably from a temporal variation of sample surface characteristics (maybe an oxidation), but for short wavelengths the first results are consistent.

4. Conclusions

A comprehensive calculation of uncertainties for a new apparatus using the comparison method has been presented. The main sources of uncertainty are the temperature measurements, especially the measurement of the surface temperature of the material. The method used for surface temperature measurements (measurement of two temperatures inside the material and extrapolation of the surface temperature) is suitable only for materials with a sufficiently high thermal conductivity.

The uncertainties on the spectral emissivity are evaluated to be better than ± 7 % for materials with a thermal conductivity above 20 W/m.K .

REFERENCES

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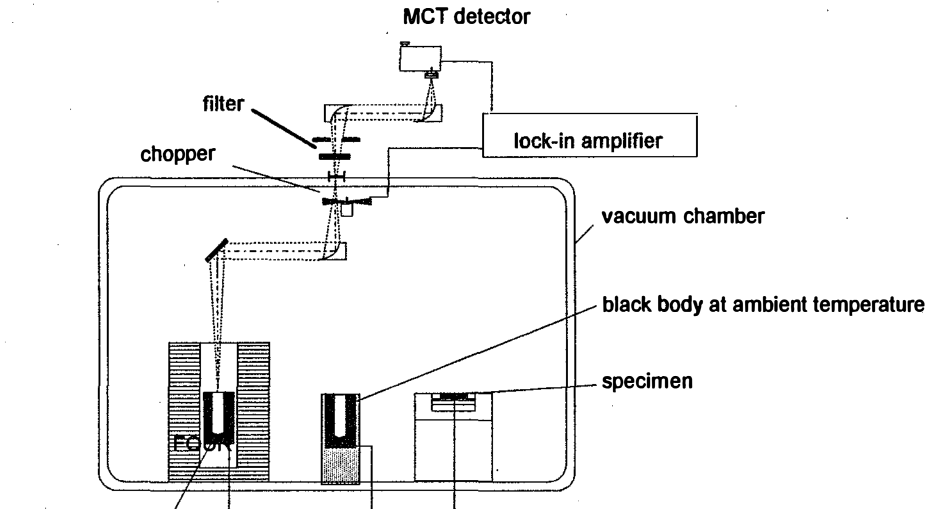

Fig. 1. - *Schematic view of the apparatus*

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emissivity of the specimen	Relative error (%)
0.05	0.5
0.1	0.3
0.2	0.2
0.3	0.1
0.4	0.05
0.5	0.04
0.6	0.03
0.7	0.02
0.8	0.01
0.9	0.005
0.95	0.005

Table1 : *Relative error on emissivity due to reflections in the chamber.*

Wavelength (µm)	Uncertainty on emissivity (± %)		
	Temperature range		
	200°C to 450°C	450°C to 600°C	600°C to 900 °C
2.5	7	4	5
3	6	3.5	4.5
4	4.5	3	4
5	3.5	2.5	3.5
6	3	2	3
7	3	2	2.5
8	2.5	1.6	2.5
9	2.5	1.6	2
10	2.5	1.5	2
11	2.5	1.5	2
12	2.5	1.5	2
13	2.5	1.5	2
14	2.5	1.5	2

Table2 : *Maximum relative uncertainty on emissivity for thermal conductivity >20 W/m.K*

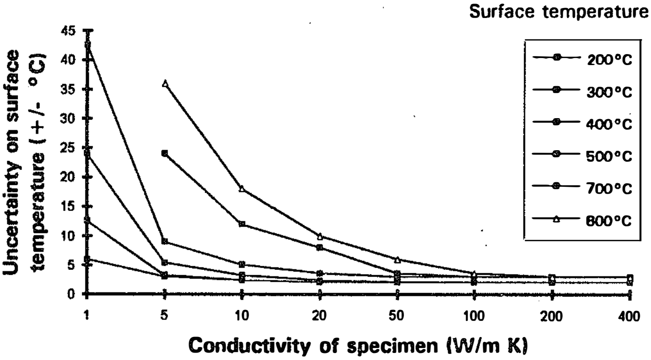

Fig 2 : *Uncertainty on surface temperature of the specimen*

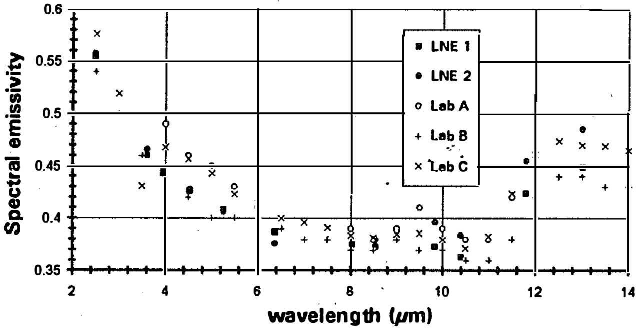

Fig 3 : *Comparison on roughened platinum at 350°C*