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Emissivity measurements at room temperature on polymeric and inorganic samples

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Abstract

An evaluation of the emissivity of polymeric and inorganic materials has been carried out in the spectral range 8-12μm using a direct technique. For polymeric samples the study focused on the dependence on the doping level. In the case of the inorganic samples measurements were aimed at the evaluation of the emissivity variations with the temperature Furthermore the effects on the emissivity of temperature differences between the sample and the background on the emissivity evaluation have been studied.

1. Introduction

Emissivity plays a key role in radiative heat transfer phenomena [1,2]; methodologies devoted to the evaluation of this parameter find applications in many industrial fields such as aerospace, optoelectronics, power generation, civil engineering, etc. Furthermore a knowledge of emissivity is fundamental for accurate temperature measurement using infrared detectors.

Direct and indirect measurement methods can be used. In the former case the emissivity is obtained by measuring the temperature of the sample, of a reference emitter applied to the sample and of the background. The latter is based on the measurement of the infrared reflectance from the sample surface; in this case the emissivity is computed as the complement to the reflectance.

2. Experimental

2.1. Experimental set-up

A sketch of the experimental set-up is shown in *figure 1*. The climatic chamber was designed and constructed to obtain a controlled, uniform temperature on the internal walls. It consists of a cubic box whose inner walls are covered by an aluminium layer painted black. The external ones were made of a suitable thermal insulator. Inside the chamber, a cooling coil reduced the background temperature and made it more uniform. The temperature distribution was monitored on-line by six Pt100 resistance thermometers placed on the inner surfaces of the chamber. A wide 10x10cm^2) thermoelectric cooling-heating system guaranteed the temperature control of the samples (typically thin pellets of 12mm diameter). An infrared mirror was placed close to the sample to measure the background temperature. In order to evaluate the sample temperature, half the surface was covered with 0.95 emissivity paint. An Inframetrics 600 infrared camera (8-12μm) was used to evaluate the apparent temperature of both the sample and the mirror. The emissivity values were then computed using the following expression [3,4]:

$$\varepsilon_S = \frac{(T_S - T_{BB})}{(T_{BK} - T_{BB})} * \varepsilon_{BB} \qquad (1)$$

where the subscripts S, BB, BK refer to the sample, its black painted portion, and the background, respectively.

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2.2. Materials

Polymeric and inorganic samples have been studied. In the first case pressed pellets of polyaniline, polytiophene, and polypirrole powder with different doping levels were considered. A significant variation of the emissivity as a function of the doping level was expected.

2.2.1. Polyaniline

Commercial, highly doped polyaniline powder was obtained from Allied Signal Inc. No information was available from the producer regarding the doping level or the type of dopant. Partial and complete undoping of the starting material was performed chemically using NH_3.

2.2.2. Polypirrole

Doped polypirrole (30%mol.) was produced according to the methodology reported in [5]. Partial undoping was accomplished using NH_3. No attempt was made to produce undoped material given the instability of polypirrole to spontaneous oxygen doping in air.

2.2.3. Polythiophene

Doped polythiophene powder (30% mol.) was produced in accordance with the method described in [6]. Partial and complete undoping was achieved using NH_3.

2.2.4. $BaTiO_3$

In the case of inorganic materials Sr doped $BaTiO_3$ pressed pellets were analyzed. A dependence of the emissivity on temperature was forecast due to the characteristic metal-insulator transition (MIT) for this material.

3. Results and Discussion

The emissivity values obtained for the polymeric samples at 20°C, 25°C, and 30°C are reported In *tables 1-3* respectively. *Table 4* lists the results for the $BaTiO_3$ sample (from 5°C to 90°C). As far as Sr-doped $BaTiO_3$ is concerned, a simultaneous evaluation on a rough surface aluminium sample was carried out to discriminate an actual temperature dependance of the emissivity from spurious effects.

Conjugate polymer samples show a lower emissivity at higher doping levels. This is related to the increase in electrical conductivity and consequently higher IR reflectivity due to free carrier absorpion.

The fact that no emissivity variation was observed in $BaTiO_3$ is due to the very low influence on reflectivity of the metal-insulator transition. In fact the MIT is associated with a variation of carrier mobility rather than an increase in carrier concentration; this, in turn, has a much smaller effect on the IR reflectivity.

The precision of the temperature value, the experimental configuration and statistical errors are the main causes of uncertainties in the emissivity measurements [7]. As far as the temperature values are concerned, their accuracy depends on the infrared camera features (characterized mainly by the Noise Equivalent Temperature Difference, the system bit resolution, the temperature range used and the non-linear response of the detector) which are well known. Thus, from their knowledge it is possible to estimate the magnitude of the uncertainties related to the temperature determination. As far as the present measurements are concerned, taking into account both statistical effects and the IR system features, the overall level of uncertainty works out (conservatively) to be about ± 0.013. On the other hand, uncertainties related to the experimental configuration (which minimised intelligent design and operation) cannot be quantified without a specific theoretical study, which is described in the following.

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Table 1

Sample	Sample temperature [°C]	Emissivity	climatic chamber average temperature
completely undoped polytiophene	20°C 25°C 30°C	0.932 0.939 0.944	16.5°C
partially doped polytiophene	20°C 25°C 30°C	0.906 0.917 0.921	16.5°C
doped polytiophene	20°C 25°C 30°C	0.760 0.770 0.761	16.5°C

Table 2

Sample	Sample temperature	Emissivity	climatic chamber average temperature
partially doped polypirrole	20°C 25°C 30°C	0.847 0.851 0.857	13.5°C
doped polypirrole	20°C 25°C 30°C	0.817 0.825 0.824	13.5°C

Table 3

Sample	Sample temperature	Emissivity	climatic chamber average temperature
completely undoped polyaniline	20°C 25°C 30°C	0.913 0.915 0.916	9.5°C
partially doped polyaniline	20°C 25°C 30°C	0.888 0.891 0.889	9.5°C
doped polyaniline	20°C 25°C 30°C	0.725 0.741 0.733	9.5°C

Table 4

Sample temperature [°C]	Emissivity $BaTiO_3$	Emissivity rough Al
5	0.937	0.143
10	0.938	0.140
15	0.932	0.124
20	0.950	0.792
25	0.939	0.180
30	0.942	0.178
35	0.936	0.198
40	0.938	0.168
45	0.932	0.164
50	0.932	0.163
55	0.929	0.159
65	0.929	0.163
75	0.927	0.159
85	0.925	0.163
90	0.926	0.163
75	0.928	0.162
65	0.924	0.160
55	0.931	0.160
45	0.935	0.160
35	0.934	0.161
30	0.933	0.166
25	0.944	0.166
15	0.934	0.138
10	0.936	0.144
5	0.940	0.147

In order to estimate the effects of the experimental set-up on the emissivity values, emissivity measurements on a layer of polyalchitiophene and on a small aluminium plate with rough surface have been carried out in the range 5°C-70°C. Polyalchitiophene was chosen for this experiment because as a polymeric material, it is able to tolerate temperature of the order of 70°C and since its emissivity is expected to be relatively insensitive to temperature variations. Small cyclic fluctuations of the emissivity as a function of the sample temperature have been observed, as is evident in *figure 2*. In order to explain this the relative position of mirror and sample has been taken into account.

As a matter of fact the sample and the mirror are in two different positions so that they "see" (notwithstanding the use of a specific reciprocal alignment procedure) two different parts of the opposite wall side. In this way the background contributions on the surface of sample and mirror are different. This difference affects the precision of the emissivity measurement. An estimation of this effect can be made as follows: We have:

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$$T_S = \varepsilon^{true}{}_S T_S + (1 - \varepsilon^{true}{}_S) T_{BI} \quad (2)$$

$$T_{BB} = \varepsilon_{BB} T_S + (1 - \varepsilon_{BB}) T_{BI} \quad (2')$$

$$T_{BK} = \varepsilon_{MI} T_{MI} + (1 - \varepsilon_{MI}) T_{B2} \quad (2'')$$

where the subscript MI refers to mirror and the superscript "true" refers to the actual sample emissivity.

Substituting these expressions into equation (1), one obtains the following relation between the sample experimental emissivity value and the above-defined temperatures and emissivities:

$$\varepsilon^{meas}{}_S = \frac{\varepsilon^{true}{}_S + \dfrac{x}{\Delta T} + \varepsilon_{MI} \dfrac{T_{B2} - T_{MI}}{\Delta T}}{1 + \dfrac{x}{\varepsilon_{BB} \Delta T} + \dfrac{\varepsilon_{MI}}{\varepsilon_{BB}} \dfrac{T_{B2} - T_{MI}}{\Delta T}} \quad (3)$$

where $x = T_{B1} - T_{B2}$ and $\Delta T = T_S - T_{B1}$. Assuming $\varepsilon_{MI} = 0$, this reduces to:

$$\varepsilon^{meas}{}_S = \frac{\varepsilon^{true}{}_S + \dfrac{x}{\Delta T}}{1 + \dfrac{x}{\varepsilon_{BB} \Delta T}} \quad (4)$$

Eq. (4) shows that the discrepancy between measured and true sample emissivity increases in function of x and decreases in function of ΔT. The reason for this is that better results are generally obtained using the direct technique with a high temperature difference between sample and background and a very uniform background surrounding the sample and the mirror. Moreover the influence of these factors is stronger if the emissivity of the sample is low.

4. Conclusion

Partially satisfactory results are obtained on the polymeric samples where a dependence of the emissivity on doping level has been found. No significant variation of emissivity with temperature has been observed for the inorganic samples.

Concerning the error sources, very good agreement between a proposed theoretical model (which takes into account measurement conditions in estimating the uncertainty in measuring emissivity) and experimental data is obtained.

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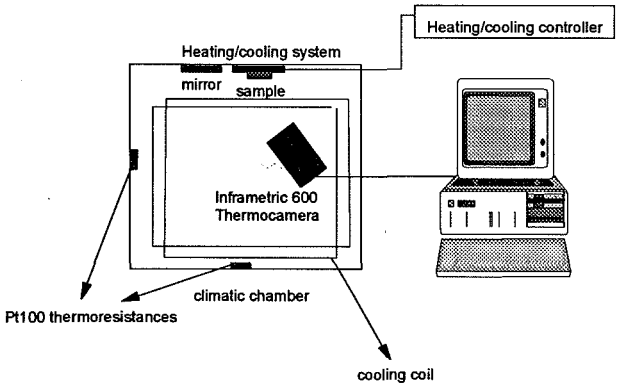

Fig.1 Experimental set-up

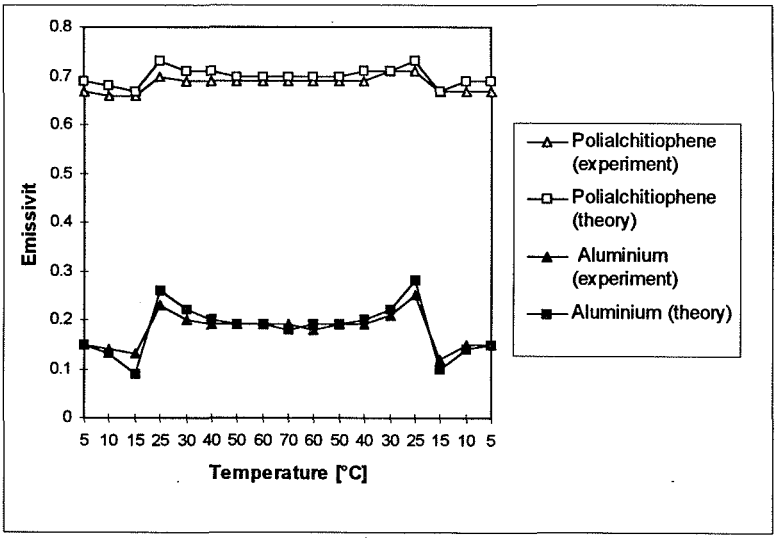

Fig. 2 Comparison between experimental data (for a polialchitiophene sample and an aluminium plate with rough surface) and the predicted effects of ΔT variations assuming an reasonable x value of 1°C taking as true emissivity the value measured at the highest temperature.