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Emissivity measurements on graphite and composite materials in the visible and infrared spectral range

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Abstract

Quantitative thermography, especially thermometry needs knowledge of emissivity values of the observed surface and careful calibration of the camera. Emissivity measurements have to be carried out either by means of the thermography system itself or by determination of the spectral emissivity and calculation of the relating emissivity by integration. A measurement device will be described, which enables both, whereby the radiation comparison technique is used. The most important details of the equipment will be described with special emphasis of the calibration procedure applying heat pipe technology in the temperature range up to 1 100°C and a graphite furnace at higher temperatures. Results of measurements on different types of graphite, carbon/carbon, and carbon/silicon carbide-composites in the temperature range 800 °C to 1 600°C will be presented.

1. Introduction

At most of the applications of thermography only relative temperature variations or temperature fields are of interest. But in principle each variation of measured intensity can be interpreted in terms of a temperature variation or of an emissivity change if we measure at objects showing some kind of reaction of the surface with the surrounding. Typical examples of such emissivity variations are oxidation of metals or erosion of materials during the reentry phase of space vehicles into the atmosphere. At all applications with true temperature evaluation of the camera signals the emissivity of the surface has to be known. Numerous methods have been proposed in the literature to measure the emissivity and temperature in situ, however, only a few of them could be successfully applied in practice of pyrometric temperature measurement at very special boundary conditions. A generally usable method of temperature measurement which is independent on the emissivity is not yet available. This is also valid for all proposed multiwavelength methods including ratio pyrometry as it could be demonstrated by means of a critical analysis [1].

Therefore knowledge of the emissivity, respectively its measurement, is the first proposition for correct radiation temperature determinations. The second important demand is a careful calibration of the used radiation detector or infrared camera system, respectively. Pyrometers usually are calibrated by the manufacturer and recalibrations are only needed to check deviations possibly caused during operation in the course of a longer period. Infrared cameras, however, are not calibrated in terms of temperature and only less experiences are available on the stability and reproducibility of a calibration.

2. Emissivity measurement technique

The radiation comparison method is well suited to measure emissivities needed for radiation temperature measurements. The radiance of the sample surface is measured by means of a calibrated detector and the surface temperature has to be determined independently. Depending on the accuracy demands the temperature can be measured very roughly, e.g. by means of a contact thermometer or by coating a small section of the surface with a blackpaint (e.g. Velvet Coating of Minnesota Mining Company or Pyromark 2500 of Helling Comp., Hamburg). The

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emissivity of such coatings is fairly well known and close to 1.0. Assuming same temperature of the coated and uncoated section, the emissivity can be determined by comparison of the corresponding signals. Higher radiation losses from the coated section may result in a temperature drop and the determined emissivity, will be too high. Other main sources of inaccuracies are reflection and radiation of surrounding areas and the characteristic of the measured detector signal related to the radiance. A more precise method is by measuring the temperature in a small hole close to the surface as shown in *figure 1*. The Linearpyrometer LP2 specially developed for accurate temperature measurements [2] enables us to realize very small target areas of up to 0.3 mm diameter. The temperature difference between the black body hole and the sample surface can be calculated by means of the heat conduction equation as presented in [3]. The accuracy of the resulting temperature difference depends on the thermal conductivity of the material which has to be known or measured [4].

For detectors with linear operation the emittance ε can be calculated as the ratio of the detector signals U_s measured at the sample to signal U_{BB} measured at the black body of the same temperature:

$$\varepsilon = U_s(T_s) / U_{BB}(T_s) \quad (1)$$

The radiation from the sample consists of the direct radiation, and a reflected radiation contribution. The contribution by reflection can be determined either by calculation or by measurement. For precise measurements it is important to have a surrounding wall with well defined temperature and emissivity as shown in *figure 2*.

In the case of radiation detectors with non linear characteristics, the emissivity determination is much more complicated. For absolute emissivity measurements the calibration procedure has to be extended to the determination of calibration curves with different simulated emissivities as demonstrated in [5]. Otherwise the built in emissivity correction has to adjusted in that way that the resulting temperature of sample radiation in equal to the black body radiation. *Figure 2* shows the schematic of the measurement device which is described in detail in [3].

- The sample is 15 mm in diameter and 3 to 6 mm thick.
- The sample is heated using an electron gun the focussed beam of which can be moved with a focus-time function resulting in an isothermal temperature distribution at the sample surface.
- The radial hole for pyrometrical temperatures measurements is 1.2 mm in diameter and 7 mm deep.
- Due to the electron beam heating method measurements only in vacuum ($<0.7 \cdot 10^{-4}$ mbar) are possible.

A Linearpyrometer LP2 [2] is used for measurements in the lower wavelength range and a thermal detector is used at wavelengths above 1.3 μm. The measurement of the total normal emittance is possible by keeping one place free in the filterwheel of the total radiation detector. The nominal wavelengths λ of the filters and the halfband widths (HBW) of the filters are:

λ(*nm*)	500	600	650	750	800	950	1 300	1 600	2 400	3 500	4 300	6 300
HBW/*nm*	10	10	10	10	10	20	180	210	400	517	680	467

The radiation detector can be installed either in the direction of the normal to the sample surface or at different angles of inclination in order to measure the angular dependent emissivity.

The imprecision of the emissivity measurements has been estimated to be ±3 % for the total emissivity and the spectral emissivities in the infrared range. At wavelength below 1 000 nm a higher imprecision in the range 5 to 10 % should be assumed. Low thermal conductivity and imperfect reproducibility of the material leads to higher inaccuracies.

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3. Calibration procedure

The calibration equipment consists of a set of tungsten ribbon lamp, standard pyrometer and black body radiator. The tungsten ribbon lamp is used as temperature standard and has to be recalibrated regularly by the Physikalisch Technische Bundesanstalt (PTB). Repeating calibrations of the lamp have shown that the stability was ±1 K during a period of more than 10 years. The Linearpyrometer LP2 has to be calibrated at only one temperature T_{Fix} at the tungsten ribbon lamp (usually 1 800 K) and due to its linearity the interpolation to higher and lower temperatures is possible by means of the Planck's equation. This linearity can only be realized by a very careful design of the pyrometer. Thereby special attention has to be directed to the blocking of the interference filter sidebands [6]. By means of this Pyrometer we measure the temperature of a variable temperature blackbody with an inaccuracy of ±1.5 K at 1 000°C and ±4 K at 2 000°C. The both following types of black bodies developed by IKE [7] are available.

a) A heat pipe black body with a cylindrical cavity of 20 mm diameter and 200 mm depth up to a maximum temperature of 1 100°C. The main advantage of this black body is the excellent temperature uniformity within the heat pipe cavity the deviation of which is below ±1 K. This temperature homogeneity can even be realized with very large diameters of the cavity or of a flat plate reference surface how it is necessary for the calibration of thermal imaging cameras especially of those using detectors arrays.
b) At higher temperatures we use a graphite cavity radiator with 15 mm diameter and 90 mm depth. This blackbody can be operated up to 3 000°C at maximum. By careful adjustment of all instruments and observation of cleanliness of all components in the optical path the imprecision of the calibration related to the radiance or the corresponding output signals U in equation (1) is better than ±1 %.

4. Measurement results

4.1. Material description

Three different materials were used to measure the spectral and total normal emissivity:

EK986: pure graphite, density = 1.75 g/cm^3, porosity = 13 %, grain size = 25 μm; the surface was ground with a roughness of a mean peak to valley depth = 25 μm;
CF322: carbon fiber/carbon matrix composite, consisting of high tensile carbon fiber bundles with 3 000 filaments of 6 μm diameter; the fiber volume content is 60 to 65%; the material was densified on the phenolic route, the final heat treatment temperature was 2 600°C; sample surface parallel to the fabric plane;
C-SiC: carbon fiber/silicon carbide matrix composite, consisting of high tensile carbon fiber bundles with 3 000 filaments of 7 μm diameter; the fiber volume content in 50%. The material was prepared by liquid impregnation of porous C/C by melted silicon (T = 1 500°C); sample surface parallel to the fabric plane.

4.2. Emissivity results

The spectral emissivity was measured at temperatures between 1 100 K and 2 000 K. No systematic dependency on the temperature could be observed. The variation of the spectral emissivity with temperature was within the inaccuracy limit of the measurements. The influence of material, however, is much more pronounced, as it can be seen in *figure 3*. Especially noticeable is the rate of decrease of the emissivity at longer wavelengths which is much higher at the composite materials compared with the pure graphite sample EK98. This is probably caused by the fiber surface which is extremely smooth and therefore has a low emissivity. The surface of fiber bundles is composed by fiber surfaces and small groves between the filaments.

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The main fiber fabric plain was parallel to the sample surface and therefore the predominating content of the radiating area is that of the fiber bundles. The surface topography is formed both by the grooves between the fibers and the roughness of the cloth structure. The grooves between the fibers are very small and therefore mainly influence the emissivity at short wavelengths in the direction to higher values.

The difference between the values of the emissivity of CF322 and C-SiC can be explained by the fact that different carbon fibers were used and the cloth structure was not the same for both materials. This distinction is much more affecting different emissivities than the matrix material, which is carbon and SiC, respectively.

The total normal emissivity increases with increasing temperature in contrary to the spectral emissivity which is nearly independent on temperature *figure 4*. Corresponding to the Wien's displacement law the radiation intensity increases with decreasing wavelength and consequently the total (integrated) emissivity increases with the temperature if the spectral emissivity increases with decreasing wavelength. The values of the CF322 sample are lower because of the difference in the carbon fibers and cloth structure as mentioned before in paragraph 4.2.

5. Conclusion

The measurement results clearly demonstrate that material and structure of composites influence the variation of the spectral emissivity with wavelength and consequently the variation of the total emissivity with temperature. This has to be taken into consideration at pyrometric temperature measurements especially if ratio pyrometry shall be applied or if a pyrometer with broad spectral band or an infrared detector is used. The emissivity ratio at two given wavelengths differs for different materials and at measurements by means of broad band pyrometers the temperature dependence of the emissivity depends on the spectral range of the pyrometer.

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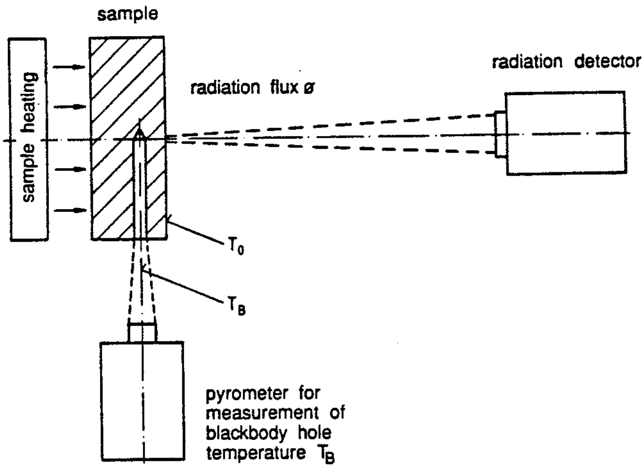

Fig. 1. - Principle of the radiation comparison method

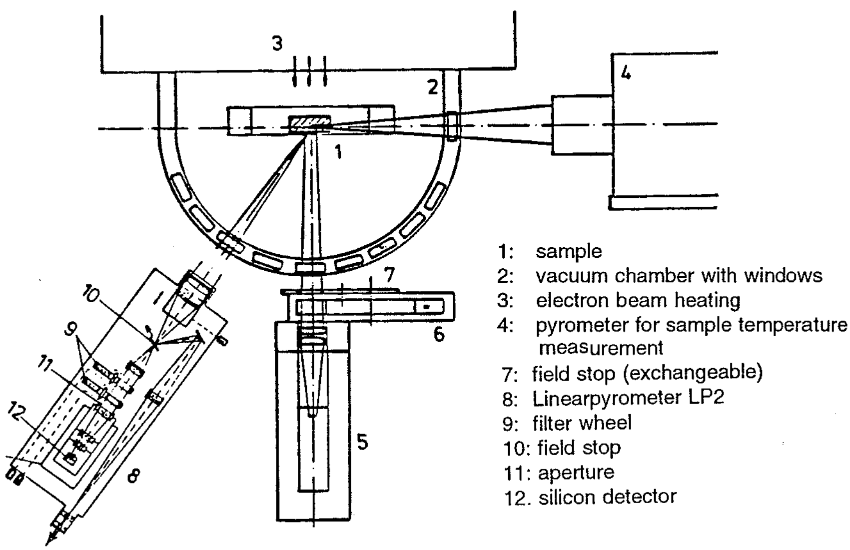

Fig. 2. - Schematic of the device for emissivity measurements

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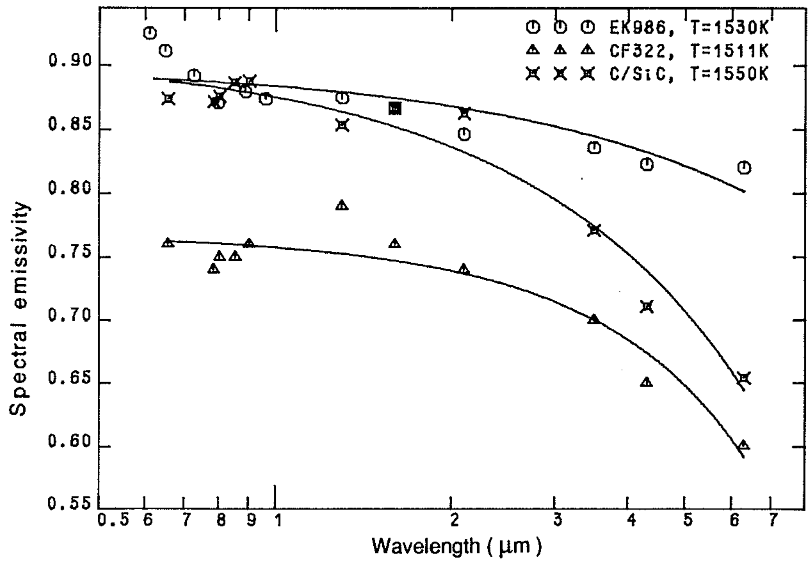

Fig. 3. - Spectral normal emissivity of graphite (EK986) carbon/carbon (CF322) and carbon/siliconcarbide (C-SIC) composites

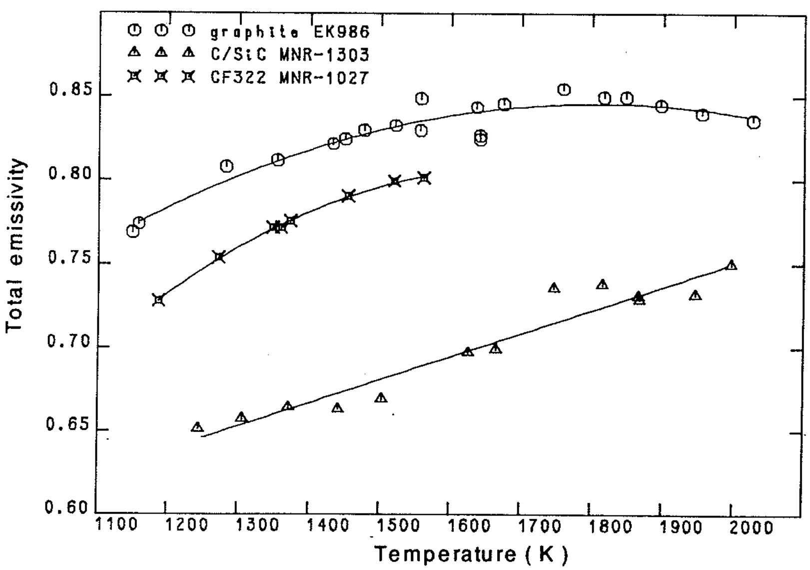

Fig. 4. - Total normal emissivity of graphite (EK986), carbon/carbon (CF322) and carbon/siliconcarbide (C-SiC) composites