Measurement of directional spectral emissivities of microstructured surfaces

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The calculation of radiative heat transfer is mostly influenced by the directional and spectral distribution of the radiation intensity in the range of infrared wavelengths. These distributions can be expressed by the directional spectral emissivity. The directional and spectral control of thermal radiation is one of the most important issues when improving efficiency and reducing energy consumption in various thermal systems. An attractive option for controlling directional and spectral radiative properties of a surface is the concept of periodic surface microstructures. Recent developments in the field of micromachining enable the manufacturing of periodic microstructures with geometrical dimensions of the same order of magnitude as the thermal radiation wavelengths. Interference effects between the structure and the electromagnetic waves are expected that are thought to increase the emissivity.

An apparatus is presented that is capable of measuring the directional spectral emissivities of solid surfaces at moderate temperatures between 330 K and 500 K. The directional distribution is studied for polar angles between 0° and 70° and for azimuth angles between 0° and 90°. The radiation intensity is detected by an FTIR-spectrometer in the wavelength range between 4 µm and 24 µm. For the evaluation of the emissivity, precise knowledge of the surface temperature is important because of its large influence on the radiation intensity. Several approaches are presented to determine the surface temperature without using a temperature probe directly on the sample surface. Experimental data will be presented for validation samples and for various microstructured surfaces.
1. INTRODUCTION

In our days a lot of technical applications depend on a good knowledge of radiative surface properties. Radiative energy transfer is important for space systems, high temperature heat transfer systems and for all kinds of solar energy systems. Because of simplicity, in engineering science it is often found that the radiative energy transfer is described with the help of hemispherical and over total range of wavelength integrated quantities of radiative surface properties. But for the detailed description of radiative energy transfer spectral and directional quantities of radiative surface properties are required. For example, the entropy content of a radiation energy flux depends on its spectral and directional distribution [3]. Therefore the aim of this work is to contribute to the data on spectral directional emissivities of technical surfaces and to present an apparatus which is capable of measuring this property. Special emphasis will be laid on microstructured surfaces. The previous work on the emittance properties of roughened or geometrically modified surfaces can be categorized into two branches. One branch is the measurement of the influence of largely random surface roughness on emission and of irregular asperities on absorption. The other is the calculation and measurement of the effect of regular surface structures on radiant emission and absorption.

Hesketh et al. [4, 5, 6, 7] extensively reported the directional and polarized spectral emissivity from one-dimensional heavily doped lamellar silicon surfaces with rectangular microstructure geometries. Selective thermal emission by electromagnetic standing waves was observed in microgrooves with repeat distances and depths of the grooves varied from 10 µm to 22 µm and from 0.7 µm to 44 µm, respectively. The measurements were carried out at temperatures of 300 °C and 400 °C and the experimental results were compared with data calculated by a geometric optics model. This research on the emission properties was extended by Wang and Zemel [8, 9, 10] to the case where the silicon is a dielectric material. Several theoretical models, for example Bloch-wave, coupled-mode, effective medium and waveguide methods, were examined. Tang and Buckius [11] reported detailed results on reflection from two dimensional metallic square grooved surfaces with repeat distances and surface heights ranging from 10 µm to 30 µm and from 1.6 µm to 20 µm, respectively. The detailed feature of thermal emissivity from microstructured surfaces was demonstrated experimentally as well as analytically. The possibility of controlling spectral reflectivity and thereby spectral emissivity from periodic microstructured metallic surfaces was shown, although the incident wavelength is restricted to the midinfrared range (2 µm -12 µm). The results from the periodic micromachined surfaces indicate that there are methods for the prediction of the emission properties, but none of the models yields a complete solution.

In this work an apparatus is presented that is capable of measuring the directional spectral emissivities of solid surfaces at moderate temperatures between 330 K and 500 K. The directional distribution is studied for polar angles between 0° and 70° and for azimuth angles between 0° and 90°. A FTIR-spectrometer is used to detected the radiation intensity in the wavelength range between 4 µm and 24 µm. For the evaluation of the emissivity, precise knowledge of the surface temperature is important because of its large influence on the radiation intensity. An useful solution is presented to determine the surface temperature without using a temperature probe directly on the sample surface. Experimental data will be presented for validation samples and for various microstructured surfaces.
2. METHOD AND EXPERIMENT

2.1 Experimental set up

The directional, spectral emissivity measurements are performed with an FTIR system. This system consists of two chambers, the vacuum chamber and the equipment chamber, as shown in Fig.1. These chambers are optically connected with a KBr window. The equipment chamber contains a Fourier transform infrared spectrometer type EQUINOX 55 manufactured by Bruker Optik GmbH. This spectrometer performs multiple wavelength measurements in the wavelength range between 0.8 and 27 µm. It has an inlet window for an external source of radiation, to which the sample chamber is connected with the help of a pipe. The spectrometer is purged with dry and carbon dioxide free air to reduce the effects of water and carbon dioxide on the measured spectrum. The used deuterated triglycine sulfate - DTGS – detector has a high sensitivity at 12°C. The radiative heat transfer between chamber walls and the detector is eliminated by tempering the sample chamber and the equipment chamber at the same constant temperature of 12°C.

The sample chamber contains different components, a sample holder system, a cylindrical cavity as a blackbody radiator and a rotatable plain mirror attachment between them. The sample chamber is evacuated with the help of a vacuum pump to exclude convective heat transfer from the sample. The inner walls of the sample chamber are painted with black lacquer with the hemispherical total emissivity of 0.94, so that the radiation striking the chamber walls from the heated components is reflected in small amounts only. Components near to the electrical heating systems in the sample holder and in the blackbody radiator are coated with polished chromium or they are made of materials with low emissivity values to reduce the emissivity of their surfaces. A rotatable mirror attachment is installed in the chamber at equidistance from the sample surface and the blackbody opening to guarantee the same optical path length for both sources. It is used to switch between the sample radiation and the blackbody radiation as an input signal for the FTIR-spectrometer. An aperture is placed between the mirror attachment and the spectrometer in order to cover the same measuring area of the radiation from the sample surface as well as from the blackbody radiator to the detector. The adjustment of the optical axes between the detector and the
sources of radiation is performed after every sample change by using an auto-collimation telescope. The sample holder is able to set the polar and the azimuthal angle of the sample to study the directional properties of the radiation from the sample surface. This is realized with the help of DC-bar armatured motors in combination with line coded absolute rotation transducers. The temperatures of the sample and the blackbody radiator are controlled by two separate PID-controlled electrical heating systems. The temperature measurement of the sample and the blackbody radiator is carried out by the use of calibrated platinum resistance thermometers Pt-100. Thermocouples are used to measure the temperature of the other components. The complete measured data is recorded with a personal computer for the following evaluation.

2.2 The microstructured samples

The microstructured silicon surfaces used in this work have been manufactured by the Fraunhofer Institut für Siliziumtechnologie (ISIT). The silicon wafers are circular in shape with a diameter of 150 mm and are 0.6 mm thick. One side of the wafer is polished and the other side is flat with clean and smooth condition. The microgrooves of different depths are etched into the polished side. The depth of the microgrooves are controlled by the etch time. The photos of rectangular microstructured silicon surfaces taken with the help of a scanning electron microscope as supplied by ISIT are shown in figure 2. These photos show the top view and the side view of the rectangular microstructured surfaces with a set of microgrooves. In the top view the grey area is the unetched flat part of the silicon wafer and the black area is the base of the groove. The rectangular shaped microgrooves are clearly seen in the side view.

![Fig. 2. Photo of rectangular microstructured silicon surfaces as supplied by ISIT](image)

The period of 10 $\mu$m and the width of 6.5 $\mu$m of the microgrooves are the same for all samples. The depth of the groove is varied to five different values: 1 $\mu$m, 3.4 $\mu$m, 11.2 $\mu$m, 21.4 $\mu$m and 34.2 $\mu$m.

2.3 FTIR -Spectrometer and its Calibration

A FTIR- spectrometer operating in external signal mode is used to measure many spectral points of the object radiance. These equipments do not provide spectra in absolute and physically meaningful units. The result of a measurement with a FTIR- spectrometer is therefore a spectrum in instrument dependent units called arbitrary units. In order to obtain a spectrum in units of the spectral radiance it is necessary to calibrate the measured spectrum.
For the calibration of a measured spectrum one should know the influencing factors on the radiation that passes from the investigated surface to the detector of the FTIR-spectrometer. Figure 2 visualizes the influences on the radiation. The detector receives not only radiation from the surface to be investigated, but also radiation emitted by parts of the chambers and the instruments. These two components are influenced by the atmosphere (e.g. moisture, carbon dioxide in air), the optical components of the spectrometer (e.g. lenses, mirrors, beam splitter, detector) and its electronics (e.g. filters, amplifiers).

\[ R(\lambda) = \frac{\varepsilon(\lambda,T) c_1}{\pi \lambda^5 \left[ \exp\left(c_2 / \lambda \cdot T \right) - 1 \right]} \]  \hspace{1cm} (1)

where L is the spectral radiance emitted by the surface \((W/m^2 \cdot \mu m^{-1})\), \(\lambda\) is the wavelength \((\mu m)\), T is the surface temperature \((K)\), \(c_1 = 3.741775 \cdot 10^8 \) \(W/m^2 \cdot \mu m^4\) is the first radiation constant, \(c_2 = 14387.7 \) \(\mu m\) \(K\) is the second radiation constant and \(\varepsilon\) is the emissivity of the surface which is equal to 1 for a blackbody surface.

The mathematical model that describes a radiance spectrum measured by a FTIR-spectrometer which receives radiation from an object as visualized in figure is given by the expression [1]

\[ S(\lambda) = R(\lambda) [L(\lambda) + I(\lambda)] \]  \hspace{1cm} (2)

where \(S(\lambda)\) is the measured spectrum that is obtained from the spectrometer, \(L(\lambda)\) is the spectral radiance of the object, \(R(\lambda)\) is the spectral response of the spectrometer which is composed of the transmittance of the atmosphere, influence of the optics and influence of the electronics, and \(I(\lambda)\) is the spectral radiance of the spectrometer and chamber inner parts.
In order to obtain spectral radiance that is emitted by the investigated object, the instrument functions $R(\lambda)$ and $I(\lambda)$ must be known. Then the calibrated spectrum in units of spectral radiance can be calculated from the measured spectrum $S(\lambda)$ using following relation:

$$L(\lambda) = \frac{S(\lambda)}{R(\lambda)} - I(\lambda)$$  \hspace{1cm} (3)

The task of the calibration procedure is the determination of the unknown parameters $R(\lambda)$ and $I(\lambda)$. Therefore it is necessary to measure spectra $S(\lambda)$ of radiation sources of known spectral radiances $L(\lambda)$. As the spectral radiance of a blackbody can be determined by using the Planck’s law (1) with an emissivity $\varepsilon = 1$, one should have to take the spectra $S_1(\lambda)$ and $S_2(\lambda)$ of two blackbodies of different temperatures $T_1$ and $T_2$, and solve them for the unknown parameters. Rewriting equation (2) for the temperatures $T_1$ and $T_2$:

$$S_1(\lambda) = R(\lambda)[L_1(\lambda) + I(\lambda)]$$  \hspace{1cm} (4)

$$S_2(\lambda) = R(\lambda)[L_2(\lambda) + I(\lambda)]$$  \hspace{1cm} (5)

where $L_1(\lambda)$ and $L_2(\lambda)$ are the spectral radiance of a blackbody at temperatures $T_1$ and $T_2$ respectively. Solving equations (4) and (5) for the unknown parameters $R(\lambda)$ and $I(\lambda)$, we get the required relations:

$$R(\lambda) = \frac{S_1(\lambda) - S_2(\lambda)}{L_1(\lambda) - L_2(\lambda)}$$  \hspace{1cm} (6)

$$I(\lambda) = \frac{S_1(\lambda)}{R(\lambda)} - L_1(\lambda)$$  \hspace{1cm} (7)

### 2.4 Determination of the surface temperature

Critical items in the measurement of emissivity are the constant tempering of the sample surface and the exact determination of the surface temperature. The emissivity measurement is based on the comparison of the radiation fluxes from the sample surface and the blackbody radiator at the same temperature. These radiation fluxes depend on the fourth power of the temperature. This means a small error in the temperature measurement of the sample surface may introduce a large error in the heat flux and consequently in the measurement of the emissivity $\varepsilon$ of the sample surface.

It is not suitable to place a contact sensor directly on the sample surface, because of the radiation exchange between the contact sensor and the detector which may introduce errors in the measurement of radiative properties of the sample surface. The determination of the surface temperature is rather based on the local energy balance for the sample.

Figure 3 illustrates an energy flow model of the real sample holder consisting of three material layers, namely the copper plate(sample holder), a heat-conductive paste film and the sample. The temperature gradients arising from heat conduction and radiation are shown in the same schematic representation. The temperature $T_m$ at the interface between the copper plate and the heat-conductive paste film is measured by using a calibrated platinum resistance thermometer (PT 100). The temperature at the interface between the heat-conductive paste film and the back of the sample is denoted by $T_g$. $T_u$ is the temperature of the sample chamber environment and the surface temperature of the sample is $T_o$ which is to be determined.
In a steady state the flow of heat flux from the back of the sample to its surface $\dot{q}_e$ is equal to the heat flux $\dot{q}_a$ from the sample surface to its environment.

$$\dot{q}_e = \dot{q}_a \quad (8)$$

The heat transfer from the back of the sample to its surface takes place only by means of conduction assuming that the heat loss from the peripheral area of the sample is negligible. The sample has the thickness $\delta_p$ and the constant thermal conductivity $\lambda_p$. Then the heat flux $\dot{q}_e$ through the sample from its backside to the surface is given by

$$\dot{q}_e = \frac{\lambda_p}{\delta_p} (T_g - T_o) \quad (9)$$

In this equation the heat-conductive paste temperature $T_g$ on the back of the sample is unknown. To replace this unknown quantity with the measured temperature $T_m$, we calculate the heat flux in the heat-conductive paste film, which must be equal to the heat flux $\dot{q}_e$ in the sample

$$\dot{q}_e = \frac{\lambda_g}{\delta_g} (T_m - T_g) \quad (10)$$

where $\lambda_g$ and $\delta_g$ are the thermal conductivity and the thickness of the heat-conductive paste film. After combining the equations (9) and (10) the result for the heat flux through the sample is

$$\dot{q}_e = \frac{T_m - T_o}{\frac{\delta_p}{\lambda_p} + \frac{\delta_g}{\lambda_g}} \quad (11)$$

The heat flux $\dot{q}_a$ from the sample surface to the chamber environment only consists of the radiation heat transfer process. The convective heat transfer is negligible, because of the vacuum of less then $10^{-2}$ mbar in the chamber. The radiative heat flux can be expressed as

$$\dot{q}_a = \varepsilon(T_o) \sigma \left( T_o^4 - T_u^4 \right) \quad (12)$$
where $\varepsilon(T_o)$ is the hemispherical total emissivity of the sample surface at the surface temperature $T_o$ and $\sigma$ is the Stefan-Boltzmann constant.

After some mathematical manipulation we get the conditional equation for $T_o$

$$T_o^4 + AT_o - B = 0$$

$$A = \frac{1}{\varepsilon(T_o)\sigma} \left(\frac{\delta_p}{\lambda_p} + \frac{\delta_g}{\lambda_g}\right)$$

$$B = \frac{T_m}{\varepsilon(T_o)\sigma} \left(\frac{\delta_p}{\lambda_p} + \frac{\delta_g}{\lambda_g}\right) + T_u^4$$

To solve this equation the function $\varepsilon(T_o)$ is required. With the help of the measured values of the directional spectral emissivity for temperatures of the blackbody in the range of the measured temperature $T_m$ it is possible to calculate the hemispherical total emissivity depending on temperature by integrating the directional spectral emissivity over all wavelengths and all directions.

$$\varepsilon(T_o) = \frac{1}{\sigma T_o^4} \int_0^{2\pi} \int_0^{\pi/2} \int_0^\infty \varepsilon(\lambda, \theta, \phi, T_o) L_{\lambda,\theta}(\lambda, T_o) \, d\lambda \, \cos \theta \, \sin \theta \, d\phi$$

The determination of $\varepsilon(T_o)$ by means of a polynomial fitting with the least square method enabled the calculation of the surface temperature $T_o$. In the last step the directional spectral emissivity for the calculated surface temperature is determined by use of linear interpolation.

For this measurement method and this determination of the surface temperature a uniformly distributed surface temperature is necessary. This distribution is checked with two methods. A FEM-model of the sample holder and the sample is constructed to calculate the surface temperature distribution. The model is build and solved by using the software package ANSYS. A result for a poorly heat conducting sample is visualized in figure 4.a. Another way to visualize the temperature distribution is to observe the sample surface with an IR-camera. One picture taken with the IR-camera AGEMA ThermoVision 570 is presented in figure 4.b. The temperature distribution as shown in figure 4.a and 4.b is constant at the measuring area in the middle of the sample. The calculated and measured distribution satisfy the requirements for this experimental method.

Fig. 4.a Simulated temperature distribution of a glass B270 sample

Fig. 4.b Observed temperature distribution of a glass B270 sample
3. RESULTS AND DISCUSSION

3.1 Validation measurement

In order to validate directional spectral emissivity results obtained in this work, the spectral emissivity of black paint with trading name Nextel-Velvet-Coating 811-21 coated on an aluminium plate is measured at different emission angles from 0° to 70°. The selected black paint is provided by MANKIEWICZ GEBR. & CO. and its physical properties are well characterized. Because its directional spectral emissivity values at different temperatures have been already investigated experimentally by different authors [12, 13] in detail, it is taken as reference material for validation of the results of the emissivity measurement.

In the present work, the surface of an aluminium plate with a diameter of 150 mm and a thickness of 5 mm is coated with Nextel-Velvet-Coating 811-21 with the help of a spraying pistol, so that the thickness of the coating can be achieved to be almost uniform over the surface. The average thickness of the coating is 0.15 mm. The backside of the plate is polished and a thin layer of heat conductive paste is applied between this aluminium plate and the sample holder plate of copper to insure a good heat conduction between them.

![Fig. 5.a Normal spectral emissivity of Nextel-Velvet-Coating 811-21 measured normal to radiating surface](image1)

![Fig. 5.b Directional total emissivity of Nextel-Velvet-Coating 811-21 measured at different emission angles](image2)

Figure 5.a represents normal spectral emissivity of Nextel-Velvet-Coating 811-21 measured at temperature 92.5 °C in the present experiment along with the data of measurements by other authors [12, 13]. It is clear from this graph that the normal spectral emissivity is almost constant at wavelengths between 8 µm and 25 µm and its value increases slightly with the wavelengths between 5 µm and 8 µm. This trend of dependence of the normal spectral emissivity with the wavelength of the radiation agrees well with both the results reported by Lohrengel from the PTB (Physikalisch-Technische Bundesanstalt [13] and Ishii et al. [12] in wavelength range 8 µm to 25 µm. Our results are closer to the results of Ishii et al. than to the results of PTB in the wavelength range 5 µm to 8 µm.

Figure 5.b graphically shows the directional total emissivity of Nextel-Velvet-Coating 811-21 at different emission angles measured in present experiment and the same quantity measured by Lohrengel et al. and reported in PTB [13]. The solid curve is the result of the present study at a temperature of 92.5 °C and the values denoted by the circles are reported in reference [13].
for temperature 90 °C. A comparison between these two curves tells that the results obtained from two different instruments are in good agreement with each other over all the angles of emission.

3.2 Periodic microstructured silicon surface

The directional spectral emissivity data presented in this section refer to measurements at different polar angles $\vartheta = 0°$ to 70° in steps of 6° and for two azimuthal angles $\varphi = 0°$ and 90° for all the microgrooves. The measurements are done at a surface temperature of 200°C.

![Fig. 6](image1.png) Directional spectral emissivity of a microgrooved undoped silicon surface with wavelength of radiation as parameter measured at an azimuthal angle $\varphi = 0°$ for a groove depth $H = 34.2 \, \mu m$ ($T = 200 °C$).

The directional spectral emissivity of the microgrooved undoped silicon surface measured at the azimuthal angle $\varphi = 0°$ for the groove depth $H = 34.2 \, \mu m$ with the wavelength of radiation

![Fig. 7](image2.png) Directional spectral emissivity of undoped silicon microgrooves at a wavelength $\lambda = 14 \, \mu m$ and an azimuthal angle $\varphi = 0°$ for different groove depth $H$ ($T = 200 °C$)
as a parameter is discussed first. In this case presented in figure 6 several pronounced emissive peaks are observed for wavelengths from 9 µm to 17 µm. A few features and some broad maxima are seen at shorter and longer wavelengths. The spacing between emission maxima increases as the wavelength increases.

![Graph](image1.png)

**Fig. 8** Directional spectral emissivity of a microgrooved undoped silicon surface with wavelength of radiation as parameter measured at an azimuthal angle $\phi = 90^\circ$ for a groove depth $H = 34.2$ µm ($T = 200$ °C).

The directional spectral emissivity at zero azimuthal angle $\phi = 0^\circ$ and a wavelength of radiation 14 µm is graphed in figure 7 for different microgroove depths. The run of the curves implies a groove depth dependence of the directional spectral emissivity of the microgrooves. It is observed that the emission peaks for the same wavelength depend on the depth of the microgrooves. The amplitude of the peaks decreases as the depth of the microgrooves decrease. A regular variation of the positions of the peaks with the depth of the microgrooves is also observed.

![Graph](image2.png)

**Fig. 9** Directional spectral emissivity of undoped silicon microgrooves at a wavelength $\lambda = 14$ µm and an azimuthal angle $\phi = 90^\circ$ for different groove depth $H$ ($T = 200$ °C).
The emissivity curves obtained at the azimuthal angle $\varphi = 90^\circ$ show a different behaviour than those obtained at the azimuthal angle $\varphi = 0^\circ$. For microgrooves with depth $H = 34.2 \, \mu\text{m}$ several pronounced emission peaks are observed at different polar angles and wavelengths as shown in figure 8. The emission decreases with the polar angle for all wave length. For larger polar angles the emissivity values reduce faster because of the shadow effect occurring in the deep microgrooves to the direction of large polar angles so that the detector can’t receive the whole emitted radiation. Some broad maxima are observed at longer wavelengths.

The directional spectral emissivity at the wavelength of the radiation $\lambda = 14 \, \mu\text{m}$ and at the azimuthal angle of $90^\circ$ for the different microgroove depths is presented in figure 9. It is observed that the emission peaks for the same wavelength depend to some extent on the depth of the microgrooves. For depth above $4 \, \mu\text{m}$ the amplitude of the peaks decreases as the depth of the microgrooves increases. The angular position of the one pronounced peak for each sample varies with the depth of the microgrooves. The physical interpretation of the spectra shown here is in progress and will be presented in subsequent papers.

4. CONCLUSION

The measurement system presented in this paper allows the measurement of the directional spectral emissivity of technical solid surfaces with different geometries and coatings. It is possible to measure at sample surface temperature up to $250^\circ\text{C}$ for polar angles from $0^\circ$ to $70^\circ$ and azimuthal angles from $0^\circ$ to $90^\circ$. With the optics and detectors used here good results are achieved for wavelength between $4 \, \mu\text{m}$ and $25 \, \mu\text{m}$. The comparison of smooth surfaces with the microstructured surfaces shows the possibility to influence the radiation characteristic of a surface with microstructures. The microstructure can increase the total hemispherical emissivity. It seems possible to adjust emissivity maxima for a specific wavelength and a specific solid angle. The physical modeling of this emissivity is an on going project at the Institute.
REFERENCES