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Characterization of high temperature solar thermal selective absorber coatings at operation temperature

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Abstract

In cooperation between the Physikalisch-Technische Bundesanstalt (PTB) and the SCHOTT Solar CSP GmbH (SCHOTT Solar) measurements have been carried out with the goal to assure, that the directional-hemispherical reflectance measurements, done as production control by SCHOTT Solar, lead to applicable results for calculating the emittance of absorber tubes for parabolic trough receivers at their operation temperatures above 350 °C.

A significant influence of the temperature on the directional spectral emittance could not be found. Due to the spectral characteristics of the emittance with a rising spectral emittance towards shorter wavelength the spectral integrated quantity: directional total emittance becomes larger with an increasing temperature due to the shift of the Planck curve.

Furthermore, the measurements of PTB confirmed the emittance values calculated from the reflection measurements and the directional-hemispherical reflectance measurements performed by SCHOTT.

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Keywords: emittance; measurement methods; calibration; solar selective absorber;

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1. Introduction

Solar thermal selective absorber coatings are currently characterized by their solar absorptance and their thermal emittance. The thermal emittance significantly influences the heat loss of the heat collecting elements in a solar power plant and so the efficiency of the plant. The consideration of the emittance is especially important at high temperatures above 350 °C occurring in parabolic trough power plants because the heat loss increases disproportionately with an increasing surface temperature of the heat collecting elements.

At SCHOTT the values for solar absorptance and thermal emittance are currently derived from reflectance measurements at room temperature in the spectral range from 0.3 μm to 25 μm. The assumed temperature independence of the emittance had to be confirmed in order to ensure that the measurements at room temperature are also valid for higher temperatures.

### Nomenclature

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\varepsilon$</td>
<td>emittance</td>
</tr>
<tr>
<td>$\varepsilon_{ds}$</td>
<td>directional spectral emittance</td>
</tr>
<tr>
<td>$\varepsilon_{dt}$</td>
<td>directional total emittance</td>
</tr>
<tr>
<td>$\varepsilon_{BB1}$</td>
<td>directional spectral effective emittance of the blackbody</td>
</tr>
<tr>
<td>$\rho_{ds}$</td>
<td>directional spectral reflectance</td>
</tr>
<tr>
<td>$\rho$</td>
<td>reflectance of the sample</td>
</tr>
<tr>
<td>$\rho_{r1}$</td>
<td>reflectance of the first reference sample</td>
</tr>
<tr>
<td>$\rho_{r2}$</td>
<td>reflectance of the second reference sample</td>
</tr>
<tr>
<td>$F_c$</td>
<td>calibration function</td>
</tr>
<tr>
<td>$L_{\Omega\lambda}$</td>
<td>directional spectral radiance</td>
</tr>
<tr>
<td>$L_{\text{Background}}$</td>
<td>spectral radiance of the thermal background of the RBCF</td>
</tr>
<tr>
<td>$L_{BB,\Omega\lambda}$</td>
<td>directional spectral radiance of a blackbody</td>
</tr>
<tr>
<td>$L_{BB1}$</td>
<td>directional spectral radiance of first reference blackbody</td>
</tr>
<tr>
<td>$L_{BB1}$</td>
<td>measured signal of directional spectral radiance of first reference blackbody</td>
</tr>
<tr>
<td>$L_{BB-LN2}$</td>
<td>measured signal of directional spectral radiance of blackbody at temperature of liquid nitrogen (second reference blackbody)</td>
</tr>
<tr>
<td>$L_{\text{Detector}}$</td>
<td>spectral radiance of the detector</td>
</tr>
<tr>
<td>$L_{\text{Planck}}$</td>
<td>spectral blackbody radiance given by Planck’s law</td>
</tr>
<tr>
<td>$L_X$</td>
<td>measured signal of radiance emitted from sample</td>
</tr>
<tr>
<td>$Q$</td>
<td>Quotient</td>
</tr>
<tr>
<td>$\Phi_D$</td>
<td>Radiation flux on the detector</td>
</tr>
<tr>
<td>$\Phi_i$</td>
<td>Radiation flux on the sample</td>
</tr>
<tr>
<td>$s$</td>
<td>detected signal</td>
</tr>
<tr>
<td>$s_x$</td>
<td>detected signal from the sample</td>
</tr>
<tr>
<td>$s_{r1}$</td>
<td>detected signal from the first reference sample</td>
</tr>
<tr>
<td>$s_{r2}$</td>
<td>detected signal from the second reference sample</td>
</tr>
<tr>
<td>$T$</td>
<td>temperature</td>
</tr>
<tr>
<td>$T_x$</td>
<td>temperature of sample</td>
</tr>
<tr>
<td>$T_{BB1}$</td>
<td>temperature of first reference blackbody</td>
</tr>
<tr>
<td>$T_{BB-LN2}$</td>
<td>temperature of blackbody at temperature of liquid nitrogen (second reference blackbody)</td>
</tr>
</tbody>
</table>
2. Determination of the directional total emittance

The directional total emittance can be determined by detecting the radiation of a heated sample or by measuring the reflectance of a cold sample and calculating the emittance by applying Kirchhoff’s law.

Detecting the radiation of a heated sample leads to results closer to ‘real life conditions’ as the sample is exposed to the same physical environment and operating conditions, e.g. the high temperature.

Measuring the reflectance at room temperature is a faster method because the rather long time for heating up the sample to a stable high temperature is not necessary. However, a prerequisite for the latter method is the assumed temperature independence of the reflectance which has to be confirmed.

2.1. Measuring the directional spectral emittance by detecting the radiation of a heated sample

Fig. 1: Principle of measuring radiation from heated sample

Measurement of radiation emitted from a heated sample is done by heating the sample to a defined temperature and detecting the emitted radiation as shown in Fig. 1. The sample (1) is heated to the desired temperature using a heating element (7). The sample temperature is measured with a sensor (5) and a controller (6) controls the heating element (7) in order to achieve a stable temperature over time. The sample (1) emits radiation (2, 3), which is detected by a direction and wavelength sensitive detector (8). Only the part (3) of the radiation leads to a signal, which is emitted towards the direction of sensitivity of the detector (8). An evaluating and recording unit (4) stores the signal including related direction and wavelength.

The detected signal \( s(\lambda) \) is related to the emitted directed spectral radiance \( L_{\Omega\lambda} \) via a calibration function \( F_c \). The directional spectral emittance could be calculated using

\[
\varepsilon_{ds}(T, \lambda) = \frac{L_{\Omega\lambda}(T, \lambda)}{L_{BB,\Omega\lambda}(T, \lambda)} = \frac{s(\lambda)}{F_c(\lambda)L_{BB,\Omega\lambda}(T, \lambda)}
\]

(1)

More details, e.g. regarding the influence of background radiation see chapter 3.1 below and [1, 2, 3, 4]
2.2. Measuring the directional spectral reflectance

As shown in Fig. 2 incident light (9) from a radiation source (10) hits the sample (1) and is reflected to a detector (8). To achieve spectral data, either the light source (10) could be a tunable monochromatic source or the detector (8) could be able to disperse the detected light and supply spectrally resolved data to the evaluating and recording unit (4).

The directional spectral reflectance is given by

$$\rho_{ds} = \frac{\phi_D}{\phi_I}$$

(2)

Fig. 2: Principle of reflectance measurement with direct irradiation and direct detection

In order to avoid the necessity of absolutely calibrated light sources and detectors, reference samples are used commonly. Using two well-known reference samples ρ can be calculated without knowledge of absolute values, with the preconditions that the lamp power is stable and the detector is linear. The sample reflectance can be calculated from the signals recorded with the reference samples and the known reflectance of the reference samples:

$$\rho_s = \rho_{r1} + (\rho_{r2} - \rho_{r1}) \frac{s_s - s_{r1}}{s_{r2} - s_{r1}}$$

(3)

For non-transparent materials, such as the solar selective absorber coating and, furthermore, neglecting the diffusely reflected components Kirchhoff’s law applies approximately:

$$\varepsilon_{ds} \approx 1 - \rho_{ds}$$

(4)

By this method the directional spectral emittance can be calculated approximately from directional reflectance measurements using equations (3) and (4).
2.3. Measuring the spectral reflectance with hemispherical irradiation

![Fig.3: Principle of reflectance measurement with diffuse irradiation](image)

If the sample is not a plane surface but a tubular body, the method of directed incidence and directed detection as described above is difficult to apply because sample, light source and detector have to be adjusted very carefully. So the measurement principle shown in Fig. 3 is preferable. Here, the tubular sample (12) is inserted into an aperture of an integrating sphere (13).

The sample (12) is hemispherically illuminated by radiation originating from the sources (10) which are diffusely reflected in the integrating sphere (14). The detector (8) detects light reflected by the sample (12) in one direction only. So here the directional-hemispherical reflectance is determined and no approximations as in section 2.2 are necessary.

To record spectrally resolved data the same alternatives as mentioned in section 2.2 may be used: either the light source or the detector must be able to provide / detect light spectrally resolved. Similar to section 2.2 the measurements are performed with respect to two reference samples. More details on the used reference samples for this method see section 3.3.

2.4. Calculating the directional total emittance

From the spectral data obtained with any of the methods described above, the directional total emittance of a sample considering the emission in the whole spectral range can be calculated using:

\[
\varepsilon_{dt} = \frac{\int e_{ds}(\lambda) \cdot L_{BB,\Omega}(\lambda) d\lambda}{\int L_{BB,\Omega}(\lambda) d\lambda}
\]

(5)

3. Experimental setups

3.1. Physikalisch-Technische Bundesanstalt (PTB)

The directional spectral emittance measurement scheme is based on a comparison of the spectral radiance of a sample inside of a temperature stabilized spherical enclosure with the spectral radiances of two reference blackbodies at different temperatures (Fig. 4). The emittance can be obtained from the ratio:
\[
Q = \frac{\tilde{L}_\lambda(T) - \tilde{L}_{BB-LN_2}(T_{BB-LN_2})}{\tilde{L}_{BB1}(T_{BB1}) - \tilde{L}_{BB-LN_2}(T_{BB-LN_2})}
\]

(6)

The first reference blackbody used is either the vacuum low-temperature blackbody (VLTBB) [5] or the vacuum medium-temperature blackbody (VMTBB) [6] depending on the temperature range. This reference blackbody is usually operated at a temperature close to the radiation temperature of the sample. The measured signal \( \tilde{L}_{BB1}(T_{BB1}) \) of this blackbody is given by:

\[
\tilde{L}_{BB1}(T_{BB1}) = F_c L_{BB1}(T_{BB1}) + L_{Background} - L_{Detector}
\]

(7)

The spectral radiance of the blackbody is given by \( L_{BB}(T_{BB1}) = \varepsilon_{BB1} L_{Planck}(T_{BB1}) \), where \( L_{Planck}(T_{BB1}) \) is the spectral blackbody radiance at the respective temperature given by Planck’s law and \( \varepsilon_{BB1} \) is the directional spectral effective emittance of the blackbody. \( L_{Background} \) denotes the spectral radiance of the thermal background of the RBCF and \( L_{Detector} \) the spectral radiance of the detector. The value \( F_c \) is the spectral responsivity of the spectrometer. The second reference source, the LN2-cooled blackbody, is mounted on top of the opto-mechanical unit (see Fig. 4) and is used for the elimination of the background radiation.

Fig. 4 shows a transparent view of the Reduced Background Calibration Facility (RBCF) with the two vacuum blackbodies VLTBB and VMTBB, the sample holder for emittance measurements, the LN2-cooled reference blackbody, the Fourier-transform spectrometer and the optical path of the radiation in the LN2-cooled beamline.

3.2. SCHOTT AG, R&D department

The measurements of the directional spectral reflectance at the R&D department of SCHOTT AG were performed using a commercially available Fourier-transform infrared spectrometer type Nexus. The instrument operates in the spectral range from 1.4 \( \mu \)m to 25 \( \mu \)m.

Measurements were made in a nitrogen atmosphere at room temperature. The instrument is calibrated using commercially available certified reflectance standards.
3.3. SCHOTT Solar

The measuring device of SCHOTT Solar comprises two integrating spheres (15, 16), each of them equipped with light sources (10) and light detectors (17, 18, 19, 20, 21) as shown in Fig. 5. The tubular shaped sample (12) is inserted in special shaped apertures at the lower sides of the spheres (15, 16). It is in close contact with the rim of the aperture, so no ambient light can enter the sphere. The light sources (10) are located in a recessed position in the side walls of the spheres, to avoid a direct illumination of the sample or the detectors and to illuminate the inner surface of the sphere only, which reflects the light in a diffuse way. The light reflected by the sample towards the sensors is detected as a measuring signal proportional to the directional-hemispherical reflectance of the sample.

Light sources, inner surface and detectors of sphere 1 (15) are optimized to detect the sample reflectance in the ultraviolet, visible and near infrared spectral range from 0.3 µm to 2.2 µm, whereas the sphere 2 (16) is optimized to detect the reflectance in the infrared spectral range from 4 µm to 14 µm.

In the spectral range from 0.3 µm to 2.2 µm the signals are detected with two spectrometers (17, 18) with spectral resolutions from 0.001 µm in the ultraviolet spectral range to 0.005 µm in the near infrared spectral range. For the infrared spectral range sphere 2 (16) contains three sensors (19, 20, 21), sensitive at 3.9 µm, 5.1 µm and in the range from 8 to 14 µm.

During the measurement procedure reference samples are used as explained in section 2.3. For the spectral range from 0.3 µm to 2.2 µm semi-cylindrical shaped reference samples made of glass are used. Their reflectance values are calculated from their refractive indices, where the refractive indices have been determined at the certified lab of SCHOTT AG, Optics Department.

The reference samples for the spectral range from 4 µm to 14 µm are stainless steel tubes ground to the same roughness as used for the absorber tubes. Instead of applying an absorber coating they are coated with pure materials whose reflectance data are available from optical handbooks and databases.

To calculate the total emittance, the parameters of an empiric formula are fitted to the measured reflectance values.

4. Samples

The different measuring setups at the PTB, SCHOTT AG and SCHOTT Solar require different sample types. Common to all sample types is the coating by SCHOTT Solar. The sample substrates for PTB were small metal plates, the sample substrates for SCHOTT AG were small glass plates and the sample substrates for SCHOTT Solar were the metal absorber tubes for the receivers.
Table 1: Samples

<table>
<thead>
<tr>
<th>Sample short name</th>
<th>Sample Type</th>
<th>Coating</th>
<th>Measured by</th>
</tr>
</thead>
<tbody>
<tr>
<td>FM</td>
<td>Flat metal</td>
<td>Coating Type 1, SCHOTT Solar, Production site 2</td>
<td>PTB</td>
</tr>
<tr>
<td>FG1</td>
<td>Flat glass</td>
<td>Coating Type 1, SCHOTT Solar, Production site 1</td>
<td>Schott AG</td>
</tr>
<tr>
<td>FG2</td>
<td>Flat glass</td>
<td>Coating Type 1, SCHOTT Solar, Production site 2</td>
<td>R&amp;D Department</td>
</tr>
<tr>
<td>TM1</td>
<td>Tubular metal</td>
<td>Coating Type 1, SCHOTT Solar, Production site 2</td>
<td>SCHOTT Solar</td>
</tr>
<tr>
<td>TM2</td>
<td>Tubular metal</td>
<td>Coating Type 2</td>
<td>SCHOTT Solar Production site 2</td>
</tr>
</tbody>
</table>

5. Results

5.1. Emittance evaluation according to PTB measurement

Fig. 7 shows the temperature dependence of the directional total emittance of all five samples. The results of PTB (labeled ‘FM’, green line) are based on actual measurements of the directional spectral emittance of the PTB samples at different sample temperatures. The directional spectral measurements have been performed under an angle of 10° and have been integrated over a spectral range from 3 µm to 25 µm.

![Fig. 7: Directional total emittance, comparison of all measurements, evaluation for a limited spectral range](image)

The other four results were calculated from reflectance measurements by SCHOTT. To compare the results, the evaluation of the directional total emittance from reflectance measurements has been done for the same wavelength...
range as the PTB measurements. This wavelength range is different to SCHOTT’s standard procedure for the determination of $\varepsilon_d$ in the production process. Hence the results are not directly comparable with SCHOTT’s published values for $\varepsilon_d$ at 400 °C.

Caused by the determination method, the uncertainty of the FM sample increases to lower temperatures, which is due to the poorer signal-to-noise ratio at lower temperatures.

The FG and TM samples were measured at room temperature only. The emittance at higher temperatures is calculated, not taking into account that the reflectance of the samples might change with temperature.

![Fig. 8: Differences in determined directional total emittance values, the FM-sample, measured by PTB, is the reference](image)

As shown Fig. 8 the directional total emittance values calculated from the reflectance measurements agree with the emittance values measured by PTB at high temperatures within a tolerance band of ±0.016.

Taking into account, that the values have been determined from different samples, coated within SCHOTT’s routine production process (where no special effort had been spent to provide equal samples), employing different measuring devices, this is a good agreement.

### 5.2. Calculation of emittance at higher temperatures

The temperature dependency of the directional total emittance for the selective absorber coating is significantly higher, if spectral data starting with lower wavelengths are considered. This is shown in Figure 9. The emittance values for the TM1 and TM2 samples are calculated considering reflectance data in the spectral range from 0.3 µm to 30.0 µm, while the PTB data is derived from measurements from 3.0 µm to 25.0 µm. The results of the FM sample, based on measurements in the spectral range from 3.0µm to 25.0µm, are displayed comparatively.

The slope of the graphs for the TM samples is significant higher than the slope of the graph for the FM sample. This is because with higher temperature a higher part of the black body radiation energy is emitted in the spectral range below 3.0 µm, whereas the spectral emittance of a solar selective absorber coating increases strongly in the spectral range below 2.0 µm. Spectral data only in the range above 3.0 µm cannot describe this effect.

Also the difference in the emittances between TM1 and TM2 are higher if the spectral range starting from 0.3 µm is considered. This is caused by the fact that the spectral emittances of coatings type 1(TM1) and type 2 (TM2) are similar in the spectral range above 3.0 µm, but are different in the spectral range below 3.0 µm.
Fig. 9: Temperature dependence of directional total emittance, calculated for TM1 and TM2 with full spectrum (results of FM with limited spectrum displayed comparatively)

6. Conclusions

The directional total emittance of a solar thermal selective absorber coating has been determined using three different methods. The results match within a tolerance band of $\pm 0.016$. The direct emittance measurements at high temperatures performed under vacuum by PTB confirm the calculation results based on reflectance measurements at room temperature in air and nitrogen performed by SCHOTT AG and SCHOTT Solar.

References

[1] Adibekyan, A.; Monte, C.; Kehrt, M.; Gutschwager, B. & Hollandt, J., Emissivity measurement under vacuum from 4 $\mu$m to 100 $\mu$m and from 40 °C to 500 °C at PTB, International Journal of Thermophysics, 2013, to be published