

## Spectral emittance and temperature determination of carbon/SiC and SiC/SiC composites

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### ABSTRACT

The radiation comparison method is used to measure the total normal and spectral emittance in the range 1000 K to 2000 K. Problems of measurement uncertainties mainly resulting from temperature measurement errors are discussed. Results of measurements on Carbon/SiC and SiC/SiC fiber/matrix composites show values in the range 0.7 to 0.9 without a systematic dependency on fiber material or orientation.

The new evaluation method for the integral six-color pyrometer is used to automatically calculate the true temperature from the measured radiance temperatures. The variation of the emittance  $\epsilon$  with wavelength can be expressed as an exponential function with one, two, three or four coefficients. An averaging method is used to find the correct surface temperature. Deviations between calculated and experimentally determined surface temperatures were found to be between 1 and 2%.

### INTRODUCTION

The knowledge of the emittance as well as the thermal conductivity is essential to calculate temperatures of thermally loaded components. These are important to estimate the lifetime of a component which is limited by the extreme temperature or to judge mechanical stresses caused by temperature gradients or rapid

temperature changes. One highly developed technique to improve the mechanical stability of materials is the reinforcement by means of fibers, whiskers or particles from the same or from a different material. Spectral emittance values are needed for temperature measurement in high temperature experiments with such materials and in the development of the material.

As is well known, the emittance of a material varies markedly both with the chemical composition and the structure of the material. In the case of fiber reinforced materials two types of materials form different kinds of surface topography and we have to expect that this influences the emittance, which depends on the surface roughness.

The accuracy of the emittance measurement essentially depends on the accuracy of the radiance measurement and of the surface temperature measurement. Because of difficulties of direct surface temperature measurement multispectral temperature measurement methods have been proposed in the literature. We will compare the results of traditional emittance measurements using separately measured temperature with results of emittance evaluation using the temperature calculated from the measured radiance temperatures.

## EMITTANCE EVALUATION WITH TEMPERATURE MEASUREMENT IN A BLACKBODY CAVITY

The method is based on the principle of radiation comparison. The radiation emitted from a disc-shaped sample is measured using thermal and/or photoelectric detectors, which were calibrated against a blackbody during separate measurements. The disc-shaped sample is in radiation exchange with cooled and blackened surroundings, the influence of which on the signal produced by the thermal radiation detector can be corrected.

The sample temperature is measured inside a small radial hole. The surface temperature  $T_0$  can be calculated by means of a heat transfer model as described earlier by Neuer (1970).

The sample is about 5 mm thick and the distance  $s$  between the radial hole and the surface, the emittance of which is to be measured, is less than 2 mm. Figure 1 shows the schematic of the measurement device. The essential characteristics are:

- 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 rotated on a circle with variable radius 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 only measurements in vacuum ( $<0.7 \cdot 10^{-4}$  mbar) are possible.

Two radiation detectors are used, each in combination with a filter wheel having provisions to place up to 12 spectral filters of 1" diameter.

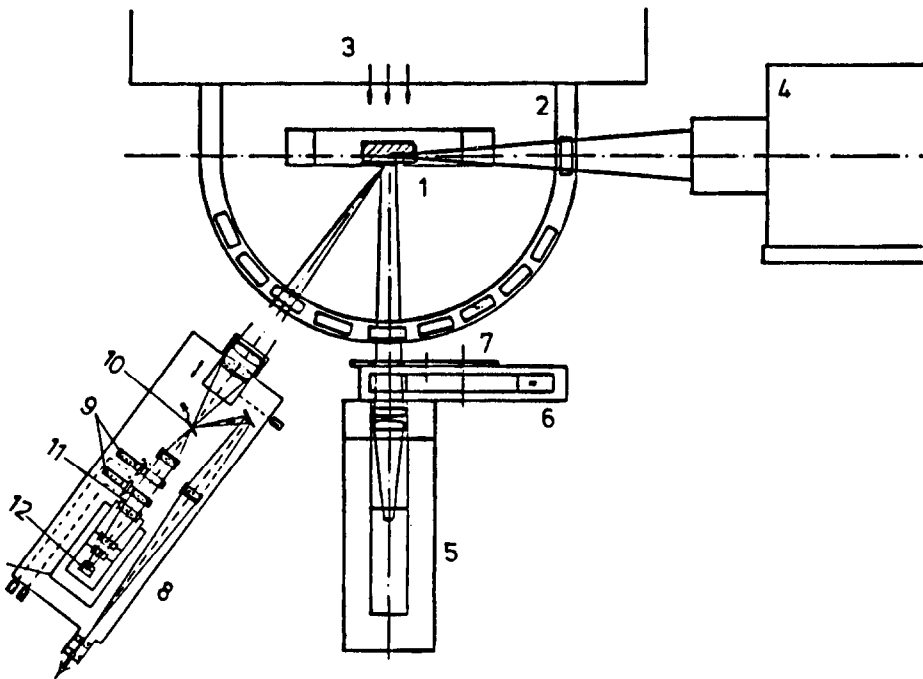


Fig. 1 Schematic of the device for emittance measurements

- |  |                              |
|--|------------------------------|
| 1: Sample  | 7: Field stop (exchangeable) |
| 2: Vacuum chamber with windows                           | 8: Linearpyrometer LP2       |
| 3: Electron beam heating                                 | 9: Filter wheel              |
| 4: Pyrometer for sample temperature measurement          | 10: Field stop               |
| 5: Thermal radiation detector with $\text{CaF}_2$ window | 11: Aperture stop            |
| 6: Filter wheel  | 12: Silicon detector         |

A Linearpyrometer LP2, described by Wörner (1982), is used for measurements in the lower wavelength range and a thermoelectric detector is used at wavelengths between 1.3  $\mu\text{m}$  and 10  $\mu\text{m}$ . The measurement of the total normal emittance is possible by keeping one place free in the filterwheel for the total radiation detector.

As shown in Fig. 1 the thermal radiation detector is installed in the direction of the normal to the sample surface. The Linearpyrometer was adjusted at an angle of inclination of  $35^\circ$  to the normal direction. This arrangement allows the measurements to be performed without an exchange of the detectors. The Linearpyrometer can also be placed in the normal direction to check whether the emittance at  $35^\circ$  differs from that at  $0^\circ$ . Additionally angular dependent emittance measurements are possible up to  $70^\circ$  using a third detector system with a bolometer combined with a mirror optic.

The imprecision of the radiance measurements has been estimated to be 3 % for the total emittance and infrared wavelength range. The inaccuracy of the calculated emittance is essentially influenced by uncertainties of the thermal conductivity which is the determining factor for the calculation of the temperature difference  $\Delta T$  between the blackbody hole and the surface (Neuer, 1970). Greater temperature differences and accordingly greater temperature errors occur with decreasing thermal conductivity, with increasing total emittance and temperature of the sample due to a higher heat flux. The spectral emittance values at short wavelengths are considerably more sensitive with temperature errors than those at longer wavelengths.

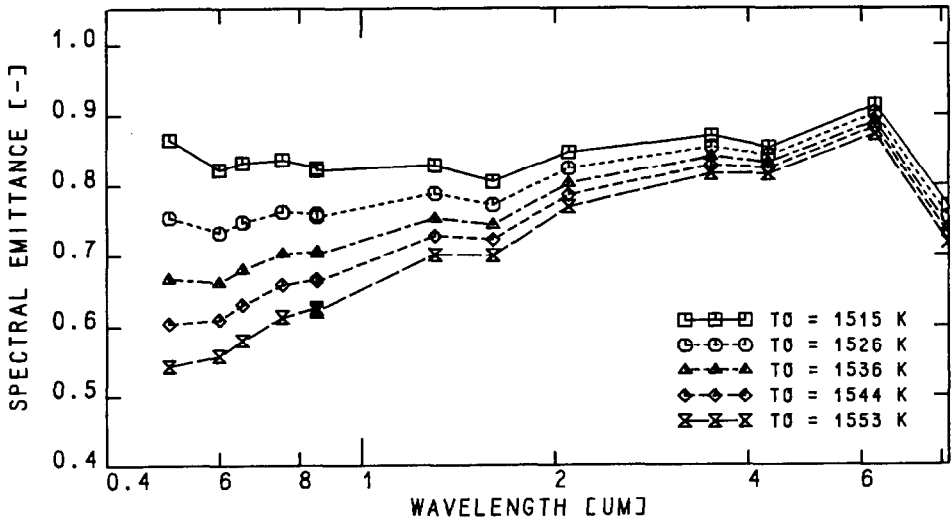


Fig. 2 Spectral emittance of a C/SiC composite evaluated with thermal conductivity values, varying between 10, 14, 22, 40 and 400 W/mK

The influence of thermal conductivity can be demonstrated by evaluation of the measurement values with varying thermal conductivity, resulting in varying surface temperatures. The result of such a parameter variation is shown in Fig. 2. The thermal conductivities corresponding to the temperatures 1515K to 1553K were 10, 14, 22, 40 and 400 W/m·K. The temperature difference increases and therewith the surface temperature with decreasing thermal conductivity.

The thermal conductivity was measured separately (Chateigner, 1990) with an inaccuracy of 20 %. The reproducibility of the material relating to its thermal conductivity is in the range 15 to 30 %. The resulting emittance inaccuracy is 5 to 20 % in the low wavelength and 3 to 6 % in the infrared range and for the total emittance. The higher inaccuracies are valid for the investigated two dimensional SiC/SiC composites with the heat flux perpendicular to the fabric plane.

#### DETERMINATION OF SURFACE TEMPERATURE BY MEANS OF MULTIWAVELENGTH TECHNIQUE

Instead of calculating the surface temperature from the measured blackbody hole temperature, we used the measured spectral radiance values by the "Integral Six-Color Pyrometer" described earlier (Hoch, 1992). It has to be noted that the spectral emittance values were not measured at the same time at all wavelengths, and that the sample temperature was not exactly stable during that time. Therefore the radiance temperatures had to be normalized by using the average blackbody hole temperature.

We only summarize the equations needed to determine the surface temperature  $T_0$  from the radiance values as measured by a *linear* photodetector.

The basic equation of optical pyrometry is

$$\lambda(\ln \epsilon_\lambda) / c_2 = (1/T - 1/T_r) \quad (1)$$

where  $\epsilon_\lambda$  is the spectral emissivity,  $T_r$  is the spectral radiance temperature,  $\lambda$  is the wavelength and  $T$  is the surface temperature,  $c_2$  is the second Planck constant.

The variation of the emissivity with wavelength can be expressed in the form

$$\ln \epsilon = a' + b'\lambda + c'\lambda^2 + d\lambda^3 + \dots \quad (2)$$

Dividing by  $c_2$ ,

$$(\ln \epsilon) / c_2 = a + b\lambda + c\lambda^2 + d\lambda^3 + \dots \quad (3)$$

where  $a = a'/c_2$ , etc.

Combining Eq. (1) and Eq. (3), multiplying by  $\lambda$ , and keeping in mind that we operate at six wavelengths, at wavelength  $m$  we have

$$-1/Tr_m = -1/T + a\lambda_m + b\lambda_m^2 + c\lambda_m^3 + d\lambda_m^4 \quad (4)$$

At another wavelength  $\lambda_n$

$$-1/Tr_n = -1/T + a\lambda_n + b\lambda_n^2 + c\lambda_n^3 + d\lambda_n^4 \quad (5)$$

In subtracting the above two equations, we obtain 15 equations, of the form

$$(-1/Tr_m + 1/Tr_n) = a(\lambda_m - \lambda_n) + b(\lambda_m^2 - \lambda_n^2) + c(\lambda_m^3 - \lambda_n^3) + d(\lambda_m^4 - \lambda_n^4) \quad (6)$$

dividing by  $(\lambda_m - \lambda_n)$ ,

$$(-1/Tr_m + 1/Tr_n) / (\lambda_m - \lambda_n) = a + b(\lambda_m + \lambda_n) + c(\lambda_m^2 + \lambda_m\lambda_n + \lambda_n^2) + d(\lambda_m^3 + \lambda_m^2\lambda_n + \lambda_m\lambda_n^2 + \lambda_n^3) \quad (7)$$

The 15 equations can be solved by regression analysis to obtain  $a$ ,  $b$ ,  $c$ , and  $d$ . We solve the 15 equations by using 1, 2, 3 or 4 coefficients. Having obtained  $a$ ,  $b$ ,  $c$  and  $d$ , each with its error  $da$ ,  $db$ ,  $dc$ , and  $dd$ , we obtain six values of the evaluated surface temperature  $Te$  from Eq. (4) at the six wavelengths:

$$1/Te = -1/Tr_m - a\lambda_m - b\lambda_m^2 - c\lambda_m^3 - d\lambda_m^4 \quad (8)$$

The evaluation of  $Te$  is carried out with 1, 2, 3 and 4 coefficients in Eq. (8). Each value of  $Te$  has an error, originating from the errors in  $a$ ,  $b$ , etc. We average the six values of  $Te$  and obtain the standard deviation, which we designate as  $dTe$ . The average of the errors in the various values of  $Te$  (due to the errors in  $a$ ,  $b$ , etc.) is defined as  $d'Te$ .

We do not know, how many coefficients are needed in Eq. (3) to represent  $(\ln\epsilon)/c_2$ ; thus we leave the decision to the six calculated values of  $Te$  and  $dTe$  as the weighting factor. The quality of the data can be detected by inspecting the  $Te$ 's with various numbers of coefficients. The less  $Te$  varies with the number of coefficients, the better the measurements.

We also carried out a linear extrapolation of the radiance temperature  $Tr$  assuming a linear function of  $Tr$  versus wavelength  $\lambda$ ,

$$Tr = Tlin + h\lambda. \quad (9)$$

## MATERIAL CHARACTERIZATION

Two types of materials have been investigated: a two dimensional (2D-SiC/SiC) and a three dimensional (3D-C/SiC) composite. The samples were manufactured by SEP, Bordeaux (Chateigner, 1990) and are characterized as follows:

a) 2D-SiC/SiC composite:

The reinforcement is an arrangement of 2D laminates. The weaves are made with silicon carbide fibers at a volume fraction of about 40%.

The silicon carbide matrix is deposited during a chemical vapour infiltration (CVI) process, and a final density of about  $2.5 \text{ g/cm}^3$  is achieved. The residual porosity volume fraction is about 10%. The thermal conductivity  $k$  depends on the cloth plane orientation.

The used sample designations are:

2D-SiC/SiC P1 (cloth plane parallel to the surface,  $k = 0.06 \text{ W/m K}$ )

2D-SiC(SiC N1 (cloth plane perpendicular to the surface),  $k = 0.12 \text{ W/m K}$ )

b) 3D-carbon/SiC composite:

The reinforcement is a three-directional structure, named NOVOLTEX (R) (SEP'S patent) made with PAN precursor carbon fibers.

The total fiber volume fraction is about 25 to 30%, with less than 10% in the third direction. In the main plane, a special arrangement gives this material a quasi isotropic behaviour.

A silicon carbide matrix is deposited by the same CVI process as for the 2D-SiC/SiC composite and a final density of about  $2.2 \text{ g/cm}^3$  was reached. The residual porosity volume fraction is quite in the same range.

The used sample designations are:

3D-C/SiC P6 (cloth plane parallel to the surface),  $k = 0.115 \text{ W/m K}$

3D-C/SiC N2 (cloth plane perpendicular to the surface),  $k = 0.152 \text{ W/m K}$

## MEASUREMENT RESULTS

The results of the total emittance measurements are plotted in Fig. 3 and the spectral emittance results are listed in Tab. 1. The spectral emittance values in Tab. 1 are given for different averaged surface temperatures  $T_o$ , calculated from the temperature measured in the blackbody cavity. In Tab. 2 the results of the multispectral evaluations are presented.  $T_e$  and  $T_{lin}$  are the surface temperatures calculated from the measured radiance temperatures corresponding to Eqs. (8) and (9), respectively. For the sake of comparison, the emittance resulting from the two different evaluation methods are plotted in Fig. 4 for a few selected surface temperatures  $T_o$ . The values of the blackbody hole method are represented by full lines and the values evaluated by means of the six wavelength method are given by broken lines.

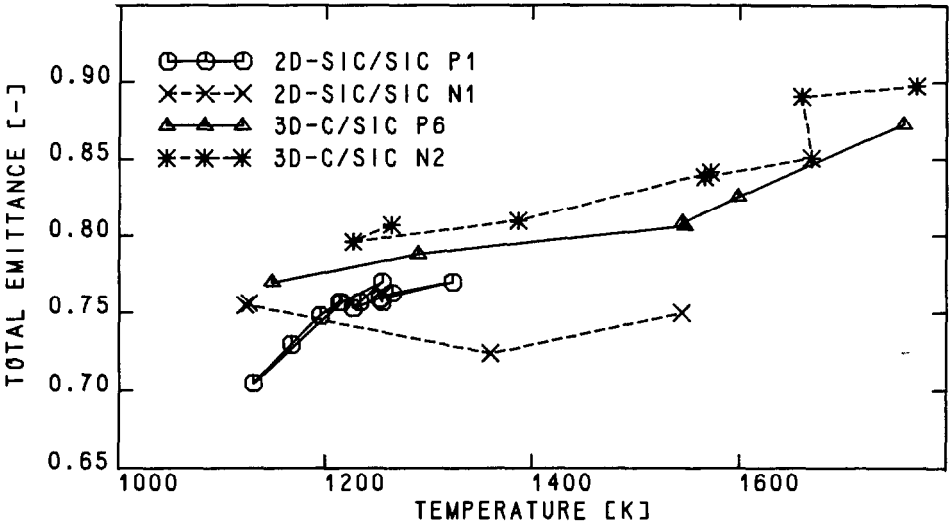


Fig. 3 Total normal emittance of SiC/SiC and C/SiC-composites with the cloth parallel to the surface (P1, P6) or perpendicular to the surface (N1, N2)

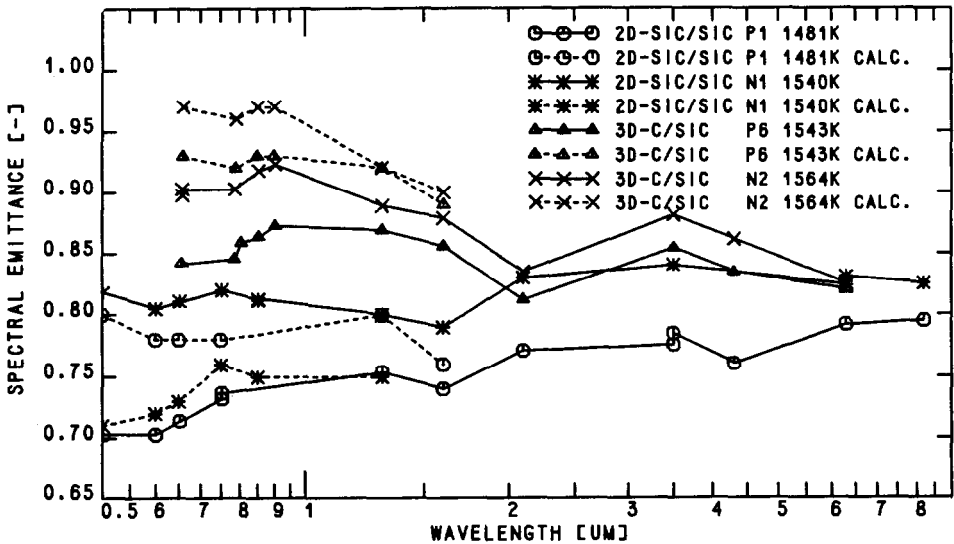


Fig. 4 Spectral emittance of various composites at similar temperatures. The dashed lines represent values evaluated by means of the six wavelength method



**TABLE 1**  
Spectral emittance of various composites, at various surface temperatures  $T_o$

Sample	2D-SiC/SiC P1		2D-SiC/SiC N1			3D-C/SiC P6		
	$T_o$ [K]		$T_o$ [K]			$T_o$ [K]		
$\lambda$ [ $\mu$ m]	1330	1481	1123	1344	1540	$\lambda$ [ $\mu$ m]	1156	1271
0.50	0.77	0.70	-	0.92	0.82	0.66	0.76	0.80
0.60	0.69	0.70	0.95	0.88	0.81	0.79	0.75	0.79
0.65	0.76	0.71	0.92	0.88	0.81	0.80	0.80	0.82
0.75	0.77	0.73	0.90	0.88	0.82	0.85	0.83	0.83
0.85	0.77	-	0.88	0.86	0.81	0.90	0.81	0.83
1.30	0.76	0.75	0.83	0.76	0.80	1.30	0.76	0.84
1.60	0.70	0.74	0.79	0.74	0.79	1.60	-	0.81
2.10	0.75	0.77	0.80	0.77	0.83	2.10	0.81	0.82
3.50	0.77	0.78	0.80	0.79	0.84	3.50	0.80	0.81
4.30	0.77	0.76	0.83	0.81	-	4.30	0.79	0.82
6.30	0.80	0.79	0.85	0.82	0.83	6.30	0.84	0.83

Sample:	3D-C/SiC P6		3D-C/SiC N2				
$\lambda$ [ $\mu$ m]	$T_o$ [K]		$T_o$ [K]				
	1543	1754	1207	1380	1564	1666	1764
0.66	0.84	0.94	0.79	0.88	0.90	0.94	0.95
0.79	0.85	0.90	0.79	0.86	0.90	0.94	0.95
0.80	0.86	0.89	0.82	0.88	0.91	0.94	0.95
0.85	0.86	0.89	0.82	0.90	0.92	0.94	0.96
0.90	0.87	0.91	0.84	0.90	0.92	0.95	0.96
1.30	0.87	0.85	0.81	0.87	0.89	0.90	0.87
1.60	0.86	0.85	0.82	0.87	0.88	0.88	0.87
2.10	0.81	0.82	0.82	0.84	0.84	0.83	0.84
3.50	0.85	0.86	0.83	0.84	0.88	0.88	0.82
4.30	0.83	0.89	0.81	0.84	0.86	0.90	0.91
6.30	0.82	0.82	0.84	0.83	0.82	0.82	0.83

A systematic deviation on the material or cloth plane orientation cannot be observed. Differences in the level of the emittance-temperature curves can also be attributed to differences in the surface roughness which was not determined.

If we compare the spectral emittance, versus wavelength at different temperatures we can find some differences between the individual samples. The level of the values is slightly higher for the C/SiC-samples and if we compare different materials at the same temperature, we find the spectral emittance to be relatively constant with wavelength. As mentioned above, the measurement accuracy in the visible spectral range is very sensible to the determined surface temperature. Therefore it cannot be clearly stated whether the scatter of the emittance at short wavelengths has to be explained by material characterization or measurement uncertainties.

TABLE 2

Surface temperatures and spectral emittance values resulting from the six-wavelength evaluation

Sample	2D-SiC/SiC P1		2D-SiC/SiC N1			3D-C/SiC P6		
	To[K] Te[K] (Tlin)[K]		To[K] Te[K] (Tlin)[K]	To[K] Te[K] (Tlin)[K]		To[K] Te[K] (Tlin)[K]		
$\lambda$ [ m]	1330 1351 (1327)	1481 1470 (1475)	1123 1139 (1143)	1344 1354 (1366)	1540 1550 (1544)	$\lambda$ [ m]	1156 1140 (1152)	1271 1254 (1272)
0.50	0.56	0.80		0.79	0.71	0.66	0.95	0.98
0.60	0.52	0.78	0.71	0.78	0.72	0.79	0.92	0.94
0.65	0.58	0.78	0.70	0.78	0.73	0.80	-	-
0.75	0.60	0.78	0.71	0.79	0.76	0.85	0.95	0.98
0.85	0.63	-	0.71	0.78	0.75	0.90	0.96	0.98
1.3	-	0.80	0.71	0.71	0.75	1.3	0.87	0.92
1.6	-	0.76	0.70			1.6	0.88	0.89

Sample	3D-C/SiC P6		3D-C/SiC N2				
	To[K] Te[K] (Tlin)[K]		To[K] Te[K] (Tlin)[K]	To[K] Te[K] (Tlin)[K]	To[K] Te[K] (Tlin)[K]	To[K] Te[K] (Tlin)[K]	To[K] Te[K] (Tlin)[K]
$\lambda$ [ m]	1543 1532 (1541)	1754 1776 (1787)	1207 1195 (1206)	1380 1371 (1386)	1564 1555 (1572)	1666 1653 (1684)	1764 1748 (1804)
0.66	0.93	0.85	0.95	0.98	0.97	1.04	1.05
0.79	0.92	0.79	0.92	0.94	0.96	1.02	1.04
0.80	-	-	-	-	-	-	-
0.85	0.93	0.79	0.95	0.98	0.97	1.02	1.04
0.90	0.93	0.81	0.96	0.98	0.97	1.02	1.04
1.3	0.92	0.77	0.87	0.92	0.92	0.94	0.91
1.6	0.89	0.77	0.88	0.89	0.90	0.92	0.87

If we compare the two evaluation methods we find differences up to 20 % between both. The values of the six wavelength evaluation are partly lower and partly higher and the variation of the emittance with wavelength is not always identical for both methods. The discrepancies are larger than the inprecision caused by thermal conductivity uncertainties, even for the samples with the lower thermal conductivity.

## CONCLUSION

The emittance values measured with the different samples are all in the range 0.7 to 0.9. No systematic dependency on fiber material or orientation could be observed. Differences in the level of the emittance-temperature curves can also be attributed to differences in the surface roughness which were not determined.

By comparing the emittance values evaluated with the surface temperature  $T_o$  calculated from the radial hole temperature (Tab. 1), with the ones evaluated with the surface temperature  $T_e$  determined from the measured radiance temperatures by means of the six wavelength theory, (Tab. 2) we can state:

- The evaluated temperature  $T_e$  is in a relatively good agreement with  $T_o$ . The deviations are in the range 1 to 2%.
- The temperature  $T_{lin}$ , found by linear extrapolation of the measured radiance temperatures to zero wavelength is generally very close to  $T_e$ .
- The emittance values show disagreements of up to 20% whereby the variation of the emittance with temperature or wavelength is partly different for both kinds of evaluation. Therefore it must be stated that multispectral evaluation is not useful for precise emittance measurements and should only be applied if - e.g. because of difficulties at surface temperature measurement - the inaccuracy limit is restricted to values above 15 %.

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