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2012 Metrologia 49 S81

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BRDF measurements of graphite used in high-temperature fixed point blackbody radiators: a multi-angle study at 405 nm and 658 nm

Heather J Patrick¹, Leonard M Hanssen¹, Jinan Zeng² and Thomas A Germer¹

¹ National Institute of Standards and Technology, Gaithersburg, MD 20899, USA

² Space Dynamics Laboratory, Utah State University, North Logan, UT 84341, USA

E-mail: heather.patrick@nist.gov

Received 16 September 2011, in final form 10 November 2011

Published 2 March 2012

Online at stacks.iop.org/Met/49/S81

Abstract

We have measured the polarization-resolved bidirectional reflectance distribution function (BRDF) for two types of graphite used in the fabrication of high-temperature fixed point blackbody cavities in and out of the plane of incidence. Measurements were made at room temperature using 405 nm and 658 nm laser sources, and the samples were illuminated at angles of incidence varying from normal incidence to 70°. All of the samples exhibited non-Lambertian behaviour, including enhanced forward scatter at high incident angles, especially for s-polarized incident light. The directional-hemispherical reflectance for unpolarized incident light, obtained by integrating the BRDF measured at individual points in a hemisphere over the sample, ranged from 0.083 to 0.101, depending upon sample and incident angle. The potential impact of these measurements on emissivity models for graphite blackbody radiators and radiance temperature scale dissemination is discussed.

1. Introduction

The eutectic alloys Co–C, Pt–C and Re–C are being considered as reference points for the dissemination of temperature at 1597 K, 2011 K and 2747 K, respectively [1]. A critical issue in their use is the knowledge of the emissivity of the graphite blackbody radiator cavities used in the measurement. Modelling of the emissivity requires characterization of the reflectance of the graphite walls of the cavity, ideally as a function of incident angle, scattering angle, polarization, wavelength and temperature. The wall reflectance is described by the bidirectional reflectance distribution function (BRDF). The cavity wall reflectance is then input to Monte Carlo-based ray tracing models to obtain the effective emissivity. The BRDF is typically approximated either as Lambertian, or as a generalized specular plus diffuse (GSD) function, because the full in-plane and out-of-plane BRDF is not well known [2, 3]. Past studies of a group of the current authors have focused on in-plane measurements of the BRDF, and

upon directional-hemispherical measurements of the diffuse reflectance using an integrating sphere, but did not measure the distribution of reflectance over the full hemisphere, including out-of-plane directions [4]. These studies, which focused on the infrared optical properties of the graphite, found that the reflectance of graphite samples varied with surface roughness and wavelength. For example, at 2 µm wavelength (the shortest wavelength for which directional-hemispherical reflectance (DHR) measurements were made in that work), the DHR varied from less than 0.1, for the sample with the roughest surface, to >0.3, for a glossy surface. In addition, the in-plane BRDF revealed non-Lambertian behaviour and an increasing specular reflection at longer infrared wavelengths. In this work, we investigate the BRDF at visible wavelengths, close to the common operating wavelengths of 400 nm and 650 nm used in filter radiometers, and measure the in-plane and out-of-plane BRDF over a hemisphere for a range of incident angles, to get more complete data for input to emissivity models. The measurements were made using the NIST Goniometric Optical

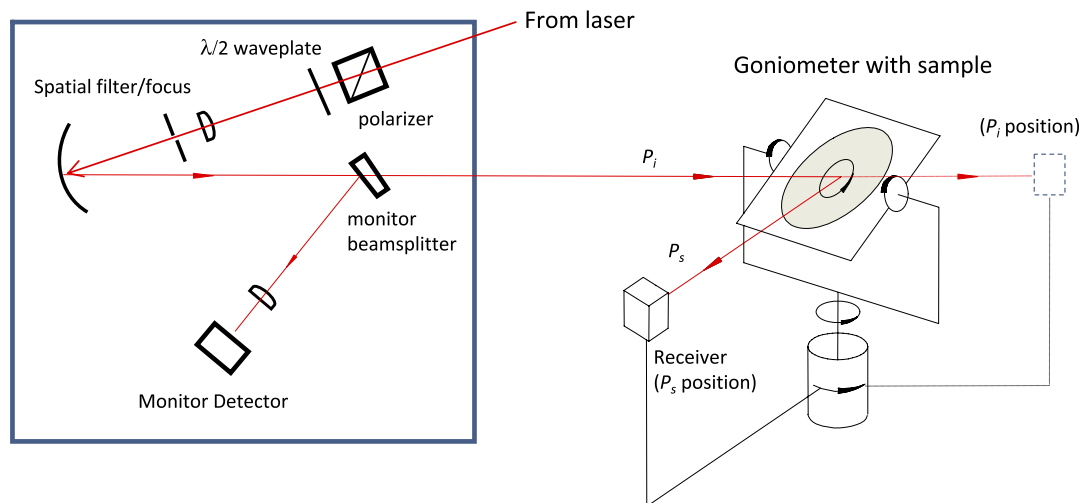


Figure 1. Layout of GOSI (the goniometric optical scatter instrument). On the left are beam conditioning optics and power monitor. The right shows the goniometer, which holds the sample, and also the receiver, which is used to measure the incident and scattered power.

Scatter Instrument (GOSI), which is capable of measuring BRDF over a full hemisphere using laser sources [5]. The results of this study will be used to guide further emissivity model development, and where possible, as inputs to current emissivity models to better estimate cavity contributions to the uncertainty in radiation thermometry.

2. Operation of the goniometric optical scatter instrument (GOSI)

BRDF measurements were made using GOSI (see figure 1) with two laser sources: one at 405 nm wavelength and the other at 658 nm. GOSI uses near-collimated laser light to illuminate a sample at a selectable angle of incidence (θ_i). A multi-axis goniometer holds the sample, and, along with a rotating detector arm, allows the scattered light to be measured at any polar and azimuthal scattering direction (θ_r and ϕ_r) in the hemisphere above the sample, except for cases where the detector blocks the incident light (near retro-reflection) and for some azimuthal cases with large incident and viewing angles (θ_i or $\theta_r > 75^\circ$) where light is blocked by the frame of the goniometer. Figure 2 shows the coordinate system with the conventions for θ_i , θ_r and ϕ_r used in BRDF measurements. The size of the incident laser spot on the sample at normal incidence was approximately 3 mm wide $\times$ 4 mm high for the 405 nm laser, and 4 mm wide $\times$ 5 mm high for the 658 nm laser. The beam is wider when $\theta_i \neq 0^\circ$, up to a maximum expansion of 2.9 times for $\theta_i = 70^\circ$, but was always small enough to underfill the sample. The receiver includes a precision aperture, a lens and a field stop, and images an area of the sample larger than the incident laser spot to a detector consisting of a silicon photodiode on an integrating sphere. When viewing the sample, the receiver collects all of the scattered power P_s in the solid angle defined by the aperture area A and the sample to aperture distance D . The receiver can also be positioned opposite the laser with the sample removed, in order to measure the incident power P_i . The BRDF, f_r , at a given θ_i , θ_r and ϕ_r

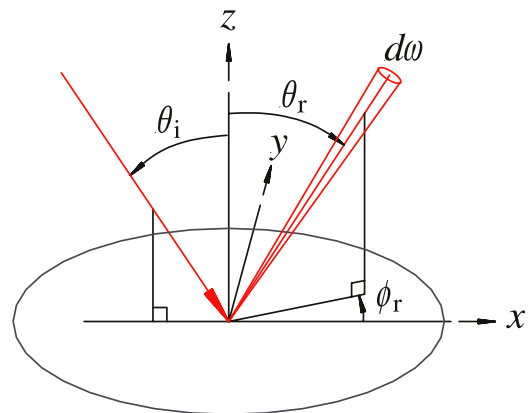


Figure 2. Coordinate system for BRDF measurements. The direction z is the normal to the sample, and the plane of incidence is the x - z plane. The incident polar angle θ_i and polar scattering angle θ_r are measured from the surface normal (z -direction). The azimuthal or out-of-plane scattering angle ϕ_r is measured from the x direction. The solid angle $d\omega$ indicates a cone of scattered rays centred on the θ_r , ϕ_r scattering direction.

is calculated from

$$f_r = \frac{P_s D^2}{P_i A \cos \theta_r}. \quad (1)$$

GOSI is fitted with multiple apertures; for this work, a 7 mm diameter aperture was used, which subtended a roughly 0.7° collection angle at the sample to aperture distance of 587 mm.

The BRDF is also a function of polarization and, if needed, GOSI can be configured for Mueller matrix polarimetry by installing polarization optics on the source and receiver. For the current work, we did not perform full polarimetric measurements, but instead used linearly polarized incident light and no polarization optics in the receiver. At each θ_i , θ_r and ϕ_r combination, two values of f_r were measured: one for s-polarization incident light (incident polarization as defined by the electric field perpendicular to the plane of incidence) and the other for p-polarization incident light (incident polarization in the plane of incidence). The polarization was varied using

a half-wave plate (see figure 1). For in-plane measurements, the sample is held vertically upright in the goniometer and rotated about a vertical axis, so that s-polarization corresponds to vertically polarized light, and p-polarization to horizontally polarized. For out-of-plane measurements, because the source laser direction is fixed while the sample orientation and detector position are varied to achieve the desired incident and scattering angles, obtaining s- and p-polarization relative to the sample plane of incidence requires rotating the incident polarization to angles that are not necessarily horizontal or vertical relative to the laboratory frame. The effect of this on normalization of the measurements of P_i and P_s using the monitor detector is discussed next.

3. Monitor detector and characterization of P_i

Because measuring BRDF over a hemisphere involves collection of many points (for example, a typical measurement with fixed θ_i and hemispherically scanned θ_r and ϕ_r required 305 scatter measurements at each polarization) we do not wish to measure the incident power P_i before every scattering measurement P_s . Instead, the incident power is measured relative to a monitor signal (see figure 1) derived by picking off a small portion of the incident beam immediately before the sample. In this way, P_i can be measured once at the beginning of a measurement sequence, or even daily, and so long as subsequent scatter measurements are also normalized to the monitor, any fluctuations in the source power will be cancelled out. The monitor method works very well. For fixed incident polarization we find that the monitor-normalized P_i is stable to within 0.05% peak-to-peak over several days at a time, even though the fractional change in the laser power may be several per cent.

A complication in the referencing scheme arises when the incident polarization is varied, however. Because the monitor beamsplitter is an angled fused silica window, the fraction of light sent to the monitor is polarization dependent. Figure 3 shows the ratio of receiver current (receiver in the incident position of figure 1) to monitor current as a function of polarization angle ζ , where $\zeta = 0^\circ$, $\zeta = 180^\circ$, etc corresponds to horizontal linear polarization in the laboratory frame, and $\zeta = 90^\circ$ and $\zeta = -90^\circ$ to vertical linear polarization. (Note that while the monitor beamsplitter reflects about 4% of the light on the first surface, and a small amount on the anti-reflection coated second surface, the ratio of receiver to monitor is not 1/0.04 because the integrating spheres used in the receiver and monitor have different efficiencies.) A fit to a cosine function is also shown. In practice, the receiver-to-monitor ratio was measured as a function of polarization angle, fitted to a cosine function, and the fit function used to calculate the value of the monitor ratio that should be used during a subsequent measurement of P_s requiring a given polarization angle. The fit function was not perfect, but we estimate the error introduced using it to be no more than $\pm 0.2\%$ over the range of polarization angles used in the measurements.

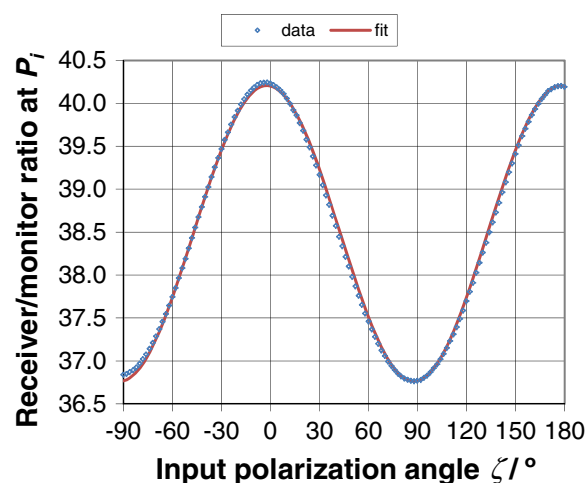


Figure 3. Ratio of detector current on receiver photodiode to that on the monitor photodiode as a function of input polarization angle, when the receiver was positioned for P_i measurement.

4. Measurements and data analysis

BRDF measurements were made using in-plane and hemispherically scanned out-of-plane geometries at the two laser wavelengths of 405 nm and 658 nm. For both in-plane and hemispherically scanned measurements, the incident angle θ_i was fixed while the scattering direction was varied. Four graphite samples, two each of two types of material used in the construction of high-temperature fixed point (HTFP) blackbody cavities, were used in this study. The sample designations were 7ST-1 and 7ST-2, for the first sample type, and SGL-1 and SGL-2, for the second sample type. Samples appeared diffuse and dark grey in colour, with little evidence of specular reflection but with some non-uniformity, such as scratches and tooling marks, on the surface. Some measurements were also made on a pressed polytetrafluoroethylene (PTFE) white reflectance standard [6] and on a white SpectralonTM Note 3 sintered PTFE sample.

For in-plane measurements, the scattering angle θ_r was varied from -80° to 80° in 2° increments, with the convention that negative θ_r corresponds to scattering angles on the incident side of the sample normal ($\phi_r = 180^\circ$). The BRDF at incident angles of 0° , 8° , 15° , 30° , 45° , 60° and 70° was measured for the four graphite samples and the pressed PTFE. To obtain the BRDF for unpolarized incident light, the measured BRDF values for s-polarized and p-polarized incident light at each θ_r were averaged. The round graphite samples had a line and writing across the back that provided a convenient fiducial mark; care was taken that these samples were always mounted into the goniometer in the same rotational orientation, and unless otherwise noted, the incident laser was centred on the sample.

Hemispherically scanned BRDF measurements were made at the same set of incident angles as the in-plane

³ Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

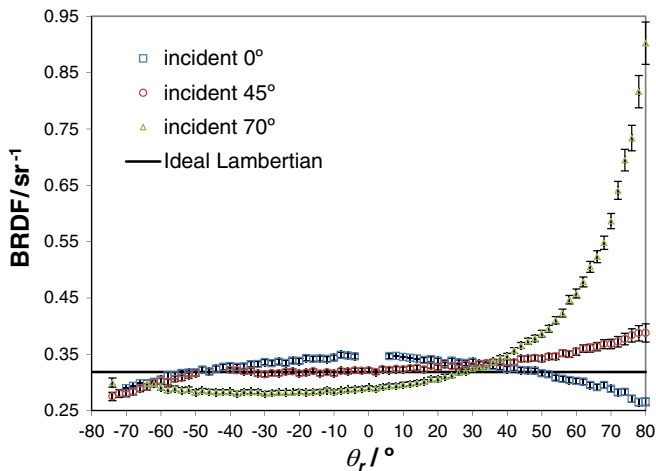


Figure 4. In-plane BRDF of a pressed PTFE sample at three incident angles, shown for unpolarized 405 nm incident light. The expected value for an ideal Lambertian reflector with unit reflectance is also shown.

measurements. Here, the BRDF was measured at points spaced by 0.1 in each direction on an x, y grid, where

$$x = \sin \theta_r \cos \phi_r \quad (2)$$

$$y = \sin \theta_r \sin \phi_r. \quad (3)$$

Regular spacing of the data in x, y coordinates is used to simplify integration of BRDF to the DHR, ρ . ρ is given by [7]

$$\rho = \iint f_r \cos \theta_r \sin \theta_r d\theta_r d\phi_r \quad (4)$$

where the integration is over the hemisphere. Like f_r , ρ is a function of θ_i , polarization and wavelength. By substituting variables to x, y we obtain

$$\rho = \iint f_r dx dy = \frac{\pi \iint f_r dx dy}{\iint dx dy} = \pi \langle f_r \rangle_{xy} \quad (5)$$

where $\langle f_r \rangle_{xy}$ represents the average f_r when sampling evenly on an x, y grid. As mentioned above, at each incident angle, two sets of measurements of f_r were made: one for s-polarized incident light, and the other for p-polarized light. To calculate ρ for unpolarized incident light, we averaged the value of ρ obtained from equation (5) using the data for f_r for s-polarized light with the value of ρ obtained using the data for f_r for p-polarized light.

5. Results: pressed PTFE sample

As part of the testing to verify GOSI's operation, we measured the in-plane and hemispherically scanned out-of-plane BRDF on a sample of pressed PTFE. This material is a standard diffuse reflector with a BRDF that is close to Lambertian, at least for small incident and scattering angles [6]. Figure 4 shows the in-plane BRDF for unpolarized incident light for three values of θ_i . Regions with no data correspond to angles where the receiver blocks the incident beam. At a glance it is clear that even this very good diffuse reflector is not

Lambertian over all incident and viewing angles. For a perfect Lambertian reflector with a reflectance factor of 1, the BRDF should be a constant with the value $1/\pi \text{ sr}^{-1}$, regardless of incident or viewing angle. In practice the BRDF deviates from the ideal, especially for $\theta_i = 70^\circ$ at large forward-scattering angles ($\theta_r > 50^\circ$). This enhanced forward scattering for large incident angles is well known [6, 8]. The estimated expanded uncertainty ($k = 2$) is shown by the error bars in the figure (see section 7). The values of BRDF in figure 4 are in reasonable agreement with previous in-plane BRDF measurements on pressed PTFE [8]. As shown by the error bars in figure 4 and discussed in more detail in section 7, the relative expanded uncertainty in BRDF for the pressed PTFE measurements is $\pm 1.3\%$ at $\theta_r = 0^\circ$ and $\pm 1.8\%$ at $\theta_r = 60^\circ$ and $\theta_r = -60^\circ$. We compared the measurements for $\theta_i = 45^\circ$ and $\theta_i = 0^\circ$ in figure 4 with measurements of BRDF for the same incident angles and the magnitude of θ_r up to 60° given in [8]. For $\theta_i = 0^\circ$ the measurements were in agreement to within their expanded uncertainties. For $\theta_i = 45^\circ$ the measurements were also in agreement, except for $\theta_r \geq 40^\circ$, where there was a fractional difference between our measurements and those of [8] of up to 4%. This may be due to a slight wavelength dependence of the BRDF; the measurements of [8] were made at 633 nm, and our measurements were made at 405 nm. While [8] reports negligible wavelength dependence of in-plane BRDF for pressed PTFE at $0^\circ/45^\circ$ (incident/viewing) and $45^\circ/0^\circ$ geometries in the 400 nm to 600 nm range, wavelength dependence of BRDF has been reported at higher incident and viewing angles [6]. The data of figure 4 also show Helmholtz reciprocity; that is, the BRDF is the same when scattering and incident angles are interchanged. For example, the BRDF for $\theta_i = 0^\circ$ and $\theta_r = 70^\circ$ is the same (within the expanded uncertainty of the measurement) as that obtained for $\theta_i = 70^\circ$ and $\theta_r = 0^\circ$, and similarly for an interchange of incident and scattering angles at 45° .

Figure 5 shows hemispherically scanned BRDF at $\theta_i = 45^\circ$ and a wavelength of 405 nm. While the data in figure 4 are for unpolarized incident light, here we consider the s- and p-polarized components separately. The BRDF is plotted as a projection of the hemisphere into the x, y plane, where x and y are given by equations (2) and (3). The line along $y = 0$ corresponds to in-plane scattering, with $\phi_r = 0^\circ$ being scattering on the specular reflection side of the sample normal, while $\phi_r = 180^\circ$ corresponds to scattering back towards the incident light. The missing data point at $(x, y) = (-0.7, 0)$ is close to the retro-reflection direction, where no light reaches the receiver because the receiver is blocking the incident beam. At any point around the circular plot, the radius from the centre corresponds to $\sin \theta_r$. Because the data were taken on a uniform (x, y) grid with spacing of 0.1, and radius < 1 , the maximum value of x for the in-plane direction is 0.9, which corresponds to $\theta_r = 64^\circ$ on the in-plane plots. As with figure 4, these plots clearly show that the BRDF deviates from the ideal Lambertian case, and this is particularly pronounced when viewing at large values of θ_r (large x) near $y = 0$. The deviation from Lambertian at large θ_r is dominated by enhanced forward scatter for s-polarized incident light. This effect has been discussed in the literature for sintered PTFE [9].

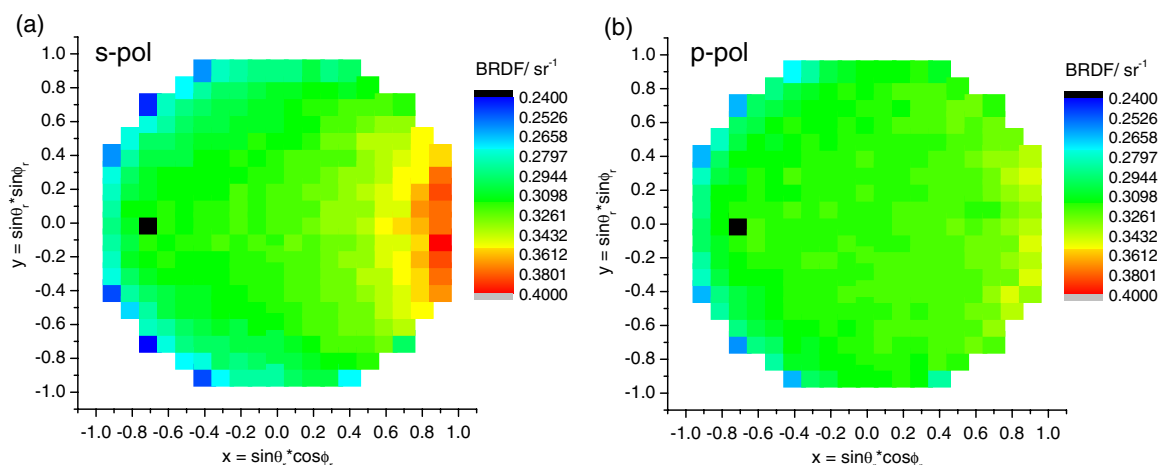


Figure 5. Hemispherically scanned BRDF on pressed PTFE sample, 405 nm light, for (a) s-polarization and (b) p-polarization at an incident angle $\theta_i = 45^\circ$. The same colour scale is used in (a) and (b).

Finally, we calculated the DHR of the pressed PTFE sample for unpolarized incident light using equation (5). For the data shown we obtained $\rho = 0.995$. This is in good agreement with accepted values. Weidner and Hsia [6], for example, report a DHR of 0.996 at 400 nm when $\theta_i = 45^\circ$. The uncertainty in ρ is discussed in more detail in section 7.

6. Results: graphite samples

In-plane and hemispherically scanned out-of-plane BRDF was measured for the four graphite samples, for s- and p-polarization and at both wavelengths, and for the seven incident angles listed in section 4. For brevity we present a subset of this data.

Figure 6 shows in-plane BRDF, for unpolarized light (average of the s and p measurements). Data in figure 6(a) were taken at 405 nm, while those in figure 6(b) were taken using 658 nm light. The same three values of θ_i used in figure 4, which span the minimum and maximum incident angles investigated, are shown, and a single sample of each graphite type (7ST-1 and SGL-1) is represented. While the samples appear diffuse, with no obvious specular features appearing in the data, there is a very strong non-Lambertian dependence of the BRDF on incident angle and scattering angle, far greater than that seen in the pressed PTFE data. While at $\theta_r = 0^\circ$ the BRDF is around 0.02 sr^{-1} to 0.03 sr^{-1} for all θ_i , the BRDF increases dramatically with increasing θ_r for $\theta_i = 70^\circ$, with nearly a $50\times$ increase seen at $\theta_r = 80^\circ$ for the 7ST-1 sample at 658 nm. We also see that while the BRDFs of the 7ST and SGL samples appear similar at $\theta_i = 0^\circ$, they start to deviate for higher incident angles. This was observed for the 7ST-2 and SGL-2 samples as well. While the 7ST-2 and SGL-2 sample BRDFs are not shown in figure 6, data from the 7ST-2 sample (not shown) roughly matched those shown by the 7ST-1 sample (shown), and similarly, SGL-2 (not shown) matched to the shown SGL-1. Where differences in BRDF between two samples of the same graphite type occurred, they were generally smaller than the differences between the two graphite types. It can likewise be seen from figure 6 that the

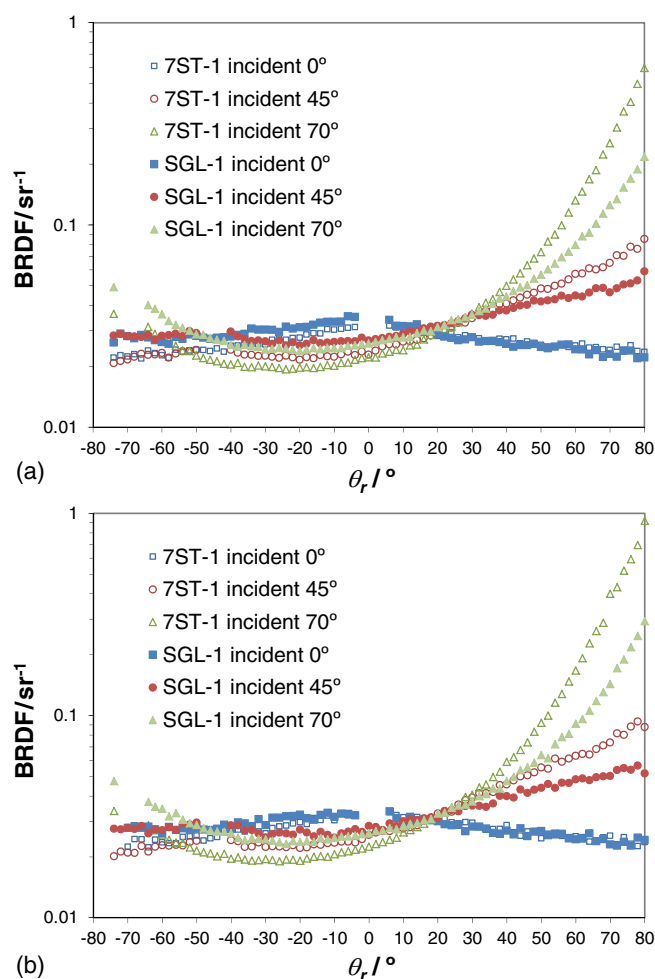


Figure 6. In-plane BRDF of graphite 7ST-1 sample and SGL-1 sample at three incident angles and unpolarized incident light. (a) BRDF at 405 nm. (b) BRDF at 658 nm. Note that BRDF is shown on a log scale.

405 nm and 658 nm BRDF results show very similar trends. The uncertainties in BRDF were larger than those for the pressed PTFE samples. There was a greater speckle noise component seen with the graphite, at about 2.5% ($k = 1$), so

Table 1. Uncertainty budget for BRDF measurements of pressed PTFE and graphite samples.

		PTFE	Graphite
Scattered power	$u(P_s)/P_s$	0.6%	2.5%
Incident power	$u(P_i)/P_i$	0.3%	0.3%
Sample distance	$u(D)/D$	0.04%	0.04%
Aperture area	$u(A)/A$	0.017%	0.017%
Scattering angle ^a	$u(\theta_r)$	0.0035 rad	0.0035 rad
Relative expanded uncertainty ($k = 2$)	$2 \times (u(f_r)/f_r)$ versus magnitude of θ_r :		
	$\theta_r = 0^\circ$	1.3%	5.0%
	$\theta_r = 20^\circ$	1.3%	5.0%
	$\theta_r = 40^\circ$	1.4%	5.1%
	$\theta_r = 60^\circ$	1.8%	5.2%
	$\theta_r = 80^\circ$	4.2%	6.4%

^a For scattering angle, the standard uncertainty, rather than relative standard uncertainty, is reported, for ease of use in equation (6).

that after combining error sources (see section 7 and table 1) the relative expanded uncertainty was up to 6.4% of the BRDF, with largest values at the largest scattering angles. Error bars are not shown in figure 6 because on this scale they are similar to the size of the symbols.

In figure 7, we focus on the hemispherically scanned BRDF for sample 7ST-1 illuminated with 405 nm light. The s-polarized and p-polarized BRDFs are shown separately in the figure. For $\theta_i = 0^\circ$ (figures 7(a) and (b)), although there is no ‘plane of incidence’, we take the convention that s-polarization is vertically polarized light, that is, incident polarization along the y direction in the figure, and p-polarization is horizontally polarized, with incident polarization along the x direction. Because the s- and p-polarization data are shown separately, it can be seen from the $\theta_i = 0^\circ$ plots that the sample has a slight preference to scatter perpendicular to the direction of polarization. Figures 7(c) and (d) show the BRDF at $\theta_i = 45^\circ$, for s- and p-polarization. Separating the results according to polarization shows that the enhanced forward scatter (BRDF at large x) is a feature produced by the s-polarized component of the incident light, but because this scatter is considerably larger than the corresponding scatter produced by the p-polarized component it dominates the unpolarized result. Figure 7(d) also shows asymmetry about $y = 0$, which corresponds to viewing from opposite sides of the plane of incidence. This asymmetry is not completely understood at present and is in contrast to the BRDF of pressed PTFE which showed symmetric BRDF around $y = 0$. However, it was reproducible from day to day and between wavelengths so long as the same area of the sample was illuminated. Asymmetry about $y = 0$ was also seen for some incident angles on three out of four of the graphite samples and was generally more observable for p-polarization. It may indicate a lay to the sample surface due to its processing and/or to surface non-uniformity. Limited testing of hemispherical BRDF versus position on the sample showed that the asymmetry could vary with illuminated position on the sample (see the discussion of figure 10). Figures 7(e) and (f) show the BRDF for $\theta_i = 70^\circ$. Again there was a very strong enhanced forward scattering seen when s-polarized light was incident on the sample.

Figure 8 shows the hemispherically scanned BRDF for the SGL-1 sample at 405 nm, with the same incident angles

shown as in figure 7. In figures 8(a) and (b), we see some polarization dependence of the scattering at $\theta_i = 0^\circ$, although the effect is not as strong as with sample 7ST-1. There is also some asymmetry about both $x = 0$ and $y = 0$ which may indicate a non-uniformity or lay to the sample surface. The $\theta_i = 45^\circ$ data for s-polarization (figure 8(c)) are similar in shape to those seen for the 7ST-1 sample, with a little less forward scatter enhancement, and also a tailing-up of the back-scattered light (around $x = -0.9$) that is not seen for the 7ST-1 sample. The p-polarization data at that incident angle (figure 8(d)) are relatively featureless. At $\theta_i = 70^\circ$, as with 7ST-1 we see a strong enhancement of the forward scatter for s-polarization, although not as pronounced as with the 7ST-1 sample. However, the back-scattered light is more prominent.

As stated earlier, differences between the two samples with the same material designation (i.e. between SGL-1 and SGL-2 or 7ST-1 and 7ST-2) were generally smaller than differences between the BRDF of the two different material types. For 7ST-2 in comparison with 7ST-1, for example, the hemispherically scanned BRDFs are matched in their polarization dependence at 0° , the enhanced forward scattering for the $\theta_i = 45^\circ$ and $\theta_i = 70^\circ$ angles, and the lack of enhanced back-scattering at those angles, as compared with what is seen for SGL-1 in figure 8. However, 7ST-2 did show one peculiar feature compared with the other samples, which was a slightly enhanced scatter around the specular peak, as shown in figure 9. Figure 9(a) shows an enhanced scattering centred upon the specular direction for sample 7ST-2 for the p-polarization BRDF. This feature was reproduced at both wavelengths and was slightly more prominent at 658 nm (not shown) than at 405 nm (shown). It was only seen when plotting p-polarization BRDF for angles of incidence between 8° and 45° . When looking at the unpolarized BRDF result (figure 9(b)), this subtle effect is obscured by the stronger forward scattering of the s-polarized component. The enhanced scattering around the specular direction for p-polarization incident light may be due to a slight difference in surface finish between samples 7ST-2 and 7ST-1, although they were not visibly different. It should be noted that none of the samples investigated (including sample 7ST-2) showed a specular peak significantly above the surrounding BRDF level in the unpolarized BRDF.

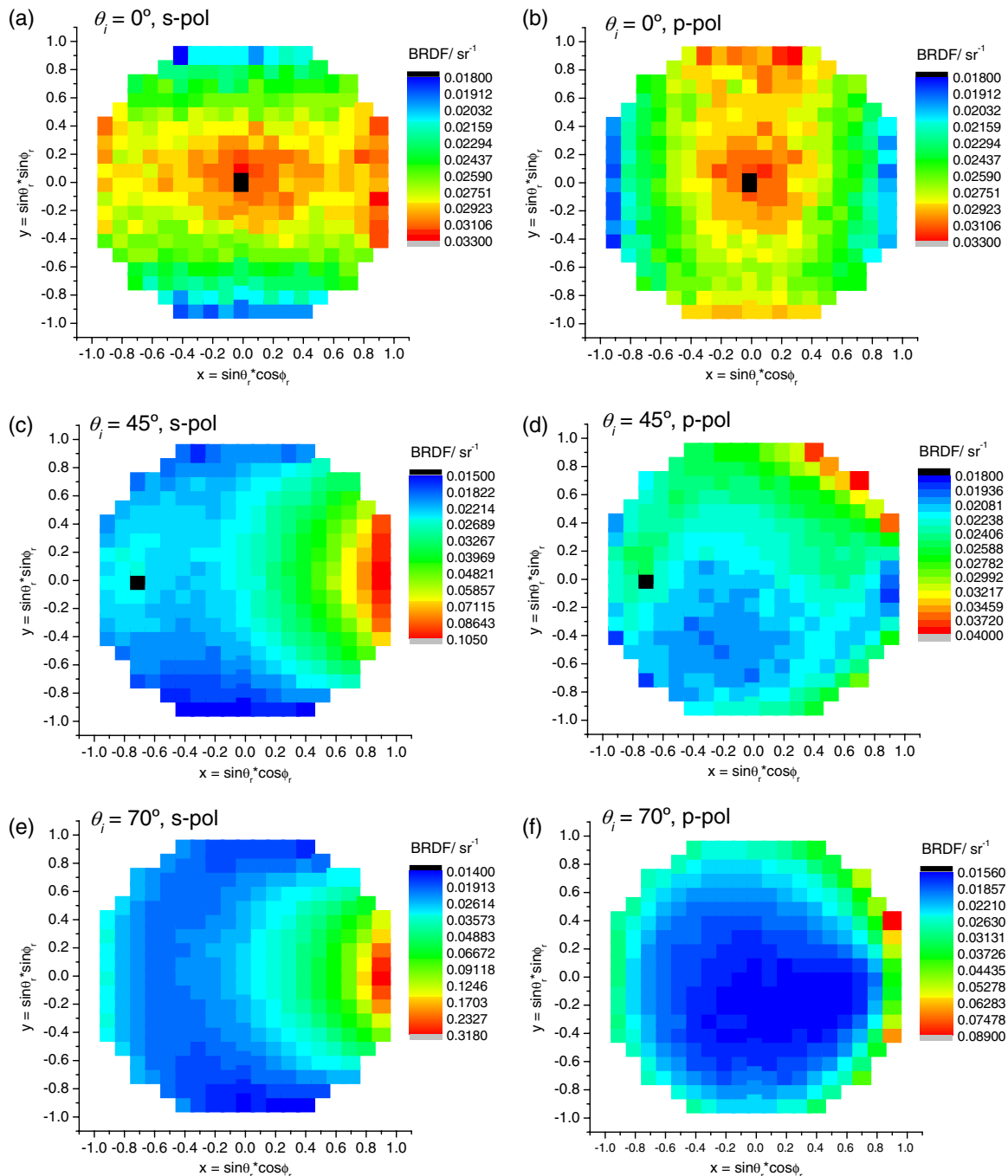


Figure 7. Hemispherically scanned BRDF on 7ST-1 sample, 405 nm light, for s-polarization and p-polarization and at three incident angles. Note the variations in colour scale from plot to plot; except for $\theta_i = 0^\circ$, where the same scale is used for s- and p-polarization, all plots have different colour mapping scales.

In figure 10, we show the calculated DHR, ρ , for all the graphite samples, incident angles and the two wavelengths investigated. We show ρ for s-polarized and p-polarized incident light, and for unpolarized incident light. As seen in the figure, the DHR for unpolarized light ranged from 0.083 to 0.101 for all the samples, incident angles and the two wavelengths used. For comparison, an ideal white Lambertian sample would have a DHR of 1. One somewhat surprising result is that despite the strong enhancement of forward scatter at the highest θ_i , the DHR for unpolarized light does not necessarily increase strongly with θ_i . While the 7ST samples

do show a somewhat increasing DHR with θ_i for unpolarized light, samples SGL-1 and SGL-2 do not show a clear trend in the DHR with θ_i . Continuing with the comparison of sample types for unpolarized incident light, we also see that while the DHRs of the two 7ST samples are not identical, and the two SGL samples are not identical, the DHRs of samples from the same material type follow similar trends with incident angle. Likewise, there is not a strong change in the DHRs between 405 nm and 658 nm; for a given sample and incident angle, the worst case change was 0.007 (for 7ST-1 at $\theta_i = 70^\circ$). Looking at the DHRs for polarized incident light more clearly reveals

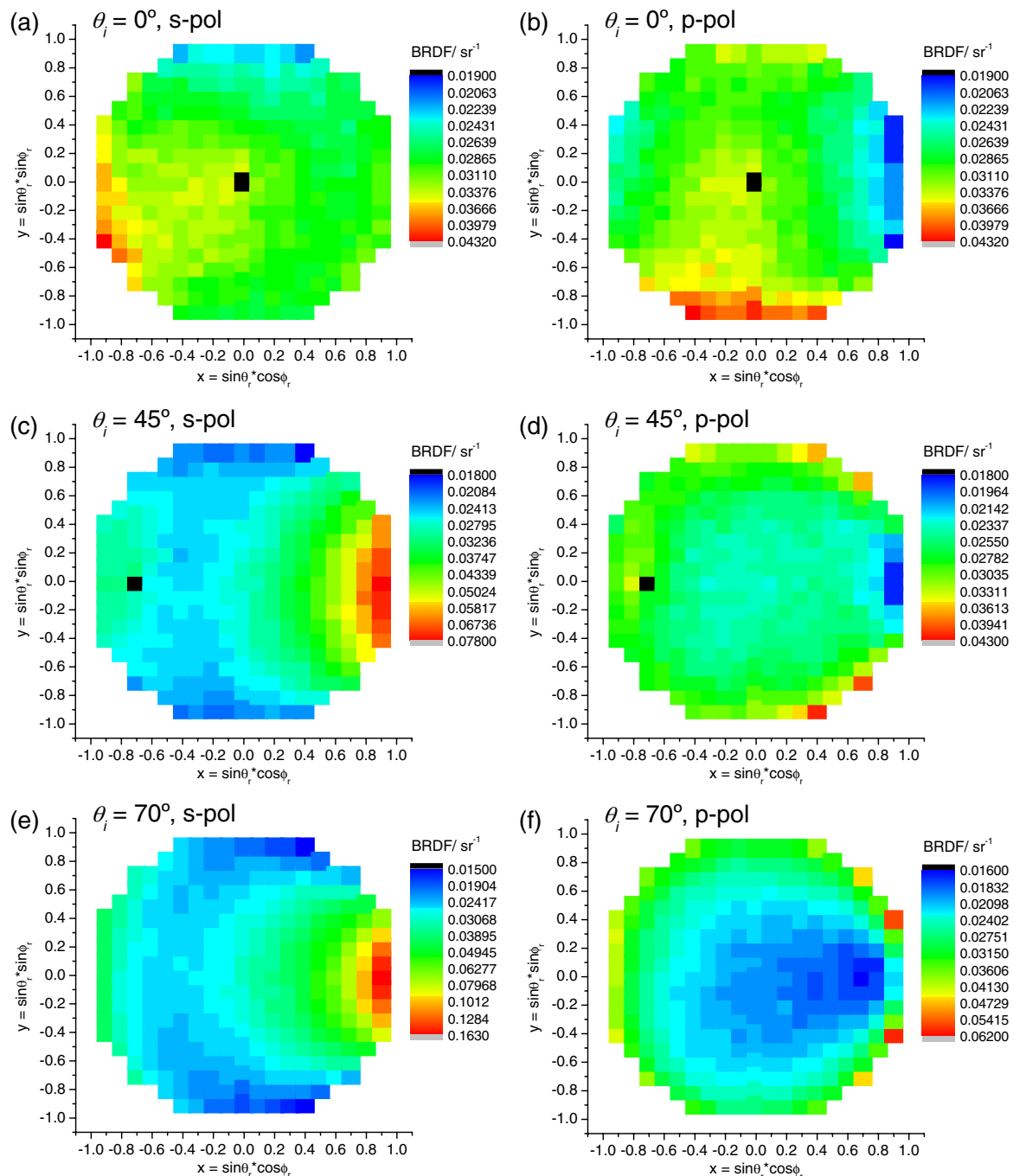


Figure 8. Hemispherically scanned BRDF on SGL-1 sample, 405 nm light, for s-polarization and p-polarization and at three incident angles. Note the variations in colour scale from plot to plot; except for $\theta_i = 0^\circ$, where the same scale is used for s- and p-polarization, all plots have different colour mapping scales.

differences between the two sample types. As θ_i increases, the 7ST samples show an increasing spread in the values of the DHR for s-polarized and p-polarized light. The 7ST-1 sample also showed the largest increase in BRDF with θ_r for non-normal incidence in figure 6, with the increase dominated by the strong forward scattering of s-polarized incident light. This is likely due to differences in surface finish between the 7ST and SGL samples. As mentioned in the introduction, differences in surface finish can cause very large changes in DHR for graphite samples [4].

Finally, we observed that the sample non-uniformity could contribute significant variability to the DHR. In a test where the hemispherically scanned BRDF was measured centred at different spots on the surface of SGL-2 at an incident angle of 0° and a wavelength of 405 nm, then integrated to obtain the DHR for unpolarized incident light, absolute changes of up to 0.005 (about 5% of the DHR) were seen. In contrast, when the same test was performed on a white Spectralon sample (which has very good surface uniformity [8] and similar uncertainty components to the pressed PTFE) with DHR close to 1, the

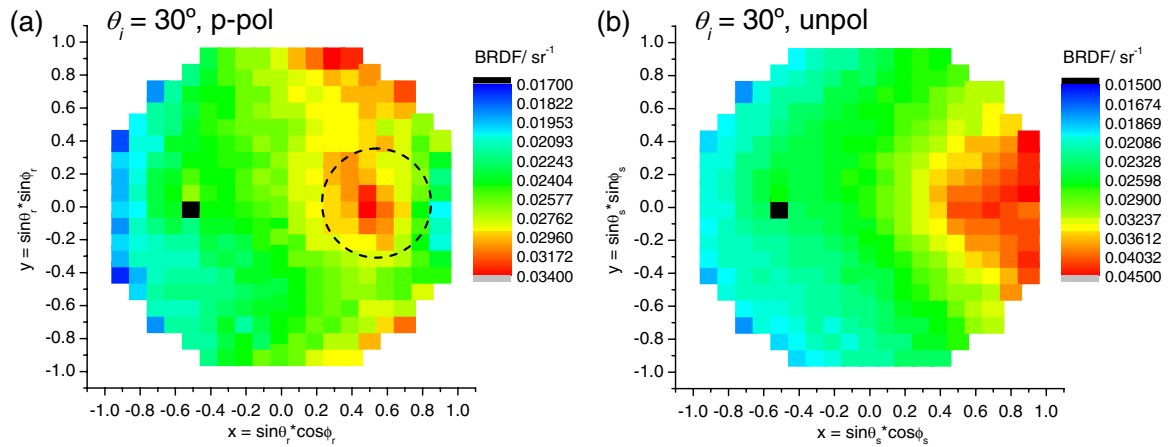


Figure 9. Hemispherically scanned BRDF of graphite 7ST-2 sample at $\theta_i = 30^\circ$ and 405 nm. This sample was unique in that it showed a slightly enhanced scatter about the specular region for p-polarized light at low incident angles. In (a) the p-polarized BRDF is shown, with the enhanced BRDF around the specular region indicated with a dashed circle. In (b) the BRDF for unpolarized incident light is shown.

absolute change in DHR for unpolarized incident light was 0.003, or about 0.3% of the DHR. While these tests were limited in scope, they indicate that surface non-uniformity of graphite should definitely be considered when assigning uncertainties to DHRs input to emissivity models.

7. Uncertainty considerations

Starting from equation (1) for the BRDF, f_r , it can be shown that the standard uncertainty in a BRDF measurement, $u(f_r)$ is found from

$$\frac{u^2(f_r)}{f_r^2} = \frac{u^2(P_s)}{P_s^2} + \frac{u^2(P_i)}{P_i^2} + \left(\frac{2}{D}\right)^2 \cdot u^2(D) + \frac{u^2(A)}{A^2} + (\tan \theta_r)^2 \cdot u^2(\theta_r) \quad (6)$$

where $u(P_s)$ is the standard uncertainty in the scattered power, P_s , $u(P_i)$ is the standard uncertainty in the incident power, P_i , $u(D)$ is the standard uncertainty in the sample to receiver aperture distance, D , $u(A)$ is the standard uncertainty in the detector aperture, A , and $u(\theta_r)$ is the standard uncertainty in the scattering polar angle θ_r . This equation does not include all possible contributions to uncertainty—for example, from a large variation of f_r with incident angle or source wavelength—but provides a good starting point for error analysis. For the purpose of this paper, the error components have been estimated as follows. For the incident power, P_i , we consider both the uncertainty in the incident power seen by the receiver, and the uncertainty inherent in fitting the incident power/monitor ratio (see section 3). Because P_i is known to vary with the exact position that the incident laser enters the aperture and the incident power/monitor ratio was measured at a single point, we include 0.2% uncertainty, combined with the 0.2% uncertainty from fitting the incident/monitor ratio versus polarization, for a total $u(P_i)/P_i$ of 0.3%. The uncertainty in P_s is dominated by laser speckle produced by the interaction of the coherent laser source and the surface roughness and is estimated from the variation of P_s with scattering angle. This was found to be lower for the pressed PTFE samples, at 0.6%, for $u(P_s)/P_s$ whereas the graphite samples had $u(P_s)/P_s =$

2.5%. The lower speckle noise in the pressed PTFE samples is probably due to a combination of the light radiating from a larger area from the PTFE sample (PTFE is somewhat translucent) and the two-dimensional (unpolarized) nature of the speckle pattern from it [10]. We have neglected sources of uncertainty arising from imperfect knowledge of the gain ratios of the receiver and monitor amplifiers, but these have been previously characterized and are expected to be small for the gain ranges used here. The uncertainty in sample to receiver aperture distance, D , was estimated to be $u(D) = 250 \mu\text{m}$ for $D = 588 \text{ mm}$; this could be improved but is generally a small contribution to the overall error. The uncertainty in the aperture area, A , is extremely small with $u(A)/A = 0.017\%$ taken from the aperture's calibration report. The component of uncertainty due to angle $u(\theta_r)$ tends to dominate the uncertainty budget at high scattering angles due to the $\tan(\theta_r)$ term, and unfortunately can be a difficult component to estimate for diffuse samples because the $\theta_i = 0^\circ$ position cannot be found from retro-reflecting the laser as with a specular sample. In practice, we aligned the diffuse samples by first aligning a specular sample in the same sample holder, then replacing the specular sample with the diffuse sample. We roughly estimate $u(\theta_r) = 0.0035 \text{ rad}$ (0.2°) from this procedure. Combining these uncertainty components in equation (6) gives a relative expanded uncertainty ($k = 2$) for PTFE samples ranging from 1.3% at $\theta_r = 0^\circ$ to 4.2% when the magnitude of $\theta_r = 80^\circ$. For graphite, the relative expanded uncertainty ranges from 5.0% at $\theta_r = 0^\circ$ to 6.4% when the magnitude of $\theta_r = 80^\circ$. The uncertainty budget is summarized in table 1.

For the calculation of DHR ρ from the BRDF measurements, we have not undertaken a full uncertainty analysis at this time, but present some of the considerations for uncertainty in ρ . DHR is the total radiant intensity over the hemisphere of the sample, relative to the incident light on the sample. Some BRDF components, like the uncertainty in P_i , represent a systematic offset in the calculated DHR, so that as a rough estimate, a given uncertainty in P_i leads directly to the same uncertainty in DHR. Other BRDF uncertainty components, however, are averaged out in the process of calculating the DHR from the many points taken

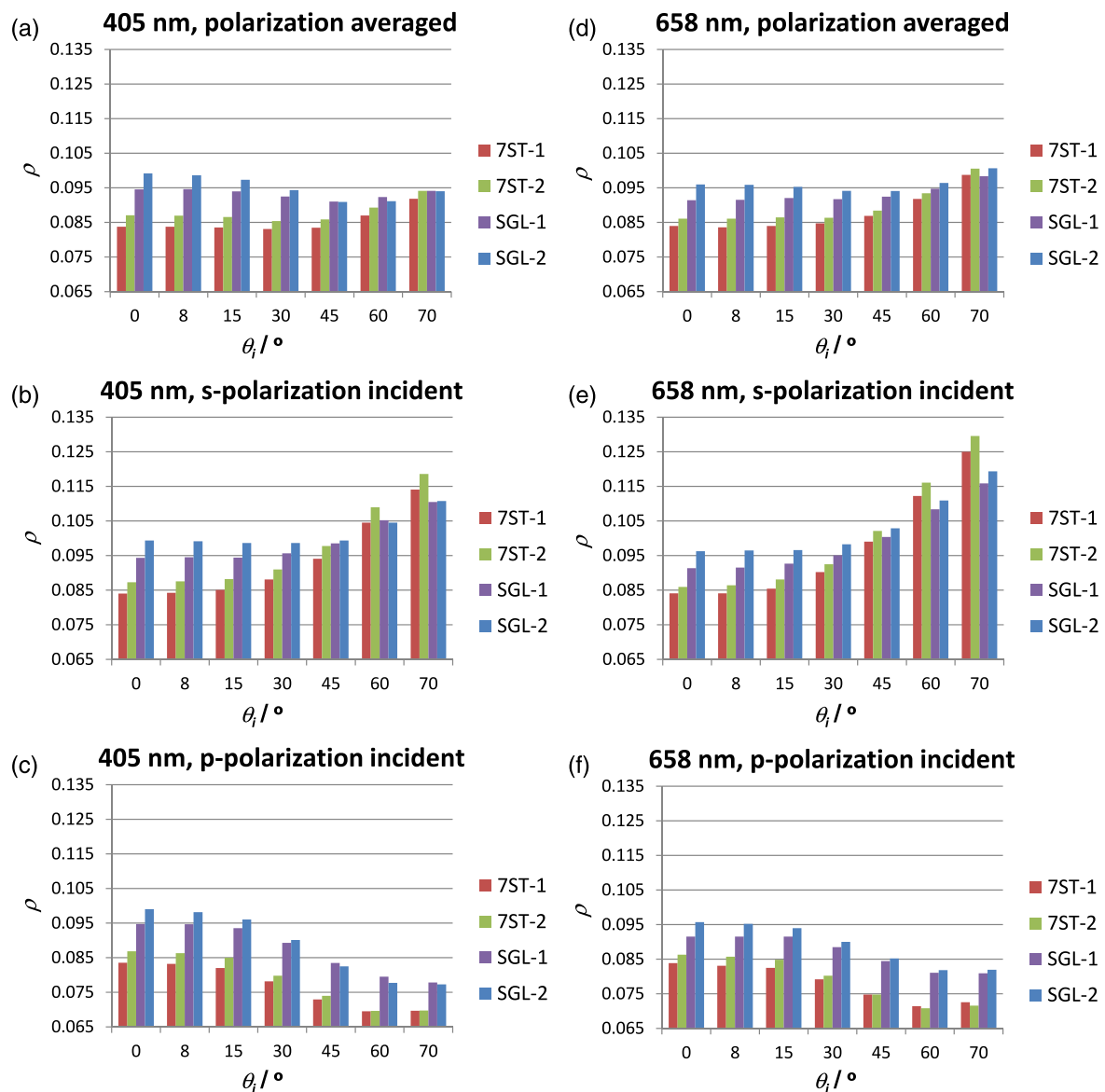


Figure 10. DHR, ρ , for all graphite samples, incident angles, and wavelengths used in the study. Left side (a)–(c): ρ for the seven values of θ_i and 405 nm light. Each colour bar represents a different graphite sample. In (a), the value of ρ for unpolarized incident light is shown. (b) and (c) show ρ for s-polarized and p-polarized incident light, respectively. Right side (d)–(f): same plot series, but for 658 nm light.

on the sphere, such that the contribution to DHR uncertainty is much lower than the uncertainty in the individual BRDF points. The speckle noise, for example, is a random source of error that reduces considerably when averaged for DHR calculations. Angular uncertainty also tends to cancel out, as does uncertainty in positioning the surface sample at the centre of rotation of the goniometer, provided that the distance D used in the calculations is the distance from receiver to goniometer centre such that a proper fraction of a hemispherical surface is calculated. Because speckle noise and angular uncertainty are significant components of the uncertainty in BRDF for the PTFE and graphite samples, and these sources of error are considerably reduced when calculating DHR, it should be possible to attain relative expanded uncertainties in DHR of a few tenths of a per cent based upon the above discussion. Because the graphite

samples exhibit strongly enhanced forward scattering at the largest θ_i , however, we also investigated the question of whether the 0.1 spacing (x, y) grid covers sufficiently large θ_r in the forward-scattering direction. Figure 11 shows the BRDF taken on the (x, y) grid with spacing 0.1, which covers θ_r up to 64° when $y = 0$ (in-plane), and the BRDF measured on an (x, y) grid with spacing 0.05, where the maximum $\theta_r = 71.8^\circ$ for in-plane. The BRDF for unpolarized incident light with $\theta_i = 70^\circ$ at 658 nm on sample 7ST-2 is shown. Top and bottom have the same colour mapping. The few black data points in figure 11(b) are where the light was blocked by either the detector or the goniometer frame, and these points were not included when calculating DHR. When the grid was extended to higher scattering angles, we saw an increase in DHR of about 6%, to 0.0106, for this sample. We also tested the finer grid at lower incident angles (not shown). The increase in

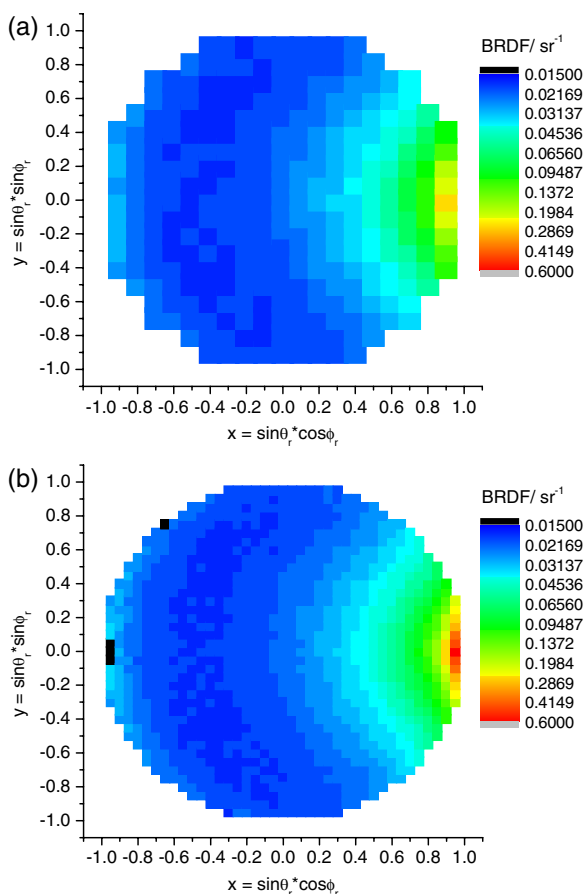


Figure 11. Hemispherically scanned BRDF, unpolarized incident light, 658 nm, $\theta_i = 70^\circ$, sample 7ST-2, measured for (a) 305 points, spaced 0.1 on the (x, y) grid and (b) 1245 points spaced 0.05 on the (x, y) grid. Top and bottom have the same colour mapping.

DHR was largest at $\theta_i = 70^\circ$ and negligible for $\theta_i < 60^\circ$. The increase was also smaller for the SGL samples. We conclude that for highly non-Lambertian samples and incident angles, the BRDF distribution in θ_r and the maximum measured θ_r should be considered when comparing DHR measurements. While it is extremely time-consuming to measure the BRDF on ever-finer grids over the hemisphere, it may be desirable to optimize the grid spacing to get higher resolution information in regions where the BRDF is changing rapidly with angle. Further efforts are needed to establish a full uncertainty budget for DHR, particularly for cases where the BRDF deviates significantly from Lambertian.

8. Discussion and future work

For the graphite samples in this study, significant deviations from Lambertian scattering were found, especially for large incident angles and forward scattering. In typical emissivity models, the BRDF of the graphite is approximated either as Lambertian or as a GSD model. In one study, the different models lead to very different behaviour of the effective emissivity when a known temperature non-uniformity was input along the cavity length [11]. The GSD model adds a specular component, which increases with increasing θ_i , to a

diffuse component. While we did not see a specular peak for unpolarized light for the samples and wavelengths investigated, it may be possible to approximate the measured behaviour using a GSD type of model, where the increasing specular peak stands in for the observed increase in forward scattering at multiple angles. It is also interesting to consider that while the BRDF of the graphite was highly non-Lambertian, there was not a large change in the total DHR with angle; that is, the total reflectance of the surface was not strongly dependent on incident angle, with a range for all the samples and incident angles of 0.083 to 0.101. While it is difficult to predict the effect of the observed BRDF on the Monte Carlo models for cavity emissivity, in one study that employed a Lambertian BRDF model, a change in graphite DHR from 0.2 to 0.3 resulted in cavity emissivity changes on the order of 0.0002, out of a typical 0.9995 emissivity, and temperature changes of around 20 mK [12]. Because preliminary uncertainty evaluations of high-temperature fixed points have source-based contributions ranging from 30 mK to 110 mK [12], this temperature change is significant. The same study included GSD models with varying degrees of specular components, and it may be possible as a first effort to adapt those models to approximate the BRDF behaviour seen here. Finally, because of the sample to sample variability in BRDF and DHR, seen in this study and others [4], and the variability seen even over different areas of the surface of the same sample in this study, future modelling efforts cannot assume fixed, highly accurate values for graphite reflectance. The magnitude of the effect on cavity emissivity from reflectance variability may point to the need for greater characterization and/or standardization of the materials used in the cavities.

In future work, we also hope to compare the DHR measurements integrated from equation (4) to DHR obtained by other techniques. This study was conducted at room temperature, while the cavities will ultimately be operated at the eutectic fixed points. While we cannot perform full hemispherically scanned BRDF at these high temperatures, we do plan to measure the BRDF in-plane using the same laser wavelengths and elevated temperatures. If the accessible points track the room temperature results, we will have more confidence in applying the hemispherically scanned BRDFs measured at room temperature to the elevated temperature cavity emissivity models. We also plan to modify the existing Monte Carlo codes to include the BRDF measurement results, and recalculate the effective cavity emissivities based upon the graphite data presented here. The results of this work will help to guide further development of emissivity models for HFTP cavities, and ultimately enable better determination of the uncertainties inherent in future HFTP temperature standards.

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