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Optics and photonics — Lasers and laser-related equipment — Test methods for specular reflectance and regular transmittance of optical laser components

Optique et photonique — Lasers et équipements associés aux lasers — Méthodes d'essai du facteur de réflexion spéculaire et du facteur de transmission des composants optiques laser

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 13697 was prepared by Technical Committee ISO/TC 172, *Optics and photonics*, Subcommittee SC 9, *Electro-optical systems*.

Introduction

Laser-based optical systems require optical components with greatly enhanced reflectance and/or transmission characteristics. It is necessary to be able to measure these characteristics precisely. The measurement procedures in this International Standard have been optimized to allow the measurement of the specular reflectance and transmittance of the optical components to a high degree of accuracy over a wide range of values.

Optics and photonics — Lasers and laser-related equipment — Test methods for specular reflectance and regular transmittance of optical laser components

1 Scope

This International Standard specifies measurement procedures for the precise determination of the specular reflectance and regular transmittance of optical laser components. The accuracy of the described test methods exceeds that of measurement procedures outlined in ISO 15368 by several orders of magnitude.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 31-6, *Quantities and units — Part 6: Light and related electromagnetic radiations*

ISO 11145, *Optics and photonics — Lasers and laser-related equipment — Vocabulary and symbols*

ISO 14644-1, *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness*

3 Terms and definitions

For the purpose of this document, the terms and definitions given in ISO 11145 and ISO 31-6 apply.

4 Symbols used and units of measure

Table 1 — Symbols used and units of measure

Symbol	Unit	Term
ρ_s		specular reflectance of sample
ρ_{ch}, ρ_m		specular reflectance of chopper, specular reflectance of deflecting mirror
τ_s		regular transmittance of sample
λ	m	wavelength
P_{av}	W	average power
β	rad	angle of incidence
d	m	beam diameter on the test sample
f_{ch}	Hz	chopper frequency
f_{am}	Hz	frequency of laser power modulation
P_r	W	power of reference beam
P_p	W	power of probe beam
ΔP	W	power difference between reference beam and probe beam
S_m		signal at frequency of laser power modulation
S_{mo}		signal at frequency of laser power modulation, probe beam blocked
ΔS		signal at frequency, which is the sum or the difference of chopper frequency and power modulation frequency
ΔS_0		signal at frequency, which is the sum or the difference of chopper frequency and power modulation frequency, probe beam blocked
C_1, C_2		arbitrary constants

5 Test and calibration principles

5.1 General

Specular reflectance and regular transmittance are determined optically as the ratio of the regularly reflected or regularly transmitted part of the reflected or transmitted power radiation to the incident power radiation.

The reflectance or the transmittance of the test sample are constant within the temperature fluctuations experienced by the component during the test and are independent of the power density of the impinging radiation.

5.2 Specular reflectance

The reflectance of optical components is determined optically by means of a measuring arrangement as shown in Figures 1 and 2.

An optically flat and highly reflective chopper mirror divides the laser beam into a probe beam and a reference beam. The probe beam is reflected by the chopper mirror and the sample, whereas the reference beam transmits without being affected. Both beams, alternating temporally, impinge upon the same spot on the rear target of the integrating sphere.

Figure 1 shows the measuring arrangement for near-normal incidence, whereas the angular dependence of reflectance can be measured in a measuring arrangement according to Figure 2. Compared with the arrangement in Figure 1, an additional mirror is used to create a double bounce permitting the measurement of the reflectance of the sample at different angles of incidence.

The powers P_p for the probe beam and P_r for the reference beam are related by

$$P_p = \rho_s^2 \rho_{ch} \rho_m P_r \quad (1)$$

where

ρ_m is the specular reflectance of the additional deflecting mirror;

ρ_s^2 is the double bounce on the sample;

ρ_s is the specular reflectance of the sample;

ρ_{ch} is the specular reflectance of the chopper mirror.

The specular reflectance ρ_s of the test sample can be expressed as

$$\rho_s = \sqrt{\frac{1}{\rho_{ch}\rho_m} \times \left(1 - \frac{\Delta P}{P_r}\right)} \quad (2)$$

where $\Delta P = P_r - P_p$

5.3 Transmittance

The transmittance of optical components is determined by means of a measuring arrangement as shown in Figure 3 using an additional mirror with known reflectance ρ_m.

For the powers P_p and P_r measured with a set-up according to Figure 3, the following relationship exists

$$P_p = \tau_s \rho_{ch} \rho_m P_r \quad (3)$$

where τ_s is the regular transmittance of the sample.

The regular transmittance τ_s of the test sample can be calculated from the following relationship:

$$\tau_s = \frac{1}{\rho_{ch}\rho_m} \times \left(1 - \frac{\Delta P}{P_r}\right) \quad (4)$$

5.4 Calibration

The reflectance of the chopper mirror has to be known for evaluation. Figure 4 shows the measurement set-up used for the calibration procedure. To determine the two unknown specular reflectances ρ_m of the additional mirror and ρ_{ch} of the chopper mirror, two sets of measurements have to be performed. One measurement is done in the set-up of the test procedure described in 8.2, while for the other measurement the integrating sphere and an additional mirror have to be replaced according to Figure 4. The beam transforming optics and the chopper mirror remain unchanged to ensure that the laser hits the chopper under identical conditions. For the set-up according to Figure 4, the following relationship for the powers P_p and P_r exists

$$\frac{P_p}{\rho_m} = \frac{P_r}{\rho_{ch}} \quad (5)$$

This set-up enables the computation of the quotient Q of the specular reflectance of the additional mirror and the chopper mirror.

$$Q = \frac{\rho_{\mathrm{m}}}{\rho_{\mathrm{ch}}} \tag{6}$$

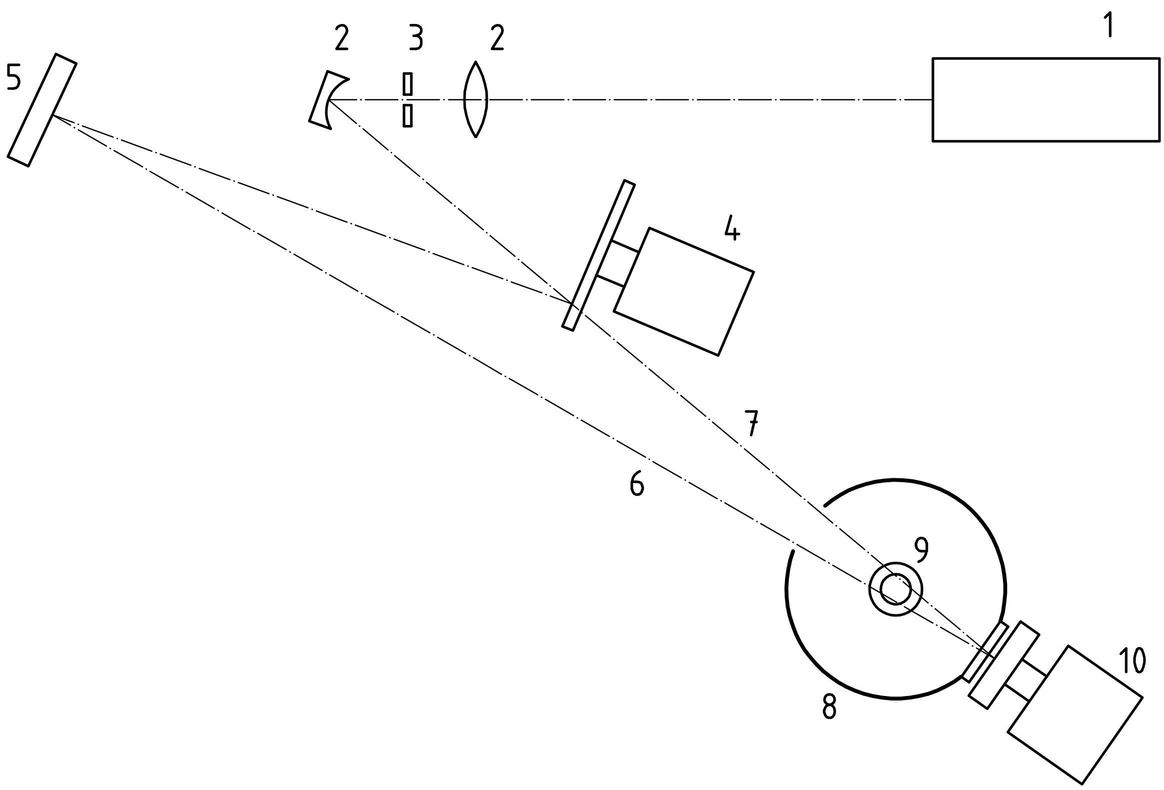
Their product P is determined according to Figure 1, where the sample is replaced by the additional mirror. In this case:

$$P = \rho_{\mathrm{m}} \times \rho_{\mathrm{ch}} \tag{7}$$

The specular reflectance of the chopper ρ_{ch} and the additional mirror ρ_{m} are given by

$$\rho_{\mathrm{ch}} = \sqrt{\frac{P}{Q}} \tag{8}$$

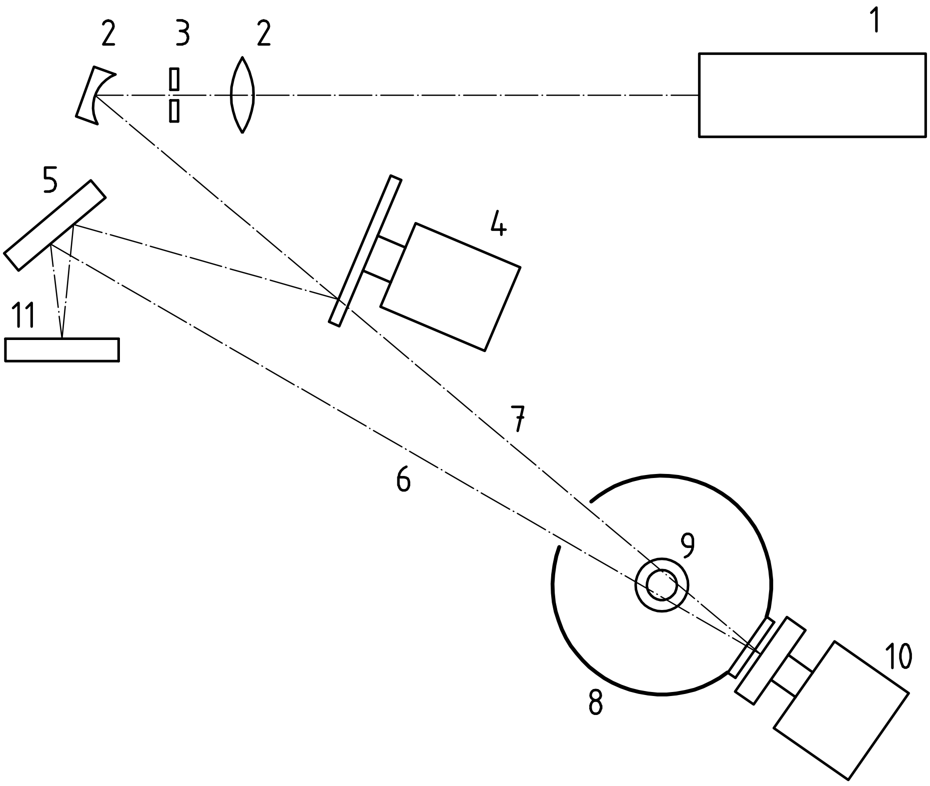
$$\rho_{\mathrm{m}} = \sqrt{P \times Q} \tag{9}$$



Key

1 laser
2 telescope
3 pinhole
4 chopper
5 sample
6 probe beam
7 reference beam
8 integrating sphere
9 detector, mounted on top of the sphere
10 rotating target

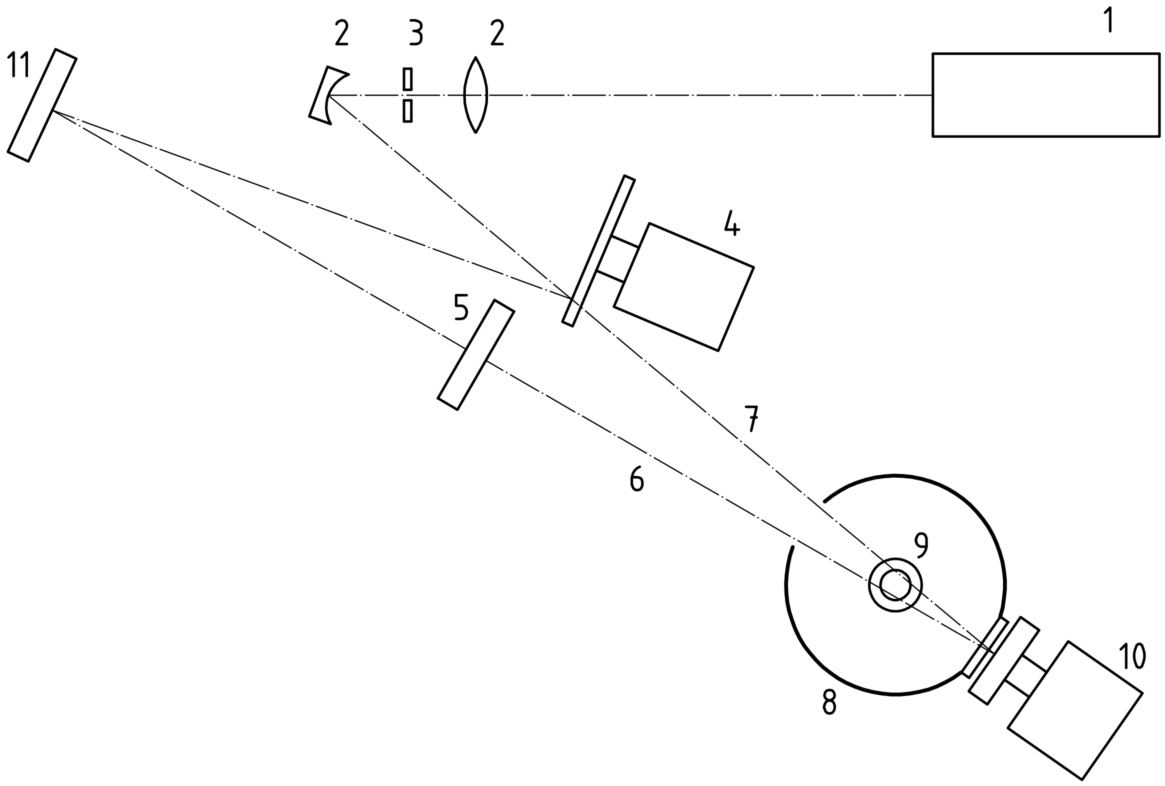
Figure 1 — Schematic measuring arrangement for specular reflectance measurement (near-normal incidence angle on sample)



Key

1 laser
2 telescope
3 pinhole
4 chopper
5 sample
6 probe beam
7 reference beam
8 integrating sphere
9 detector, mounted on top of the sphere
10 rotating target
11 additional mirror

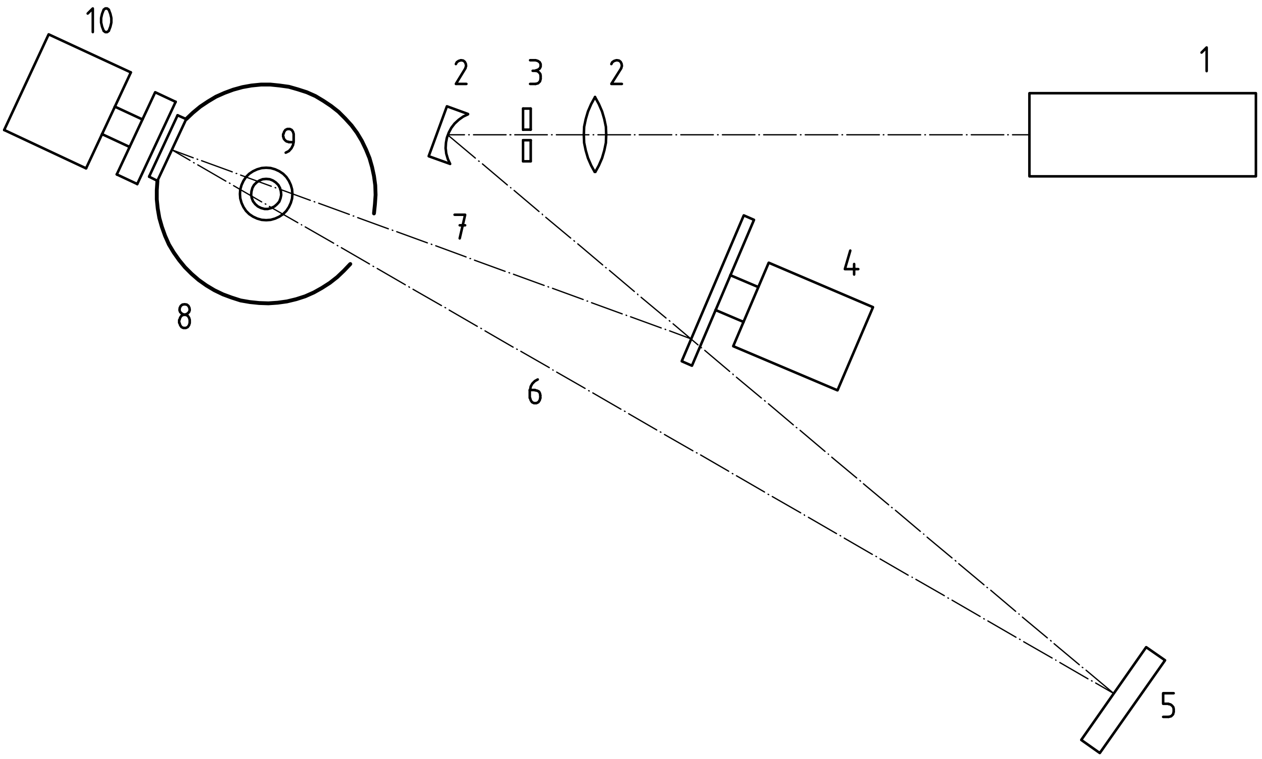
Figure 2 — Schematic measuring arrangement for specular reflectance measurement (arbitrarily chosen angle of incidence on sample)



Key

1 laser
2 telescope
3 pinhole
4 chopper
5 sample
6 probe beam
7 reference beam
8 integrating sphere
9 detector, mounted on top of the sphere
10 rotating target
11 additional mirror

Figure 3 — Schematic measuring arrangement for measuring the transmittance (arbitrarily chosen angle of incidence on sample)



Key

1	laser	6	probe beam
2	telescope	7	reference beam
3	pinhole	8	integrating sphere
4	chopper	9	detector, mounted on top of the sphere
5	additional mirror	10	rotating target

Figure 4 — Schematic measuring arrangement for calibrating the chopper mirror

6 Preparation of test sample and measuring arrangement

6.1 General

Storage, cleaning and the preparation of the test samples shall be carried out in accordance with the manufacturer's instructions for normal use.

The environment of the testing place shall consist of dust-free filtered air with less than 60 % relative humidity. The residual dust shall be reduced in accordance with the clean-room Class 7 as specified in ISO 14644-1.

A laser shall be used as the radiation source. The laser-beam propagation ratio shall be nearly unity and the beam power stability shall be as high as possible.

Wavelength, angle of incidence and state of polarization of the laser irradiation used for the measurement shall correspond to the values specified by the manufacturer for the use of the test sample. If ranges are accepted for these three quantities, any combination of wavelength, angle of incidence and state of polarization may be chosen out of these ranges.

6.2 Laser beam preparation

All stray radiation and radiation scattered from optical components in the beam path has to be separated from the laser beam in order to ensure that a well defined beam enters the measuring sphere either after passing the chopper mirror (reference beam) or after being reflected from the chopper mirror (probe beam). Do this by focussing and recollimating the beam using a spatial filter in the focal plane (beam-transforming optics in Figures 1 to 4). It is recommended that the beam is filtered at least twice and to use only reflective optics after the last spatial filter, in order to minimize scattered radiation. The distance between the last optical component of the beam transforming optics and the rest of the test set-up shall be as large as possible. The beam transforming optics, shown in Figure 1, can be made either using reflecting or transmitting components which permits the optimization of the beam parameters (focus position, diameter, divergence, Rayleigh length). This is of special importance for the 10,6 µm wavelength. For shorter wavelengths the beam transforming optics are not required.

Image the laser out-coupling window onto the entrance port of the integrating sphere. This minimizes diffraction patterns in the aperture plane. A focal point is present near the plane of the chopper mirror and this reduces the effect of the chopper mirror edges passing through the beam. Imaging cannot be done exactly for both the reference and probe beams simultaneously except for the special case where the sample compensates for the increased path length so, in general, a compromise may be required.

To minimize beam-clipping effects at the entrance of the integrating sphere, it is necessary to ensure that the reference and the probe beams have the same beam diameter at the entrance of the sphere and that both beams should enter the sphere with the same displacement from the centre of the entrance port.

6.3 Chopper

The beam propagation ratio and beam-pointing stability of the reflected beam shall not be affected by the chopper mirror during one revolution. Radiation shall not be transmitted through, or scattered by, the chopper mirror.

The application of the lock-in technique with an amplifier locked to the chopper frequency f_{ch} permits the detection of very small differences in power levels that correspond to small differences in reflection. The detection limit is given by the relative intensity noise of the laser source, the ratio of the noise equivalent power to the incident power of the detector at f_{ch}, and the measurement time. Relative intensity noise typically shows a $1/f$-dependence, the chopper frequency shall be as high as possible.

6.4 Detector arrangement

The detector arrangement consists of an integrating sphere with a detector, appropriate for the wavelength at which the measurement will be performed, and a lock-in amplifier.

To ensure that the entire reference and probe beam enter the sphere the entrance port shall be as large as possible, at least five times the beam diameter. The entire energy in both the reference and the probe beams shall enter the sphere. Both beams shall be at as small an angle as possible with equal angles on each side of the normal centreline.

For reducing the amount of scattered radiation returned by the mirrors into the aperture of the integrating sphere, the distance between the mirrors and the integrating sphere has to be as large as possible.

NOTE The accuracy of the test procedure is limited by the amount of scattered radiation of the mirrors used.

The target and integrating sphere surfaces shall be coated with a material that is highly diffusive and reflecting at the laser beam probe wavelength.

Speckle patterns may be caused by the coherence of the beams in the integrating sphere leading to a greatly increased noise in the measurement of the difference signal. For suppressing slowly moving speckles on the detector surface, either the sphere is equipped with a rotating target as shown in the Figures 1, 2, and 3 or the laser beam source is frequency modulated at a high frequency. Both methods result in a fast-moving speckle pattern on the detector smoothing out the random speckle signal noise.

It is recommended to use a detector without d.c.-sensitivity (e.g. a pyroelectric detector), which only detects the difference $\Delta P = P_r - P_p$ between the probe beam power P_p and the reference beam power P_r, which is directly proportional to the reflection losses of the chopper mirror and the sample.

A high dynamic range is required for the low noise detector since this directly influences the resolution. The detector characteristics shall be linear over a wide signal range since this directly influences the accuracy of the measurements.

Silicon detectors for the visible and near infrared spectral range as well as pyroelectric detectors for the infrared spectral range shall meet these specifications.

7 Characteristic features of the laser beam

The following physical quantities are needed for characterizing the laser radiation used for the test:

— wavelength λ;

— angle of incidence β;

— state and degree of polarization;

— beam diameter on the test sample d;

— average laser power P_{av};

— frequency of laser power modulation f_{am} (if used);

— frequency of laser frequency modulation (if used).

The beam transforming optics enable the diameters of the reference and probe beams to be the same at the entrance port of the sphere. The noise of the laser source is one of the accuracy limiting factors of the measurements, the laser noise shall be as low as possible. Frequencies of the chopper and the laser modulation (if used, see Clauses 8 and 9) have to be chosen so that the noise is minimal.

8 Test procedure

8.1 Calibration of the chopper mirror

8.1.1 Calibration with reduced accuracy

The quotient Q of the specular reflectance of the additional mirror and the chopper is determined with two sequential measurements:

a) $\Delta P = P_r - P_p$ is measured according to Figure 4;

b) P_r is measured in the same way but with the probe beam blocked.

Since a lock-in amplifier measures only the absolute value of ΔP, the phase information has to be used as well. A phase shift of 180° occurring between the open and the blocked probe beam, the specular reflectance of the additional mirror will be higher than the specular reflectance of the chopper mirror. The quotient Q is determined as

$$Q = \frac{\rho_m}{\rho_{ch}} = 1 - \frac{|\Delta P|}{P_r} \text{ for } \rho_m < \rho_{ch} \quad (10)$$

$$Q = \frac{\rho_m}{\rho_{ch}} = 1 + \frac{|\Delta P|}{P_r} \text{ for } \rho_m > \rho_{ch} \quad (11)$$

8.1.2 Calibration with increased accuracy

If higher accuracy or long term stability of the laser source is required, an optional power modulation of the laser source with frequency $f_{am} \geqslant 2f_{ch}$ as described in 8.2.2 shall be used. The quotient Q of the specular reflectance of the additional mirror and the chopper is determined with two sequential measurements:

a) S_m and ΔS are measured with the set-up according to Figure 4;

b) S_{m0} and ΔS_0 are measured in the same way but with the probe beam blocked.

Since the lock-in amplifier measures only the absolute value of ΔS and ΔS_0, respectively, the phase information has to be used as well. If a phase shift of 180° occurs at the lock-in amplifier locked to $f_{am} \pm f_{ch}$ between the open and the blocked probe beam, the specular reflectance of the additional mirror is higher than the specular reflectance of the chopper mirror. The quotient Q is therefore determined as

$$Q = \frac{\rho_m}{\rho_{ch}} = \frac{\frac{S_m}{|\Delta S|} - \frac{S_{m0}}{|\Delta S_0|}}{\frac{S_m}{|\Delta S|} + \frac{S_{m0}}{|\Delta S_0|}} \text{ for } \rho_m < \rho_{ch} \quad (12)$$

$$Q = \frac{\rho_m}{\rho_{ch}} = \frac{\frac{S_m}{|\Delta S|} + \frac{S_{m0}}{|\Delta S_0|}}{\frac{S_m}{|\Delta S|} - \frac{S_{m0}}{|\Delta S_0|}} \text{ for } \rho_m > \rho_{ch} \quad (13)$$

8.2 Specular reflectance for near-normal incidence

8.2.1 Measurement with reduced accuracy

Two sequential measurements are necessary to determine the reflectance of the sample:

a) $\Delta P = P_r - P_p$ is measured with a set-up according to Figure 1;

b) P_r is measured in the same way but with the probe beam blocked.

NOTE For this measurement the long-term (typically 1 min) instability of the laser power is an additional source of error.

8.2.2 Measurement with increased accuracy

If higher accuracy or long term stability of the laser source is required, an optional power modulation of the laser source with frequency $f_{am} \geqslant 2f_{ch}$ shall be used. The modulation frequency f_{am} shall be chosen as high as possible, so that the noise level of the laser is minimal. This allows the simultaneous determination of the signal S_m proportional to the mean power $P_m = (P_r + P_p)/2$ and the signal ΔS proportional to the power difference ΔP with two lock-in-amplifiers locked to f_{am} and to $f_{am} + f_{ch}$ (or $f_{am} - f_{ch}$), respectively.

For the calculation of P_r, it is necessary to at least once determine the signals S_{m0} and ΔS_0, which are related to the signals S_m and ΔS by blocking the probe beam.

For this modulation of the laser power, the measured signal S_m at f_{am} is proportional to the sum of P_r and P_p (reference and probe beam)

$$S_m = C_1(P_r + P_p) \tag{14}$$

A lock-in amplifier is locked to the frequency f_{am} for measuring S_m. Blocking the probe beam gives

$$S_m = C_1 \times P_r \tag{15}$$

Similarly, the measured signal ΔS at $f_{am} + f_{ch}$ (or $f_{am} - f_{ch}$) is proportional to the difference of P_r and P_p,

$$\Delta S = C_2(P_r - P_p) \tag{16}$$

A second lock-in amplifier is locked to the frequency $f_{am} + f_{ch}$ (or $f_{am} - f_{ch}$) for measuring ΔS. Blocking the probe beam gives

$$\Delta S_0 = C_2 \times P_r \tag{17}$$

Combining Equations (14) to (17) and the relationship that corresponds to the measuring arrangement in Figure 1 ($P_p = \rho_s \rho_{ch} P_r$), the reflectance of the sample is given by

$$\rho_s = \frac{1}{\rho_{ch}} \times \frac{\dfrac{S_m}{\Delta S} - \dfrac{S_{m0}}{\Delta S_0}}{\dfrac{S_m}{\Delta S} + \dfrac{S_{m0}}{\Delta S_0}} \tag{18}$$

Therefore, the following two measurements are necessary to determine the reflectance of the sample:

a) S_m and ΔS are measured with a set-up according to Figure 1 using two lock-in amplifiers.

b) S_{m0} and ΔS_0 have to be determined by blocking the probe beam in the same set-up. The ratio $S_{m0}/\Delta S_0$ is a constant of the set-up and has to be measured only once.

8.3 Angular dependence of reflectance

8.3.1 General

The angular dependence of the specular reflectance can be measured in a measuring arrangement described in Figure 2. Compared with the arrangement in Figure 1, an additional mirror is used to realize a double bounce set-up allowing to measure the reflectance of the sample at different angles of incidence.

8.3.2 Measurement with reduced accuracy

For the powers P_p and P_r measured according to Figure 2 the following relationship holds:

$$P_p = \rho_s^2 \rho_{ch} \rho_m P_r \tag{19}$$

where ρ_m is the specular reflectance of the additional deflecting mirror.

The reflectance ρ_s of the test sample can be calculated from the following relation:

$$\rho_s = \sqrt{\frac{1}{\rho_{ch}\rho_m} \times \left(1 - \frac{\Delta P}{P_r}\right)} \tag{20}$$

So, again two measurements are necessary to determine the reflectance of the sample:

a) ΔP is measured with a set-up according to Figure 2;

b) P_r is measured in the same way but with the probe beam blocked.

8.3.3 Measurement with increased accuracy

As described in 8.2.2 the accuracy of the measurement can be increased by amplitude modulation of the laser beam. Similar to the method given in 8.2.2, the signals S_m, ΔS, S_{m0}, ΔS_0 have to be determined using two lock-in amplifiers.

The reflectance of the sample is given by

$$\rho_s = \frac{1}{\sqrt{\rho_{ch}\rho_m}} \times \frac{\sqrt{\frac{S_m}{\Delta S} - \frac{S_{m0}}{\Delta S_0}}}{\sqrt{\frac{S_m}{\Delta S} + \frac{S_{m0}}{\Delta S_0}}} \tag{21}$$

Two measurements are necessary to determine the reflectance of the sample:

a) S_m and ΔS are measured with a set-up according to Figure 2 using two lock-in amplifiers;

b) S_{m0} and ΔS_0 have to be determined by blocking the probe beam in the same set-up. The ratio $S_{m0}/\Delta S_0$ is a constant of the set-up and has to be measured only once.

The powers ΔP and P_r or the signals S_m, S_{m0}, ΔS and ΔS_0 (refer to 8.2.2), are determined in the same way as for the reflectance measurements under near normal incidence and the reflectance of the beam deflecting mirror has to be determined according to 8.2.2.

8.4 Transmittance

8.4.1 General

The arrangement is similar to the one for the reflectance measurements. The powers ΔP and P_r or the signals S_m, S_{m0}, ΔS and ΔS_0 (refer to 8.2.2) are determined in the same way as for the reflectance measurements. The reflectance of the beam deflecting mirror has to be determined according to 8.2.1 or 8.2.2, respectively. The angular dependence of transmittance can be measured by tilting the sample and correcting the beam displacement and by readjusting the reflecting mirror.

8.4.2 Measurement with reduced accuracy

Two measurements are necessary to determine the transmittance of the sample:

a) ΔP is measured with a set-up according to Figure 3;

b) P_r is measured in the same way but with the probe beam blocked.

8.4.3 Measurement with increased accuracy

As described in 8.2.2, the accuracy of the measurement can be increased by amplitude modulation of the laser beam. Similar to the method described in 8.2.2 the signals S_m, ΔS, S_{m0}, ΔS_0 have to be determined using two lock-in amplifiers.

The transmittance of the sample is given by

$$\tau_s = \frac{1}{\rho_{ch}\rho_m} \times \frac{\frac{S_m}{\Delta S} - \frac{S_{m0}}{\Delta S_0}}{\frac{S_m}{\Delta S} + \frac{S_{m0}}{\Delta S_0}} \tag{22}$$

So, again two measurements are necessary to determine the transmittance of the sample:

a) S_m and ΔS are measured according to Figure 3 using two lock-in amplifiers.

b) S_{m0} and ΔS_0 have to be determined by blocking the probe beam in Figure 3. The ratio $S_{m0}/\Delta S_0$ is a constant and has to be measured only once.

9 Evaluation

9.1 Specular reflectance for near-normal incidence

9.1.1 Measurement with reduced accuracy

ρ_s calculated using Equation (2), from the measurements described in 8.2.1.

9.1.2 Measurement with increased accuracy

ρ_s calculated using Equation (18), from the measurements described in 8.2.2.

9.2 Angular dependence of reflectance

9.2.1 Measurement with reduced accuracy

ρ_s calculated using Equation (20), from the measurements described in 8.3.2.

9.2.2 Measurement with increased accuracy

ρ_s calculated using Equation (21), from the measurements described in 8.3.3.

9.3 Transmittance

9.3.1 Measurement with reduced accuracy

τ_s calculated using Equation (4), from the measurements described in 8.4.2.

9.3.2 Measurement with increased accuracy

τ_s calculated using Equation (22), from the measurements described in 8.4.3.

10 Test report

The following information shall be included in the test report:

a) General information

1) test has been performed in accordance with ISO 13697:2006;

2) date of test, time;

3) name and address of test organization;

4) name of individual performing the test.

b) Information concerning the test sample

1) type of test sample;

2) manufacturer of test sample;

3) part ID, date of production;

4) specifications by the manufacturer concerning storage, cleaning, etc.;

5) specifications by the manufacturer for normal use.

c) Information concerning the test facility

1) beam source;

— type of beam source;

— manufacturer;

— manufacturer’s model designation;

2) description of other relevant test equipment.

d) Test conditions

1) wavelength used for test;

2) operating mode cw/pulsed;

3) source parameter settings;

— output power or energy;

— current or energy input;

— pulse energy;

— pulse duration;

— pulse repetition rate;

4) beam propagation ratio;

5) polarization;

6) environmental conditions (temperature, humidity, vibration);

7) cleaning;

8) method of mounting of optical component.

e) Information concerning testing and evaluation

1) test method used reduced accuracy/increased accuracy;

2) detector and sampling system;

— integration time of the lock-in amplifier;

3) other optical components and devices used for the test (polarizer, monochromator, etc.);

4) surface quality/imperfections/contamination (if inspected);

5) other relevant parameters or characteristics of the test;

— beam diameter at the test sample;

— location of beam on the sample;

— chopper frequency f_{ch};

— frequency of laser power modulation f_{am}.

f) Error budget

g) Test results

— specular reflectance ρ_s;

— regular transmittance τ_s.

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