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Standardization in optical coating characterisation

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Abstract : In the rapid development course of laser technology and modern optics , optical metrology continuously gains importance for the quality management in the industrial production environment and also for research in optical coatings. Besides absorption and scatter losses , the spectral characteristics and laser induced damage thresholds are considered as common quality factors for coated optical components and often define the optimization targets for new products and applications. Also , these quality parameters are the basis for the comparison of commercial optics and can be found in the product catalogues of most manufacturers of optical components. As a consequence , standardization of characterisation procedures for these fundamental properties evolved to a crucial point for the optics industry. During the last decade , adapted standard measurement techniques have been elaborated and discussed by representatives from many industrial companies and research institutes within working groups of the International Organisation for Standardization (ISO). In this contribution , the current state of standardized characterisation techniques for optical coatings is summarised. Selected standards for the measurement of absorption (ISO 11551) , scattering (ISO 13696) and laser induced damage thresholds (ISO 11254 , Parts 1 and 2) will be described and discussed in view of their applicability and reproducibility. The report will be concluded by an outlook on the current projects and future tasks of standardization in optics characterisation.

Key words : optical coating ; standardization ; optics characterisation ; absorption ; scattering ; laser induced damage threshold ; round-robin test

1 Introduction

The production of optical thin films with advanced quality is an enabling technology for many innovative applications of lasers and modern optics. Prominent examples in industrial production environments can be found in laser material processing or micro-lithography , where the economic efficiency is directly coupled to the optical losses and the lifetime of the beam steering or imaging systems. Also in research and development of laser systems for medical applications , optical metrology , and information technology , optical components have to fulfil a variety of demanding requirements , which can be on-

ly achieved on the basis of the newest optical production techniques. Prominent examples can be found in laser fusion experiments , where optical coatings with extremely high damage thresholds have to be realised on large area components , or in fs-technology , which can be only transferred to economical applications on the basis of optical coatings with highest degree of complexity to manage the dispersion effects in the laser systems. Standardized characterisation methods with high precision and sensitivity are a key position for these application fields and challenges of optical thin film technology. Standards provide the means for a comparison of the quality factors during optimization of optical components and for their commercialisation within the

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global market. Therefore, in most optics companies and optical thin film centres, standardized characterisation techniques are maintained as a major tool for the quality management within the development procedures and the production lines, and they are developed further to keep pace with progresses in laser technology and modern optics.

In the present contribution a brief review on quality parameters of optical coatings is given, and selected examples are presented in order to illustrate the typical development and testing procedures for International Standards in optics characterisation. Besides measurement protocols for laser induced damage thresholds, standard methods for the determination of optical absorptance and total scattering will be discussed. In addition to practicability and efficiency, success-

ful round-robin tests are considered as a major prerequisite for the qualification of draft standards in optics characterisation. Therefore, several international experiments have been co-ordinated on selected standardization projects, which are elaborated and discussed in the Standardization Committee ISO/TC 172/SC 9. Most of these round-robin tests were conducted within the framework of the European Project EUREKA EUROLASER CHOCLAB, which has been initiated by the German Ministry of Science and Technology in the year 1995. Results of selected round-robin tests will be summarised and evaluated in respect to the practicability of the existing standard measurement procedures. Finally, some current as well as future trends in laser technology and their influence on optics characterisation will be considered.

Tab. 1 Selected quality parameters of optical laser components and the corresponding characterisation techniques. For the stability of optical components in respect to mechanical, chemical and other environmental influences, a variety of national and international standard measurement procedures is available.

Specification	Parameter/ Unit	Standard/ Measurement principle
Laser induced	CW-LIDT W/cm	ISO 11254-1: CW-laser irradiation
Damage threshold (LIDT)	I on 1-LIDT J/cm ²	ISO 11254-1: irradiation with single pulses
	S on 1-LIDT J/cm ²	ISO 11254-2: repetitive irradiation with pulses
	Certification J/cm ²	DIS 11254-3: irradiation sequence
Optical losses	Absorptance 1 × 10 ⁻⁶	ISO 11551: laser calorimetry
	Total scattering 1 × 10 ⁻⁶	ISO 13696: integration of scattered radiation
Transfer function	Reflectance %	DIS 13697: precise laser radiometric method
	Transmittance %	DIS 15368: spectrophotometry
Surface quality	Form tolerances /N	ISO 10110: part 5, interferometry
	Scratch/ Digs	ISO 10110:13 parts containing different
	Roughness	types of imperfections
Stability	Abrasion	ISO 9211: different test methods
	Environmental	ISO 9022:21 parts containing a variety of conditioning
	Stability	methods

2 Quality parameters for optical coatings

Some of the quality parameters frequently

considered in optical coating technology are compiled in Tab. 1 in conjunction with the corresponding measurement techniques. For the surface properties and the environmental stability of optical coatings, a variety of national and international standards are available and under con-

tinuous discussion in different committees of ISO and also national working groups. The basis of this development work, which was performed during several decades, is formed by the classical applications of optical coatings in imaging systems, consumer optics and ophthalmology. In contrast to this, standardization of coatings for laser technology is subjected to a stronger dynamism driven by the rapid development of this emerging field. As a consequence of the massive engagement of industrial companies, which are interested in an accelerated transfer of their innovations in commercial products, many of the important standards for optical laser coatings could be implemented within a few years. The EUREKA Project EUROLASER CHOCLAB (Characterisation of Optics and Laser Beams), which contributed fundamental research aspects and especially round-robin experiments for investigations in the practicability of the discussed standard measurement procedures, was also a substantial factor for this fast progress. In the characterisation of optical coatings, measurement procedures for absorptance (ISO 11551^[11]) and total scatter (ISO 13696^[12]) have been tested in several international campaigns. Furthermore, several measurement activities were initialised to examine the general applicability and to extend the current standards for the measurement of laser induced damage thresholds (ISO 11254-1, -2,^[3]) to fs-regime. In the following, the fundamentals of these standard measurement procedures will be introduced and illustrated. Also, selected results of the interlaboratory tests will be summarised and discussed in view of the applicability of the investigated standard measurement procedures.

3 Laser induced damage thresholds

The laser induced damage thresholds (LIDT) of an optical component is one of the most important quality parameters in the devel-

opment and application of high power laser systems. Even today, the power handling capability of optical components is a limiting factor in many laser applications including nuclear fusion technology, material processing or laser medicine. During the intense scientific activities of the last forty years^[4-5], dedicated to an improvement of laser induced damage thresholds, a broad spectrum of different damage mechanisms had been discovered and investigated for miscellaneous laser operation conditions and wavelengths. The continuous discussions on the research results within in the international scientific community clearly revealed the need for comparable or standardized damage threshold measurements. In a first approach, an extended international round-robin experiment for the wavelength of $1.064\ \mu\text{m}$ ^[6] has been conducted at the beginning of the 1980-es. This experiment clearly indicated that severe restrictions in the beam parameters of the test facility and the evaluation algorithms have to be inserted in order to achieve comparable measurement results. Therefore, in the present standard procedure for LIDT measurements, ISO 11254^[3], laser systems operating in single transversal and longitudinal modes are recommended. Also, a precise beam diagnostic package has to be installed in order to characterise the beam parameters at the specimen or a conjugate location. Besides online damage detection systems, an inspection using a differential interference contrast microscope is prescribed for a reliable identification of the damage state of the irradiated sites. For the evaluation of the raw data, a technique based on the determination of a survival curve is preferred which was introduced by Seitel and Porteus^[7] and further developed by other working groups^[8]. Besides 1 on 1- and CW-tests, which are outlined in the first part ISO 11254-1, irradiation sequences with repetitive laser pulses (S on 1-tests) are also considered in part 2 of the standard. S on 1-tests represent the typical operation condition of a com-

ponent in practical applications. They also provide the means for a rough estimation of the component lifetime by an assessment of the threshold behaviour as a function of the number of pulses necessary to damage the surface. A corresponding uncomplicated evaluation and extrapolation method on the basis of the characteristic damage curve is also described in the second part of ISO 11254.

Even though 1 on 1-damage tests are not representative for the operation conditions of optical components in typical applications, they are often listed in catalogues of optics manufacturers and considered for a comparison of the power handling capabilities of competing products. Therefore, the standard measurement procedure for 1 on 1-LIDT has been tested in round-robin tests. As an example, the outcome of a national German measurement campaign with Nd:YAG laser facilities is illustrated in Fig. 1. This experiment^[9] was concentrated on laser components which are routinely applied in Nd-laser systems. The specimens were produced in individual production runs to ensure nearly identical properties for each of the six optics types. Every partner received an original sample set containing three samples of each component type for his LIDT-measurements. Three partners were involved operating Nd:YAG laser systems with pulse durations between 10 ns and 15 ns and beam diameters adjusted to values ranging from 160 μm to 600 μm , respectively. The uncertainty of the measurement values is influenced by the reproducibility of the spatial and temporal beam parameters as well as the calibration of the beam diagnostic system. In most experimental set-ups, these contributions accumulate to a total error budget in the range of 10% ~ 20%. Besides these error margins completely pertaining to the apparatus, also the statistical nature of the damage mechanisms, which are mainly initiated by inclusions at the wavelength of 1.064 μm , have to be taken into account. In considera-

tion of these uncertainties, the agreement between the measurement results is good for the majority of the samples. The raw data of this preliminary experiment were discussed in detail and processed further for the conception of the current standard^[10-11].

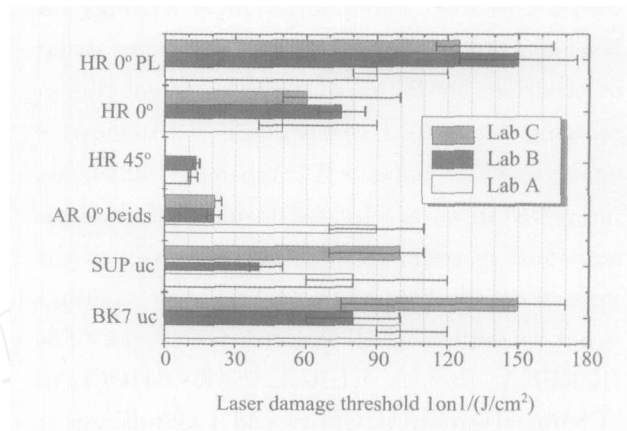
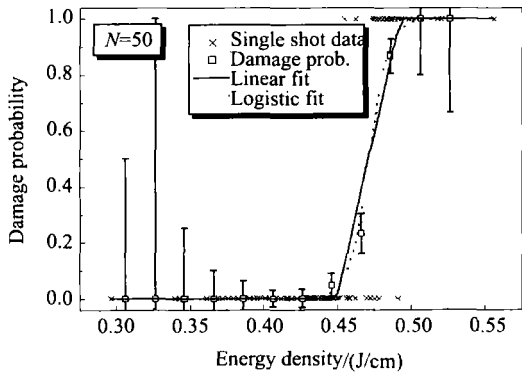


Fig. 1 Summary of results for a national measurement campaign on laser induced damage thresholds at 1.064 μm . The component types are indicated by the following notations: HR0° PL: dielectric mirrors with protection layer, HR0°: standard dielectric mirror, HR45°: standard deflection mirror, AR 0°beids.: laser window with antireflective coating on both sides, SUP uc: substrate of SUPRASIL, uncoated, BK uc: substrate of BK7 uncoated.

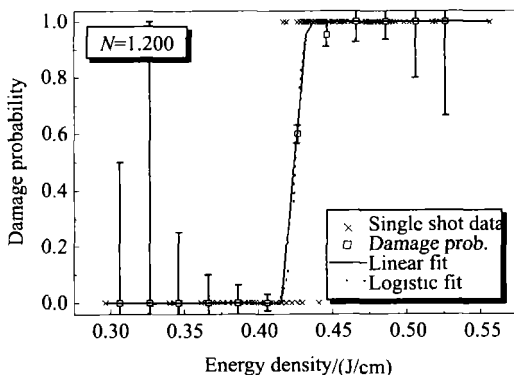
For a further evaluation and qualification of the LIDT standards, several national and international round-robin tests have been carried out also at the wavelengths 10.6 μm and 248 nm. All tests were based on optical component types selected from practical applications in laser technology and performed with different laser systems by partners of several countries. Although a broad spectrum of laser beam parameters was employed by the partners, good agreement could be achieved for most component types. The results demonstrated the versatility of the measurement procedure described in ISO 11254-1, which is now accepted by most optical companies and research institutes.

Further research work was carried out to qualify ISO 11254 for applications of excimer lasers operating with high pulse repetition rates in the DUV/VUV-spectral range (193 nm and 157 nm) as well as for the rapid development of ultra short pulse lasers, which operate in the pulse length regime below 1 ps. As the most prominent pacesetter for the present research activities in the DUV/VUV-spectral range, semiconductor lithography on its way towards integrated circuits with structures below 90 nm can be considered. The future potentiality of fs-laser technology is mainly attributed to high precision laser material processing, laser medicine and ultra fast diagnostics.

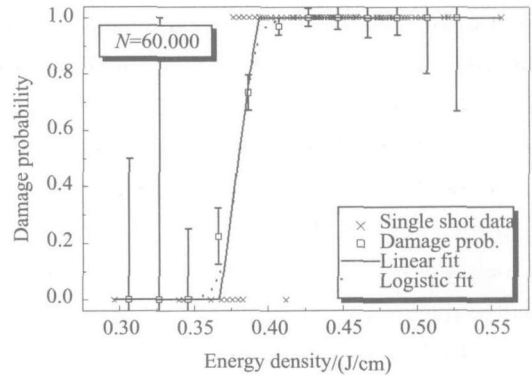
For these laser sources, which operate at high pulse repetition rates up to the kHz-range, the S on I-testing routine described ISO 11254-2



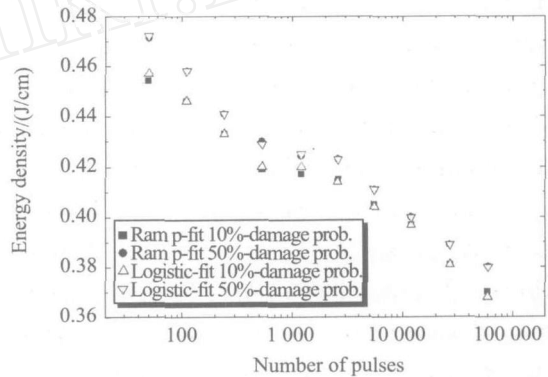
(a)



(b)



(c)



(d)

Fig. 2 Example for the evaluation of a damage threshold measurement according to ISO 11254-2. The test was performed using a CPA-Ti:sapphire laser (repetition rate 1 kHz) with a pulse duration of approx. 100 fs. The diameter of the near Gaussian beam profile was adjusted to 100 μm at the sample surface. Results are presented for a dielectric broadband mirror of $\text{TiO}_2/\text{SiO}_2$, which is optimised for applications in fs-systems. The dielectric mirror was deposited by an adapted ion beam sputtering process on a fused silica substrate. The specific evaluation methods used for a further refinement of the damage probability curves are described elsewhere^[12].

was analysed in several experiments and international measurement campaigns. The S on I measurement protocol prescribes an interroga-

tion of the test surface with a train of pulses, which have identical beam parameters. In order to avoid catastrophic damage of the specimen and to determine the number of pulses until damage occurs, an online damage detection system, which interrupts the laser system after damage, has to be employed for advanced S on I-testing. An example for the data reduction technique outlined in the Draft Standard is illustrated in Fig. 2 for a laser induced damage threshold measurement with a commercial ultra-short pulse laser^[12]. In Fig. 2a-c damage probability plots are depicted as a function of the energy density for selected pulse numbers. These survival curves can be extracted from the raw data by calculating the ratio of damaged sites to the total number of sites tested for a certain energy interval and pulse number. The survival curves are constructed by repeating the described fundamental step for all energy intervals tested. The error bars in Fig. a-c are a consequence of the fact, that different numbers of events are available for the evaluation in a certain energy interval. If only few interrogation sequences have been performed within a defined energy interval, the uncertainty of the damage probability is accordingly high. In Fig. 2 (d) the energy density values of certain damage probabilities values (10%, 50%) are plotted as a function of the number of pulses. This diagram represents the characteristic damage curve, which gives an overview of the damage behaviour for the specimen. Considering the achieved maximum damage threshold value of 0.5 J/cm^2 , optical components for ultra-short pulse lasers have to be significantly improved for present and future applications.

4 Laser calorimetric measurement of absorptance

Absorption in optical coatings is mainly induced by deficiencies in stoichiometry, absorbing

inclusions and contaminants, or by special effects at the interfaces between the layers. In most optical component, absorption leads to a conversion of a fraction of the impinging laser power into heat, which dissipates in the bulk of the component and induces distortion effects. For example in laser material processing, these thermal distortion effects can induce a shift of the focal plane on the work piece, which in turn, deteriorates the quality of the process. Furthermore, the loss of laser power in the optical components of an application is also an economical aspect, because additional expenditures are necessary for the corresponding compensation by a higher output power of the laser system. In the course of the massive implementation of high power laser systems for material processing in industrial production, optical companies constantly optimise their coatings in respect to optical absorption during the last two decades. Initially, several measurement principles based on photothermal deflection, thermal imaging and laser calorimetry were applied for the determination of absorption losses^[13]. During the further evolution of low loss coatings, a clear trend towards laser calorimetric methods could be observed, which was driven by the practical advantages of laser calorimetry. In contrast to the photothermal deflection methods, which are based on a rather complicated signal generation process involving a variety of parameters, laser calorimetry provides the means for absolute measurements by uncomplicated calibration techniques. Early comparison tests on calorimetry, which were initiated as a consequence of the slow progress in the development and the increasing commercial demand for low loss components, revealed severe deviations of the measured absorptance values. In the course of these preliminary measurement campaigns, a standardization project on optical absorptance (ISO 11551) based on the laser calorimetric technique had been started. As pacesetters for the development of ISO

11551, coating companies active in the field of CO₂-laser components introduced calorimetric absorption measurements in their quality management systems first. As a consequence of the introduction of the measurement technique, a tremendous improvement of commercial components for 10.6 μm could be observed. During a few years, the absorptance values of laser windows and lenses for material processing applications had been more than halved on the basis of reliable measurement facilities.

The fundamental measurement principle described in ISO 11551 is relatively uncomplicated. A temperature sensor is attached to the specimen, which is located in a thermally isolating calorimetric chamber. After thermal equilibrium between the sample and the environment is reached, the sample is subjected to a laser beam with known power starting at the time t_1 for a heating period with a duration t_B . During this heating period, the energy of the laser beam is partly converted into heat by absorptance, and the temperature of the specimen increases. At a defined instant t_2 , the laser is switched off, and the temperature of the specimen decreases as a consequence of heat dissipation to its environment. For the evaluation of the calorimetric measurement, the recorded temperature curves of the heating and cooling cycles are considered. The pulse method (see Fig. 3) is based on the assumption, that the total amount of absorbed heat is deposited at the time $(t_1 + t_2)/2$ by an instantaneous irradiation. Under this condition, the absorptance can be deduced by extrapolation of the cooling curve to the time $t_1 + t_B/2$

$$= \frac{(T_{Ext} - T(t_1))}{t_B P} \sum_i m_i C_{p,i}, \quad (1)$$

where the sum $\sum_i m_i C_{p,i}$ represents the heat ca-

capacity of the sample unit including the specimen, the mount, and the temperature sensor. For most applications, the specific heat data $C_{p,i}$ can be directly taken from tables, meanwhile the masses m_i of the sample and the other parts of the sample holder are determined by weighing. For measurements, where the available laser power or the absorptance of the specimen is not sufficient to achieve a high temperature in a short time interval, an extrapolation method may be applied for the evaluation of the temperature curve (see Fig. 4). In this case, the temperature behaviour is modelled directly on the basis of a solution for the heat conduction equation with the boundary conditions according to the sample geometry. For a sample with infinite thermal conductivity and small temperature increases, the heat equation can be reduced to

$$\frac{dT}{dt} = \frac{P}{C_{eff}} - T, \quad (2)$$

where γ represents the coefficient for heat losses induced by radiation and convection. The effective heat capacity C_{eff} is combined of the contributions from the sample and the holder in conjunction with additional heat contact effects to arrangements in the calorimetric chamber. The differential equation 2 can be solved by exponential function for the heating ($P \neq 0, t_1 \leq t \leq t_2$, eq. 3) and the cooling curve ($P=0, t > t_2$, eq. 4):

$$T(t) = T(t_1) + \frac{P}{C_{eff}} (1 - \exp(-\gamma(t - t_1))), \quad (3)$$

$$T(t) = T(t_2) + \frac{P}{C_{eff}} (\exp(-\gamma(t - t_2)) - \exp(-\gamma(t - t_1))), \quad (4)$$

For the determination of the absorptance, the recorded temperature curves are employed for a

fit of the functions with respect to the parameters in eq. 3 and 4. An example for an evaluation of a calorimetric measurement according to this exponential method is depicted in Fig. 4. The advantage of this data reduction technique, which is recommended for lower laser powers and exposure times longer than 60 s, is the involvement of all measured data points in the procedure.

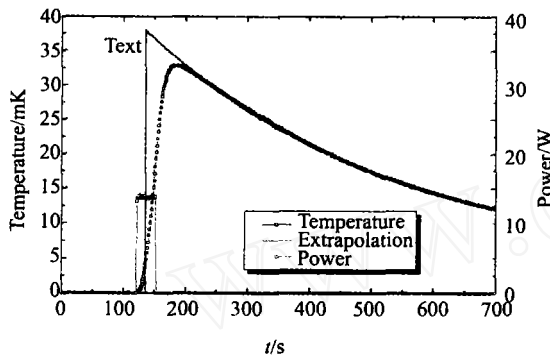


Fig. 3 Pulse methods for the evaluation of temperature curves recorded during a calorimetric absorptance measurement according to ISO 11551. The irradiation time starting at t_1 and ending at t_2 is indicated by the rectangular graphs at bottom of the diagram.

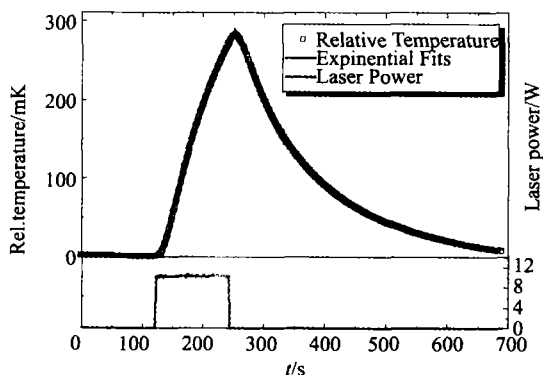


Fig. 4 Exponential methods for the evaluation of temperature curves recorded during a calorimetric absorptance measurement according to ISO 11551.

The measured temperature curve and the graphical presentation of the data reduction

method are mandatory parts of the test report of ISO 11551 and can be assessed for more sophisticated evaluation methods. The described evaluation methods are well adapted to nearly ideal conditions, i. e. the measurement of specimens with high thermal conductivity and absorptance values exceeding the sensitivity limit of the calorimetric facility by orders of magnitude. This situation is characteristic for most optical components applied for high power CO₂-lasers operating at a wavelength of 10.6 μm . Nowadays, all products for these applications are routinely certified in respect to absorption. In order to assess the practicability of the standard especially for these types of optical components, extended international round-robin tests^[14-15] have been conducted during the elaboration phase of ISO 11551. As an example, selected results of a parallel measurement campaign dedicated to optical components often used in CO₂-laser systems are illustrated in Fig. 5. In this experimental approach, a batch of identical samples was distributed to the laboratories which performed their measurements in parallel at a certain time instant. The major problem of this approach is the realization of identical properties and the proof, that the deviation of the absorptance values within the sample set is small compared to the uncertainties of the applied measurement facilities. In the present round-robin test, the sample set consisted of only two types: a high power laser window with advanced antireflective coating and a metal mirror of a special aluminium-alloy designed for improved environmental stability. Three specimens of each sample type were distributed to the laboratories after a pre-inspection cycle at the co-ordinating laboratory. Then the specimens were measured in a first cycle (cycle 1), circulated in a path which avoids long shipment distances, and re-measured in a second cycle (cycle 2) at the participating laboratories. Finally, a post inspection was performed by the co-ordinating partner in order to assess possible

degradation and ageing effects within the samples. The results of the 11 partners involved in the experiment are summarized in Fig. 5 for the laser windows. Besides the average values, which are determined from the absorptance data measured for the three ZnSe-windows by each facility, absorptance values combined from both measurement cycles and the path of the sample sets are indicated. The error bars are assigned to the standard deviation calculated from the available measurement data. Most of the data points are located near the total average absorptance value, which amounts to 0.21 % and is slightly higher than the average value determined in the pre-screening experiment. With the exception of three partners, absorptance values within an error limit of 25 % were obtained by the laboratories. By following the sample sets along their path through the laboratories, deviations from the total average value can be considered as typical for the individual laboratories and are nearly independent of the sample sets. Therefore, in first order, the deviations detected in the current experiment originate predominantly from specific measurement problems of the facilities. In this experiment, a clear aberration of laboratory L K toward extremely high absorptances and of laboratory L G towards low values can be detected, respectively. Also, a slight departure from the 25 %-limit of partner L I is apparent. Most of the causes for these tendencies, which were also observed for the metal mirrors investigated in the test, could be identified during the evaluation phase. Deviations of individual measurement facilities could be attributed to specific calibration errors, effects on the temperature sensors by scattered radiation, or not sufficient output power of the measurement laser system^[15].

The results of the round-robin experiments, which involved more than 400 single measurements and approximately 100 samples, clearly indicate the practicability of the measurement procedure described in ISO 11551 for the wave-

length of 10.6 μm .

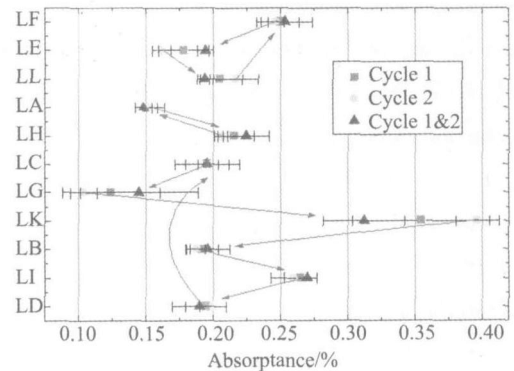


Fig. 5 Mean absorptance values for the laser windows measured by the participants in cycle 1 and cycle 2. The combined average values are indicated by cycle 1 and 2; meanwhile the path of the sample sets is illustrated by arrows. The participating laboratories are indicated by the capital letters.

As a consequence of the low thermal conductivity of glass and quartz materials routinely employed for the VIS- and NIR-wavelength region, the uncomplicated evaluation schemes illustrated in Fig. 3 and 4 can not always be applied in this range. In order to evaluate the detailed evolution of the temperature field in such optical components, the heat transport equation has to be solved considering the boundary conditions for the calorimetric measurement procedure^[16-17]. An other difficulty of calorimetric absorptance measurements in the NIR/ VIS-spectral range is the low level of absolute absorption, which ranges between 1×10^{-5} and 1×10^{-4} for conventional components and can even reach values below 1×10^{-6} for layer systems deposited by advanced ion beam techniques^[18-20]. In consideration of the output power level achievable by the presently available laser systems, calorimetric facilities have to reach temperature sensitivities in the mK-region, to assess the absorption behaviour of modern low loss components. Also, special means have to be

considered to shield the system from the environment and to improve the sensitivity of the temperature measurement. Within the European EUREKA-project CHOCLAB several technical approaches have been investigated and optimised for measurements in the sub-ppm-regime^[17,21]. The achieved results were integrated in a revised version of ISO 11551 which was published in 2003 and reflects also the specific sources of errors of calorimetric measurements with increased sensitivity. An example for a sensitive laser calorimetric measurement on an uncoated fused silica substrate is depicted in Fig. 6. Recent progresses in high sensitivity laser calorimetry allow for temperature detection limits below $50 \mu\text{K}$ corresponding to absorptance levels well below 0.1×10^{-6} .

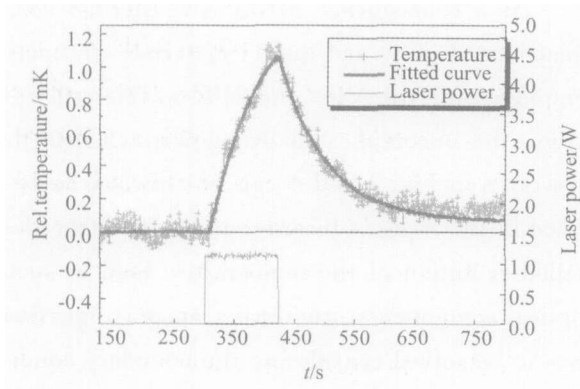


Fig. 6 Example for a laser calorimetric measurement with high sensitivity on an uncoated fused silica substrate (thickness: 1 mm, measured absorptance: 3×10^{-6}).

Further aspects, which were identified during the testing of ISO 11551 with excimer lasers in the DUV/VUV-spectral range, include non-linear effects appearing in materials with band gap energies near to the photon energy of the applied laser systems. Besides these non-linear effects in bulk materials and coatings, which could be directly measured^[22-25], degradation and conditioning mechanisms have to be considered. Recent investigations also indicate an essential

influence of the environment of the calorimetric facility as well as the handling, storage, and cleaning conditions of the samples on the absorptance results in the UV-range. High energetic photons in the DUV/VUV-range may also excite electronic transitions in the materials with fluorescent decay channels which dissipate the adsorbed radiation energy without the generation of heat. Obviously, the calorimetric measurement scheme is not sensitive to these absorption phenomena, and therefore, additional measurement techniques on the basis of fluorescence spectroscopy have to be combined with laser calorimetry to acquire the total power absorbed by the optical component.

5 Measurement of total scattering

Total scattering is a loss channel in optical components, which is mainly induced by microstructural defects in the bulk and the surface roughness of the coatings and the substrates. The radiation impinging onto the optical component is deflected by these imperfections out of the specular direction and is no longer available for the intended application. Thus, the amount of radiation scattered by the optical components is of major concern because it influences directly the efficiency of a product or an application. Especially in the UV-spectral range, losses by optical scattering impair the economic efficiency, because the production of UV-laser photons is expensive compared to most other prominent wavelengths. Also, laser safety aspects have to be considered in high power laser applications, where even low optical scattering may lead to dangerous laser power levels in the environment of the laser and beam steering systems. As a consequence an ISO-Standard has been developed during the last years^[2], which is dedicated to the

measurement of the total scattering (TS) value of optical laser components. In this standard procedure, which was derived from national standards developed in the United States for the measurement of total integrated^[26] and angle resolved^[27] scattering, the radiation scattered by the specimen is collected and integrated by an Ulbricht-sphere^[28] or a Coblentz-hemisphere^[29]. The power of the scattered radiation is measured by a detector and related to the corresponding power of a 100 % diffuse reflecting standard. Depending on the half space of integration, total backward and total forward scattering are distinguished. These measurement values are directly connected to the application in laser systems and are not related to the reflectance of the test surface as the total integrated scatter value defined in ASTM F 1048.

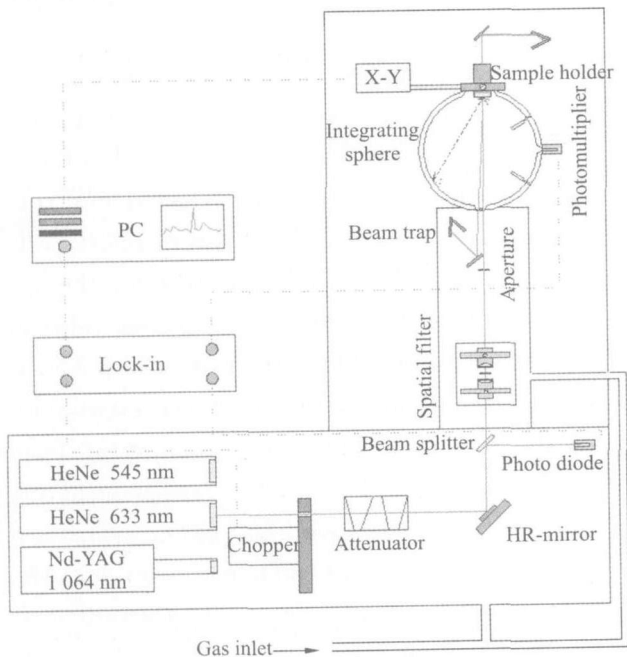


Fig. 7 Example of a measurement facility for total scattering according to ISO 13696 with an Ulbricht-sphere for the visible and near infrared spectral range^[31]. The measurement facility has a sensitivity of less than 1×10^{-6} for the installed laser wavelengths.

A typical measurement facility, which is equipped with an Ulbricht-sphere for the visible and near infrared spectral range, is depicted in Fig. 7. This apparatus consists of separate chambers for the beam preparation and the scatter measurement device, which can be flushed with gases in order to reduce the contribution of Rayleigh scattering to the zero signal of the system. For the quality management in industrial production, set-ups with sensitivities of a few 1×10^{-5} are sufficient and can be built up with low financial and experimental expenditure. Meanwhile the Ulbricht-sphere offers a variety of advantages in respect to handling, alignment, and geometry of the specimens; specific disadvantages have to be considered for shorter wavelengths. Since the fundamental principle of the Ulbricht-sphere is based on multiple diffuse reflections of the scattered radiation, the absolute diffuse reflectance of the inner walls is of primary importance for its efficiency. For wavelengths below 200 nm, appropriate materials with high diffuse reflectance are not available, or they exhibit radiation induced ageing effects. Therefore, Coblentz-hemispheres coated with adapted high reflecting metal and protection layers are preferred for TS-measurement in the deep and vacuum UV-spectral range. In comparison to the integration principle of the Ulbricht-sphere, the collection effect of the Coblentz-sphere can be considered as an optical imaging of the scattered radiation onto the detector, which are positioned at conjugate points. In this model, the Coblentz-hemisphere forms a spherical mirror, and the typical restrictions of such an imaging device have to be examined for an estimation of the measurement accuracy^[30].

Tab. 2 Properties of the experimental facilities employed within an international round-robin test on total backward scattering (D : sphere diameter, ROA : range of acceptance angle, d_r : spot diameter on the sample surface, S_b : sensitivity limit, E_r : relative error).

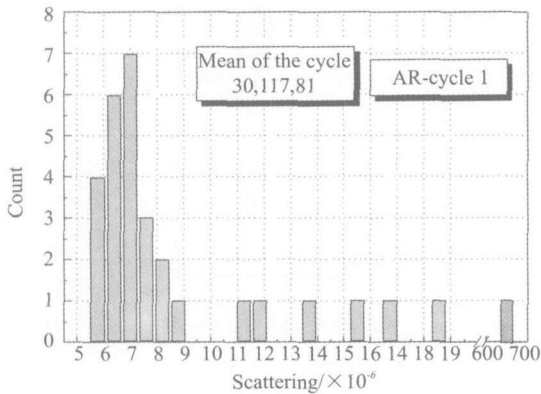
Lab.	Type	D/ mm	ROA (°)	d_r / mm	$S_b/ \times 10^{-6}$	$E_r/ \%$
1	Coblentz	254	2.50 - 70.0	2.5	5.0	2-3
2	Coblentz	350	2.00 - 85.0	0.3	< 0.1	< 10
3	Ulbricht	220	2.00 - 85.0	1.0	1.4 - 4.0	< 10
4	Ulbricht	150	2.90 - 70.0	1.5	< 5.0	-
5	Ulbricht	250	2.00 - 88.0	0.4	< 0.5	< 10
6	Coblentz	220	2.85 - 80.0	1.0	20.0	3
7	Ulbricht	150	2.00 - 82.0	1.5	< 5.0	< 10
8	Ulbricht	250	-	1.0	9.0	-
9	Ulbricht	152	1.90 - 82.0	1.0	2.0	< 10

Sources of error and the practicability of the standard measurement procedure have been investigated within the EUREKA-project EURO-LASER CHOCLAB at the wavelength 633 nm of the He-Ne laser. On the basis of the experience gathered during an extended national pilot test^[31], a parallel international round-robin test had been initiated involving a sample set, which represents optical component types often applied in laser technology^[32]. Every partner received three separately sealed specimens of a high reflecting mirror, a beam splitter and a laser window which were produced in batches especially ordered for the experiment in order to achieve identical properties for each sample type. More than 100 samples were distributed among the ten partners after a pre-screening procedure at the co-ordinating laboratory in an initial cycle. In the second phase, total backward scatter values of the specimens were measured by the contributing partners according to ISO 13696 during a relatively short time period. A broad spectrum of different measurement parameters was available within the round-robin test (see Tab. 2). Six laboratories performed total backward scatter measurements with Ulbricht-spheres, and three partners employed arrangements with Coblentz-hemispheres. As in other extended international experiments, a double blind approach was preferred, where neither the co-ordinator, nor the

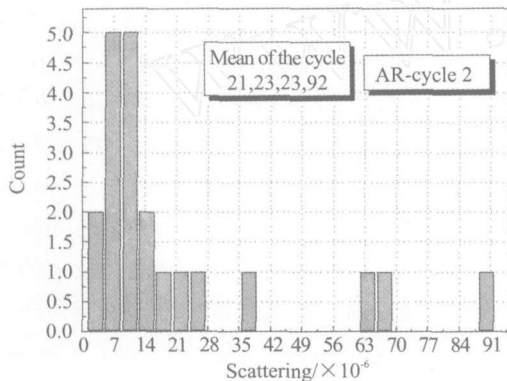
participants were informed about the measurement results until termination of the measurement campaign. After completion of the measurement sequence at the partners, all sample sets were collected by the co-ordinating laboratory and inspected in two scatter measurement runs in order to monitor contamination problems of the surfaces.

As an example, measured backward scatter values for the laser windows with antireflective coatings on both surfaces are illustrated in Fig. 8 (a) and 8(b). In these diagrams a statistical survey of the measured total backward scatter values is presented in units of counts for the appearance of surface sites with a scatter value in an interval indicated by the x -axis, which is calibrated in units of scattering. The concentration of the measured values in the interval from 5.5×10^{-6} to 9×10^{-6} in the histogram of the pre-screening cycle (see Fig. 8(a)), indicates a good quality of the production run for the AR-coated laser windows. The total backward scatter values attained from the round-robin cycle exhibit a broadening of the statistical distribution (see Fig. 8(b)), which reflects the deviations of the values measured by the contributing partners. In contrast to the significant broadening, the statistical mean values of the two distributions differ only by a few percent. A detailed summary and discussion of all data accu-

mulated within this extended campaign^[32] demonstrates a good practicability of the standard ISO 13696.



(a)



(b)

Fig. 8 Statistical distribution of total backward scatter values determined during the pre-screening (a) and the round-robin cycle (b). The samples (laser windows with AR-coatings of Ta₂O₅/SiO₂, V-coating for 633 nm, AOI 0°) were coated in one deposition run on laser grade fused silica substrates.

Present trends in optical scatter measurements are concentrated on lower wavelengths in the DUV/VUV spectral range, which impose additional difficulties related to the handling of the measurement radiation in appropriate beam preparation systems and to the properties of the collecting element. In addition, specific problems are expected which are induced by Rayleigh scattering of the environmental atmosphere,

which increases with the wavelength according to a $1/\lambda^4$ -law. Therefore, scatter measurements in the DUV/VUV-spectral range have to be performed in vacuum or an atmosphere of inert gas, which is transparent for the measurement radiation. An experimental arrangement, which has been qualified for the measurement of total scattering at the wavelengths 193 nm and 157 nm, is depicted in Fig. 9. Besides the technical problems connected with the high photon energy of the measurement radiation, the topic of calibration with an appropriate diffuse reflecting standard had to be addressed during the adaptation of the measurement procedure described in ISO 13696 to the DUV/VUV-range. A special laser processed diffuse reflecting standard with a Lambertian scatter characteristic had been developed and qualified for the DUV/VUV-measurement. In addition, enhanced influences of the sample contamination and radiation induced effects in the specimens have to be taken into account. Recent experiments indicate that ISO 13696 can be transferred to the DUV/VUV-spectral range without significant amendments.

6 Reflectance and transmittance

The measurement of spectral reflectance and transmittance according to ISO 15368 is a routine task in an optical thin film laboratory. For the wavelength-range from 175 nm to 50 μm a variety of commercial spectrophotometers are available, which can be adapted to the specific tasks of optical thin film technology with low expense. As a consequence of recent progresses of DUV/VUV-laser-systems for applications in semiconductor lithography, material processing and conditioning of technical surfaces, new demands are imposed on optical thin film technology in the production of stable and power resistant coatings for the DUV/VUV-spectral range. For the determination of the fundamental thin film properties in this spectral region, appropri-

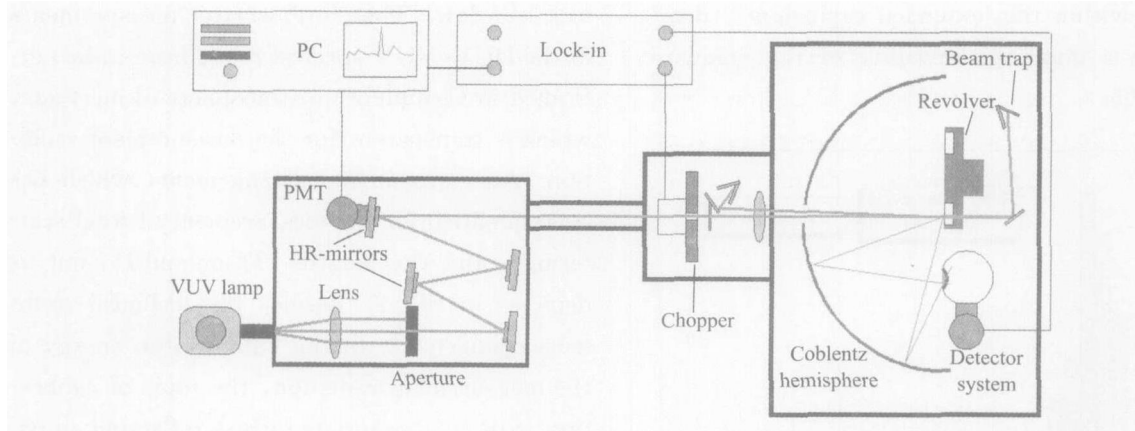


Fig. 9 Set-up for TS-measurements in DUV/VUV-spectral range with a D_2 discharge lamp as light source and a Coblenz sphere as collecting element^[33]. The scatter measurement is operated under vacuum conditions with a phase sensitive detection scheme.

ate spectrophotometric devices have to be developed and qualified. The fundamental principle of a UV/VUV-spectrophotometer designed especially for optical thin film optimization^[34] is illustrated in Fig. 10. The system consists of four functional compartments which are operated under vacuum conditions or flushing with pure nitrogen. The source section contains two D_2 -discharge lamps with different exit windows which can be alternatively selected for the measurements by a switching mirror (mirror 1 in Fig. 10). For measurements in the wavelength range between 115 nm and 230 nm, the D_2 -discharge lamp with the MgF_2 exit window is coupled into the monochromator unit. To surpass the second order limit of the spectrometer above 230 nm, the second D_2 -discharge lamps is equipped with a quartz exit window which acts as a second order filter for the measurement range between 150 nm and 300 nm. The chamber behind the lamp unit contains the monochromator system with a holographic grating arranged in Seya Namioka configuration. As an advantage of the Seya Namioka configuration, the wavelength can be tuned by a simple rotation of the grating with a computer controlled stepping motor. A mirror is installed behind the exit slit of the monochromator to direct the monochromatic radiation into the polar-

izer compartment and to shape the measurement beam. After passing the polarizer unit, the beam enters the sample compartment where a part of the beam is deflected onto a photomultiplier tube (PMT) by a scraper mirror. This PMT acts as the reference detector for a ratiometric measurement compensating for power fluctuations or drifts of the light source. The remaining part of the beam impinges onto the sample which is mounted in a 4-axis positioning system. The second PMT for signal detection can be positioned behind the sample for transmittance measurements or in the beam reflected by the sample for reflectance measurements. In this configuration the sample compartment offers the advantage to measure the reflectance of the sample directly without any special reflectance units which are most common for commercial systems at other wavelengths. Furthermore, transmittance and reflectance can be measured under fixed conditions, because no realignment of the sample is necessary for switching between both measurement modes. The position of the signal PMT in respect to the sample is adjusted automatically in the measurement routines. For controlling and read out of the functional parts, the spectrophotometer is equipped with a computer system containing all necessary motor driving and converter

modules. The control software features a menu of measurements methods including plain spectrophotometric scans and surface mappings as well as measurement cycles for recording the reflectance or transmittance as a function of the angle of incidence or cycles involving polarised radiation.

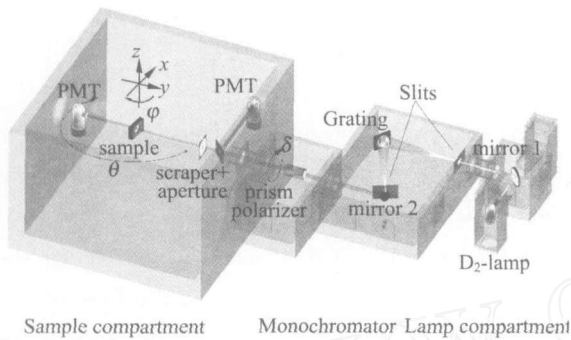


Fig. 10 Illustration of the fundamental principle of the developed spectrophotometer.

Another task in optical coating technology is often the determination of reflectance and transmittance values with a higher precision above 10^{-3} which is beyond the capability of conventional spectrophotometers. Especially for laser wavelengths, the cavity ring down (CRD) technique^[35] can be applied for the accurate determination of transmittance values near zero or reflectance values near unity. Experiments in the VIS- and NIR-spectral range on the comparability of the CRD-technique with combined loss measurements demonstrate the versatility of the CRD-technique^[36]. The major disadvantage of the CRD-technique is the limitation to optics with losses resulting in ring down times which are sufficiently long for an accurate measurement. As an alternative, the measurement procedure described in ISO/DIS 13697 for an accurate determination of reflectance and transmittance at a single wavelength has been elaborated^[37]. The principle of this ratiometric method is illustrated in Fig. 11. A laser beam with well defined and stable beam parameters is switched by a rotating chopper mirror (C) between a direct path or a reflection by the specimen into an

integrating sphere (US). Thus the signal attained by the detector (D), which is attached to the inner wall of integrating sphere, turns between two levels corresponding to the incident beam power or to the power reflected by the specimen, respectively. The modulation depth S , which can be determined using a lock-in amplifier (LI) tuned to switching frequency of the chopper blade, is directly related to reflectivity of the specimen by the equation.

$$R_s = \frac{1}{R_c} \left(1 - \frac{S}{S_0} \right), \quad (5)$$

The parameters R_s and R_c are the reflectance values of the specimen and the chopper blade, respectively. S_0 denotes the signal according to the incident power, which can be determined by blocking the beam path to the specimen.

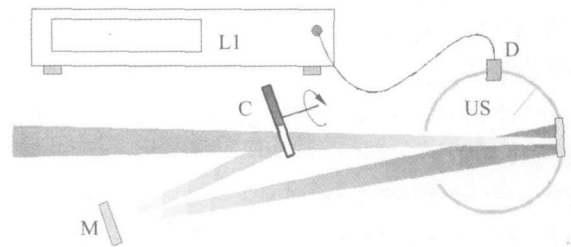


Fig. 11 Basic principle of a reflectance measurement facility according to ISO/DIS 13697. For transmittance measurements, the specimen is positioned between the reflector (M), which is replaced by a mirror of known reflectance and the Ulbricht-sphere (US). The laser source, which emits the beam at the left side of Fig. 11, is not depicted

The measurement principle had been tested and optimized within an international measurement campaign^[39] resulting in a high versatility for the measurement of coated components. Self-containing absolute calibration procedures have been developed, and an accuracy of 10^{-4} has been demonstrated for the wavelength of the CO₂-laser. For the Nd:YAG laser (1,064 μm) an even better precision below 10^{-5} could be achieved^[40].

7 Conclusions and outlook

During the last decades, laser technology emerged from research laboratories to innovative applications in many technology fields with high future potential. In the course of this development, optical coatings advanced to a key technology with essential impact on progresses in modern optics. Besides the improvement of high quality deposition processes, the characterisation of optical coatings is an essential prerequisite for a continuous economical and technical success of optical coating technology. In view of the demanding specifications of laser technology, International Standards have been developed and qualified for the key parameters of modern laser optics. Besides measurement techniques for laser induced damage thresholds, ISO-Standards were developed and tested for the determination of absorption and scatter losses in optical components. Furthermore, for the measurement of the transfer properties of optical laser components approaches were investigated and will be published as standard during the next few months. The present work of the Standardization Committees is concentrated on the measurement of phase shifts induced by optical coatings as well as on the topics of contamination and cleaning of optical surfaces, which are considered as a milestone for the development of high quality coatings for the DUV/VUV-spectral range. Further challenges will be imposed by the rapid growths of ultra short pulse laser technology on the standardization of measurement techniques for the group delay dispersion of chirped mirrors and other dispersive optical elements. A further standardization problem of highest interest is the lifetime of optical coatings, which is far from being solved at the moment. Finally, also the trend towards shorter wavelengths in the EUV-spectral range should be mentioned, which is driven by next generation lithography in a wave-

length range around 13 nm. The solution of these new tasks and the revision of present standards in optics characterisation will foster the modern optics community and contribute essentially to further progresses and economical success in the field.

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