

High-performance infrared optical coatings for the LWIR range by magnetron sputtering

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Abstract

Optical coatings for the infrared spectral range from 4.5 μm to 14 μm require materials with low absorption, sufficient refractive-index contrast and suitable mechanical stability. In this work, Ge, ZnS and several metal fluorides are investigated as coating materials deposited by RF magnetron sputtering. Non-reactive sputtering with Ar is compared with reactive fluoride deposition from metallic targets using NF_3 . Ge provides a stable high-index material with a refractive index above 4. In contrast, non-reactively sputtered fluorides are limited by low deposition rates, insufficient reproducibility or humidity sensitivity. Reactive sputtering with NF_3 improves the deposition behaviour of the fluoride materials. Reactively sputtered SrF_2 shows optical constants close to literature data, although its deposition rate remains limited. Reactive DyF_3 provides a higher deposition rate and a refractive index of 1.32 at 14 μm . Optical emission spectroscopy is used to monitor the reactive DyF_3 process through the Dy-to-Ar emission ratio. Infrared demonstrator coatings are designed with Ge and DyF_3 , including anti-reflection and a dichroic coating. The results confirm the feasibility of the material combination, while deviations from the theoretical spectrum indicate that further improvement of the deposition-rate control is required.

Keywords: long-wave infrared; RF magnetron sputtering; infrared optical coatings; germanium; dysprosium fluoride; reactive sputtering; NF_3 ; optical emission spectroscopy; film stress; demonstrator coating

1. INTRODUCTION

High-performance optical coatings for the infrared spectral range are required for spectroscopy, thermal imaging, environmental monitoring and space instrumentation. These applications require broad-band optical performance with low absorption, low scatter and sufficient mechanical robustness. This is particularly relevant for space applications, where optical performance must be maintained under demanding environmental conditions [1-4].

For the spectral range from 4.5 μm to 14 μm , anti-reflection and dichroic thin-film coatings can be designed using established interference-coating principles. The design requires suitable high-index and low-index materials. The total layer thickness increases with wavelength, which makes IR coatings sensitive to material absorption and film stress. A high refractive-index contrast is therefore required to reduce the total layer thickness. Low absorption is equally important, because even small losses can become relevant in thick multilayer systems [1,2,5].

Common material systems for infrared thin films include Ge, ZnS, ZnSe and low-index metal fluorides. Published optical data are available for evaporated Ge, ZnS, YbF_3 , LaF_3 and DyF_3 [2,7,8]. These data provide an important reference for sputtered films. However, sputtered metal fluorides are not yet established as standard materials for IR coating stacks. Their deposition behaviour, optical constants and environmental stability therefore have to be assessed for the intended process route [5,7,8,12].

Conventional deposition routes for IR coatings include evaporative processes and high-temperature processes with or without plasma assistance. These methods can restrict the choice of coating and substrate materials. This is relevant for temperature-sensitive infrared materials such as ZnS and ZnSe. Fluoride materials introduce an additional constraint, because plasma-assisted processes can lead to process-induced absorption. A further limitation is the restricted control of intrinsic film stress. In many conventional processes, the final stress state is strongly influenced by the thermal expansion mismatch between the coating and substrate materials [9-12].

These limitations affect coating reliability. Porous or mechanically weak films can reduce environmental stability. High intrinsic stress can cause substrate curvature, cracking or delamination in thick IR multilayer systems. These effects limit the usable material combinations and reduce the process window for broad-band infrared coatings [10-12].

RF magnetron sputtering is investigated as an alternative deposition route. The method can produce dense thin films and allows film properties to be adjusted through deposition parameters [9,12]. This is relevant for IR multilayer coatings, where absorption, stress and environmental stability must be controlled within the same material system. In this work, the focus is placed on reactively sputtered metal fluoride layers. In the following, the investigated materials are motivated and the results discussed based on sputtering experiments. Finally, a demonstrator coating based on Ge as high-index material and DyF_3 as low-index material is fabricated and discussed.

2. COATING MATERIALS AND EXPERIMENTAL METHODS

2.1 Coating and substrate materials for IR

The selection of coating materials is determined by the spectral range from 4.5 μm to 14 μm . Suitable materials must provide low absorption over this range and sufficient environmental stability. In addition, a high refractive-index contrast is required to limit the total thickness of coatings for broad-band antireflection and dichroic beam splitters. Often, Ge together with ZnS is used as an established material combination for infrared multilayer systems. Metal fluorides provide lower refractive indices and are therefore relevant as complementary materials [2,3,5].

Germanium is the most prominent high-index material for this spectral range. Published optical data for evaporated Ge films show a refractive index above 4 and low absorption in the relevant infrared range [2]. Ge/ZnS multilayer systems have also been demonstrated for mid-infrared anti-reflection and dichroic coatings [3]. ZnS has a refractive index of about 2.2 and a broad infrared transmission range. However, its refractive index is substantially higher than that of fluoride materials. Therefore, ZnS is included as an established medium-index reference material rather than as the preferred low-index material.

Rare-earth fluorides are relevant low-index candidates because they combine a refractive index of approximately 1.4 with broad infrared transparency. Optical constants have been reported for several fluoride materials, including YbF_3 and DyF_3 [2,5,7]. YbF_3 is well documented for infrared coating applications and has also been used in YbF_3/ZnS multilayer systems [6]. DyF_3 is of particular interest because published measurements show transparency up to 16 μm .

SrF_2 and BaF_2 are included into the material development as additional low-index materials. Both materials provide broad infrared transparency and allow comparison between rare-earth and alkaline-earth fluorides. Published thin-film optical data for SrF_2 and BaF_2 can be found in Refs. [4,14]. MgF_2 has a particularly low refractive index, with reported values of $n = 1.23$ at 10 μm . However, its reported transmission range is less suitable for the complete spectral range up to 14 μm . MgF_2 is therefore not investigated further in the present work [7].

Oxides such as Y_2O_3 , HfO_2 and Sc_2O_3 may provide improved environmental stability and can be used as adhesion-promoting layers. However, their transmission ranges are less favourable for the complete range from 4.5 μm to 14 μm . They are therefore retained as possible auxiliary materials, but not selected as the primary low-index materials.

Based on these considerations, the experimental investigation focuses on Ge, ZnS, YbF_3 , DyF_3 , SrF_2 and BaF_2 . Ge provides the high-index basis. The fluorides are investigated as low-index candidates.

2.2 RF magnetron sputtering

All coating materials investigated in this work are deposited by RF magnetron sputtering in a flexible coating system at Fraunhofer IST. The system provides several magnetron positions and allows different coating materials to be deposited sequentially without opening the process chamber between individual layers. As substrates are introduced without a loadlock, target conditioning and pre-sputtering are required before each deposition to establish reproducible process conditions. The principal deposition parameters for each target material are RF power, working pressure and gas flow. All processes are performed without additional substrate heating.

The process development distinguishes between two process alternatives: non-reactive sputtering in pure Ar and reactive sputtering with additional NF_3 . Non-reactive sputtering is applied to metallic and ceramic targets, including Ge, ZnS and selected metal fluorides. For each material, the RF power, Ar flow and working pressure are varied systematically to identify process conditions that provide a sufficient deposition rate and stable film properties. Since changes in the target surface can affect the deposition behaviour, the targets are conditioned before each process.

Reactive sputtering with NF_3 is investigated as an alternative process for depositing metal fluorides from metallic targets. NF_3 is added to Ar as the reactive gas, while NF_3 flow, Ar flow and RF power are varied to determine the accessible

process window. As the target surface state changes during reactive sputtering, the deposition behaviour cannot be described by the gas flows alone. Optical emission spectroscopy is therefore used to monitor the plasma and assess changes in the reactive state of the process. For reactive DyF₃ deposition, the ratio between the Dy and Ar emission signals is used as the process indicator. Changes in this ratio are linked to the NF₃ flow and to changes in the Dy target surface. The OES signal therefore provides the basis for stabilising the reactive process.

The use of NF₃ requires a dedicated safety concept. The laboratory is equipped with gas detectors, local ventilation and filtered process exhaust. The gas supply is integrated into the equipment safety controls so that the reactive gas flow can be interrupted if a potentially dangerous condition is detected. H₂S is also available as a reactive process gas in the sputtering system, but H₂S-based processes are not included in the present work. The following section presents the results for non-reactive and reactive sputtering separately.

2.3 Optical, mechanical and environmental characterisation

Optical characterisation is based on spectroscopic transmission and reflection measurements over an extended spectral range. Measurements in the visible and near-infrared range are performed with a Cary 5000 spectrophotometer up to 3.3 μm . An Essent Optics Photon RT spectrophotometer is used to extend the measurement range to the mid-infrared up to 5.2 μm .

The spectral range is further extended into the infrared using FTIR spectroscopy. Several Thermo Fisher FTIR spectrometers are used for transmission and reflection measurements between 2.5 and 25 μm wavelength. The FTIR configuration allows reflection measurements at an incidence angle of 10°.

In the following, the combined transmission and reflection data are used to evaluate film transparency, absorption and spectral losses and film thickness. In selected cases, IR ellipsometry complements the transmission and reflection measurements.

Film thickness and curvature are measured by profilometry using a Dektak system. These measurements serve to verify the film thickness determined from the transmission and reflection fit for selected samples and showed good agreement. Film stress is assessed from the curvature of 280 μm thick silicon substrates. The stress is calculated from the curvature change using Stoney's equation [10].

Environmental stability is assessed by cleanability, tape adhesion, abrasion and humidity testing. Transmission and reflection are measured before and after environmental exposure to identify changes in the optical performance.

3. RESULTS AND DISCUSSION

3.1 Non-reactive sputtering with pure Ar

Previous work, carried out in the same sputtering system at Fraunhofer IST, reported mainly on non-reactive sputtering [12]. This included Ge as high-index material basis for the planned IR multilayer systems, as well as ZnS.

For this purpose, process parameters like Ar flow and sputtering power are systematically varied and the samples subsequently analysed optically by spectrophotometry.

Next, the refractive index and absorption coefficient (n , k) are extracted from transmittance and reflectance measurements. For this purpose, the combined transmission and reflection spectra were fitted using a Cauchy model for the refractive index and an exponential model for the extinction coefficient [12]. To motivate the approach in the material development, the results on non-reactive sputtering of Ge, ZnS, YbF₃, DyF₃, SrF₂ and BaF₂ are briefly summarized in the following. More detailed process data can be found in Table 1.

For Ge, the approach described above provided optical constants ($n > 4$ in the whole IR range, negligible absorption) that agreed with literature data, where Duris et al. report $n \approx 4.17$ at 10 μm [3]. Furthermore, a deposition rate of approximately 0.5 nm/s was obtained in the investigated process window. The parameter studies show that changes in RF power and Ar flow affect film stress, optical behaviour and microstructure of the films. Thus, the window of 300-400 W RF power and 50 sccm Ar flow was found to be preferable. Ge layers produced with the above-mentioned process passed the cleanability, adhesion, abrasion and humidity tests.

Furthermore, for metal fluorides, non-reactive sputtering was carried out from ceramic targets for YbF₃, DyF₃, SrF₂ and BaF₂ in pure Ar atmosphere [12]. Here, the Cauchy dispersion model did not provide a good basis. Therefore, the optical

constants of these materials were modelled using an arbitrary function in the software OTF Studio. The resulting values showed good agreement with literature data and indicated low absorption in the infrared spectral range. Nevertheless, k values smaller than 0.01 cannot be reliably determined from the optical measurements described above. Thus, the given k values present an upper limit.

For YbF_3 , the fitted refractive index was approximately $n \approx 1.5$ at $10\ \mu\text{m}$, which is higher compared with a literature value of $n \approx 1.37$ at $10\ \mu\text{m}$ [14]. For YbF_3 and also DyF_3 , a very low deposition rate ($\ll 0.01\ \text{nm/s}$ for YbF_3 , $\ll 0.01\ \text{nm/s}$ for DyF_3) was found that makes practical use impossible. Furthermore, the limited film thicknesses made the reliable fitting of n and k for DyF_3 not possible. For all the fluoride materials, the limited film thickness resulted in weak spectral features, which reduces the reliability of the extracted optical constants above $10\ \mu\text{m}$.

For SrF_2 , the fitted value of $n = 1.52$ at $10\ \mu\text{m}$ is again higher compared with a literature value of $n = 1.3655$ at $10\ \mu\text{m}$ for crystalline SrF_2 [15]. For BaF_2 , a refractive index of $n = 1.39$ at $10\ \mu\text{m}$ was fitted, which falls into the range found in literature with $n \approx 1.33$ - 1.42 at $10\ \mu\text{m}$ [17]. BaF_2 layers seem to be more moisture sensitive, though. The deposition showed to be not reproducible due to the moisture uptake in the target after sample exchange.

Apart from YbF_3 , the sputtered fluoride layers were not stable after exposure to humidity. Therefore, and due to the partly impractical deposition rates, the non-reactive sputtering of fluorides can be considered unsuitable.

Additionally, non-reactive sputtering of ZnS was carried out. The fitted value of $n = 2.19$ at $10\ \mu\text{m}$ agrees well with a literature value of $n \approx 2.18$ at $10\ \mu\text{m}$ derived from the Cauchy parameters reported by Duris et al. [3]. Therefore, the sputtered layers show promising optical properties, but the stable process window was narrow. High compressive stress and delamination were observed, and the samples were not stable after humidity exposure. A reactive ZnS process using H_2S as reactive gas may provide an alternative route to improve environmental stability.

Table 1: Summary of available process and optical data for IR coating materials from non-reactive sputtering. Note that the determination of reliable n and k values for DyF_3 was not possible due to the low layer thickness.

Material	Target / process	RF power (W)	Ar flow (sccm)	Dep. rate (nm/s)	n (10 μm)	k (10 μm)	n (14 μm)	k (14 μm)
Ge	metallic Ge, non-reactive	400	50	~ 0.5	4.19	< 0.01	4.19	< 0.01
ZnS	ceramic ZnS, non-reactive	100	75	~ 0.1	2.19	< 0.01	2.19	< 0.01
YbF_3	ceramic YbF_3 , non-reactive	175	30	$\ll 0.01$	~ 1.5	< 0.1	~ 1.5	< 0.1
DyF_3	ceramic DyF_3 , non-reactive	250	50	$\ll 0.01$	--	--	--	--
SrF_2	ceramic SrF_2 , non-reactive	120	130	~ 0.03	1.52	< 0.01	1.49	< 0.02
BaF_2	ceramic BaF_2 , non-reactive	120	130	~ 0.08	1.39	< 0.01	1.30	< 0.01

Table 2: Summary of available process and optical data for metal fluoride coatings from reactive sputtering.

Material	Target / process	RF power (W)	Ar flow (sccm)	NF_3 flow (sccm)	Dep. rate (nm/s)	n (10 μm)	k (10 μm)	n (14 μm)	k (14 μm)
SrF_2	metallic Sr, reactive	250	80	3.0	~ 0.03	1.38	< 0.01	1.28	< 0.02
DyF_3	metallic Dy, reactive	200-250	80	2.0-2.9	0.1	1.47	< 0.01	1.32	< 0.02

3.2 Reactive fluoride sputtering with NF₃

Due to the limitations described above, reactive sputtering with NF₃ in addition to Ar is investigated as an alternative process for the deposition of metal fluorides from metallic targets. The process is first evaluated with SrF₂ and DyF₃. Similarly to the procedure described above, process parameters for the sputtering are systematically varied to find a suitable process. As can be expected for reactive sputtering, the additional NF₃ flow serves as crucial process parameter. In principle, stable process conditions are established with an NF₃ flow up to 3 sccm, and an additional Ar flow around 50-80 sccm.

As described above, the optical constants (n , k) are extracted from measurements of transmittance and reflectance of single layers. Fig. 1 shows the respective measurements for DyF₃ and demonstrates the procedure exemplarily. The measurement curves are fitted with an arbitrary function in OTF Studio, and an exponential model to derive n and k , respectively. For DyF₃, the measured data align well at the transition from the MIR to the FTIR range, and the fitted dispersion values show a reasonable trend. The fitted values are $n = 1.47$ at $10\text{ }\mu\text{m}$ and $n = 1.32$ at $14\text{ }\mu\text{m}$. These values are compared in Fig. 1 with literature data from Hahner et al. [7] for the refractive index, but no corresponding k values are reported. The fitted value reported here remains below 0.01 at $10\text{ }\mu\text{m}$ and below 0.02 at $14\text{ }\mu\text{m}$, and is used as an upper-limit estimate for coating design.

For SrF₂, the measured data are shown in Fig. 2, together with the extracted n and k . Although there is a small offset at the transition between the NIR and FTIR measurements in the order of 1%, the dispersion is comparable to the literature curve used for the fit. The fitted values are $n = 1.38$ and $k < 0.01$ at $10\text{ }\mu\text{m}$ and $n = 1.28$ and $k < 0.02$ at $14\text{ }\mu\text{m}$. At $10\text{ }\mu\text{m}$, $n = 1.3655$ and $k = 5.28 \times 10^{-4}$ for crystalline SrF₂ are reported [15], which is in good agreement with the fitted values presented here. The lower refractive index compared with non-reactively sputtered SrF₂ indicates process-dependent optical constants, though [12]. Even for the reactively sputtered SrF₂, the deposition rate of about 0.03 nm/s remains a limiting factor, though.

Thus, reactive DyF₃ sputtering seems to be more promising with a higher deposition rate, a comparable refractive index and even lower absorption at $14\text{ }\mu\text{m}$.

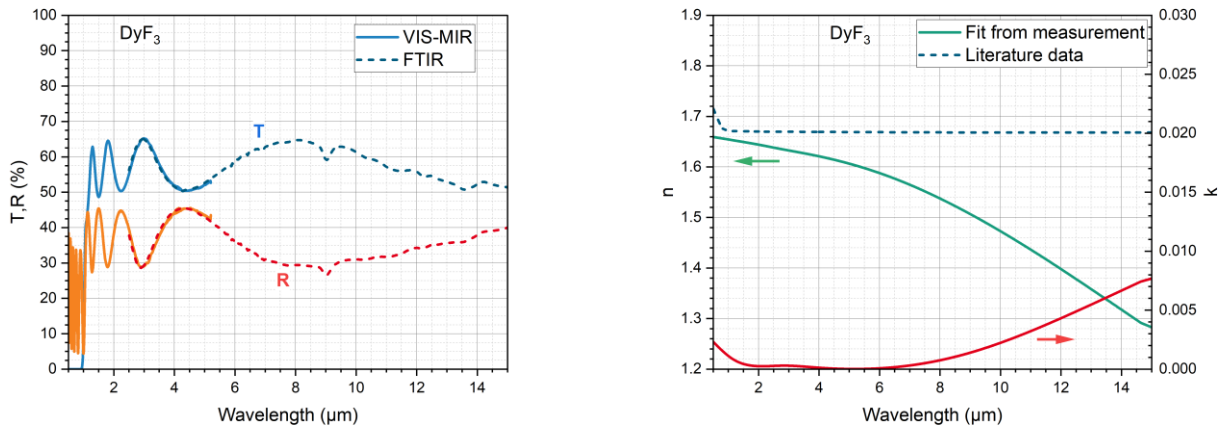


Figure 1: Optical data for reactively sputtered DyF₃. Left: transmittance and reflectance measurements from two different setups (Photon RT for VIS-MIR, Thermo Fisher FTIR). Right: refractive index (n) and absorption coefficient (k) fitted from the measurements and compared to literature data [7].

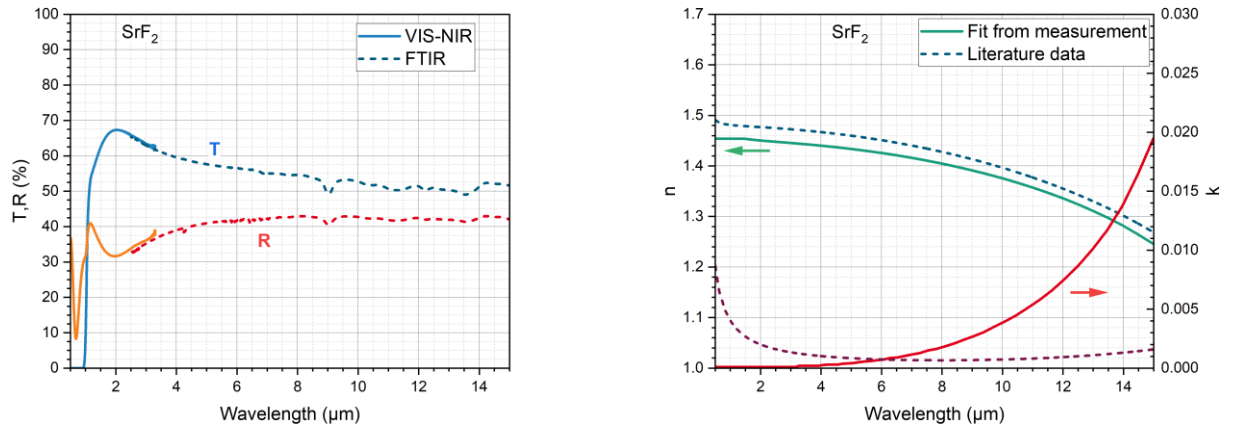


Figure 2: Optical data for reactively sputtered SrF₂. Left: transmittance and reflectance measurements from two different setups (Agilent Cary for VIS-NIR, Thermo Fisher FTIR). Right: refractive index (n) and absorption coefficient (k) fitted from the measurements and compared to literature data [15].

In order to make the reactive sputtering process more reliable and reproducible, optical emission spectroscopy (Emicon Plasma Monitor by Plasus) is used. From the collected spectra, the well-separated and strong Dy line at 405 nm, and the Ar line at 434 nm are taken as relevant signal.

The ratio of the Dy peak to the Ar peak height is taken for the process control. A more direct access to the NF₃-driven process was not possible, as no direct fluorine lines are visible, or at least not visible in presence of the strong Ar lines. When introducing NF₃ to the process, the Dy signal decreases. Without stabilization, i.e. adjustment of the NF₃ flow, the Dy/Ar ratio drifts to even lower values during the process. The decrease of the Dy signal is consistent with a change of the Dy target surface during reactive sputtering. This behaviour is interpreted as possible target poisoning. Thereby, the amount of sputtered Dy is reduced when the highly reactive fluorine forms DyF₃ on the surface of the Dy target. In the present experiments, the ratio is manually stabilised by NF₃ flow adjustment at approximately 1.1–1.5.

Nevertheless, these results show that DyF₃ is the most promising low-index material for the respective spectral range. With the process control, implemented by adjustment of the NF₃ flow, a Ge/DyF₃ multilayer coating is realized in the next step.

3.3 Demonstrator coatings

From the above finding, Ge/DyF₃ is the preferred material combination, with Ge as high-index material and reactively sputtered DyF₃ as low-index material. With this combination, several demonstrator coatings are designed, shown in Figs 3 and 4, namely a dichroic coating that shall be used as beam splitter at 40° angle of incidence, as well as an antireflective coating in two variants with either four or seven layers. The dichroic coating is designed for high reflectance in the 4.5 to 6.3 μm range, and high transmittance in the 6.7 to 14 μm range. The AR coatings are designed for the whole transmission range between 6.7 and 14 μm.

The 4-layer AR coating is deposited on a Si substrate, and measurements of transmittance and reflectance are shown in Fig. 3 (right). The comparison to the theoretical design reveals blue-shifted spectra in the experiment of approximately 7 %, which indicates layer thickness deviations. In fact, a reverse engineering with the design software OTF Studio supports that due to a lower deposition rate, the SrF₂ layers are generally 10 % too thin to meet the expected spectrum. Further improvements are expected when changing the OES-based process control from a manual to an automatic mode. This way, the 7-layer AR and the 14-layer dichroic design shall be realized as immediately next steps.

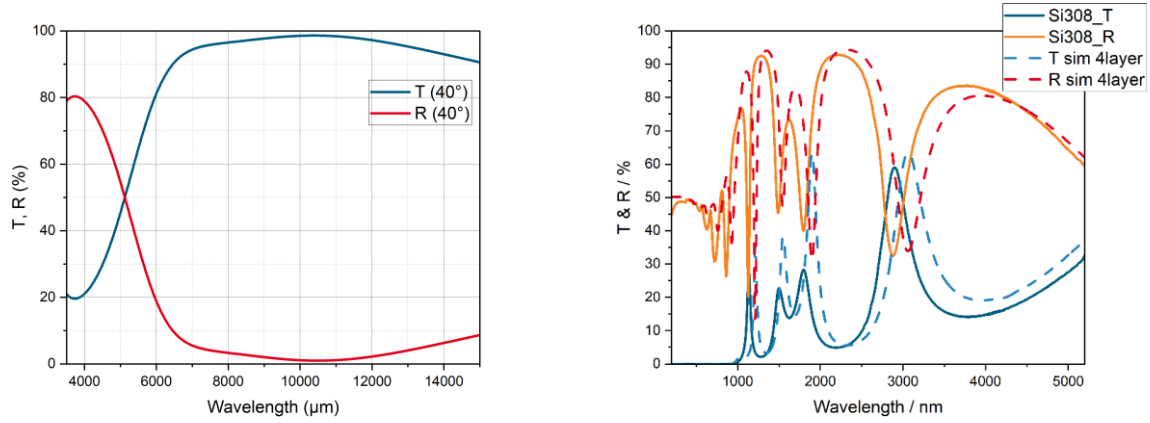


Figure 3: Left: Theoretical design of a 4-layer antireflective coating based on Ge/DyF₃ as layer materials on a Si substrate. Note that the backside reflection is excluded. The goal is to maximize transmittance at 40° angle of incidence in the whole 6.7 μm to 14 μm range. Right: Measured transmittance and reflectance of the coating, showing a slight blue-shift that can be attributed to deviations in the deposition rate.

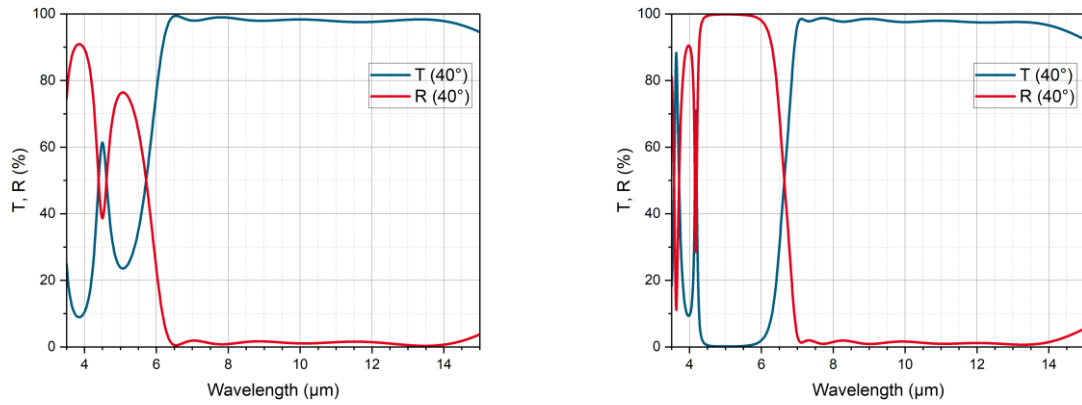


Figure 4: Left: Theoretical design of a 7-layer antireflective coating based on Ge/DyF₃ as layer materials on a ZnSe substrate, designed for highest transmittance in the 6.7 μm to 14 μm range at 40° angle of incidence. Right: Theoretical design of the corresponding dichroic beam splitter coating with 13 layers based on the same material combination. It is designed for high transmittance in the range 4.5 to 6.3 μm and high reflectance in the 6.7 to 14 μm range. Note that the backside reflection is excluded in both cases.

5. CONCLUSIONS

RF magnetron sputtering was investigated for the deposition of optical coatings in the infrared spectral range from 4.5 μm to 14 μm . Ge was identified as a suitable high-index material, while the non-reactive deposition of ZnS and several metal fluorides was limited by film stress, low deposition rates or insufficient environmental stability. Reactive sputtering of metal fluorides with NF₃ improved the deposition behaviour. Among the investigated materials, DyF₃ provided a suitable combination of low refractive index, low absorption and a deposition rate that allows multilayer fabrication. Optical emission spectroscopy was used to monitor the reactive process, with the Dy/Ar emission ratio serving as the process indicator.

Based on these results, demonstrator coatings with Ge and reactively sputtered DyF₃ were designed and fabricated. A four-layer antireflection coating confirmed the feasibility of this material combination, although the spectral shift indicated deviations in the deposited layer thicknesses. Further work will therefore focus on automated OES-based process control, improved layer-thickness accuracy and environmental qualification of the DyF₃-based multilayer coatings.

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