Optical multilayer anti-reflecting coating in the infrared range

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Abstract. The constant expansion of the applicable scope of optical technologies has led to the need to create optical coatings not only with certain frequency characteristics, but also high performance properties. This article contains the results of a research of a thin optical film from an advanced material SrYF₄. On the basis of this film, a multilayer coating was synthesized, which has high transparency in the near and middle infrared range. The coating has successfully passed the climatic tests on resistance to cold and heat.

1. Introduction
When designing systems of the near and middle infrared (IR) range, it is necessary to ensure not only high efficiency of antireflection optical coatings but also their physical integrity over a wide temperature range and moisture resistance.

Compared with well-studied oxide glasses, fluoride glasses with antireflection coatings have a number of advantages: wider transmission spectrum and lower losses [1]. This raises the problem of obtaining new stable fluoride compounds, including for the implementation of optical structures with film coatings [2-4].

To implement high-performance coatings, films with a refractive index of n ≈ 1,4-1,5 or less are required. The most commonly used films are those of yttrium fluoride YF₃, characterized by a small refractive index. For example, at a wavelength of 10 microns n ≈ 1,28-1,30 [5]. However, coatings containing layers of YF₃ with a total thickness of several micrometers are characterized by poor reliability when working in a real atmosphere. When the temperature changes, they often peel off from the optical antenna surface due to mechanical stresses occurring in YF₃ films.

Currently, work on the production of film coatings is led in two directions: synthesis of completely new coatings and improvement of the characteristics of known compounds by selecting the composition of optimal structures. However, it is the development of composite structures based on various fluoride compounds that can often improve both optical and performance characteristics [6]. To create optical components with coatings, a promising direction is the use of films of doped fluorides, mixtures and solid solutions of binary metal fluorides. In particular, the properties of optical films in BaF₂ – YF₃ and BaF₂ – MgF₂ systems have been studied in works [7].

It is known that YF₃-based films are destroyed by temperature changes. This disadvantage can be eliminated by creating a new film-forming material YF₃ with SrF₂ since SrF₂ demonstrates the preservation of optical properties in a wide temperature range. It is also recommended for the creation of optical components of laser systems [6]. This article presents the results of studies of a new film-forming mixture SrYF₄, which has shown to be promising for the development of reliable coatings that
can operate in real atmospheric conditions with changes in temperature and humidity of the environment.

2. Purpose and results of the research
In this paper, we research a new SrYF$_4$ film in the range of 1.3 microns to 12 microns. YF$_3$ films with high transparency in the visible and IR spectral ranges are promising in the considered frequency range. Their disadvantages noted above can be compensated by combining YF$_3$ and SrF$_2$. Such a compound should not lead to deterioration of the optical properties of the new film since the transparency range and refractive index of the SrF$_2$ film are close to the parameters of the YF$_3$ film.

In our work, polished discs made of single-crystal zinc selenide ZnSe of brand SVD with a thickness of 4 mm were used as a substrate. It allows us to study films in the near and middle IR spectral region since it is most transparent in the range up to 12-15 microns [6,7].

The coating of SrYF$_4$ film on a single-crystal ZnSe substrate was performed at Balzers BAK-760 (Liechtenstein). The spectra of substrates and films were measured by Fourier spectrophotometer FSM-1201. Previously, the spectrophotometer was adjusted according to tabulated reflection standards. Measurement errors were +0.2%.

Figure 1 shows the results of the substrate study: the calculated refractive index ($n_s$, curve 1), which was determined through the optical constant search program Film Manager [8], the measured transmittance spectrum ($T_s$, curve 2), reflection spectrum ($R_s$, curve 3), as well as the absorption coefficient spectrum ($A_s$, curve 4). In addition, the program [8] was introduced with the found values of the substrate $n_s$ and $k_s$, and the transmission, reflection and absorption spectra of the substrate were calculated. The calculated spectra coincided with the experimentally measured ones with high accuracy.

![Graph showing refractive index, transmittance, reflection, and absorption spectra for ZnSe substrate.](image)

Figure 1. Charts of refractive index (1); of spectrum: transmittance (2), reflection (3) and absorption (4) for ZnSe substrate of wavelength.

Figure 2 shows the measured transmission spectra $T$ (curve 1) and reflection spectra $R$ (curve 2) from the upper side of the plate as solid lines, and the absorption spectrum $A$ (curve 3) is shown dot-and-dash line. In the same figure, dashed lines show the transmission spectra $T_{corr}$ (curve 4) and reflection spectra $R_{corr}$ (curve 5) found for the SrYF$_4$ film, taking into account their correction for absorption in the substrate by the method presented in [6].
The formula was used to calculate the absorption

$$R+T+A=1.$$  

The refractive index ($n$) of the corrected SrYF$_4$ film spectrum was calculated by the step-by-step method described in [7,8]. Initially, refractive indices and geometric film thickness were determined in the absorption-free regions. Then the calculation was performed on the adjacent section of the spectrum. The average value of the refractive index in the shorter wavelength region was taken as the initial approximation. After that, we moved on to the next section of the spectrum.

The absorption coefficient ($k$) was determined numerically. To do this, using the program Film Manager, the absorption at a given wavelength was calculated by varying the absorption coefficient. Those values of the absorption coefficient were calculated, for which the absorption had the value closest to the experimentally measured. Figure 3 and figure 4 show the results of calculations of refractive and absorption indices of a new film-forming material SrYF$_4$. From figure 4 it is visible that at wavelengths of 3.2 microns and 6 microns, water absorption in the pores of the film does not exceed 1%.
To develop a multi-layer anti-reflection coating, the choice of substrate material was performed. Zinc selenide (ZnSe) is known to be used as a material for the production of optical elements: windows, lenses, mirrors, prisms, beam splitters, etc. Its distinctive feature is the low absorption in the infrared region. The material, although being polycrystalline, is characterized by a homogeneous structure, high transmission in the IR region and low internal losses associated with absorption and scattering. Zinc selenide is most commonly used for the production of components for CO₂ lasers (including high-power) and broadband spectral devices operating in the range from 0.6 to 17 microns. It has a high refractive index \( (n=2.4) \). The transparency range is from 0.5 to 22 microns.

The SrYF₄-based film has a low refractive index in the near and mid-IR spectral region, has low absorption and can be used as part of a complex coating. So on its basis, the structure of the anti-reflective coating of 7 layers was synthesized. The dependence of its transmittance on the wavelength is shown in figure 5.

The resulting coating is a broadband anti-reflection coating in the near and middle infrared range with an average transmittance of more than 81%. It has high mechanical strength and hardness, chemical resistance, and it is non-hygroscopic.
Conclusion
In this work, a 7-layer anti-reflection coating for the infrared range based on SrYF$_4$ film in the range of 1.2-12 microns was synthesized. This film with a thickness of up to 1-2 microns in comparison with pure YF$_3$ has high performance properties: rather low refractive index and small absorption, low stress, lower hygroscopicity, high mechanical strength, including cases with temperature changes. Low and high temperature climatic tests have been performed successfully.

Thus, SrYF$_4$ film can be recommended for the development of multi-layer high-performance anti-reflection coatings designed to work in all weather conditions. Theoretical and experimental studies have confirmed its minimal losses and high transparency in the range from 1.2 to 12 microns.

Acknowledgement
The work was supported by the Russian Science Foundation (RSF, Gr. No. 19-79-10110).

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