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Search for high-strength multilayer free-standing film filters with high transmittance in the wavelength range of the „water window“ (2.3−4.4 nm)

© M.M. Barysheva, S.A. Garakhin, A.Ya. Lopatin, V.I. Luchin, I.V. Malyshev, N.N. Salashchenko, N.N. Tsybin, N.I. Chkhalo

Institute of Physics of Microstructures, Russian Academy of Sciences,
603950 Nizhny Novgorod, Russia
e-mail: tsybin@ipmras.ru

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Some variants of the composition of multilayer absorption film filters with a high transmittance in the spectral region of the „water window“ (2.3−4.4 nm) have been considered. Having created an ultimate pressure difference between the sides of free-standing films at which they are damaged, we compared the strength of 100 nm thick Ti-based multilayer filters with Al, Be, C interlayers and 100 nm thick V-based multilayer filters with Al interlayers. Sc and Cr was also considered as interlayers. Among the tested periodic multilayer structures, the best strength characteristics were demonstrated by Ti/Be (with a fraction of Ti in a period of about 0.6) and V/Al (with a fraction of V in a period of about 0.4) multilayer filters. Despite the fact that Ti/Be and V/Al filters are inferior in strength to Ti and V monolayer filters of the same thickness, these multilayer filters may be of interest, since they have either a higher transmittance in the „water window“ (Ti/Be) or more high level of blocking of visible radiation (V/Al).

Keywords: spectral region of the „water window“ multilayer free-standing filters, ultimate pressure difference, film filter strength, transmittance in the soft x-ray, visible light blocking level.

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Introduction

The soft X-ray (SXR) range (wavelength range $\sim 1-10$ nm) is of interest for various applications, such as X-ray microscopy in the „water transparency window“ [1–3], X-ray lithography at wavelengths shorter than 13.5 nm [3–5], X-ray spectroscopy [6,7].

Effective radiation sources are needed to conduct research in the SXR range. Such sources are synchrotron sources and free electron X-ray lasers. However, in laboratories, more affordable and compact plasma sources are usually used for operational measurements — laser plasma or gas discharge [6,8–10]. Since plasma sources have a wide spectrum of radiation, it is usually necessary to filter out radiation outside the working band. The easiest way to achieve this in the case when the working wavelengths lie in the SXR and extreme ultraviolet (EUV) wavelength range, — use free-standing absorption film filters [10–12]. By selecting the material and thickness of the film filter, it is possible to achieve the necessary level of blocking of out-of-band long-wave radiation at a high (tens of percent) transmittance at operating wavelengths.

Depending on the type of plasma source, optical system and detector, the requirements for the blocking characteristics of film filters (wavelength ranges that need to be suppressed, the degree of their suppression) may vary markedly. Thus, in the case of using multilayer mirror coatings in the optical scheme, a high level of blocking of long-wave radiation is usually required, which is effectively reflected by these mirrors (visible, near infrared range).

For experiments in the SXR range, film filters with the highest possible transmittance in the operating range are needed, since the brightness of most laboratory plasma sources in the SXR wavelength range is small and the radiation intensity is usually not enough. The problem is complicated by the absence of normal line incidence mirrors with a high reflection coefficient in the SXR range. For example, in the spectral range „of the water window“ (2.3−4.4 nm) the maximum reflection coefficient achieved to date near the normal is only slightly greater than 20% [13,14].

A high transmittance is also required when using free-standing films as holders for living biological samples. In this case, a wet sample is placed between two films, which reduces the evaporation rate of the liquid, but due to the passage of radiation through two films, losses at working wavelengths increase. Most often, in microscopy in the spectral range „of the water window“ Si_3N_4-membranes with a thickness of 100 nm and a transmittance of 50−70% in one pass [15] are used for this purpose. As will be shown below, by choosing a more transparent material with the same film thickness, a transmittance of up to 85% can be achieved.

It is possible to increase the transmission coefficient of the film filter at working wavelengths by reducing the thickness of the film, but at the same time the level of suppression of long-wave radiation decreases, and also, which may be more significant, the mechanical strength of the filter significantly decreases (with a constant aperture). Film filters must have sufficient strength to remain intact both during their manufacture and during subsequent manipulations with them (transportation, installation in a vacuum volume, pumping). A certain level of strength is required when films are used as X-ray windows separating volumes with different pressures [16,17], or when films are used as holders for biological samples in SXR microscopy [18]. In the latter case, the film must be strong enough to remain intact when the biological object under study is placed on it.

In some cases, it is possible to overcome the above dilemma by switching to multilayer periodic structures [19], when the composition of the film filter includes several materials whose layers alternate. With the same total thickness, a multilayer film structure may be stronger than a single-layer one. Hardening can be caused, for example, by the fact that the boundaries between the layers prevent the development of cracks in the direction across the layers when the film is stretched.

The reverse situation is also possible, when multilayer films turn out to be less durable than single-layer ones. The same interlayer boundaries can be sources of microcracks when the elastic characteristics of the materials of the layers differ significantly.

Everything is not exhausted by the mechanisms of hardening or softening in multilayer film structures given here, information about other possible mechanisms of hardening is given, for example, in [20,21].

Multilayer structures may be of interest for other reasons. By selecting the second material (interlayer), the blocking properties of the film filter can be improved. Since the thickness ratio in the multilayer structure period depends on the magnitude of internal stresses in the deposited film [22], it becomes possible (if necessary) to select the thickness ratio so as to minimize the magnitude of internal stresses. This is important because large internal stresses can lead to deformation and damage of the films (for example, during their separation from the substrate).

However, since it is impossible to predict in advance what the strength of a particular multilayer structure will be, whether the values of internal stresses in the film will be critical, experimental studies are necessary.

In this paper, we investigated the strength and optical characteristics of multilayer filters of various compositions based on transparent in the range of „water window“ materials, primarily titanium-based. As can be seen from the calculated transmission spectrum (Fig. 1), titanium is one of the most transparent materials in the SXR range. Titanium filters (usually 200 nm thick) are used as long-wave radiation blocking filters in plasma radiation sources of the SXR band [23,24], in the SXR band spectrometers [6,25].

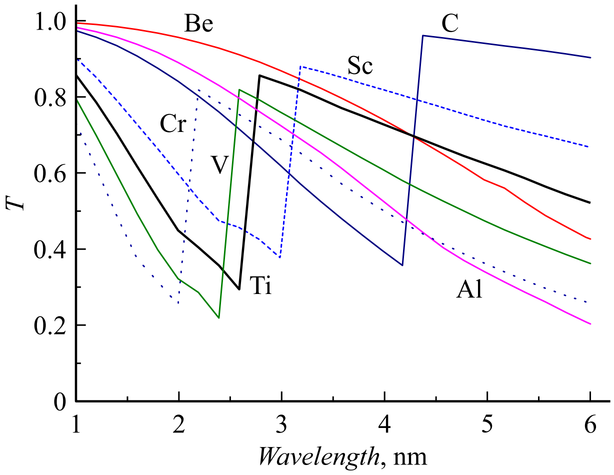

Figure 1. Calculated transmission spectra of filters with a thickness of 100 nm from materials transparent in the wavelength range „of the water window“. The optical constants are taken from site [26].

In addition to titanium, spectral filters made of beryllium, scandium, vanadium, aluminum, chromium may be of interest in the range „of the water window“ (Fig. 1). Some variants of multilayer film structures containing these materials are also investigated in this study.

1. Research methods

All film samples were produced at the magnetron sputtering facility [3]. Deposition of targets Ti, Be, Cr, Sc, V, Al and C magnetron targets (as well as Mg or Y for sublayers) were sputtered in an argon atmosphere at a pressure of $7 \cdot 10^{-4}$ Torr. The pressure of the residual gases in the vacuum volume before deposition was better than 10^{-6} Torr. The films were deposited on polished silicon substrates with a diameter of 100 mm, the uniformity of the distribution of layer thicknesses over the area was not worse than 2%. Free-standing films were made using a metal sublayer (Mg or Y layer deposited on a silicon substrate), which was dissolved in the process of selective liquid etching (an aqueous solution of acetic, nitric or hydrochloric acid was used as a selective etcher). The sublayer gradually dissolved from the edge to the center of the substrate, the substrate sank to the bottom, and the film remained floating on the surface of the etcher. To prevent acid vapor deposition on the film surface and their penetration into the sublayer through the pores in the film, continuous air blowing of the film surface was carried out during the etching process.

Layer thicknesses in multilayer films were determined from preliminary calibrations. For this purpose, two multilayer periodic structures of the same composition, but differing in the thickness of the layers of one of the materials (under study) in the period, were sequentially deposited onto a silicon substrate. The change in the thickness

Table 1. The ultimate pressure drop averaged over N attempts ($\langle\Delta p\rangle$) between the sides of the film with a thickness of H, at which the film is damaged

Structure, nm	H, nm	N	$\langle\Delta p\rangle$, atm	σ, atm	$\Delta p_{\max}$, atm	T (633 nm)
(Ti−3.1/Be−1.05)×25	103.75	9	0.08	0.02	0.11	–
(Ti−3.1/Be−2.1)×20	104.0	10	0.165	0.057	0.24	$4.1\cdot10^{-4}$
(Ti−2.05/Be−3.15)×20	104.0	11	0.12	0.09	0.40	$5.7\cdot10^{-4}$
(Ti−1.3/Be−3.9)×20	104.0	11	0.12	0.032	0.19	$8.4\cdot10^{-4}$
(Ti−4.1/Al−1.05)×20	103.0	9	0.08	0.042	0.18	$1.3\cdot10^{-4}$
(Ti−3.1/Al−2.1)×20	104.0	10	0.085	0.059	0.19	$3.8\cdot10^{-5}$
(Ti−2.05/Al−3.1)×20	103.0	9	0.07	0.025	0.12	$2.1\cdot10^{-5}$
(Ti−1.3/Al−3.85)×20	103.0	8	0.075	0.016	0.10	$5.4\cdot10^{-6}$
(Ti−2.05/C−3.1)×20	103.0	5	0.02	0.019	0.06	–
(Ti−3.1/C−1.95)×20	101.0	8	0.035	0.024	0.09	$4.4\cdot10^{-3}$
Ti−103	103.0	11	0.2	0.063	0.26	$2.5\cdot10^{-4}$

Note. The diameter of the hole in the silicon frame is 2.5 mm, σ — standard deviation, $\Delta p_{\max}$ — the maximum pressure drop among N attempts. T (633 nm) — filter transmittance measured at wavelength 633 nm.

of the layer in the period was due to a change in the speed of passage of the substrate over the corresponding magnetron target. Assuming that the thickness of the layer depends linearly on the time the substrate is above the magnetron target (which means it is inversely proportional to the speed of uniform motion of the substrate), it is possible to determine the periods of both multilayer structures by measuring the angular dependence of the reflection coefficient at grazing angles (on the PANalitical X'Pert PRO diffractometer, $\lambda = 0.154$ nm), calculate the inverse proportionality coefficient between the speed of passage of the substrate over the magnetron target and the thickness of the layer of the test material deposited in one pass.

Additionally, the accuracy of calibration was checked on homogeneous films with a thickness of 20−30 nm by comparing the measured angular dependence of the reflection coefficient at a wavelength of 0.154 nm with the calculated curve.

For comparative testing of filters for strength, films of the same composition were mounted on polished silicon frames with a round through hole with a diameter of 2.5mm in the center. Then the samples were sequentially placed on a stand, on which a pressure drop was created between the sides of the film due to the gradual pumping of air from one side, and with the help of a mechanical pressure gauge VP4-UU2 (scale step — 0.01 atm), the limit drop at which the film samples were damage was measured. Since the magnitude of the drop at which the rupture occurs depends, among other things, on the presence of defects in the free-standing film (pinholes, folds, etc.), several (6−12) tests were carried out, and the results were averaged.

To measure the transmittance coefficients in the visible and SXR wavelength ranges, films were mounted on silicon frames with a hole with a diameter of 8−10 mm. The transmission coefficients at a wavelength of 633 nm (red light) were measured at a stand using a He−Ne laser (LGN-207A) and an FD-24K photodiode with an adjustable signal amplifier. The relative error of the transmission coefficient measurement is — 10%.

Spectral measurements of transmission coefficients in the SXR wavelength range were carried out on a laboratory reflectometer [12], which includes an X-ray tube with replaceable anodes, a monochromator spectrometer RSM-500 and a receiver (photocathode from CsI). Titanium and carbon with emission lines $\lambda = 3.14$ and 4.47 nm, respectively, were used as anodes.

2. Results

For comparative studies, films with a total layer thickness of about 100 nm were produced. Filters of this thickness make it possible to obtain a high, more than 70%, transmittance in the spectral range „of the water window “ with a sufficiently high level of visible radiation suppression (better than 10^3).

It should be noted here that it would be more correct to compare the strength of film filters of different compositions having similar transmission coefficients in the working (SXR) wavelength range (in this case, their thicknesses may differ noticeably). However, at the initial stage, when many different variants of structures are considered and the most durable ones are identified, an easier-to-implement, though

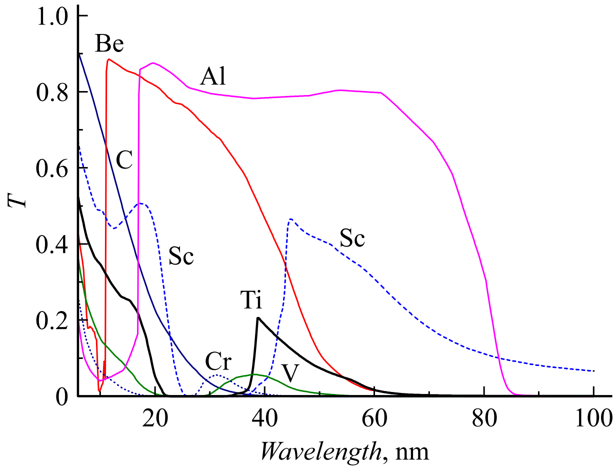

Figure 2. Calculated transmission spectra of filters with a thickness of 100 nm in the EUV wavelength range. The optical constants are taken from site [26].

not entirely correct comparison of film filters of the same thickness seems acceptable.

Beryllium, aluminum and carbon were proposed as interlayers to the titanium filter. The interlayer materials were selected for the following reasons. Beryllium at the same thickness is more transparent in the spectral range „of the water window“than titanium (Fig. 1), and the substitution of titanium with beryllium increases the transparency of the filter. The introduction of aluminum layers into the Ti-containing filter improves its blocking properties in the visible and infrared wavelength ranges. Carbon interlayers are interesting because their presence reduces the transparency of the filter in the EUV range, including in the titanium bandwidth in the wavelength range of 38–60 nm (Fig. 2). The presence of this band in the transmission spectrum of the filter may be undesirable.

Table 1 shows the results of testing Ti/Be, Ti/Al and Ti/C multilayer structures with different thickness ratios in the period. In addition to the structure of the films and their thicknesses (H), the number of tested film samples (N) and the average pressure drop at break ($\langle \Delta p \rangle$), the table also shows the values of the standard deviation (σ), the maximum pressure drop among all tests of this structure ($\Delta p_{\max}$) and the measured transmission coefficients of filters at a wavelength of 633 nm.

As already noted, microscopic defects in the films (for example, pinholes appearing at the stage of separation of the film from the substrate or caused by the penetration of acid vapors from the surface through the surface of the film and the release of hydrogen during their reaction with the sublayer) have a significant effect on the limiting value of the pressure drop at which the film breaks. In addition to the defects of the film itself, the size of the spread may be influenced by the heterogeneity of the distribution of the film material over the aperture of the hole, the features of the edge of the holes, etc. Since we are interested in the strength of the film filter, which is determined by both the strength of the film material and the defects present in it, as a criterion for comparison, we use the pressure drop averaged over several tests for rupture. Taking into account that the maximum pressure drop is sustained by films with a minimum number of defects, the values of the maximum pressure drop at break ($\Delta p_{\max}$) should be proportional (approximately, since the number of attempts is limited, and in addition, for different film compositions, the deflection value may differ) to the stress at break in films of this composition.

Table 1 shows that Ti/C multilayer filters are the least durable. This is presumably due to the high fragility of the carbon layers. Also, the strength is probably influenced by large internal stresses in these structures (large internal stresses can lead to microcracks and ruptures). Apparently, it was due to high internal stresses that it was not possible to produce free-standing (Ti–4.1 nm/C–0.95 nm)×20 multilayer films with a thickness of carbon layers in a period of about 1 nm (films in the process of dissolution the sublayers collapsed and crumbled).

Ti/Al structures showed slightly better results compared to Ti/C. It should be noted that in the Ti/Al pair, the average pressure drop to rupture weakly depends on the ratio of layers in the structure (Table 1). It is not excluded that this behavior may be due to a significant influence on the strength of interlayer boundaries. Nevertheless, with an increase in the titanium content, the strength of the Ti/Al films themselves increases somewhat, judging by the values of the maximum differences in rupture.

The greatest strength of the considered multilayer variants was demonstrated by Ti/Be filters at a fraction of the thickness of Ti in a period of about 0.6. With a decrease in the proportion of titanium in the period, the strength of the filter decreases, which is probably due to an increase in the volume of brittle beryllium. However, with a decrease in the thickness of beryllium layers, the strength of Ti/Be filters also decreases. The reasons for this remain unclear, but it is possible that, as in the case of carbon interlayers, with a layer thickness Be close to 1 nm, internal stresses in the film increase.

A somewhat unexpected result was shown by a filter made of a homogeneous titanium film removed from the Y-sublayer, which, with the same thickness, turned out to be stronger than all tested variants of multilayer Ti-containing filters.

From the point of view of the lowest transmittance in red light, as expected, Ti/Al filters with a large proportion of Al content are the best. Ti/C filters are the least effective in terms of blocking visible radiation due to the transparency of C layers. Ti and Ti/Be filters demonstrate intermediate results, providing, at a thickness of about 100 nm, a decrease in the intensity of transmitted light in the visible range by more than 1000 times.

In addition to beryllium, another material that is calculated (Fig. 1) to have a lower absorption coefficient than titanium is scandium (at wavelengths beyond $L_{2,3}$

Table 2. The ultimate pressure drop averaged over N attempts ($\langle\Delta p\rangle$) between the sides of the film with a thickness of H, at which the film is damaged

Structure, nm	H, nm	N	$\langle\Delta p\rangle$, atm	σ, atm	$\Delta p_{\max}$, atm	T (633 nm)
(Ti−3/Sc−2)×20, Ti−2	102.0	6	0.087	0.077	0.24	$4.2 \cdot 10^{-4}$
Ti−3, (Cr−1.95/Ti−3)×20	102.0	–	–	–	–	–
Cr−100.6	100.6	6	0.153	0.106	0.28	$9.5 \cdot 10^{-6}$
(V−1/Al−4.02)×20	100.4	9	0.10	0.036	0.16	–
(V−2/Al−3)×20	100.0	10	0.163	0.087	0.38	$1.6 \cdot 10^{-5}$
(V−3/Al−2)×20	100.0	7	0.136	0.071	0.27	$4.2 \cdot 10^{-5}$
(V−3.97/Al−1)×20	99.4	6	0.127	0.048	0.17	$2.0 \cdot 10^{-4}$
V−110	110.0	11	0.213	0.092	0.40	$2.0 \cdot 10^{-4}$
Al−150	150.0	26	0.057	0.03	0.12	–
Be−150	150.0	11	0.092	0.069	0.24	–

Note. The diameter of the hole in the silicon frame is 2.5 mm, σ — standard deviation, $\Delta p_{\max}$ — the maximum pressure drop among N attempts. T (633 nm) — filter transmittance measured at wavelength 633 nm.

absorption edge ($\lambda > 3.15$ nm)). But scandium has a significant drawback — high chemical activity, which does not allow using the available technology (using a metal sublayer) to produce homogeneous Sc films. Nevertheless, if Sc is layered with a chemically more resistant material, then with small proportions of scandium content in the period, it is possible to produce a multilayer Sc-containing film structure. In particular, we managed to produce samples (Ti−3 nm/Sc−2 nm)×20 of a multilayer filter. As can be seen from Table 2, the Ti/Sc filter demonstrates relatively low mechanical strength. The presence of areas with pinholes on Ti/Sc films indicates that in part the low strength may be due to partial etching of scandium layers during the dissolution of the sublayer.

An attempt was made to manufacture a Cr/Ti-multilayer filter with a thickness of chromium layers about 2 nm. This film structure twisted and collapsed during the separation of the film from the substrate, which is apparently caused by large internal stresses in the Cr/Ti film and the fragility of the Cr layers. The relatively high fragility of chromium films is also indicated by the presence of cracks along the edges of the sample observed during the manufacture of filters from a homogeneous Cr film with a thickness of about 100 nm. Despite the presence of cracks, it was possible to produce small aperture test samples from Cr and tests them (Table 2). We note that chromium filters with the same thickness better suppress radiation at a wavelength of 633 nm than titanium filters.

Interesting results were obtained on V-containing filters. Like titanium, vanadium is a fairly ductile metal. As can be seen from Table. 2, V/Al-multilayer filters show the greatest strength at a fraction of vanadium in the period of about 0.4. In terms of tensile strength, such filters are comparable to the best Ti/Be filters. Nevertheless, homogeneous vanadium films of comparable thickness removed from the Y sublayer turned out to be more durable (as in the case of titanium).

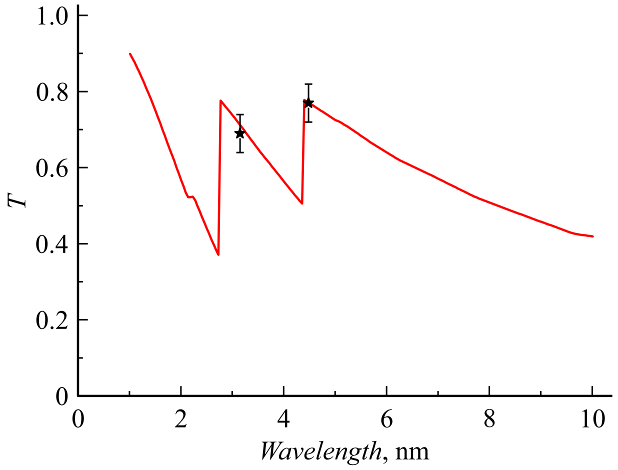

Figure 3. Calculated (solid line) and measured (asterisks) at wavelengths of 3.14 and 4.47 nm on the laboratory reflectometer transmittance of the multilayer (Ti−3.1 nm/C−1.95 nm) × 20 filter.

In Table. 2, for comparison, the previously obtained test data for the strength of homogeneous Al and Be filters with a thickness of 150 nm are presented. As can be seen, even with a thickness of one and a half times greater, their tensile strength is generally inferior to the strength of most of the filters considered here, including Ti/Be and V/Al. The disadvantages of the Be and Al filters include high transparency in the bandwidth in the EUV range (Fig. 2).

For several film structures, the transmission coefficients of filters at some wavelengths in the SXR range were measured. As can be seen from Figs. 3 and 4, the values

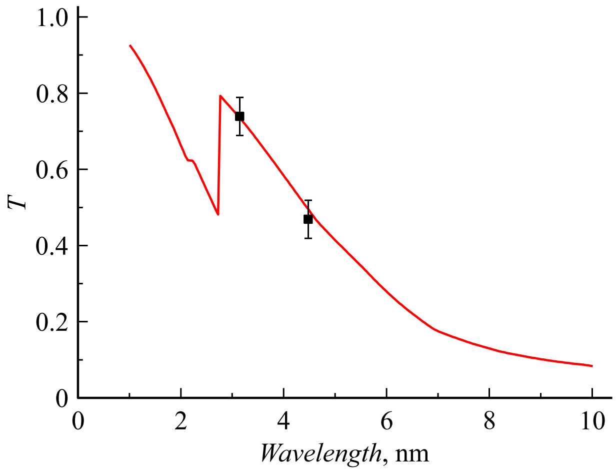

Figure 4. Calculated (solid line) and measured (squares) at wavelengths of 3.14 and 4.47 nm on the laboratory reflectometer transmittance of the multilayer (Ti$-$2.1 nm/Al$-$3.1 nm) $\times$ 20 filters.

of the transmission coefficients of Ti/C and Ti/Al filters measured in the spectral range „of the water window“ are close to the calculated ones. This could be expected, since the impurities usually present in the film (oxygen in the form of oxides or carbon in the form of hydrocarbon contaminants) have almost no effect on the transmission coefficient of the filter in this region of the spectrum.

Conclusion

In this paper, we considered some variants of the composition of multilayer film filters with a thickness of about 100 nm with a high transmittance in the spectral range „of the water window“ (2.3$-$4.4 nm). The main attention was paid to the tensile strength of the filters and their blocking properties in the visible wavelength range. From the point of view of mechanical strength, filters made of homogeneous titanium and vanadium films showed the best result among the tested structures. Considering that the transmission coefficients of vanadium films with a thickness of 100 nm and titanium films with a thickness of 160 nm are close in the SXR range, titanium filters (from the point of view of strength and blocking properties) look preferable. Vanadium filters may be of interest if it is also necessary to transmit radiation in the wavelength range between the absorption edges of vanadium and titanium (in the wavelength range of 2.5$-$2.75 nm).

Among multilayer filters, Ti/Be with a titanium fraction in the period of about 0.6 and V/Al with a vanadium fraction in the period of about 0.4 demonstrated high tensile strength. Ti/Be filters with a thickness of 100 nm have a higher calculated transmittance in the spectral range „of the water window“ than V/Al (65$-$85% against 45$-$75%), however , they are inferior to the latter in degree blocking light in the visible range ($4.1 \cdot 10^{-4}$ vs. $1.6 \cdot 10^{-5}$). We note that both structures have a disadvantage associated with the presence of a bandwidth in the UV range with an estimated transmission coefficient reaching 20%.

Compared with filters made of homogeneous Ti and V films of the same thickness, multilayer Ti/Be filters have a higher calculated transmittance in the spectral region „of the water window“, and a multilayer V/Al filter — a better degree of radiation suppression in the visible and infrared wavelength range.

Of the materials considered for filters, aluminum and (to a lesser extent) chromium have high blocking properties in the optical wavelength range. However, homogeneous aluminum films are fragile, and chromium — are rather brittle, so it is preferable to use these materials as interlayers.

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Conflict of interest

The authors declare that they have no conflict of interest.

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