Development of multilayer X-Ray optics and it's application in physical experiments and scientific instruments in IPM RAS

N.I. Chkhalo, N.N. Salashchenko

1. Introduction

Researches in the field of multilayer x-ray optics in our collective have been started in 1978 within the limits of Institute of applied physics AS USSR, and in this we are very lucky. Greater role in development of these works support academician ***A.V. Gaponov-Grekhov*** has played. If members of the staff at the beginning more clearly represent the requirements for multilayer structures, to the substrate and to the technology, most of all, think about it, would continue his research in the field of ultra-thin semiconductor films and superlattices. However, in the "courage of ignorance" we took on the job. The important role played publications in 1972. of ***E. Spiller*** and in 1977 of ***A.V. Vinogradov*** and ***B.Ya. Zeldovich*** in which the reality of practical manufacturing of multilayer X-ray mirrors has been shown. In 1991 for a cycle of researches in this direction a number of IPM employees have been awarded the State prize of the USSR. In the group of the prize winners 6 persons were from IPM: ***A.D. Akhsakhalyan, S.V. Gaponov, V.M. Genkin, B.M. Luskin, Yu.Ya. Platonov*** and ***N.N. Salashchenko***, from FI AN - ***A.V. Vinogradov*** and from the Kharkov polytechnic institute - ***A.I. Fedorenko***.

Currently, four out of the team are in foreign countries (***VM Genkin, BM Luskin, YY Platonov, A. Fedorenko***), and only one of them, ***Yu.Ya. Platonov***, continues to deal with the problems of X-ray optics. On behalf of *Yu.Ya. Platonov* our collective has grown up very qualified and demanded researcher for the USA. The expression "grow up", of course, is conditional, *Yu.Ya*. grew with us, and we grew with him, all with feedback. In this case was positive feedback. It is a pity that it was grown for the United States. Talented ***Yu.Ya. Platonov*** could be useful to us in our country.

Winners of last State prize of the USSR (1991), employees of IPM AS USSR, for a cycle of works on multilayer X-ray optics.
From left to right: S.V. Gaponov, N.N. Salashchenko, A.D. Akhsakhalyan top row; V.M. Genkin, B.M. Luskin, Yu.Ya. Platonov bottom row.

Important role in the development of multilayer X-ray optics in IPM RAS played A.A. Fraerman (left) and S.A. Gusev.

However, as always, a great contribution to the development of multilayer X-ray optics gave not only the employees who are awarded by the State Prize, but a number of other staff who, in principle, could also be qualified by the award, but as it is know, authors number is directive limited. The contribution of some of them, especially at the initial stage of the work, when laying the physical and technological basis of multilayer X-ray optics, it is difficult to overestimate. And today they are ready to come to the aid practically in any questions and, that is important, come. In particular, ***A.A. Fraerman*** contributed greatly to the establishment the theoretical foundations for diagnostic of multilayer structures (MLS). ***S.A. Gusev*** developed methods of electron microscopy to study the surface roughness, which are at least potentially could be used as substrates for the MLS (at that time we even did not know whether there are substrates with subnanometer surface roughness, in principle), methods of cross-section preparation and study the MLS by electron microscopy. He took a part in studying properties of the first multilayer X-ray mirrors at synchrotron VEPP-2M, Novosibirsk.

S.V. Gaponov and N.N. Salashchenko.

Earlier they often sat so together and discussed plans of the works, new offers and received results. Only were a little bit younger.

In the years the works of the team in the field of multilayer X-ray optics and it’s applications in various physical experiments and scientific instruments in 2008 were awarded by the prize A.G. Stoletov of the Russian Academy of Sciences. The prize winners were ***N.N. Salashchenko*** and ***N.I. Chkhalo***.

The important role in development and popularizations of researches in the field of X-ray optics have played at the beginning (since 1997) Workshop on X-ray optics, and then a section in Symposium “Nanophysics and Nanoelectronics” which have been organized in IPM RAS under ***S.V. Gaponov's*** initiative.

Yu.Ya. Platonov, S.V. Gaponov, N.N. Salashchenko. September 2012, IPM RAS.

They are again, after many years together (at least in the photo).

Yu.Ya. Platonov, a U.S. citizen and a member of the Japanese company Rigaku, is now uncommon.

In the early years we had no measuring base and measured characteristics of multilayer mirrors in soft X-rays in Institute of Nuclear Physics, Novosibirsk and partially in NPO "Burevestnik", Leningrad. Only later, approximately, in 2-3 years, we have understood, that without own measurements we can not developing further more technology because operative measurements of characteristics of the mirrors on working wavelengths are necessary. And it was a push for creation of own measuring base. It is worth noting that as a result we are perhaps the only one group in the world in the field of X-ray optics, which has its own technology and measuring base for certification of the elements of X-ray optics, both on reflection, and on transition on operating or close to them wavelengths. Recently, this also applies to precision measurements of roughness and shape of the substrate surfaces and mirrors. In this connection it would be desirable to note ***I.G. Zabrodin*** big contribution and who participated in development of the technological and measuring equipments at all stages of development of multilayer X-ray optics in IAP and in IPM, and who continues to work actively in our collective now.

Andreev
Sergey Sergeevich

A little bit later (in 1990) in our collective has came ***S.S. Andreev*** who too early left (in 2011) us, but its contribution to adaptation of magnetron sputtering technology for MLS deposition for X-ray optics, in development of the process equipment and, the main thing, in training of young technologists, impossible to overestimate. The natural successor of ***S.S. Andreev*** is its student ***V.N. Polkovnikov***, on whom it imposes great responsibilities, not only to obtain their own scientific results, but also to educate the next generation of technologists.

Practice shows that good technology is growing very slowly, 7-10 years, and that we all need to consider, when summing up the intermediate results and ask, what did you do when your peers (theorists or researchers working on standard equipment) already published so much work.

From the start the studies were conducted in a large scientific cooperation, in which initially interaction with employees of BINP, ***E.S. Gluskin*** and ***G.N. Kulipanov***, played important role. After some time in the INP appeared ***N.I. Chkhalo*** whom ***E.P. Kruglyakov*** in 1986, sent to us in the IAP, on training, just on the subject of laser deposition of MLS for X-ray optics. Now ***N.I. Chkhalo*** is our leading scientific employee who appreciably defines directions of works in the field of multilayer X-ray optics in IPM RAS.

In the further the big participation in researches taken employees of A.F. Yoffe PTI RAS (***S.V. Bobashev, L.A. Shmaenok***, etc.), P.N. Lebedev FI AN (***I.I. Sobelman, V.A. Slemzin, A.P. Shevelko***, etc.), A.V. Shubnikov IC RAS (***M.V. Kovalchuk, S.I. Zheludeva***, etc.), the Moscow State University (***A.V. Andreev, Y.V. Ponomarev***, etc.), NPO "Burevestnik" (***I.A. Brytov, A.Y. Grudsky***), Plasma physics Institute, the Netherlands (***F. Bijkerk***), Synchrotron center BESSY, Germany (***F. Schaefers***), etc.

Actually before, and now all of the works, the results obtained and the problems are extensive and in-depth are discussed in the team. And first of all, especially the current issues are discussed on so called “council of elders”. Such “council of elders” can have variable composition, depending on the subject matter, but as a rule, a quasi-permanent staff includes leading members of the department. One of the current sessions of the “Council of Elders” is shown in the photo.

“Meeting” of quasi-constant “councils of elders” in a direction of multilayer X-ray optics.

E.B. Kluenkov, N.N. Salashchenko, A.D. Akhsakhalyan, N.I. Chkhalo, V.I. Luchin.

It is necessary to recognize, that our work is mainly male, experimental, frequently connected with development of the special equipment. Therefore for work we, as a rule, take mainly a male staff. But it turned out that it is not so. Below is a separate photo of our women. First, their amount is not too small, and secondly, their utility in the work no one can dispute.

Women of our department (from left to right):

sit - E.N. Sadova,
Z.L. Kozhevnikova,
M.M. Barysheva;

stand - M.V. Zorina,
N.A. Korotkova,
V.A. Tyurina.

In this paper, we present the most interesting and important research results in the development of multilayer optics for short-wavelength range (λ=0.01 - 60 nm) and its applications in various physical experiments and scientific instruments, obtained in IPM over the past 20 years, i.e. over the years of existence of the IPM RAS.

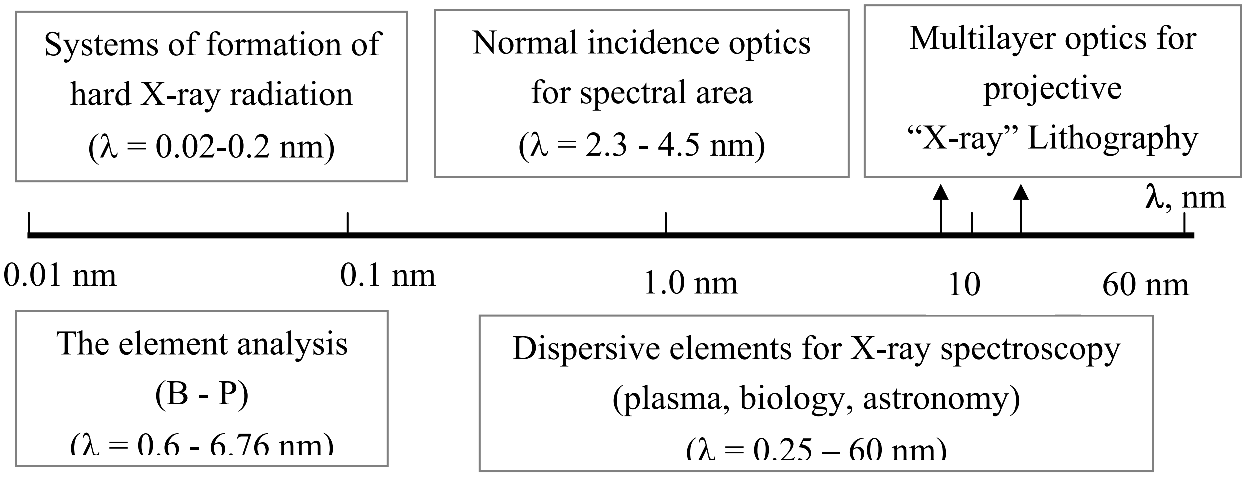
Note, that the multilayer X-ray optics with the MLS periods d $\approx$1–30 nm is a typical nanotechnology product. As an example, multilayer dispersive elements and imaging optics with the periods d =1-2 nm can serve at a number of effectively reflecting periods N = 300-1000 when the deviation of the MLS period value on depth and in a plane should not exceed $\delta d \approx d/N \approx (1\text{-}5)\cdot 10^{-3}$ nm. It is clear, that such accuracy define requirements to stability of technological process of MLS deposition.

For perfection of the technology it is necessary to know well such internal parameters of multilayer structure, as the period and the ratio of the thickness of the layers in the period, real density of the layer materials, roughness and width of interfaces between layers. In the cases when thickness of the layers are close to crystallographic parameters of materials (h$\approx$0.3-0.7 nm), it is difficult to speak about width of the interfaces and on the specific parameters of the layers themselves. The short period MLS diagnostics is an independent and important problem.

Directions of researches of our team are connected with creation of element optical base for a spectral range λ≈0.01 - 100 nm and include following sections.

- Perfection of multilayer "X-ray" optics
  - ✓ MLS optimization for various spectral ranges
  - ✓ Development of MLS diagnostics methods and studying of physical processes in MLS
- Development of manufacturing techniques of "X-ray" optics elements
  - ✓ Development of the process equipment and technology of MLS deposition
  - ✓ Perfection of focusing and collimating systems for "hard" (λ≈0.05-0.2 nm) X-ray radiation
  - ✓ Development of methods of precision correction and metrology of the surface shape
  - ✓ Development of manufacturing techniques of free standing (without substrates) X-ray optics elements (multilayer splitters, polarizer, phase shifters and absorption filters)
- The perspective instrument making connected with development of the equipment for diagnostics of multilayer film structures and devices based on multilayer X-ray optics elements
  - ✓ The new diagnostic equipment
  - ✓ The equipment for the control and corrections of the surface shape
  - ✓ Stands of projective "X-ray" lithography and microscopy.

It is necessary to note, that the research directions are appreciably defined by needs of various practical applications. In particular, suggest the development of multilayer optics for use as multilayer dispersion elements for X-ray plasma diagnostics and X-ray fluorescence elemental analysis, for formation of hard X-ray beams (λ = 0.01-0.2 nm) to increase (in magnitude) efficiency of X-ray tubes, for equipment of channels on synchrotrons. In particular, assume development of multilayer optics for applications as multilayer dispersive elements for plasma X-ray diagnostics and for the X-ray fluorescent element analysis, for shaping of hard X-ray radiation beams (λ = 0.01-0.2 nm) with the purpose of increase (in tens times) efficiency of X-ray tubes use, to equip channels of synchrotrons etc. In the last decade, a special place, we began to engage in research, to develop high-precision normal incidence optics of soft X-ray range for various problems of modern physics and engineering. For example, for creation on their basis of objectives for projective EUV lithographers (wavelengths of 13.5 nm, or 6.7 nm) or for biological microscopes in spectral areas of “water and carbon” transparency windows (λ=2-5 nm), for development space telescopes for problems of X-ray astronomy.



The basic applications of the multilayer “X-ray” optics developed in IPM RAS.

A key parameter characterizing the imaging system is the spatial resolution, which is limited by the diffraction of light at the exit aperture of the optical system. According to Rayleigh criterion the diffraction limit of optical system resolution $\delta x = k \cdot \lambda / NA$ is defined by wavelength λ and the objective numerical aperture NA ($NA = n \cdot \sin\alpha$, n - index of refraction of environment between an objective and an image plane, α - half of exit angular aperture of an objective), where k - factor of proportionality. The factor k is defined by coherent properties of light and way of object illumination and changes within the limits from 0.25 up to 0.77. For not coherent irradiation it makes k =0.61. Thus, the resolution of image systems makes about half of wavelength, or for an optical range - more than 100 nm. This restriction came into a conflict with the development of nano-world (the characteristic dimensions less than 100 nm), which, in particular, at one time threatened violation of Moore's law - doubling the density of electronic components (transistors) on a chip every eighteen months.

In the production of microchips (photolithography), this was the reason for seeking alternative ways of formation of microstructures, for example, by scanning probe microscopes. Extensive research carried out in the direction of multi-beam and electron and ion projection lithography, technology of nano-printing. All of these methods have already found their niche and continue to evolve, however, for the high volume production of chips traditional photolithography was no competition. This is made possible by the widespread use of so-called resolution enhancement techniques. Among them are purely optical, reducing the coefficient of *k*, for example, by oblique illumination and optimize the coherence properties of the radiation incident on the mask, the application of phase correcting or absorbing layers on the mask. Almost one and a half were able to improve the resolution by using immersion liquids. Immersion potential has not been exhausted, and the search for new liquids with high refractive index is continued. Not optical resolution enhancement methods include technologies that use physical and chemical properties of the photoresist, and stage-by-stage patterning, when, for example, the elements are formed through a period (double patterning).

Modern scanners with working wavelength of 193 nm and standard technological processes already provide the resolution down to 22 nm. Practically five times “overcoming” the diffraction limit of resolution related to more complicated and, therefore, cost of equipment and production of chips. Instead of a simple patterning procedure that includes deposition a photoresist on the wafer, exposure and development - there are dozens of new operations.

If in lithography still it is possible to promote noticeably in the area of nanometer resolution, in microscopy of all methods of the resolution improvement, in fact, is only immersion. In achieving the ultimate spatial resolution the classical microscopy actually already exhausted its possibilities. An alternative is the optical near-field microscopy, reaching the resolution in the tens of nanometers. However, as in lithography, the price for this is complex scanning system and a number of functional limitations.

Achievement nanometer resolution in classical schemes of microscopy and photolithography is possible if to use shorter wavelengths (10 nm and even shorter). Efficiency of such approach is well visible on the results received in the field of projective EUV lithography at wavelength of 13.5 nm. Already on the first experimental lithographic facilities the resolution was better than that obtained with the “advanced” lithographers, operating at a wavelength of 193 nm. Recently, for the next generation lithography a new wavelength $\lambda \approx 6.7$ nm is actively discussed.

Alongside with prospect of increase in the spatial resolution the ranges of soft X-ray and extreme ultra-violet radiation (λ ~1-60 nm) for some reasons are rather interesting for experimental physics, astronomy, chemistry and biology. Energy levels of the majority of atoms lay in this spectral range that causes resonant character of interaction of the radiation with substance, providing, thus, researchers by unique and a trustworthy information about an internal structure of atoms and their interaction with the neighbor atoms. Maxima of radiation of laboratory plasma and emission lines of the basic impurity ions lay in this range that does X-ray spectroscopy of the plasma most convenient for diagnostics. Emission lines of radiation of the Sun crown is one of the most authentic sources of information on the physical processes proceeding on the Sun. Weak scattering and large enough penetration into the substance allow tomography study of “thick” objects, which in combination with nanometer resolution opens new perspectives for studies of condensed matter in physics, chemistry and microbiology. Weak absorption of radiation with energy less than the ionization potential and absorption jump in excess of the binding energy in some cases provide such a high absorption contrast images (for example, proteins in the aquatic environment), that to obtain high-quality images of the object required dose by several orders of magnitude smaller than that when use “non-resonant” X-rays or fast electrons. In some cases it allows studying “alive” biological samples with resolution in tens of nanometers and organic objects being in “thick” (> 1 μm) matrix, for example, in water suspension, with units of nanometers resolution.

The combination of classical X-ray methods (diffractometry, X-ray and photoelectronic spectroscopy, small-angle scattering, etc.) with nanometer spatial resolution opens unique prospects in nanophysics of the condensed matter.

The serious restriction until recently constraining possible advantageous use of short wavelength radiation for achievement of nanometer spatial resolution was absence of diffraction quality (the resolution is limited by the diffraction of light) high aperture optics. In this direction last years our collective carries out wide enough researches (the breadth of the researches is defined not only by our limited forces, but also by financial opportunities) in the field of manufacturing, metrology and application of the precision X-ray optics. Much attention is paid to short-period multilayer mirrors, which are the basis of the optics, as well as methods for studying super-smooth surface roughness of substrates for multilayer mirrors in the whole spectral range of spatial frequencies that determines the formation of images. Alongside with obvious applications in lithography and microscopy, we consider opportunities of application of precision X-ray optics for surface diagnostics with nanometer spatial resolution and for formation of super-strong electromagnetic fields.

Last years the big attention was given to researches on optimization of multilayer optics for projective lithography on the wavelength of 13.5 nm. These researches include increase of mirrors reflectivity, development of methods for stress compensation in Mo/Si reflecting MLS, creation precision aspherical imaging optics, development of a quality control of surface roughness, development of methods of restoration extremely expensive aspherical substrates which is needed after unsuccessful deposition of multilayer covering. The failure of coating is very likely, because manufacturing of optical elements for schemes projection lithography requires deposition of MLS with a given distribution of periods over the aperture of the substrate with an accuracy of 0.01 nm. The next generation of nanolithographs is supposed to be developed on shorter wavelength of radiation, about 6.7 nm that, at least twice, raises requirements to the accuracy of the optical elements.

In the field of multilayer optics for the solar investigations it is possible to note, that practically all Russian space stations with specialized telescopes for studying the Sun in the spectral range of 13-30 nm, have been equipped with multilayer mirrors made in IPM RAS. For example, station “CORONAS-F” is equipped by the mirrors made in IPM RAS and in Optics Institute, Paris. For this project, an aspherization process of spherical substrates with using of additional buffer multilayer coating with a required film thickness distribution over the surface was developed. Subsequently reflection coating with a period corresponding to the working wavelength is deposited onto the substrate. By such technology off-axis parabolic mirrors with multilayer *Mo/Si* MLS for the spectral ranges of 17.5 and 30.4 nm having an angular resolution around 1 arc. second have been done. In these spectrum parts there are the most intensive lines of ions *Fe IX* - *Fe XI* and *He II*, irradiated by the solar plasma in a wide range of temperatures from

$2 \cdot 10^4$ up to $1.3 \cdot 10^6$ K, and high registration efficiency is reached when using normal incidence multilayer mirrors in combination with thin-film filters. New tasks of “X-ray” astronomy (the nearest 5-10 years) assume the further essential development of manufacturing techniques of precision image optics and new generation of spectral filters which about would be told below in detail.

As a result of extensive researches, including the member’s of the IPM team, the field of application of the multilayer “X-ray” optics repeatedly extended, most of the methods of traditional optics (collimation, focusing, imaging, polarization of the radiation) have become available for the extreme ultraviolet and X-ray spectral regions. In fact, we are on the threshold of a new technological jump associated with the development of short-wavelength range. Although a number of important issues remain to be resolved, this range is extensively developed. In many ways, the current state of affairs in the field of multilayer optics associated with the work of the team of researchers from IPM RAS.

IPM staff which actively work (or worked earlier) in the field of multilayer "X-ray" optics (from the left-to the right)

<u>sitting</u>: A.I. Kuzmichev, A.D. Akhsakhalyan, S.V. Gaponov, N.N. Salashchenko, V.I. Luchin, E.B. Kluenkov, A.A. Fraerman.

<u>standing</u>: Z.L. Kozhevnikova, S.D. Starikov, V.A. Tyurina, V.V. Pogov, B.A. Volodin, I.A. Kaskov, P.K. Gajkovich, N.A. Korotkova, S.A. Gusev, V.N. Polkovnikov, I.G. Zabrodin, M.N. Drozdov, L.A. Mazo, M.N. Toropov, M.V. Zorina, S.A. Churin, A.Ye. Pestov, Ye.N. Sadova, N.N. Tsybin, D.Ye. Paryev, M.M. Barysheva, S.Yu. Zuev, A.Yu. Klimov, N.I. Chkhalo, A.I. Kharitonov, A.A. Akhsakhalyan, A.Ya. Lopatin, Yu.A. Vainer.

2. Technological and research support of works in the field of multilayer XEUV optics

Basis of works in the field of multilayer X-ray optics is their technological and research support. In our group in first ten years the manufacturing techniques of multilayer X-ray optics elements were based on a pulsed laser deposition and developed in parallel with development of this method. This activity was within the frame of IAP AS. We must bear in mind that, because the laser deposition allows targets as small as 1×1 cm^2, this technique is very useful when you need to search for the best materials for MLS, which should work in a specific spectral range. And while the size of the mirrors was limited by 5 cm, the laser deposition technique completely satisfied the researchers. But when there was a need of mirror substrates for larger sizes, up to 20-30 cm, it became clear that it was necessary to develop the technology of magnetron sputtering. For a while it was a combination of both technologies, and then we completely passed to the magnetron sputtering.

Photo of technological group for MLS deposition. From left to the right: V.N. Polkovnikov, B.A. Volodin, N.N. Tsybin, L.A. Suslov, D.E. Paryev, L.A. Mazo, P.K .Gajkovich, E.B. Kluenkov, S.D. Starikov.

The transition to the technology of magnetron sputtering has taken about three years. In development of own magnetron installations and technologies the big persistence have shown ***E.B. Klyuenkov, S.S. Andreev, B.A. Zakalov, L.A. Suslov, I.G.***

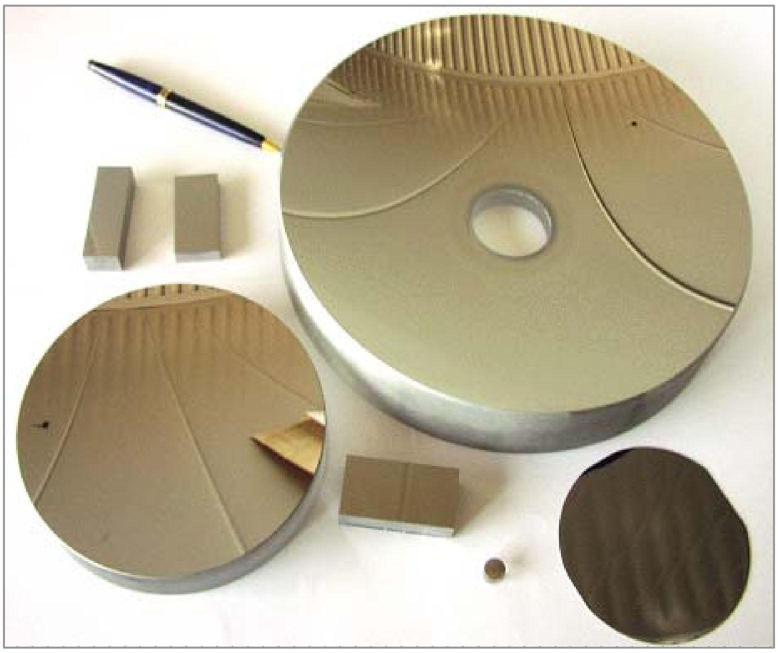
Zabrodin and I.A. Kas'kov. Almost immediately, we stopped at 150 mm diameter magnetrons, which allows at erosion zone of 100-120 mm reliably deposit MLSs with thickness uniformity or with a given distribution of layer thickness up to ≈ 0.1-0.2% at a substrate diameter up to 200 mm. To deposit MLSs onto substrates with a diameter up to 350 mm in parallel the magnetrons with target size of 160×350 mm have been developed.

It should be noted that these were the years of 1993-1994, and we made equipment, which at that moment was for us appropriate, not only on the forces, but also on the financial resources. Therefore we began with installations on the basis of two magnetrons and vacuum chambers with a diameter of ≈ 50 cm. Further, of course, and the number of magnetrons and the vacuum chambers became too small.

Technological installation with six magnetrons for MLSs deposition.
In the foreground shown "owner" of this installation B.A. Volodin.

The most interesting results regarding manufacturing of MLSs for X-ray optics all over the world for today are received with magnetron sputtering technology.

The use of installations with a larger number of simultaneously working magnetrons allows deposition MLSs, in which besides the main reflective layers in a single vacuum cycle "sacrificial", necessary for making free standing MLSs, or stress-compensating, or anti-diffusion layers can be deposited. In particular, technological installations in which simultaneously can work up to six magnetrons have been developed and manufactured.



Typical products of multilayer X-ray optics including a flat mirror in diameter of 100 mm, the concave and convex spheres with the maximal diameter of 260 mm, parabolic and elliptic cylinders.

It is hoped that the magnetron sputtering technology opportunities are not yet fully exhausted, but one has to admit that today, some stagnation in obtaining of new results in multilayer X-ray optics takes place. This stagnation is observed, for example, in the manufacture of short-period MLSs (period d = 1-3 nm), in demand as dispersive elements for X-ray plasma diagnostics, or as imaging optics in the wavelength range λ = 2-5 nm. Great problems occur when deposit materials with high chemical and/or diffusion activity. In this case, as a rule, there are abnormally blurred interfaces and, as a consequence, reducing reflectivity of the mirrors.

In order to study the possibility of controlling the width of the interfaces in the MLSs a technological stand, which combines magnetron (stand includes four magnetron sources) and ion-beam sputtering (there are two ion sources), was developed and produced. Simultaneous application of both sputtering methods may be of interest when MLS consists of materials whose rate of sputtering by magnetron is very low. For example, magnetron sputtering is very difficult apply to pure boron target, interest to which today is very high in relation to the development of optics for the spectral range of 6.7 nm based on the *La/B* materials, potentially interesting for the development of the next generation projection lithography. There are problems with sputtering and other targets on the basis of light elements such as C and B_4C.

The multipurpose stand for MLS deposition by magnetron and ion-beam sputtering methods, including an opportunity of layer surfaces polishing by low energy ions.

Considering, that boron and carbon have anomalously large chemical and diffusion interaction with many materials, in the stand there is an opportunity of ion-beam polishing of each layer surface. For this purpose two low energy ion beam guns are used. We proceeded from the assumption that the “polishing” will remove the top low density (porous) layer and thus preventing (reducing) mixing of films. Developed scale-model multi-technology stand can be seen as a testing ground for the further development of equipment for deposition of multilayer X-ray elements.

In parallel with development of multilayer X-ray optics element deposition technology the methods of diagnostics and the measuring equipment for MLS characterization and certifications are developed. A basis or the first approach of the X-ray diagnostics of MLS, deposited on flat substrates, is the measurement of angular dependences of mirror reflection by small-angle X-ray diffractometry. For such measurements for a long time we used domestic diffractometers DRON-3 and DRON-4 types. And it seemed to us, that everything is all right, that such equipment allows studying even short period MLSs. But about 10 years ago we have bought diffractometers from Philips (then Panalytical) companies and practice of using them showed that only now we can characterize authentically enough the MLS, especially it concerns short period mirrors.

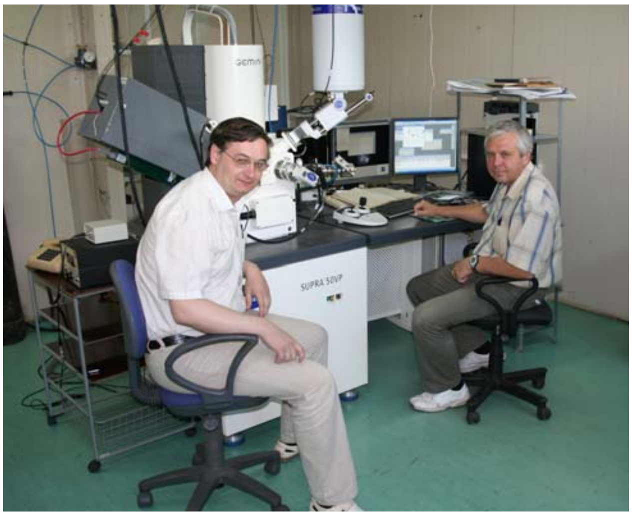
A team of diagnostics and characterization of elements of X-ray optics.

From left to right: S.Y. Zuev, A.E. Pestov, N.I. Chkhalo, Yu.A. Vainer, M.V. Zorina, M.N. Toropov. On the background it is possible to see the reflectometer for studying X-ray characteristics of mirrors in spectral area of 0.6 - 20 nm.

For measurements of characteristics of mirrors in soft X-ray and in VUV ranges (λ=0.6-200 nm) where traditionally synchrotrons are used, we have developed original laboratory raflectometrs on the basis of own (very successful) dismountable X-ray tubes (λ=0.6-20 nm) and gas-discharge sources of radiation (also own) for longer wavelength area. In development of the reflectometers the greater role belongs to ***S.Yu. Zuev***. He alone has developed, assembled and adjusted the first reflectometer. The following upgraded version of the reflectometer has been developed under direction of ***N.I. Chkhalo***, and thus we now can certificate any multilayer optics elements both on reflection, and transmission practically on the working wavelengths, not resorting to the synchrotron help. However, comparative (our and synchrotron BESSY-2, Berlin) measurements of test samples, which have shown good coincidence, have been lead and were recognized by experts. It seems appropriate to mention that sometimes we need in precise measurements of characteristics of multilayer optics elements, which are held periodically on synchrotron BESSY-2. Usually the measurements does our old friend ***Dr. Franz Schäfers***, maybe the best in the world experimentalist in the field of synchrotron researches of the soft X-ray optics.

Drs. F. Schäfers and N. Salashchenko. 2003

In addition, using opportunities IPM RAS and, in particular, the center of the collective using, the full complex of MLS properties studying includes atomic force and electron microscopy, and level-by-level mass-spectroscopy of secondary ions.



Investigation of multilayer structures by electron microscope (at the left) carried out by S.A. Gusev and V.N. Polkovnikov, and by SIMS - M.N. Drozdov and N.N. Tsybin.

Earlier with application of a method of standing X-ray waves (together with employees of IC RAS), X-ray diffractometry, EXAFS and Mössbayer spectroscopy (together with employees of the Moscow State University and the St.-Petersburg University), MLS internally structure, processes on interfaces and in layers practically of all types MLSs which are of interest from the point of view of manufacturing of multilayer optics for X-ray range have been studied. These works laid the foundation for the complex study of MLSs and became the basis of modern concepts of the physics and technology of multilayer X-ray mirrors.

Creation of technological and research base of multilayer X-ray optics was accompanied by obtaining of new physical results from which it is possible to recognize as the most significant the following.

- The methods of determination of MLS structural parameters by reflectometry in X-ray spectral range were developed.
- The effect of the self-consistency of MLS interfaces, leading to increasing of spectral resolution of mirrors was predicted and studied.
- Diffusion processes in MLS were studied in details.
- X-ray waveguide modes in MLS were investigated.
- Effects of resonant amplification of diffusion scattering in waveguide type heterostructures were detected and studied.
- New *Cr/Sc* MLS, became the base for the spectral area of the “water window”, was proposed and studied.

It is possible to note, that by the capabilities and technical characteristics created metrological base is unique even at a world level and are widely used both by domestic, and foreign developers and researchers for calibration of X-ray optical elements, spectral equipment and detectors of X-ray radiation.

In conclusion of this section it is necessary to note a great role of talented (and practically unique) designer ***B.A. Zakalov***, collective of a workshop led by ***N.G. Gus’kov*** and a head of technological laboratory ***E.B. Kluenkov*** in creation, together with employees of “Multilayer X-ray optics” division, all variety of the technological and metrological equipment. The equipment practically could no be bought and became as a basis for our scientific and technological researches and achievements.

Developers of the process equipment. At the left-to the right: B.A. Zakalov, V.N. Polkovnikov, I.A. Kas'kov, E.B. Kluenkov, I.G. Zabrodin, N.G. Gus'kov.

3. Physics, technology and diagnostics of multilayer structures for soft X-ray and extreme ultra-violet ranges

With development of technology of synthesis of MLSs with ultra-short ($d \approx 1$-3 nm) periods in X-ray optics have appeared real alternative to the zone plates for imaging with high spatial resolution. It is possible to consider occurrence of multilayer X-ray optics one of revolutionary nanotechnology the end of 20-th century, and for last 30 years the area of its application has repeatedly extended, and the majority of methods of traditional optics (collimation, focusing, imaging, polarization of radiation) became accessible and for soft X-ray (SXR) and extreme ultra-violet (EUV) ranges.

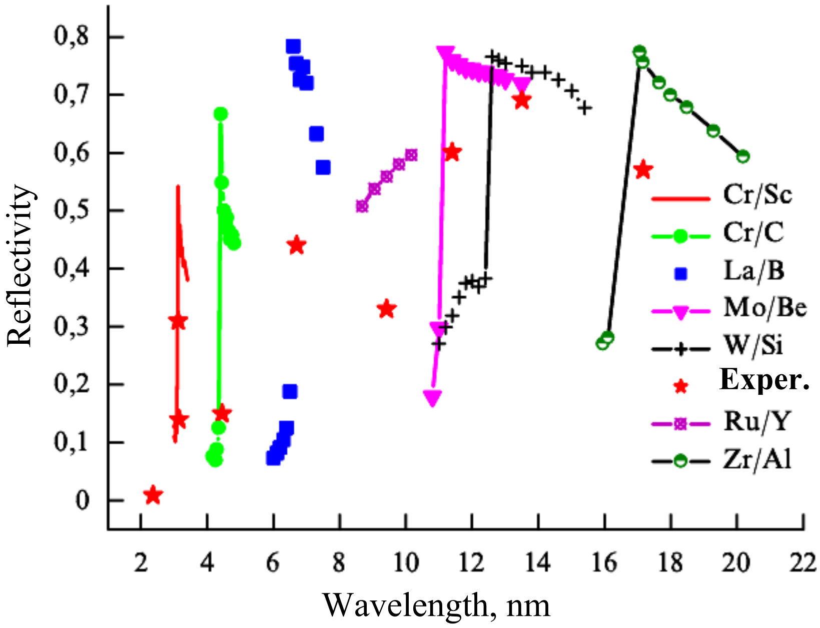
Multilayer X-ray mirrors represent the periodic structures consisting of layers of materials with various optical constants, by analogy with interference mirrors for an optical range. Specificity of X-ray mirrors are extremely small periods (according to Bragg condition at normal incidence the MLS period consisting from two films is about $\lambda/2$), down to 1 nm, big number, up to 1000, of the periods and strong absorption practically of all materials. Necessity of the huge number of the periods is caused by low reflection factors from one border $R_{1,2} \approx |(\varepsilon_1-\varepsilon_2)/4|^2$, where $\varepsilon_{1,2}$ - dielectric permeability of film materials. For example, the reflection factor from a border vacuum-silicon at a wavelength of 1 nm makes $R=2\cdot10^{-8}$, and even for such heavy material as gold it does not exceed $R=6\cdot10^{-7}$.

When choosing materials for MLS for the decision of the specific targets it is necessary to be guided by following basic conditions. First, choose materials with optimal optical constants, providing the maximal reflection on the working wavelength. Secondly, materials of the layers should minimally chemically interact with each other to keep strong optical contrast on the borders. Thirdly, materials of layers should be deposited onto substrates by vacuum methods, and their growth should occur without development of a roughness of the growing film surface. The problem is complicated by the fact that physical and chemical properties of super-thin layers can noticeably differ from properties of a bulk material. So a choice of a new material requires a complex research.

In the first works the MLS materials chosen such a way to provide maximal jump of optical densities (a material with greater Z) / (a material with low Z), types *Au/C*, *W/C*. In the further it has appeared, that the approach when as low absorbing material is used such one whose *K*- or *L*- of absorption edge is a little bit shortly than the working wavelength, is more fruitful. Optimum condition is when the working wavelength corresponds to the area of an abnormal dispersion of the material: in this case the minimal absorption and maximal, in some cases exceeding unit, index of refraction is observed. The choice of strongly absorbing material represents the compromise between absorption of radiation and jump of electronic density on a border. Such approach well illustrates the *Cr/Sc* MLS. Chrome (Z=24) and scandium (Z=21) are close in periodic system of elements, and their difference of electronic densities is small. However in the field of an abnormal dispersion of scandium in a vicinity of L_{III} - edge of absorption (λ_L=3.11 nm) where the real part of the dielectric permeability can be more units, and chrome absorbs poorly, for this pair of materials the maximal reflection coefficients are observed. Since the first works on *Sc*-containing mirrors, which then had wide continuation, as pair material chrome was chosen, as the least interacting with scandium.

By present time, there is a limited set of pairs the materials corresponding above listed requirements. From the most perfect short period mirrors it is possible to note mirrors on the basis of W/B_4C which minimal periods reaches about 1 nm. On the basis of such MLSs it is possible to make mirrors of normal incidence for radiation with the minimal wavelength ≈ 2 nm that can be of interest, for example, for reception the image of objects in spectral area close for *K*-edge of oxygen absorption ($\lambda \geq 2.36$ nm). However already in the field of $\lambda > 3.1$ nm essentially higher reflectivity have MLS on the basis of scandium.

In figure the calculated and experimentally received reflection coefficients of some the most perspective MLSs in the spectral range of 2.3-20 nm, demonstrating the modern state of affairs in technology of synthesis of normal incidence multilayer mirrors for SXR and EUV ranges, are given. For short-wave border of the considered range, λ=2.3 nm, experimental reflection coefficient do not exceed 1%, that essentially differs from calculations for ideal structures with zero interface roughness. Nevertheless, the reached reflection coefficients of the mirrors already allow applying them, at least, in EUV range. However it is necessary to consider, that optical systems with the high spatial resolution usually include at least two mirrors, and frequently even more, and their efficiency sharply decreases even at insignificant reduction reflection of a single mirror. In the short-wave range reflection coefficients of the mirrors are in a fraction of the theoretical maximum. Therefore the key problem of multilayer X-ray optics is connected with increase of MLS reflectivity.

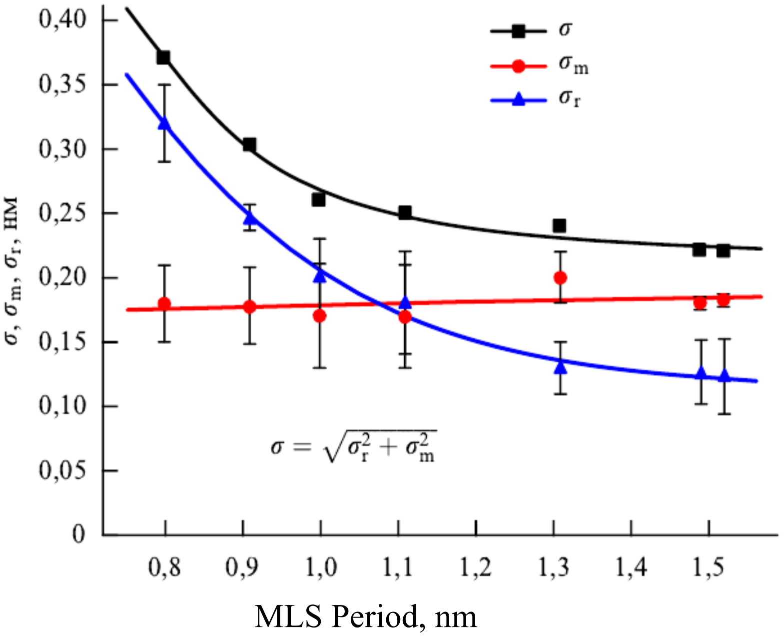


Calculated (lines with symbols) and experimentally received (asterisks) reflection coefficients of the most perspective multilayer mirrors at normal incidence.

The strong impact on reflective characteristics of the short period MLSs gives the interface borders roughness, σ. The amplitude reflection coefficient for "nonideal" border in Bragg resonance can be written down in the form of $r_F \cong r_{Fid} \cdot \exp(-2\pi^2\sigma^2 / d^2)$, where r_{Fid} - factor of reflection from ideal border, d - the MLS period. Presence of the transition region leads to increase in extinction length of X-ray radiation in MLS and, as consequence, to increase in number of reflecting layers, narrowing a spectral band of reflection and, accordingly, to additional decrease in integral reflection coefficient. The transition layer in MLS is formed as truly geometrical roughness, and due to diffusion and chemical interaction of layer materials. In turn geometrical roughness is defined by initial roughness of substrate and growing roughness which depend on materials of layers, and on technology of MLS deposition.

Thus, increase of MLS reflectivity and progress toward the shorter wavelengths is closely connected with a problem of quality of the "interfaces". For solution of this problem it is necessary to develop technological processes of MLS deposition with as much as possible sharp interfaces. In parallel it is required to develop methods of diagnostics, allowing studying the interfaces and separation of contributions of layer material mixing and truly geometrical roughness, to study correlations of interface roughness and substrate roughness in all range of the spatial frequencies defining reflection coefficients and imaging properties of the mirrors.

In this connection it is possible to note our work where on the basis of the analysis of angular dependences of specular reflection and diffuse scattering of hard X-ray radiation we divided contributions of diffusion mixing and interface roughness in short period W/B_4C MLS. In spite of the fact that the applied technique "works" only for MLS with fully correlated interfaces, it has allowed to receive a number of interesting physical results.



Dependences of transition area length σ, interface roughness σ_r and depths of layer mixing σ_m from period value for series W/B_4C mirrors.

In figure results of measurements of full length of the transition area, σ, the geometrical roughness, σ_r, and the length of mixing layer, σ_m, depending on MLS period value are shown. From the received dependences the following is visible. First, predictably, the depth of the mixing defined by interdifussion and "ballistic" processes (in a condensate there are atoms and ions with energy more than 10 eV) practically does not depend on value of MLS period. Secondly, the roughness σ_r poorly varies at greater periods and starts to increase sharply since the periods of $d \approx 1.1 \div 1.2$ nm. The explanation of such dynamics of the roughness the most probable is connected with infringement of the layers continuity when their average thickness corresponds to 0.4-0.6 nm. Thirdly, for

MLS with the periods d>1.1 nm the greater contribution to the interface width gives the layer materials mixing, basically defined by energy and a chemical compound of a condensate, instead of a geometrical roughness as it was supposed earlier. As a whole the carried out researches have shown, that the short period W/B_4C MLS have perfect borders. Similar properties have *W/Si* MLS with the periods d>2 nm. Carried out researches have allowed to promote noticeably in the area of short periods MLS.

Another important feature of MLS consisting of ultra-thin films is that due to the difference in physical and chemical properties of the materials, the parameters of interfaces in the unit cell of the heterostructures can vary widely. In many MLS used as X-ray optics elements the interfaces asymmetry is insignificant, and methods of diagnostics developed for MLS with symmetric borders do not lead to significant mistakes at definition of thickness and density films, and effective interface roughness in such MLSs. However in some cases asymmetry of interfaces cannot be neglected. In particular, as examples are the Mo/Si MLSs, on the basis of which is already under construction multilayer optics for projection lithography at a wavelength of 13.5 nm, or La/B_4C mirrors for the spectral region of 6.7 nm, promising for the next generation lithography. As a joke, it is for these MLS so bright asymmetry of the electron density profile at the interfaces between the layers is observed, that the standard diagnostic methods based on X-ray reflectometry do not allow adequately describe the inner structure of the MLSs and predict reflection coefficients of the mirrors in wide range of X-ray radiation.

The adequate technique of X-ray diagnostics of MLS with asymmetrical interfaces has been developed by ***M.M. Barysheva***. It allows confirming by non-invasive diagnosis methods the strong asymmetry of interfaces in Mo/Si MLSs and explaining low reflectivity of La/B_4C MLSs in SXR range observed by the researchers engaged in developing of multilayer optics on the basis of this pair of materials. It was found out, that the main reason of that are abnormal high chemical and diffusion interaction lanthanum and boron. Development of MLS diagnostics with asymmetrical interfaces has appeared practically useful and for development of technology of MLS deposition, and for search of the super-thin barrier layers preventing interaction of lanthanum and boron.

With development of works on X-ray lithography in last 2-3 years the new direction was appeared - creation of effective multilayer optics for spectral area $\lambda \approx 6.7$ nm where by estimations it is possible to receive the space resolution down to 8 nm. In development of technology of MLS deposition for the spectral area of 6.7 nm the basic participation are accepted by young employees ***V.N. Polkovnikov*** and ***S.D. Starikov***. It is a complex and important problem and it would be desirable to hope, that at its decision the employees will professionally grow, and we would be proud by received results.

MLSs La/B in this area have a calculated peak reflection coefficient up to 80%, however, with a spectral width of the reflection curve of 0.5-0.6 nm, integral reflection coefficient is not very big. Increasing the integral reflection up to 2 times is possible using pair of materials U/B, but it is for today absolutely a fantasy. Because of extremely low sputtering rate of the boron target by magnetron sputtering in the experiments the pure boron is replaced with it carbides, B_4C and B_9C, though thus even the calculation reflection coefficient hardly reaches 70%. However really received reflection coefficients for La/B_4C mirrors at normal incidence (thickness of the period of 3.4-3.5 nm at number of the periods 130-150) are far from the theoretical limit and do not exceed 45-47%. Development of the lithographer based on the mirrors with such reflection coefficients when a number of the mirrors can be more than ten, simply it is not profitable. For today it is clear, that the main reason of insufficient reflectivity of the mirrors is connected with interface blurring owing to processes of interdiffusion and formation of their chemical compounds, and also owing to difference of density of lanthanum layers from tabular values (80-90 % from density of a bulk material).

To reduce the layer interdiffusion so-called antidiffusion barrier layers are applied desirable transparent on working wavelength of radiation. Search of materials for the antidiffusion layers do not give cardinal successes for a long time, the increase in peak reflection was at a level of units of percents, though also it somehow goes to our “coin box”. Only recently experiments with carbon barrier layers have allowed reaching practically 60% of reflection coefficients for the normal incidence mirrors.

Not being limited by optimization of the antidiffusion layers, we in parallel consider some additional opportunities to increase reflectivity of the MLSs, in particular La/B_4C. For example, to reduce the effects of layer material mixing, it is proposed to realize an opportunity of “sealing” of a surface of each deposited layer with using “polishing” by low energy ions to remove the top part of the layers with lower density. For sputtering of targets of pure boron, as well as other targets of light materials, it is possible to apply the ion beam sputtering technology. In the new technological stand which was described above, there exist an opportunity in a single work cycle simultaneously to sputter targets by magnetron and ion beam methods.

It is possible to note, that though still there is a number of problems both in diagnostics and in technology of MLS deposition of normal incidence multilayer mirrors, already now with their help the majority of listed above problems, at least, in the field of wavelengths $\lambda > 3$ nm are solving. Short period mirrors ($d \sim 1$-2 nm) represent the big interest for applications as polarizers and phase shifters of X-ray radiation, image elements for high resolution microscopes in a spectral range «water window» ($\lambda = 2.3$-4.5 nm), focusing and collimating mirrors for hard X-ray range. Peculiarities of the short period MLSs are connected with greater number of in-phase reflecting layers (up to 10^3 and even more) which minimal thickness can reach 0.3-0.4 nm, and that their reflective ability is extremely sensitive to the interfaces quality. In our works the technology of deposition has been optimized and methods of diagnostics of short period MLS are developed, mechanisms of interface formation are studied. The basic result of these researches became development of technology of MLS deposition with the periods of 1-2 nm with reflection coefficients at normal incidence in a range of soft X-ray radiation at a level of 10-20 %. The most part of the received reflection coefficients or were, or till now remain on record level.

Practically in a separate scientifically-technological direction in the field of multilayer X-ray optics pass the researches connected with development of free standing film structures, or with transfer them onto new substrates which functionally more approach for carrying out of concrete experiment or application. This direction now “grows” and as we hope, can soon take a worthy place among other directions of X-ray optics. It is possible to consider that such researches begun with already old manufacturing of the epitaxial super-thin semiconductor films for studying effects of dimensional quantization of exitons. Work was carried out together with ***V.S. Bagaev*** group (FI AN) under ***L.V. Keldysh's*** initiative. At that time the films of semiconductors were deposition on substrates of various alkali-halide crystals and in order to prevent their destruction because of the big difference in temperature expansion coefficients at cryogenic experiments they were transferred onto MgF_2 substrates. Results have been obtained, published, and the technology have postponed till the best times, when still it is required.

The technology required in multilayer X-ray optics. In our first works on film polarizers for the X-ray range the multilayer mirrors deposited onto thin (≈100 nm) Si_3N_4 membranes. The root-mean-square roughness of the surface of the membranes usually is about $\sigma \approx 0.6$ nm that is defined by high-temperature technology of their manufacturing. It is inadmissible great roughness for MLS with the period of 2-3 nm, thus membranes bring additional absorption of radiation. For these reasons, being based on old experience, the technique of manufacturing of free standing mirrors (without any supporting membrane) was developed that allows essential reducing the MLS interface roughness down to $\sigma \approx 0.3$-0.4 nm.

Samples of film polarizers with diameter of working zone up to 10 mm have been made and tested on synchrotrons. Experimentally at synchrotrons SPRING-8 and BESSY-2 was shown, that on the basis of *Cr/Sc* MLS on wavelengths of 3.1-3.3 nm and 4.3-4.47 nm quarter-wave plates were made. In these works except for technologists ***S.S. Andreev*** and ***K.A. Prokhorov***, which deposited the MLS for polarizers, and their quality should be essentially above, than that for the typical reflecting mirrors, a greater role in manufacturing the free standing films have played ***V.I. Luchin*** and ***A.Ya. Lopatin***. Especially it would be desirable to note ***A.Ya. Lopatin's*** greater role in development of laboratory manufacturing techniques of all free standing film structures, including described below.

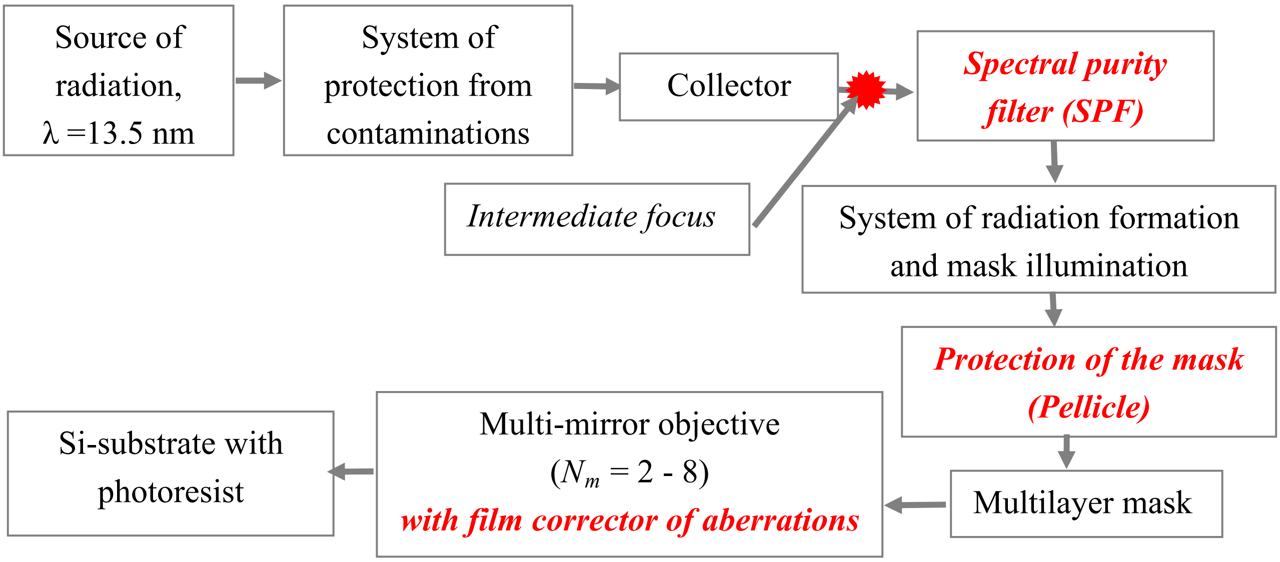
Later the technology of transfer the film structures onto new substrates was used for deposition of multilayer structures on curved mica crystal. The matter is that thin mica can be curved, with radius of curvature in units of centimeters. Therefore already for a long time curved crystals of mica are applied as focusing dispersive elements for diagnostics of high-temperature plasma with good spectral resolution. However, the period of the unit cell of mica, as a layered crystal, is about 1 nm. Therefore the spectral range of work of such dispersive element is physically limited by wavelength no more than 2 nm.

In principle it is possible, so we thought at first, to deposit a multilayer mirror on mica, and then, bend it at the specified radius, to get a new focusing dispersive element, which working spectral range is now determined by the properties of the artificial multilayer mirror and can be significantly increased as compared to any natural crystals. But it turned out that due to internal stress the multilayer coating detached from the mica surface, taking with it the last layer of the crystal, and also the same occurs at the bending of the crystal coated with a multilayer mirror.

The problem was resolved by transfer of a multilayer film from a substrate onto which it was deposited, on a surface of mica. Adhesion such transferred MLS with mica surface was such that it was possible to bend mica down to small radii of curvature. It has allowed solving a problem of manufacturing focusing, high aperture dispersive elements for the X-ray range. In particular multilayer dispersive elements based on W/B_4C and *Cr/Sc*, transferred on cylindrically bend mica crystal with radius of curvature of 20 mm, have been applied in Hamosh spectrometer for X-ray diagnostics of laser plasma. Researches in the field of development of technology of high aperture bend dispersive elements and their applications for plasma diagnostics have been lead under the initiative and together with FI AN employee ***A.P. Shevelko***.

Thus, the laboratory manufacturing techniques of free standing MLSs which are applied as polarizers, phase-shifting and splitting plates, and dispersive elements have been developed.

Free standing multilayer films of not resonant type (in them multilayer structure is used not for Bragg's reflection or transmission but only for mechanical strength) have found application as absorbing spectral filters for X-ray diagnostics of laboratory and space plasma. It has appeared that such free standing films are necessary in stands of projective lithography with working wavelength of 13.5 nm. In particular, these are absorbing spectral filters, films for protection of masks from contamination, correctors of objective aberrations, which are placed in the scheme of the scanner as shown in figure.

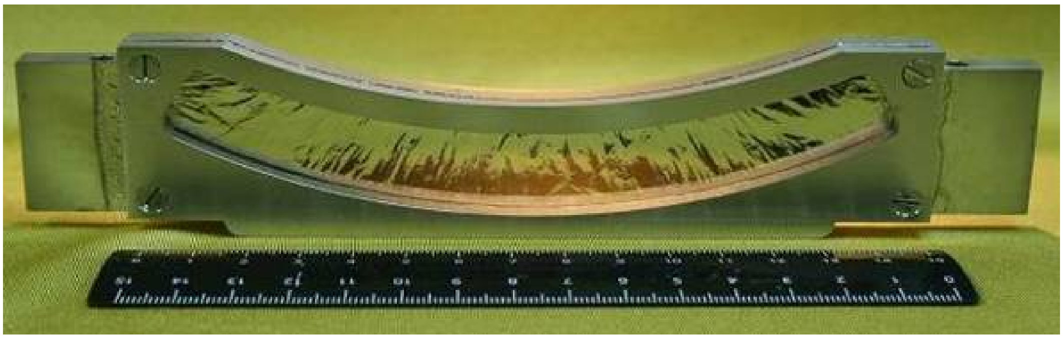


The typical scheme of the scanner with free standing films structures shown by red color.

The first free standing multilayer structure placed in the scheme after the intermediate focus is the spectral purity filter (SPF). Their necessity is the fact that for any type of radiation sources conversion of excitation energy into the energy of “good” light with a wavelength of λ=13.5 nm in the spectral band width determined by the multimirror optical system, $\Delta\lambda/\lambda \approx \pm$ 1%, is, as a rule, less than 1%, and in the long term can reach a few percents. The rest of the excitation energy is mainly highlighted in the form of longer-wave background radiation, which brings a negative impact on the elements of the optical system, and the process of exposing the photoresist, and therefore must be filtered from the useful radiation.

Now in scanners the laser-plasma sources of radiation (λ=13.5 nm) on the basis of pulsed CO_2 lasers with high, up to tens of kilowatt, average power of radiation with wavelength of 10.6 μm are used. This radiation in turn effectively scatters on plasma and also turns to additional background radiation. Average power of this additional background radiation can be not only comparable with background radiation of plasma, but also essential exceeds it. All background radiation must be filtered from the useful radiation. For these reasons the *SPFs* are applied in scanners which should satisfy a number of specific requirements. In particular, the *SPF* transmission of radiation with λ=13.5 nm should be at a level 70-80 % and no more 1% of background radiation. These conditions it is possible to satisfy at *SPF* thickness about 40-50 nm.

At *SPF* diameter of 160-200 mm the total density of power of the absorbed radiation in the filter reaches 4-8 W/cm^2. At such thickness of a film the filter is cooled only due to own thermal radiation, and the temperature of a film can reach a value up to 1000°C. The additional requirement to the *SPF* is providing protection of elements of the scheme against bombardment by corpuscular particles of the various nature of formation.



The first SPF on the basis of Zr/Si (with the sizes 140×20 мм^2 at thickness of 53 nm) developed especially for scanner ADM of ASML Company. The filter kept long-term working capacity at the absorbed power of radiation up to ≈1 W/cm^2.

Accompanying requirements to the spectral filters are connected with features of technological process of photoresist exposure in the scanner and operating conditions of the scanner. So to the important features relates the exposing by burst of radiation at a repetition packs ≈3 Hz (relative pulse duration is 1.5) and the frequency of the pulses of a few kilohertz, and eventually up to ~ 50 kHz. And it is desirable that the process continued steadily without changing the filter for at least a few thousand hours. The scanner has a fundamentally technological hydrogen atmosphere, and of course, the presence of residual gases - water, oxygen, and carbon compounds. All this affects the long-term operation of the filter under pulsating heat.

In the further requirements to *SPF* have quickly and cardinally changed. Now the *SPF* should bear, even in the long term, the absorbed power of radiation up to 10 W/cm^2 and to serve as protection of elements of the scheme against bombardment by corpuscular particles with the micron and sub-micron sizes. Diameter of the *SPF* working zone has essentially increase also, up to *D*=160 mm. On *SPF* surfaces at the total thickness of the film ≈55 nm have appeared coverings of $MoSi_2$ protecting the film from oxidation at heat.

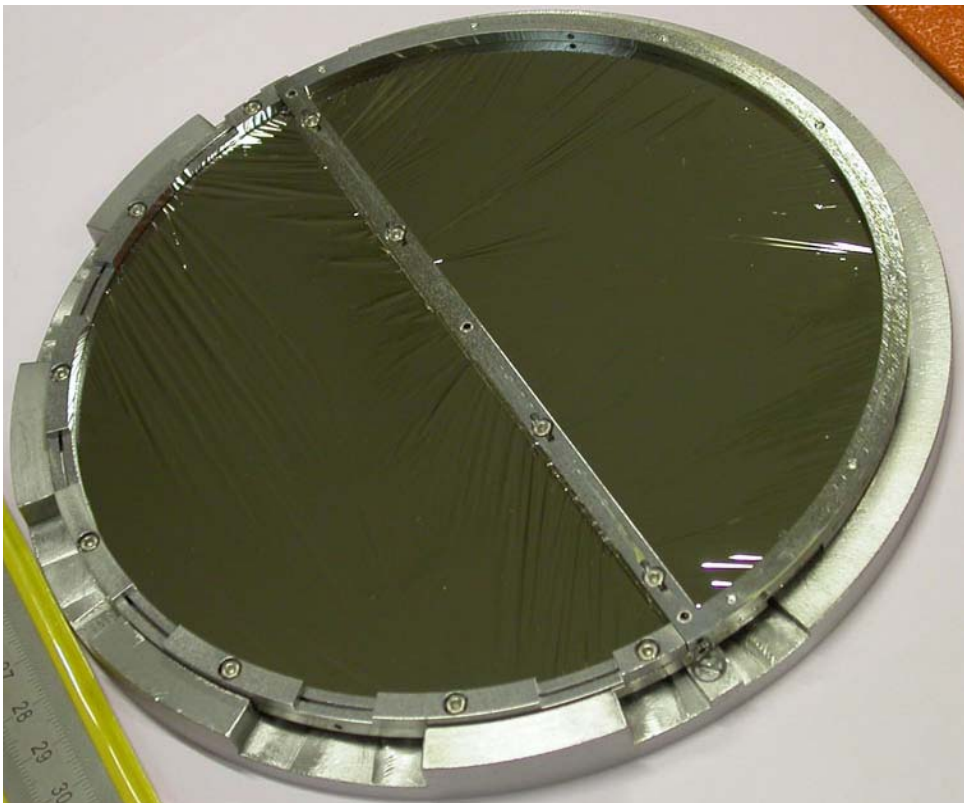
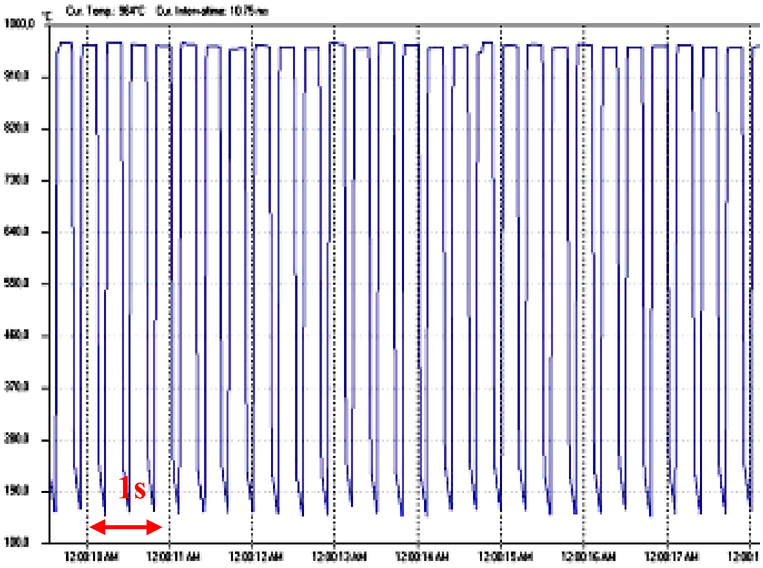


Photo of a free standing SPF for applications in conditions when absorbed power of the radiation is up to 5 W/cm^2. Diameter of working zone D=160 mm, total thickness of the film is of 55 nm.



Typical time dependence of SPF temperature during exposure at laboratory researches.

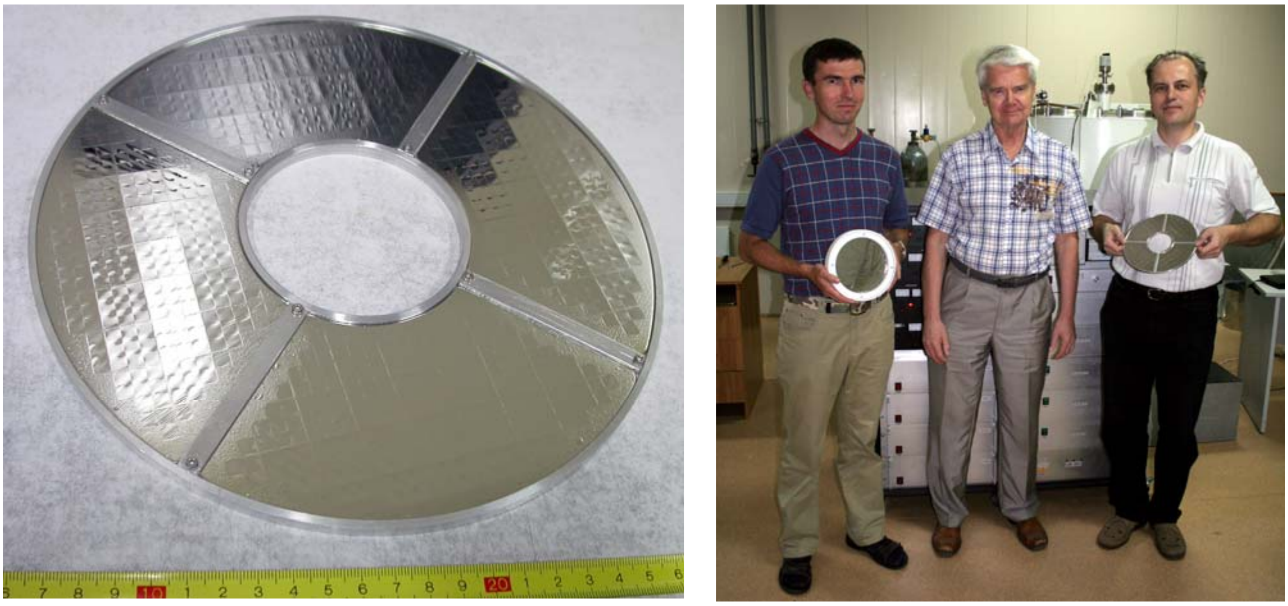
T_{max}=964°C; T_{min}≈175°C; repetition frequency of exposure F=3.3 Hz.

The SPF composition:

$MoSi_2$(3.5 nm), [Zr(2.8nm)/ $ZrSi_2$(1.2nm)]×15, Zr(2.8nm), $MoSi_2$(3.5nm)

To the pulse mode of the irradiation of the spectral purity filter corresponds also time dependence of its temperature. Typical time dependence of temperature during an irradiation at laboratory researches is shown in figure. The maximal temperature of the filter during the radiation is equal T_{max} = 964°C, and in a pause, when radiation is absent, the filter is cooled due to own thermal radiation and the temperature decreases down to T_{min}≈175°C.

More recently, requirements to the spectral filters for space researches have grown so that almost equal to the requirements for such elements for the EUV projection lithography. Features of requirements to the space filters are caused by brightness of the Sun practically in all spectral ranges and, as consequence, necessity of reliable suppression of background radiation, and the raised mechanical strength of the films. The filters should sustain complex conditions of launching of a rockets connected with vibration, acoustics and acceleration. Thus the working aperture of the individual elements of the filter reaches 170 mm. Solution of technological problems of manufacturing filters for space applications mostly fell on the shoulders of ***A.Ya. Lopatin*** and ***N.N. Tsybin***. As the result all these problems have been successfully solved. The photo shows the filter (the whole filter is composed of four equal parts) on the basis of *Al/Si* structure, intended for the telescope operation at wavelengths of 17.1 and 30.4 nm.



Photos of the multilayer Al/Si filter for a spectral range of 17-35 nm for a solar telescope and its creators - N.N. Tsybin, V.I. Luchin and A.Ya. Lopatin.

The second element based on the free standing film in the lithograph scheme serves for protection of a multilayer mask against contamination (*Pellicle*) by particles in the size more than 30 nm. These particles always, though in very small amounts, can appear in a vacuum chamber. The protective film is close, on a distance about 1 mm, from a mask and simply blocks a mask from flying particles. In such scheme radiation should pass two times through the pellicle on the way to objective, and a total transparency after two passes must be not less than 70%. All power of radiation absorbed in a film will go on its heating which also is desirable for minimizing. Therefore the transparency of such film should be not less than 85% that can be realized at thickness of a pellicle of 20-25 nm. For placing into the scanner diameter of the protective film in form o circle should be about 170 mm. Now for preliminary experiments a laboratory technology is developed and free standing multilayer films with internal (working) diameter of 80 mm are fabricated. At film thickness of 25 nm the transparency on the wavelength of 13.5 nm is equal 84%.

The film in the scanner is irradiated on “stitching” through a diaphragm with the “banana” form and with the sizes ≈ 140×10 $мм^2$ before full exposure of the chip. Thus, at scanner operation essentially non-uniform heating of the free standing film is supposed and this fact should be taking into account to prevent sagging of the film and its contact with a diaphragm. The density of the absorbed power of radiation in a film can make up to 5 W/cm^2. It corresponds to temperature of a film up to 800-900°C. In other conditions of operation of the “*Pellicle*” do not differ from conditions for the *SPF*. The important parameter of the "*Pellicles*" is a time of life which at the best (if the film will not fail that considered as an accident) is defined by its possible contaminations during work in the scanner chamber. Therefore, apparently, well to think up ways of its clearing, to restore and prolong service life of the film. But this is something for the future.

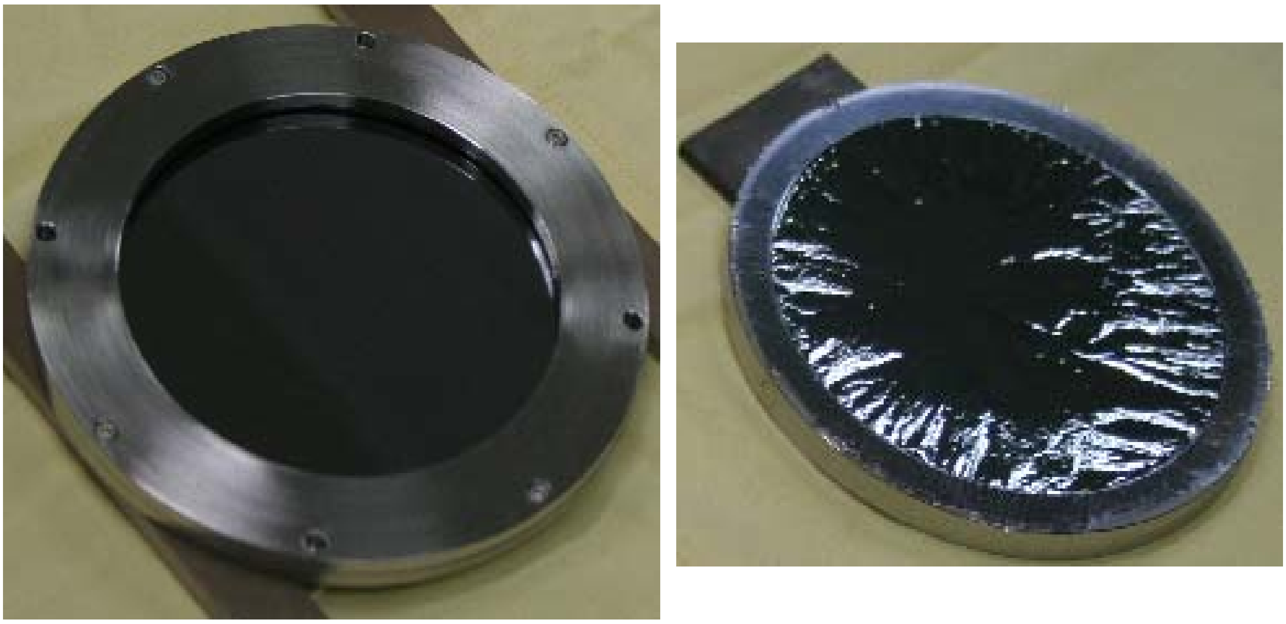
The preliminary composition of the film with a transparency ≈85% on the wavelength of 13.5 nm is determined and the laboratory manufacturing techniques of the free standing protective films with a thickness of 20-25 nm are developed. While this first samples had a diameter of 80 mm, as need to determine the requirements for the quality of the films when they are used in the real conditions in an operating scanner. With this purpose a manufacturing technologies of free standing films (it is technically easier and cheaper) which always have wrinkles (wrinkles are determined by the specific characteristics of the technological methods), and stretched films, which have almost no wrinkles, have been developed. At the next stage testing the protective films on survivability under actual operating conditions to be carried out, and how the films (and which – simply free or stretched) affect on the spatial resolution of the scanner.

Group of technology of free standing X-ray optics elements.

Sitting
B.A. Volodin and V.I. Luchin.

Standing
A.Ya. Lopatin and N.N. Tsybin.

The third type of the free standing film is the film corrector of phase and amplitude aberrations of an objective of the scanner. In spite on fundamental differences in functional properties of the correctors from the mentioned above free standing films, in their production there is a lot in common. In the photo the free standing films with working diameter of 80 mm, including a sample of a stretched film, for correctors of aberrations are shown. With such samples processes of objective aberration correction are studied now.



Photos of some free sanding films with working diameter of 80 mm for the "combined" phase and amplitude correctors. On the left the sample of stretched film is shown.

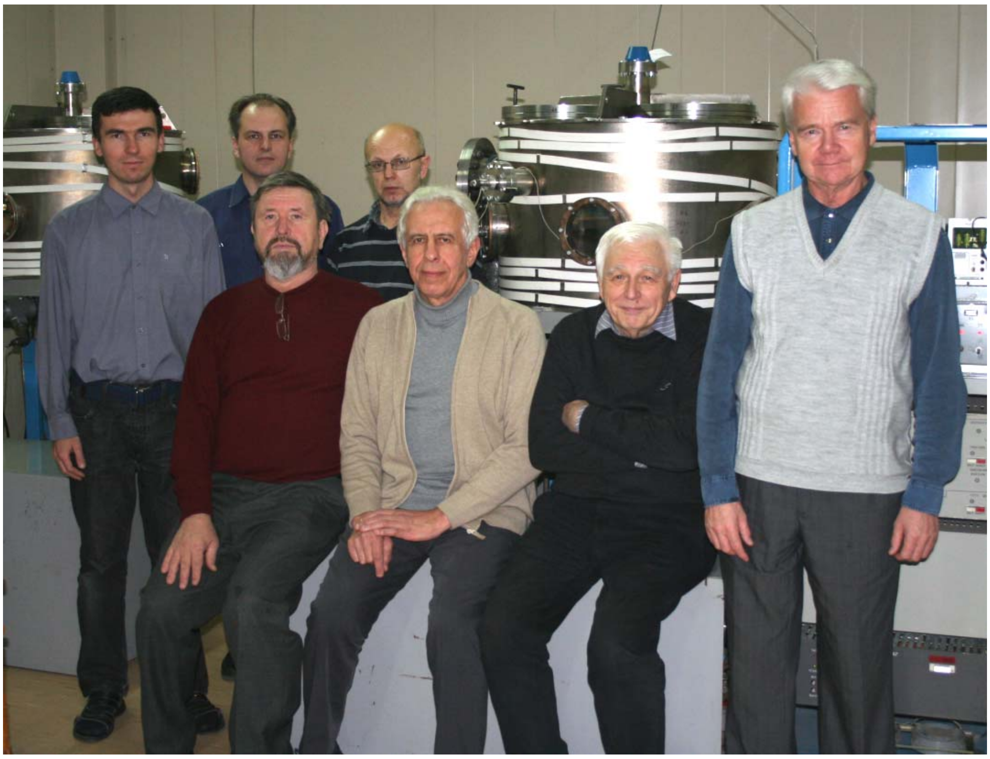
Numerous tests of the free standing film structures developed in IPM for stands of projective lithography are appreciably spent by ***L.A. Shmaenk*** who though is now the Russian-Dutch scientist and formally never there was an employee of IPM RAS, for last thirty years of versatile teamwork in the field of applications of elements of multilayer X-ray optics actually for a long time became the present employee of our department. In researches of samples before and after tests by the method of secondary ion mass-spectroscopy active participation accepts ***M.N. Drozdov***.

In summary it is possible to note, that rather "fresh" direction - free standing film structures - undergoes rapid development. They are of interest and as free standing multilayer mirrors, and as not resonant film structures which application has been described above. But recently is seen one more application of the free standing films from materials containing hydrogen (for example, films of polymers) or carbon, connected with development of laser-plasma sources of protons. It is possible to hope that in this direction free standing structures will be useful too.

It is possible to carry to the most significant physical results of this section:

- Correlation properties of the interface roughness in multilayer structures are studied, that, in particular, for the first time allowed to divide contributions of diffusion mixing of the layer materials and geometrical interface roughness in total depth of a transition layer.
- Pairs of materials and technological conditions at which films in MLS keep the continuity down to thickness of 0.3-0.4 nm are found out. These materials were a basis for creation of high reflectivity short period multilayer mirrors.
- Short period multilayer mirrors are applied in a number of experiments with laser-plasma and synchrotron sources of radiation as dispersive elements with the spectral resolution up to $\lambda/\delta\lambda \approx 500$ in spectral range of 1-3 nm, as polarizers and phase shifters of radiation in spectral range of 1-4.5 nm.
- Various short period MLS which have found applications in physical experiments and in scientific instrument making in various scientific laboratories and the industrial companies of the world were synthesized and studied.

- The laboratory manufacturing techniques of free standing film structures are developed. The free standing films have found application as multilayer mirrors, polarizers, phase-shifting and splitting plates, dispersive elements, as optical elements of stands of projective nanolithography - absorption spectral filters, films for protection of masks against contaminations and correctors of objective aberrations, for X-ray diagnostics of laboratory and space plasma.



They invent, develop manufacturing technology, manufacture and test free standing elements of X-ray optics.

From left to the right: N.N. Tsybin, A.Ya. Lopatin, E.B. Kluenkov, B.A. Volodin, L.A. Shmaenok, N.N. Salashchenko, V.I. Luchin.

4. Precision multilayer “X-ray” optics

The main factors constraining for a long time the development of precision optics for SXR and EUV spectral ranges was hard to satisfy the requirements to the surface quality of mirrors and lens aberrations in general. In order to achieve diffraction-limited images, when the spatial resolution is limited only by the wavelength of the radiation, in this range interfacial roughness in the MLS and, accordingly, the surface roughness of substrates for the mirrors shall be at the atomic level (less than 0.3 nm). The accuracy of the shape of the surfaces, often aspheric, on the parameter of the standard deviation (RMS), depending on the spectral range is between ≈ 0.2-1 nm.

For the decision of this problem it was necessary to solve two problems. The first is a development of adequate methods of studying of a roughness and the surface shape, the second - development of methods of correction of the substrate shape up to set.

Increased by 1-2 orders surface quality requirements demanded revision of real possibilities of the existing methods used in the optical industry. In particular, it has appeared, that traditional interferometric methods of the optical surface shape quality control in which as a reference the wave front formed by reflection from a reference surface is used, possess high ($\lambda/1000$ and above) sensitivity to variations of the wave fronts, however an absolute accuracy of measurements does not exceed $\lambda/20$-$\lambda/50$, where λ - working wavelength of the interferometer. It is related to the quality of the reference surface shape and design features of the interferometer in which the light beams pass through a series of optical elements, uncontrolled gathering additional phase shifts.

Fully these problems manifested themselves in the measurement of roughness of ultra-smooth surfaces. As a whole the practice has shown, that for studying properties of super-smooth and ultra-precise surfaces it is necessary to refuse traditional calibration of devices by means of various standards, and to develop the methods based on the first principles, i.e. working on the basis of fundamental physical laws when the basic characteristics of the device (method) can be measured in physically transparent experiment with an opportunity to lead an authentic estimation of errors of the measurements. Following this ideology in IPM the complex of techniques including the analysis of angular dependences of the diffusion and specular reflection of soft and hard

X-rays, atomic-force and white-light interference microscopy allowing reliably measure surface roughness with any dimensions and shape has been created.

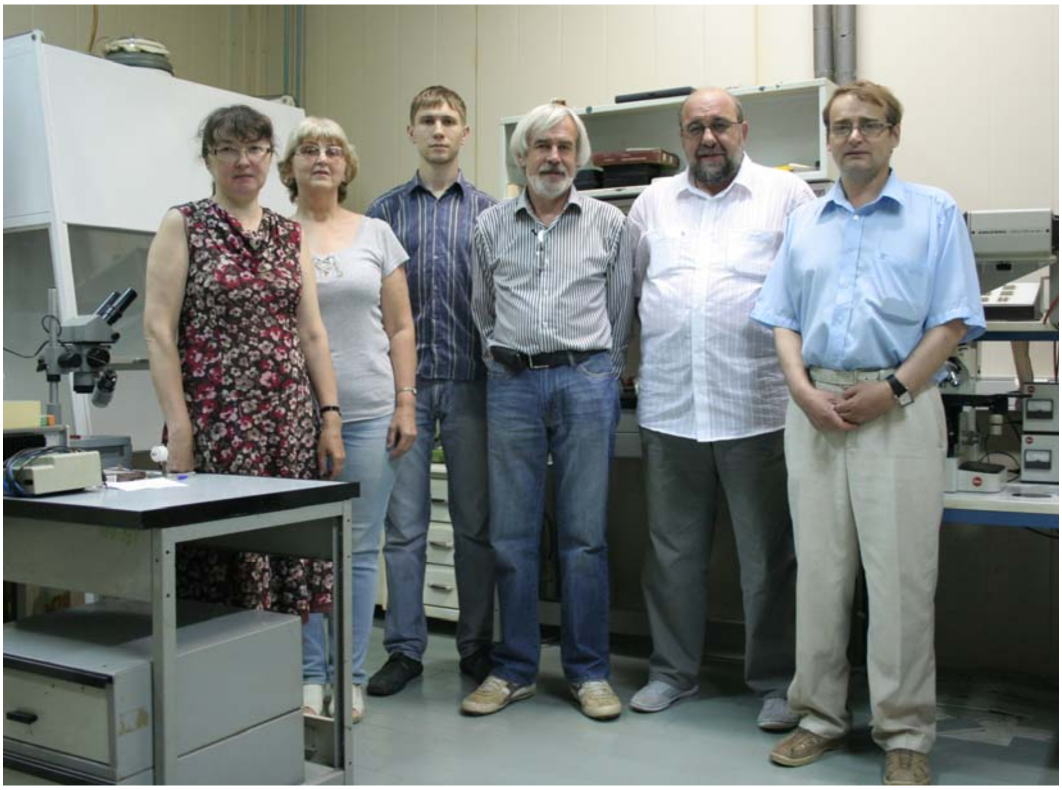
In development of the complex of measurement techniques for super-smooth surfaces roughness measurements active participation accepted

M.M. Barysheva and M.V. Zorina, B.A. Gribkov and Yu.A. Vainer.

For certification the surface shape and aberrations of optical systems such method became the interferometry with diffraction reference wave, proposed in 1933 by ***V.P. Linnik***. The basis of the method consists in formation of a reference spherical wave as a result of light diffraction on the small pin-hole. The solution of a classical task of light diffraction on an pin-hole in a screen with infinite conductivity and zero thickness shows, that within the limits of diffraction maximum, which angular width makes $\pm \lambda/d$, d – pin-hole diameter, a phase surface of the diffracted wave represents an “ideal” sphere.

Essential jump in measurements accuracy of interferometers with diffraction reference wave has been made owing to development in our institute in 2008 of the source of the reference spherical wave based on a single-mode optical fiber with the sub-wave exit aperture. Its appearance was made possible by the addition of a number of developments in the field of wave-front aberration metrology and in the manufacturing of fiber probes for near-field microscopy.

Based on the source of the spherical wave a **vacuum interferometer,** already successfully working in IPM RAS more than four years, has been made. Quality of measurements of the optical surfaces shape is those, that on measurements already gravitational field of the Earth effects. Therefore the optical elements shape in our interferometer is studied in that orientation, in relation to the gravity of the Earth, as the element would be installed in working device. To satisfy this condition the interferometer is made on a “push-pushing” manner when the upper and lower parts of the interferometer are mirror symmetric.



Developers of manufacturing techniques of spherical wave sources based on a single-mode optical fiber with the sub-wave exit aperture:

E.D. Chkhalo, L.V. Klimenko, M.N. Toropov, V.V. Rogov, A.Yu. Klimov, N.I. Chkhalo.

Occurrence of metrology became stimulus for development of manufacturing methods of optical elements with sub-nanometer accuracy of the surface shape. Manufacturing of the optics occurs in two stages. At the first stage with use of standard methods of polishing and metrology the super-smooth surface is made. In the second stage using the methods of local ion-beam etching and/or vacuum deposition of thin films the surface shape is adjusted (corrected) to sub-nanometer deviations from the required.

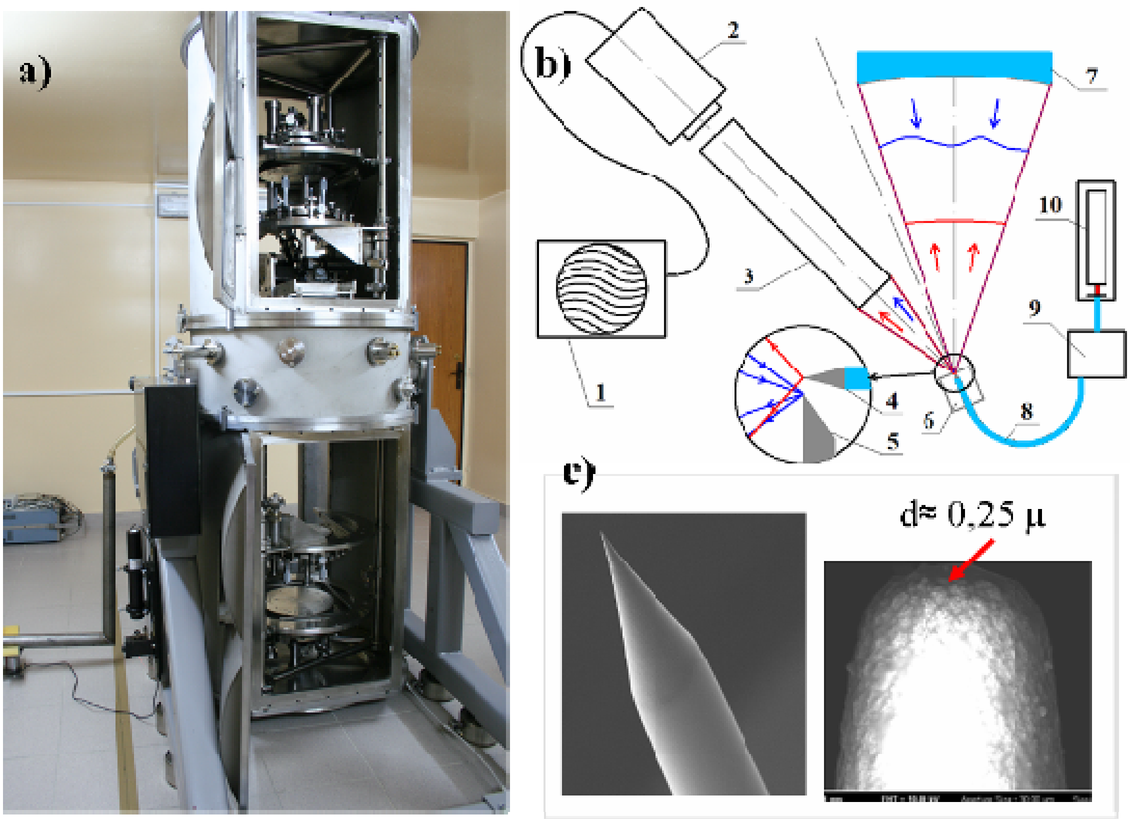


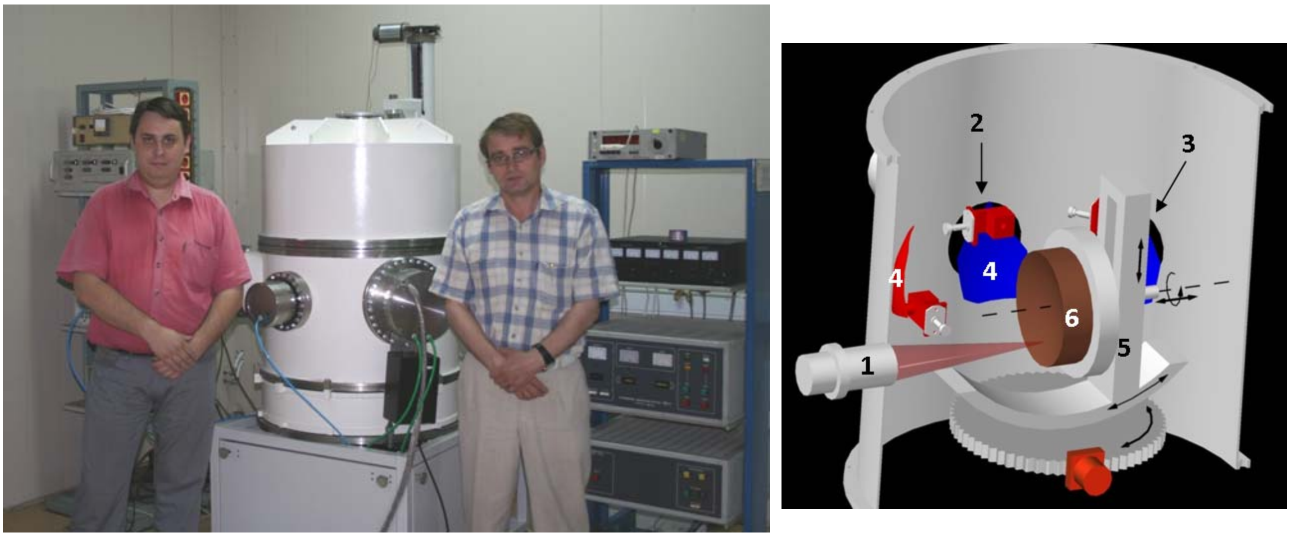
Photo (a) and optical scheme (b) of the vacuum interferometer with diffraction reference wave and electron-microscopic images of the reference spherical wave source based on single-mode tipped optical fiber (c). 1 - PC, 2 - CCD camera, 3 - lens, 4 - the source of the spherical wave, 5 - flat mirror, 6 – 3D table, 7 - investigated concave surface, 8 - optical fiber, 9 - polarization controller, 10 - laser.

Photo of the vacuum interferometer with diffraction reference wave, from left to right:

M.N. Toropov, B.A. Zakalov, N.I. Chkhalo, D.G. Volgunov, N.N. Salashchenko.

An employee of “Composite” company (Moscow) ***I.L. Strulya*** together with employees of IPM RAS a laboratory technology of superpolishing of optical surfaces, including aspherical, from fused quartz and monocrystalline silicon have developed. For ion-beam precision correction of the super-smooth surface shape in IPM the facility equipped with one ion gun was developed, allowing working only with inert gases. As the key requirement to the correction process is keeping surface roughness on initial, atomic smooth, level the complex of researches on study the influence of parameters of technological processes on a roughness of a surface using this installation has been lead. As a result of these researches the technology of ion beam etching of materials mostly used in X-ray optics has been developed: *Cr/Sc* MLS, fused quartz, glass ceramic and zerodur.

On the basis of experience of working with the experimental stand a new installation for ion beam correction of substrates with a diameter up to 300 mm and equipped already with three ion guns, allowing to work with active gases too, and to retouch a surface by focused down to 1 mm ion beam, has been developed.



The stand for precision correction of surface shape of optical elements by the method of ion beam etching, developed in IPM RAS. Authors of the development are A.E. Pestov and N.I. Chkhalo. At the right the scheme of the stand is shown: 1 - the source with focused ion beam, 2 –RF source with (aperture 150 mm), 3 - source with thermocathode (the aperture 60x90 mm^2*), 4 - shutter measuring an ion beam current, 5 - five-axis goniometer, 6 - the processed sample.*

With use of the developed complex of the equipment and technologies in IPM RAS are made supersmooth spherical and aspherical surfaces with sub-nanometer accuracy. The typical mirror in a frame protecting a surface from deformation at mounting the mirror in a device is shown in a photo. The RMS of errors of the surface shape makes 0.6 nm.

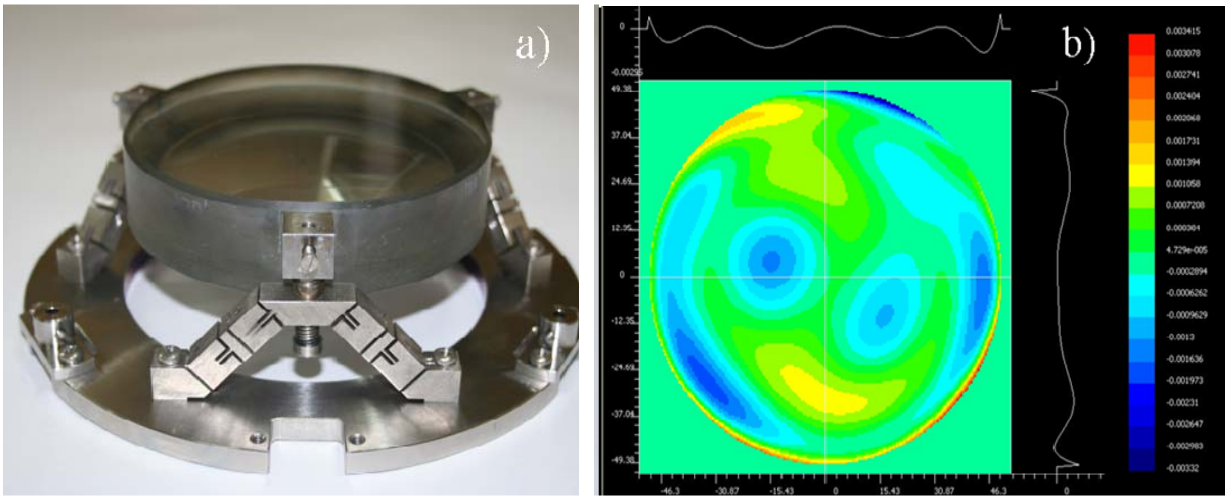
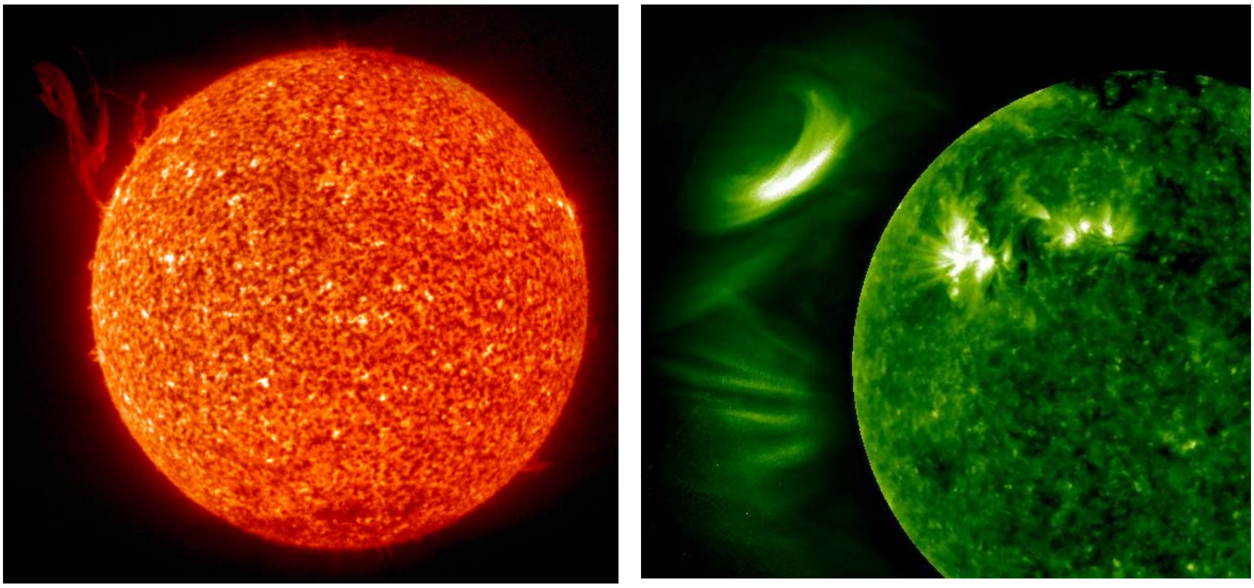


Photo of a concave spherical substrate in diameter of 130 mm and with numerical aperture of NA = 0.25 and the map of deviations of the surface shape from the nearest sphere: P-V=7.3 nm, RMS=0.6 nm.

Thus, in a relatively short period of time in IPM was established technological and metrological complex, including an interferometer with a diffraction reference wave, facilities for ion-beam etching and deposition of thin films, precision reflectometers, allowing us to create ultra-smooth and high-precision optics. Thus, although in a laboratory version, we got into a prestigious club of high-tech companies, consisting of ZEISS (Germany) and, in part, General Optics (USA) and Nikon (Japan) with the amount of technologies needed to create precision optics and ultrahigh resolution projection lenses.

Currently developed in IPM technologies are used for the manufacture of optics for the Russian program to study the Sun, using satellites. The figure shows images of the solar corona at wavelengths of 30.4 and 17.1 nm, obtained in 2009 as part of an experiment TESIS.



Eruption of the protuberance on east limb of the Sun of 23.04.2009.
Supervision of TESIS telescope in line HeII 30.4 nm. Temperature of plasma is nearby 80000 K.

The face of the modern civilization in many respects is defined by development of the micro-nano-electronics. Thus key technology of microelectronics, basically defining the degree of integration of electronic elements (transistors) into the chip, is the projective photolithography. The future of the nanolithography is connected with the working wavelengths laying in EUV and even SXR ranges. With use of working wavelength of 6.7 nm already in the foreseeable future it is possible to expect achievement of critical dimensions at a level of 10 nm and even less.

The leading world companies have begun researches in the field of EUV lithography in the middle of 90ths of the last century. Unfortunately, despite of significant reserves of a domestic science in the field of element base of EUV lithography, first of all in multilayer interference optics and sources of EUV radiation, due attention from the Russian elites this direction has not received. It is possible to note, that in the middle of 90ths in FOM Plasma Physics Institute (Netherlands) firstly in the Europe an installation for demonstration of principle of EUV-lithography with a laser-plasma source (***F. Bijkerk, L. Shmaenok, C. Bruineman***) has been constructed. Optical elements for installation have been made in IPM RAS that has ensured in photosist the pattern elements with the sizes 100 nm that was impressing for that time. Already at that time in our collective the spherical mirror with big diameter up to 250 mm with gradient distribution of the period of multilayer structure over the surface has been made. As a result of the scientific cooperation with ASMLithography, the Netherlands, on the issue of EUV lithography with ***L.A. Shmaenok*** was proposed and developed a compact double mirror device to characterize “in-band” sources in the spectral range of 13.5 nm for the ADT scanner. In the device pair of mirrors with specially designed spectral dependences of their reflection coefficients matches transmission spectral dependence of multimirror (up to 11 mirrors) optical system of the scanner. The developed device has been chosen in 2004-2006 by consortium SEMATECH for the program of comparative metrology of EUV sources in Europe, USA and Japan.

Photo of the nanolithographer-multiplier developed in IPM RAS.

The basic developers of the nanolithographer-multiplier (from the left-to the right): Sitting: B.A. Zakalov, N.N. Salashchenko, I.G. Zabrodin. Standing: V.N. Polkovnikov, L.A. Suslov, A.E. Pestov, D.G. Volgunov, N.G. Guskov, M.N. Toropov, N.I. Chkhalo, S.Yu. Zuev, I.A. Kas’kov.

With understanding of all importance and actuality for the country of this direction, and with the hope, that Russia in due course will take its true place in the world innovative process, within the frame of scanty, in comparison with foreign corporations, financial resources, at financial support by the RAS (***Zh.I. Alfyorov's*** Program), and RosAtom (here a greater role minister of the nuclear industry academician ***A.Yu. Rumyantsev*** has played at that time), and the RFBR, from the beginning of 2000ths we are dealing with this problem. As already it was mentioned above, within the limits of these researches crucial technologies of a world level in the field of precision optics have been created. In the field of free standing multilayer structures we managed to outstrip the nearest competitors. As a result in IPM the first in Russia has been developed and run in 2011 the stand of projective lithography on wavelength of 13.5 nm with the designed resolution of 30 nm. Now the first microstructures by the method are already received.

One of appreciable scientific results in the field of EUV lithography, received together with employees of Institute of chemistry in N.I. Lobachevsky University, is development of the domestic photoresists for wavelength of 13.5 nm. These works have been begun in the beginning of 2000, and the big collective of researchers participated in them from both parties. From developers of a chemical compound of the photoresists there is a leader, qualified and recognized in our collective, ***S.A. Bulgakova***. On radiation sensitivity at 13.5 nm and contrast of the images the photoresists correspond to the best world analogues. Now there are begun researches of photoresists resolution and in the near future will be studied their plasma-strength.

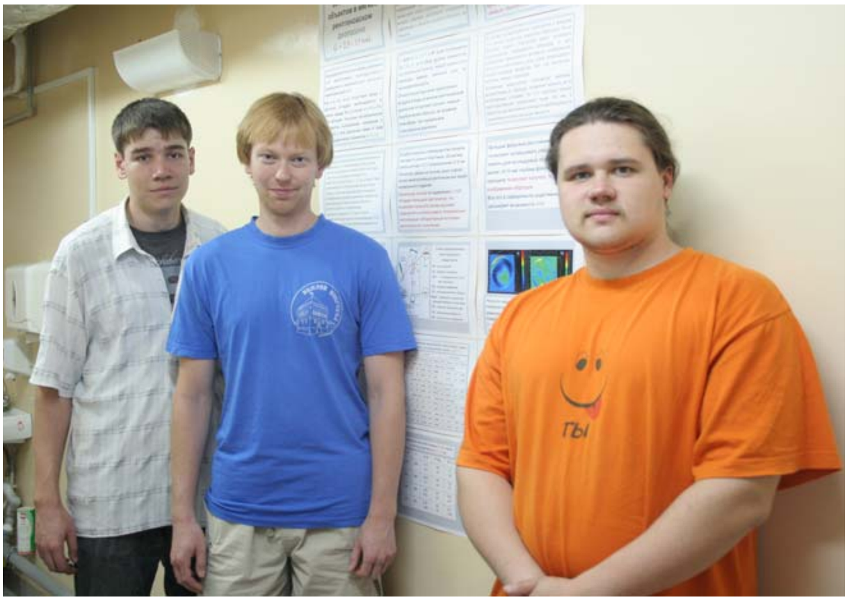
The basic developers of the photoresists for radiation with wavelength of 13.5 nm.
From the left-to the right: M.M. Johns, S.A. Bulgakova, V.I. Luchin, A.Ya. Lopatin, A.E. Pestov, M.N. Toropov.

Recently, microelectronic community began active preparations for the transition to the next generation lithography, after 13.5 nm. It should be noted that when a new wavelength of 6.7 nm chosen, also played a role proposal made in ASML in 2006 by ***N.N. Salashchenko*** and ***N.I. Chkhalo***. We continue developing technological aspects of manufacturing of effective multilayer optics for this spectral range. In 2012 have been synthesized *La/*B_4*C/C* multilayer X-ray mirrors of normal incidence with barrier layers from super-thin carbon films with record, up to 60 %, reflection coefficients. The received result can form an experimental basis of optics for the next generation nanolithography with the spatial resolution down to 8 nm.

Relatively new area, the sources of which we are in, is the **X-ray microscopy of the ultrahigh spatial resolution** based on normal incidence multilayer optics. Due to the resonant interaction of radiation with atoms in the range (photon energy equal to intra-atomic transitions and the ionization potentials of low levels), extremely high absorption contrast of images is observed. In particular, in a number of cases it leads to that necessary absorbed in the sample doze for reliable registration of images in some orders of magnitude is less, than in case of electron or hard X-ray microscopy. The last, for example, allows studying even alive objects with the resolution in some tens of nanometers.

Use of magnetic and electric dichroism effects, observable for some materials in the soft X-ray range, in aggregate with the microscopy opens to researchers of nano-(electro) magnetism enormous prospects.

It is possible to note, that researches in the field of the high resolution soft X-ray microscopy became possible only owing to progress in technology deposition of short period normal incidence multilayer X-ray mirrors, and in metrology and manufacturing of ultra-precise imaging optics. It is possible to hope, that owing to high reflectivity of multilayer mirrors, X-ray microscopes will be used in biological laboratories and in due course become same accessible, as atomic-force or electron microscopes. Greater role in this break employees of our department have played, and in the further (*it would be desirable to hope*) the leader of this direction becomes our today's post-graduate student ***Shcherbakov A.V.***



P.K. Gajkovich, D.E. Paryev, A.V.Shcherbakov - them and start working on the X-ray microscope for biological research.
Who better than a young start a new direction!

It is possible to carry the following to the basic scientific and technological results of this section:

- The technologically-measuring complex for manufacturing and certifications of optical elements with sub-nanometer RMS surface shape errors and optical systems wave front aberrations is created. Creation of the complex opens opportunities for development in Russia of optics for projective nanolithography, X-ray microscopy and astronomy of the ultrahigh resolution. With application of the complex the projective objective for nanolithograph with working wavelength 13.5 nm was developed and made.
- An interferometer with diffraction reference wave, providing certification of the high aperture optical elements shape and wave aberrations of complex systems with accuracy at a level of 0.2-0.3 nm have been developed. Physical methods of surface shape correction, including aspherical ones, with sub-nanometer accuracy were developed. These works are the basis for development in Russia high technologies in the field of creation of the lithographic equipment of new generation, X-ray microscopes and telescopes of ultrahigh resolution, etc.

5. Focusing and collimation of hard X-ray radiation

One of well enough demanded directions of multilayer X-ray optics is connected with development of elements and units for formation of hard X-ray radiation beams. This direction is based on very simple idea, which was proposed and patented by the German scientist ***H. Göbel***, who offered the possibility to connect multilayer X-ray optics and elementary geometrical figures, for example cylindrical paraboloids or ellipsoids to form X-ray beams shape. In the case of linear X-ray source such shapers let one to get or a parallel X-ray beam, or build up in the ellipsoid second focus a linear image with a magnification factor.

A feature of multilayer structures deposition onto such shapers of radiation is connected with necessity to satisfy the Bragg condition ($2d \cdot n \cdot sin\ \theta \approx \lambda$, n – an index of refraction, θ – a grazing angle, d – a period of MLS) in each point of the surface. Such mirrors have found wide application as in serial devices (diffractometers, X-ray fluorescence analyzers) of leading world manufacturers of the X-ray equipment, and in synchrotron laboratories. The use of the curved multilayer mirrors allows increasing the devices aperture ratio in tens-hundreds times.

For carrying out X-ray structural analysis, as a rule, of low-dimensional biological objects, for example, proteins, point sources of X-ray radiation are used. For formation of radiation beams from the point sources traditionally various crossed Kirkpatrik-Baez systems are used. Among them the greatest aperture ratios have the symmetric angular systems consisting of two identical crossed multilayer mirrors in the form of the elliptic cylinder with mutually perpendicular element of cylinder. The manufacturing techniques of angular systems are based on well developed manufacturing techniques of cylindrical reflectors. However, significantly larger (in magnitude) aperture ratio have the mirrors with shape of ellipsoid of rotation. Needs for such shapers of radiation of the point sources now exist, especially for studying biological objects. In laboratory variants the shapers with a figure of ellipsoid of revolution exist, perspectives of their application is shown, however there are also greater technological problems of their manufacturing.

The manufacturing techniques of multilayer elliptic and parabolic cylinders are well developed in a number of the organizations in the world, including IPM RAS, in ***A.D. Akhsakhalyan*** group. The basic achievements of the group are connected with complex, at a world level, development of full manufacturing techniques of cylindrical shapers of radiation, including methods of manufacturing of the precision dies defining the shape of the cylinders, technology of MLS deposition with the gradient distribution of the period, methods of assembly and certification of the shapers. The developed shapers of radiation find application both in X-ray diffractometers leading world manufacturers, and basically domestic diffractometers.

The aperture ratio of Kirkpatrik-Bajez systems can be increased by increasing the number of reflecting elements around of an optical axis. In IPM for the first time in the world the four-angular system has been made and studied, the aperture ratio which, accordingly, twice surpassed the aperture ratio of the symmetric angular systems of the same length.

Group of development of multilayer shapers of "hard" X-ray radiation.
A.A. Akhsakhalyan, A.D. Akhsakhalyan, A.I. Kharitonov, L.A. Mazo.

More complicated is the situation with development of the shapers with figures of rotation, the aperture ratio which, basically, can surpass in tens times the aperture ratio of the angular systems. Two techniques of mirrors manufacturing with the shape of rotation figures and a quality monitoring of the shape have been developed. In the first technique the surface of rotation is formed by a replicas method, in the second – by the method of plasma-chemical etching. By these techniques the mirrors which have shown the reflective characteristics close to the calculated ones are made and investigated. It is possible to consider it as a good technological reserve for the future, but, certainly, the basic works still ahead.

It is possible to carry to the basic scientific and technological results:

- Development of methods of calculation and optimization of mirrors characteristics, in view of the real sizes of X-ray radiation sources, technological opportunities on manufacturing of multilayer structures and requirements on characteristics of formed beams of X-ray radiation.
- Development of manufacturing techniques of focusing and collimating mirrors, including the methods of certification of reflective and geometrical characteristics of a surface and formed X-ray beams.

6. Conclusions

Within the frame of the given historical review we have attempted to describe the current state of researches in IPM RAS in the field of manufacturing and applications of a new class of optics - multilayer optics for X-ray and extreme ultra-violet spectral ranges. It is possible to note, that owing to enormous progress in the world last decades in technology of deposition of multilayer coatings for "X-ray" mirrors, in manufacturing and certification of diffraction qualities optical elements and systems for SXR and EUV ranges, there was the real opportunity appreciably already realized, to transfer practically all arsenal of traditional methods of light beams controlling (*monochromatization, focusing, collimation, imaging, beam splitting, polarization, phase-shifting and analyzing, etc*.) in short-wave (nanometers) spectral range. It opens essentially new opportunities in microscopy of the condensed matter, for researches in biology, astrophysics and in other areas, in nanotechnologies, first of all in lithography and in nanodiagnostics.

This progress became as a result of wide researches in a number of adjacent areas of solid state physics, of surface physics, thin-film growing, physical optics, etc. It has demanded creation of perfect technologies of growing of multilayer structures, new technologies and methods of manufacturing and measurement of optical elements and systems with unprecedented accuracy. And in this connection it is necessary to note important role of the IPM RAS in creation of this direction. Of the main results of 20 years of development of direction of the multilayer X-ray optics in IPM, which will subsequently affect the future of the creative life of our community, are the following.

In IPM RAS technologies of deposition of multilayer structures from super-thin films, optimized for elements of multilayer optics for X-ray and extreme ultra-violet ranges are developed. A set of specialized equipment for deposition of multilayer structures with a given distribution of period on the surface of the substrates with a diameter of 350 mm with almost any surface shape is created.

The technologically-measuring COMPLEX for manufacturing and certifications of optical elements with sub-nanometer level RMS surface shape distortions and optical systems wave front aberrations is created. Creation of the complex opens opportunities for development in Russia precision optics for ultrahigh resolution projective nanolithography, X-ray microscopy and astronomy, and for formation of super-strong electromagnetic fields optical and soft X-ray ranges.

Metrological basis of the complex is developed in IPM interferometer with diffraction reference wave, providing certification of the high aperture optical elements shape and wave aberrations of complex systems with accuracy at a level of 0.2-0.3 nm. The base model of the existing interferometer serves as the prototype for the further development of the whole scale interferometers and methods of precision interferometry. Physical methods of the surface shape correction, including aspherical ones, with sub-nanometer accuracy are developed.

On the basis of the researches in the nanotechnology field for the first time in Russia the nanolithography stand (λ=13.5 nm) with the designed resolution of 30 nm is created. The first samples of microstructures are received. Creating of the stand showed the emergence technologies to develop and manufacture in Russia advanced lithography equipment, which in coming years will be a key in the production of chips with topological norms of 22-8 nm, the optical systems to produce ultra-high spatial resolution for astronomy and microscopy in short wavelength range. This development, together with other national developments in the field of high-power gas discharge EUV radiation sources can be the basis for the national program for the production of components of nanoelectronics.

Based on original technique of free standing multilayer films a number of the X-ray optical elements with new properties are developed. Periodic structures with a small interface roughness ($\sigma \approx 0.3$ nm) are applied as phase-shifters for an interval of wavelengths of 1.5-4.5 nm. For the first time in this spectral range a quarter-wave plate was made. Multilayer mirrors with small radiuses of curvature in the shape ellipsoid of rotation as focusing reflectors for hard X-ray range ($\lambda \sim$ 0.1 nm) and cylindrical dispersive elements for the aperture ratio spectrometer for the soft X-ray range are created. Manufacturing techniques of heat-resistant free standing spectral purity filters and elements for protection of masks against of particles (pellicles) with record characteristics (the aperture up to 160 mm, film thickness of 20-50 nm and transmission at λ=13.5 nm $\geq$

70 %) which have found application in stands of projective nanolithography (λ=13.5 nm) are developed.

Manufacturing techniques and methods of certification of multilayer shapers of hard X-ray radiation, including focusing (elliptic) and collimating (parabolic) cylindrical mirrors are developed.

Now we can show imagination on possible ways of development of scientific direction ***«Multilayer X-ray optics»***.

Familiar optical range, when viewed in the spectral range from 0.1 μm to 1 mm, and includes extensive ultraviolet range, including vacuum ultraviolet, visible relatively narrow range, near and far-infrared range, and even the submillimeter range, or, as it is now called, the terahertz range. I.e. this broad spectral range includes much, even not very related, sub ranges. In this sub ranges, there are lots of physical effects, which employ specialists completely different specialties, are not always understand each other.

Almost as wide spectral range covers an area of the hard X-rays to long-wavelength edge of extreme ultraviolet radiation, λ≈0.01-100 nm. But since at present there is only development of this short-range, then we as developers of optical components and optical systems based on these elements, and the optics are effective can only be multilayered, consider the whole range as one. And we try not only to develop the optics, but also to apply it to the physical experiments and instrumentation in the whole spectral region. Of course, and in this spectral region have already taken place a separation of researchers on “narrow” spetsilists. For example, developers of the sources (discharge and laser-plasma sources, free-electron lasers) are some majors. There are spectroscopists, astronomers, etc. They, not well connected each other, too. But they can not do anything without appropriate optical elements and, therefore, without us. So while the experts in “our” band is not “run away”, we can continue to work with all and for all.

Our participation in the further development of multilayer optics, of course, connected with our developments in previous years in the fields of deposition technology and metrology different multilayer structures for the elements of X-ray optics, in manufacturing and metrology of the precision imaging optics. On this basis, is expected to continue to deal with problems in which these developments are key ones. Consider some ways of our development.

The first way can be associated with the choice of one or two scientific and technical fields, in the long term or practically useful for humanity, or for our country, and it is better if it coincides. The solution of these problems must be associated with the formulation and resolution of numerous fundamental and applied problems. The completion of the great practical importance problem is not necessarily just only using own resources, the main thing for us is to address fundamental issues in the stated problem. Examples of such problems can be following:

- Development of laboratory stand projection lithography at a wavelength of 6.7 nm or at a different wavelength, which may in the future provide a resolution of 8-10 nm with a greater or equal performance compared to the EUV lithography at a wavelength of 13.5 nm.
- Development of X-ray microscope with the spatial resolution of 5-10 nm for studying biological objects in lines of the concrete chemical elements interesting for biologiests.

Solution of these problems requires further and significant development of multilayer optics. In particular, the development of production technology and metrology of aspherical elements for imaging optics made with angstrom level surface roughness and shape precision. Further researches and developments in free standing optical element and development of methods controlling the spectral and angular characteristics of multilayer mirrors are required.

Researches in the field of optics for nanolithography and microscopy closely relate with the researches directed on development of the equipment (telescopes) for “X-ray” astronomy. Requirements to the optical elements for the space telescopes are practically the same level, as to the optics for nanolithography on a wavelength of 13.5 nm.

Regarding of our involvement in solving problems of X-ray lithography and microscopy it is possible to make the following remarks. In our opinion, development of the domestic scanner looks not very real. Undoubtedly, that for understanding of problems it is useful to develop model samples either projective schemes, or the elementary scanners. But what about development of domestic scanner for high volume manufacturing of chips? This is a very expensive project, which should perform together industrial and academic organizations, but define the work program should industry. This should engage the most advanced groups, as it is done in the West and in Japan. Even if the scanner will be designed and manufactured, it should be build into a production line of micro-nano-electronics. Currently existing in our country advanced lines of microelectronics are purchased in the West. That means that the scanner should be inserted into the purchased production line. Designed scanner must be competitive product, should be replicated and sold in conditions when the global market of the scanners rigidly divided. But we can make the world's first projection scheme at a wavelength of 6.7 nm, if we solve the problems with precision multilayer optics. A prerequisite to this we have only appeared recently. By EUV sources and discharge (IS RAS), and gyrotron (IAP RAS), and free electron X-ray lasers (BINP SB RAS), one hopes, in the country there are good developments. Then the Russian side would contribute to the world community in the form of optical components and radiation sources for the next-generation scanners.

As to the X-ray microscope for biology, it, certainly, more chamber problem. At proper interaction with biologists it is possible to develop useful tool which as a while it is represented, can have and commercial prospects. But also in this question there are many concrete problems. It is clear, that for financing development of the microscope, it is necessary to show all over again even the laboratory sample of the microscope and preliminary results of studying of biological samples. And it looks real.

The second way of our development can be connected with development of manufacturing techniques, methods of diagnostics and certification of optical element for a spectral range of 0.01-100 nm. It will demand the decision of many fundamental scientific and technological problems, the further development of technological, research and measuring base. Development in such direction assumes close interaction with leading groups and organizations in the world to understand what concrete problems in development of “our” spectral range are. Really such collaboration with the world could be if we at least in some questions, let not in everything, are better than others and it would be known in the world. I.e. publications should be a high level, and basic samples of optical elements or units have passed testing in leading groups.

Apparently, symbiosis from two ways of development of multilayer X-ray optics is most real. It is necessary to have the “greater” problem, for example, development of laboratory samples of the nanolithographer on wavelength of 6.7 nm and the X-ray microscope. Thus in parallel necessary to go on the second way - to develop element base for X-ray and EUV ranges and for own development, and for the foreign scientific organizations with attraction of additional means. In such strategy of development can be entered and problems of the X-ray astronomy.

In summary we shall try to present more concretely the basic directions of researches in the field of multilayer “X-ray” optics in IPM RAS.

1) Development of optics for XEUV spectral range ($\lambda \sim 0.01$-100 nm)

1.1. Development of the technological and metrological equipment and technology of MLS deposition for elements of multilayer X-ray optics. Apparently, the special attention would be given to development of the stand for MLS deposition by methods of magnetron and ion beam sputtering (separately or in common) with an opportunity of ion beam polishing of the surface of each deposited layer.

1.2. Development of diagnostic methods for measurements of roughness of all lateral sizes of super-smooth surfaces, the development of appropriate laboratory equipment.

1.3. Development of a technologically-measuring complex for manufacturing of the imaging precision optics.

1.3.1. Development of interferometers with the diffraction reference wave, including optimization of correctors of the spherical wave for measurement of aspherical surfaces.

1.3.2. Development of methods and equipment for precision correction of the surface shape. Studying of an opportunity of strong (on tens of micrometers) aspherization of the super-smooth surfaces by methods of ion beam etching.

1.3.3. Development of methods of the MLS stresses control and its compensation.

1.4. Development of methods of manufacturing and diagnostics of free standing film structures (*multilayer mirrors: dispersive elements, polarizers, splitters; multilayer spectral filters for application in various schemes – nanolithographers, space telescopes, X-ray diagnostics of high-temperature plasma*).

1.5. Development of methods of manufacturing and certification of multilayer optics for formation of “hard” X-ray radiation beams, including shapers with a figure of rotation.

1.6. Development of manufacturing techniques and certification of multilayer dispersive elements.

2) Creation of element base and participation (in Russia and in the world) in creation projective XEUV lithographers on wavelengths of 13.5 nm and 6.7 nm, or on alternative wavelengths

2.1. Creation of laboratory manufacturing techniques of effective multilayer optics for spectral area of 6.7 nm and search for alternative wavelengths for the next generation lithography.

2.2. Development of free standing film structures for stands of projective nanolithography on a wavelength of 6.7 nm, or on an alternative wavelength.

2.3. Creation of the model sample of the projective scheme on the wavelength of 6.7 nm or on the alternative one, optimization of the photorisists for chosen spectral area.

2.4. Development of the laboratory sample of the nanolithographer-multiplier on the wavelength of 13.5 nm. Optimization of the photoresists for spectral area of 13.5 nm.

2.5. Initiating and participating in the development of short-wavelength lithography program in Russia.

3) **Creation of element base and development of X-ray microscopes with the spatial resolution of 5-20 nm**, *including for researches of biological objects in the ranges of "windows of transparency of water and carbon"*

4) Innovative activity.

. *We consider the results of researches representing innovative interest in Russia and in the world. On all innovations need appropriate patents.*

4.1. Multilayer dispersive elements, including focusing elements.

4.2. Elements for controlling hard X-ray radiation.

4.3. Elements of precision imaging optics, including reference surfaces (including aspherical) for schemes of the traditional interferometers.

4.4. Free standing film structures.

4.5. Optical elements for "X-ray" astronomy.

4.6. Sources of the spherical wave for point diffraction interferometers.

4.7. Interferometers with diffraction reference wave. In cooperation with industry development of an interferometer for industrial applications is supposed.

Selected publications 1993 - 2013 years

A.I. Chumakov, G.V. Smirnov, J. Arthur, S.L. Rude, D.E. Brown, A.Q.R. Raron, G.S. Rroun, N.N. Salashchenko. *Resonant Diffraction of Synchrotron Radiation by a Niclcar multilayer.* // Phys. Rev. Lett., V.71, №15, 1993, p. 2489-2492.

S.I. Zheludeva, M.V. Kovalchyk, N.N. Novikova, A.N. Sosphenov, N.E. Malysheva, N.N. Salashchenko, A.D. Akhsakhalyan, Yu.Ya. Platonov. *New modification of XRSW a both the surface of layered substrate under total external reflection conditions for structure characterization of organic layers.* // Thin Solid Films, 1993, V.232, p.252-256.

A.D. Akhsakhalyan, N.N. Kolachevsky, M.M. Mitropolsky, E.M. Ragozin, N.N. Salashchenko, V.A. Slemzin. *Fabrication and Investigation of imaging Normal-Incidennce Multilayer Mirrors with a Narrov-Band Reflection in the Range 4,5nm.* // Phisica Scripta, V. 48, 566-570, 1993.

V.A. Slemzin, I.A. Zhitnik, E.N. Ragozin, A.A. Andreev, N.N. Salashchenko, Yu.Ya. Platonov. *Aspherical imaging multilayer mirrors with sub-areseconnd resolution for solar XUV-telescope.* // SPIE Proc., V.2279, 1994, p. 234.

N.N. Salashchenko, Yu.Ya. Platonov, S.Yu. Zuev. *Multilayer x-ray optics for synchrotron radiation.* // NIM, 1995, A359, p.114-120.

N.N. Salashchenko, E.A. Shamov. *Short - period X - ray multilayers based on Cr/Sc.* //Optics communication, **134**, N 1-6, p.7-10 (1997).

A.V. Andreev, Yu.V. Ponomarev, I.R. Prednikov, N.N. Salashchenko. *Resonance amplification of diffusion scattering of the X-rays in heterostructures of waveguide type* // JETP Letters, **66**, no. 4, pp. 219-223, 1997.

F. Schaefers, H.-Ch. Mertins, I. Packe, F. Schmolla, N.N. Salashchenko, E.A. Shamov. *Cr/Sc - Multilayers for the Water Window.* //Applied Optics, **37**, 719-728 (1998).

S.S.Andreev, H.-Ch.Mertins, Yu.Ya. Platonov, N.N.Salashchenko, F. Schaefers, L.A. Shmaenok. *Multilayer dispersion optics for x-ray radiation* // Nucl. Instrum. and Meth. 2000, A448, 133-141.

L.A. Shmaenok, S.V. Golovkin, V.N. Govorun, A.V. Ekimov, N.N. Salashchenko, V.V. Pickalov, V.P. Belik, F.C. Schüller, A.J.H. Donné, A.A.M. Oomens, K.A. Prokhorov, S.S. Andreev, A.A. Sorokin, B.G. Podlaskin, L.V. Khasanov. *Novel Instrumentation for Spectrally Resolved Soft X-Ray Plasma Tomography: Development and Pilot Results on TEXTOR* // Review of Scientific Instruments, Vol. 72, No. 2, 1411-1415, 2001.

S.Yu. Zuev, E.B. Kluenkov, K.A. Prokhorov, N.N. Salashchenko. *Multilayer dispersion elements based on* B_4C *for the spectral range* $\lambda=6.7\text{-}8$ *nm //* Journal of Surface Investigation. X-ray, Synchrotron and Neutron Techniques, 2002, , no. 1, pp. 27-31.

S.S. Andreev, A.D. Akhsakhalyan, M.A. Bibishkin, N.I. Chkhalo, S.V. Gaponov, S.A. Gusev, E.B. Kluenkov, K.A. Prokhorov, N.N. Salashchenko, F. Schäfers, S.Yu. Zuev. *Multilayer optics for XUV spectral region: technology fabrication and applications* // Centr. Europ. Journ.of Phys. 2003. 1, p.191-209.

S.S. Andreev, M.S. Bibishkin, N.I. Chkhalo, E.B. Kluenkov, K.A. Prokhorov, N.N. Salashchenko, M.V. Zorina, F. Schaefers and L.A. Shmaenok. *Short - period multilayer X - ray mirrors* // Journal of Synchrotron Radiation Vol 10, Part 5, (2003) 358-360.

A.A. Akhsakhalyan, A.D. Akhsakhalyan, A.I. Kharitonov, E.B. Kluenkov, V.A. Murav'ev, N.N. Salashchenko. *Multilayer mirror systems to form hard X-ray beams.* // Central European Journal of Physics 3(2) 2005 163–177.

Yu.A. Vainer, A.E. Pestov, K.A. Prokhorov, N.N. Salashchenko, A.A. Fraerman, V.V. Chernov, and N.I. Chkhalo. *Analysis of Cross-Correlation of Interface Roughness in Multilayer Structures with Ultrashort Periods* // Journal of Experimental and Theoretical Physics. 2006. Vol.103. No. 3. Pp. 346-353.

N.N. Salashchenko, N.I. Chkhalo. *Short-wavelength projective lithography* // Bulletin of the Russian Academy of Sciences. V. 78. №5. 2008. Pp.13-20.

N.I. Chkhalo, A.Yu. Klimov, V.V. Rogov, N.N. Salashchenko, and M.N. Toropov. *A source of a reference spherical wave based on a single mode optical fiber with a narrowed exit aperture.* // Rev. Sci. Instrum. V.79, Issue 3**.** 2008.

A.V. Vodopyanov, S.V. Golubev, D.A. Mansfeld, A.G. Nikolaev, K.P. Savkin, N.N. Salashchenko, N.I. Chkhalo, and G.Yu. Yushkov. *Extreme-Ultraviolet Source Based on the Electron-Cyclotron-Resonance Discharge* // JETP Letters Vol. 88. No.2. Pp. 95-98. 2008.

E.B. Kluyenkov, A.E. Pestov, V.N. Polkovnikov, D.G. Raskin, M.N. Toropov, N.N. Salashchenko, and N.I. Chkhalo. *Testing and Correction of Optical Elements with Subnanometer Precision. Nanotechnologies in Russia* // Vol. 3. Nos. 9-10. Pp. 602-610. 2008.

Yu.E. Borozdin, E.D. Kazakov, V.I. Luchin, N.N. Salashchenko, V.V. Chernov, N.I. Chkhalo, A.P. Shevel'ko. *X-ray and VUV spectroscopy of plasma with using of new focusing multilayer structures* // JETP Letters Vol. 87. No.1. Pp. 33-35 (2008).

S.S. Andreev, M.M. Barysheva, N.I. Chkhalo, S.A. Gusev, A.E. Pestov, V.N. Polkovnikov, N.N. Salashchenko, L. A. Shmaenok, Y.A. Vainer, S.Y. Zuev. *Multilayered structures based on La/B4C (B9C) for projection XUV-lithography at wavelength of 6.7 nm.* Nuclear Instruments and Methods in Physics Research A. V.603. Issues 1-2. 2009. P. 80-82.

N.I. Chkhalo, E.B.Kluenkov, A.E.Pestov, V.N. Polkovnikov, N.N. Salashchenko, M.N. Toropov. *Manufacturing of XEUV mirrors with sub-nanometer surface shape accuracy.* Nuclear Instruments and Methods in Physics Research A. V.603. 2009. Issues 1-2. 2009. P. 62-65.

S. S. Andreev, M. M. Barysheva, N. I. Chkhalo, S. A. Gusev, A. E. Pestov, V. N. Polkovnikov, D. N. Rogachev, N. N. Salashchenko, Yu. A. Vainer, and S. Yu. Zuev. *Multilayer X_Ray Mirrors Based on La/B4C and La/B9C* // Technical Physics. Vol. 55. No. 8. 2010. Pp. 1168–1174.

N.I. Chkhalo, A.E. Pestov, N.N., Salashchenko and M.N. Toropov (2010). *Manufacturing and Investigating Objective Lens for Ultrahigh Resolution Lithography Facilities*, Lithography, Michael Wang (Ed.), ISBN: 978-953-307-064-3, pp. 656, (p. 71-114) INTECH, Available from: http://sciyo.com/articles/show/title/manufacturing-and-investigating-objective-lens-for-ultrahigh-resolution-lithography-facilities

M.M. Barysheva, B.A. Gribkov Yu. A. Vainer, M.V. Zorina, A.E. Pestov, Yu. Ya. Platonov, D.N. Rogachev, N.N. Salashchenko, N.I. Chkhalo. *Problem of roughness detection for supersmooth surfaces* // Proc. of SPIE. V. 8076. P. 80760M-1-10. 2011.

N.I. Chkhalo, M.N. Drozdov, S.A. Gusev, E.B. Kluenkov, A.Ya. Lopatin, V.I. Luchin, N.N. Salashchenko, L.A. Shmaenok, N.N. Tsybin, B.A. Volodin. *Freestanding multilayer films for application as phase retarders and spectral purity filters in the soft X-ray and EUV ranges* // Proc. of SPIE. V. 8076. P. 80760O-1-11. 2011.

N.I. Chkhalo, M.M. Barysheva, A.E. Pestov, N.N. Salashchenko, M.N. Toropov. *Manufacturing and characterization the diffraction quality normal incidence optics for the XEUV range.* // Proc. of SPIE. V. 8076. P. 80760P-1-13. 2011.

S.Yu. Zuev, E.B. Kluenkov, A.E. Pestov, V.N. Polkovnikov, N.N. Salashchenko, L.A. Suslov, M.N. Toropov, N.I. Chkhalo. *Technological complex for manufacturing of precision imaging optics //* Bulletin of the Russian Academy of Sciences: Physics. 2011. Vol. 75, No. 1, pp. 57–60.

Barysheva M M, Pestov A E, Salashchenko N N, Toropov M N, Chkhalo N I. *Precision imaging multilayer optics for soft X-rays and extreme ultraviolet* // Physics-Uspekhi. 55 (7) 681-699 (2012).

N.I. Chkhalo, M.N. Drozdov, E.B. Kluenkov, A.Ya. Lopatin, V.I. Luchin, N.N. Salashchenko, N.N. Tsybin, L.A. Shmaenok, V.E. Banine, A.M. Yakunin. *Free-standing spectral purity filters for EUV lithography //* Journal of Micro/Nanolithography, MEMS, and MOEMS, 11(2), 021115-1-7. 2012.

N.I. Chkhalo, S.V. Golubev, D.A. Mansfeld, N.N. Salashchenko, L.A. Shmaenok and A.V. Vodopyanov. *Source for EUVL based on plasma sustained by millimeter-wave Gyrotron radiation* // Journal of Micro/Nanolithography, MEMS, and MOEMS. 11(2), 021123 (1-7).2012.

N.I. Chkhalo, S. Künstner, V.N. Polkovnikov□, N.N. Salashchenko, F. Schäfers, S.D. Starikov. *High performance La/B4C multilayer mirrors with barrier layers for the next generation lithography* // Appl. Phys. Lett. 2013. V.1020. P.011602.