Development of a vacuum system for a compact soft X-ray source

K A Sergushichev, A A Smirnov, A A Samokhvalov, D B Belskiy, E P Bolshakov and V A Burtsev

1 Center of diagnosis and microtechnology, Saint Petersburg Electrotechnical University "LETI", Saint Petersburg 197376, Russia
2 Department of Laser Photonics and optoelectronics, ITMO University, 190000, Saint Petersburg, Russia
3 "Burtsev Laboratory" Limited Company, Saint Petersburg 197022, Russia

E-mail: samokhvalov.itmo@gmail.com

Abstract. The results of the development and experimental investigation of vacuum system with micromembrane element for a compact soft X-ray source and their implementation are presented.

Today compact sources of soft X-ray radiation (SXRs) have many scientific and technical applications, such as projection lithography, photoelectron spectroscopy, “water window” microscopy, nanostructuring of materials, etc. A significant problem of working with radiation of this wavelength range (2-15 nm) is its strong absorption in all media, given that the SXR sources have a low average power. The task arises of optimizing vacuum systems for the most efficient output of an SXR from its generation to the area its use.

The most efficient generation of SXR radiation is realized by creating a dense high-temperature plasma in the geometry of a hollow cathode, for example, in a ceramic capillary [1, 2]. In this case, the radiation source has an extended structure, namely, the length of the emitting plasma is ~ 1-2 mm, diameter ~ 500 μm, the divergence of the SXR beam from the capillary is ~ 30 mrad with a typical internal diameter of the capillary ~ 3 mm [3]. The radiation from the plasma region spreads to a cold working gas (Ar, Xe, N₂), whose typical pressures are 0.2-0.3 mbar, which corresponds to a particle concentration of ~ 10¹⁵ cm⁻³.

Thus, there are specific requirements for vacuum systems in the design and use of the sources of SXR: reducing the volume of vacuum chambers to ensure rapid pumping and implementation of the high-frequency operation of SXR sources, pumping out residual gases to ensure high vacuum (up to 10⁻⁶ mbar) and minimizing the losses of SXR in gas.

Such problems are solved with the help of differential pumping systems, i.e. multi-step vacuum systems. If it is necessary to pump out from an initial pressure of 10⁻¹ to 10⁻⁶ mbar, then at least three stages of pumping are required. At the output of each of the steps (chambers), a special vacuum separator (scimmer) is installed, the geometry and dimensions of which are determined by the required operating conditions of the system. Common disadvantages of differential pumping systems when working with plasma sources, in particular with sources of SXR are: the presence of residual gases in almost the entire path of the vacuum system, strong absorption of SXR in residual gases, the need for a
large number of powerful pumps, large dimensions of the entire system. In addition, when using SXR sources based on high-temperature plasma, the problem of filtering radiation arises, i.e. it is necessary to install a filter in the vacuum system after the radiation source that would reflect the radiation of the visible range and transmit the SXR, and as a rule, these are thin freely hanging metal foils with a thickness of less than 1 micron.

In our design of the compact SXR source, an attempt was made to combine vacuum separation and filtration of the desired SXR range using a micromembrane element. In our development, the micromembrane element was a multilayer structure (figure 1) obtained on a silicon substrate, in which a transparent window of SiO2 of 3x3 mm in size and 200 nm thick was formed, a thin film of aluminum was deposited on both membrane surfaces, the film thickness on both sides of the membrane was 200 nm. This design has increased strength and withstands pressures up to ~ 400 mbar. A thin metal film provides full reflection of visible radiation and transmission of SXR, and by combining films of different metals such as Al, Ti, you can select a narrow spectral region of SXR, for example, "water window" (2.2–4.4 nm), or create a different selective filter for interference lithography in the frequencies of SXR [4].

![Figure 1](image.png)

**Figure 1.** The cross section of micromembrane element and its working area photograph.

Figure 2 shows the layout of the vacuum assembly of a compact SXR source with a micromembrane element. The design for connecting a forevacuum pump and a pressure sensor provides two standard KF-16 series flanges, the pump and the sensor are connected to the flanges using a standard vacuum fastener (centering ring and hinged clamp), which allows for a fast installaton of the SXR source. For further installation with other vacuum systems, bellows hoses or standard vacuum flanges are used. Note that in SXR sources based on capillary plasma, the operating pressure range is 0.1-0.3 mbar and pumping to high vacuum is not required, but due to the existing structures of traditional differential pumping systems, the entire volume must be pumped out at once. In the case of using a micromembrane element with a thin metal film (SXR filter), this is not required, which is evident from the presented model.

Let's determine the time of pumping out the volume of the designed chamber (figure 3) using the well-known formula [5]:

\[ t = \frac{V}{S} \left( \ln \frac{p_a}{p_e} \right) F, \]

where \( t \) is the pumping time, \( h \), \( V \) is the volume of the pumped capacity, m\(^3\), \( S \) is the pump capacity, m\(^3\)/h, \( p_a \) is the initial vacuum level, mbar, \( p_e \) is the required vacuum level, mbar, \( F \) is the pumping curve coefficient, which for the range of pressure 0.1–1 mbar is assumed to be 3. Taking the pump capacity of 15 m\(^3\)/h (typical value for a series of spiral pumps), the pumping time to a pressure of 0.1 mbar will be ~5 s, which is a good result, since working pressures for Ar, Xe, N\(_2\) are in the range of 0.2–0.3 mbar. In the calculation, the pumping curve coefficient was taken equal to 3, although it largely depends on the volume of the pumped-out chamber and the design features of the system.
(chamber geometry, type and number of seals), however in [5] its upper value is assumed to be 10, which can worsen our estimates of pumping time insignificantly.

**Figure 2.** General view of the vacuum mode of a compact source of SXR.

![General view of the vacuum mode of a compact source of SXR.](image)

**Figure 3.** General view of the vacuum unit of the compact SXR source: 1 - gas inlet, 2 - capillary unit and return conductor (and its photo in the upper left corner), 3 - micromembrane element, 4 - flanges for connecting the pressure sensor and pump.

At the same time, the flow of gas in the sources of SXR in the area of plasma formation is not laminar due to the dynamics of ionization processes [6], pinch formation, plasma front movement [7], and ablation of the capillary walls. The nature of the flow of rarefied gases is determined by the Knudsen number: $Kn = \lambda/L$, where $\lambda$ is the mean free path, $L$ is the characteristic size of the gas flow region. The case of $Kn \gg 1$ corresponds to the free flow of gas, which is realized in the bulk of the chamber (figure 3); in the case of $Kn \ll 1$, the gas flow is described by hydrodynamic models, this variant is realized during the breakdown of a thin capillary. However, the intermediate value of $10^{-3} < Kn < 1$ can be realized at the boundary of plasma and non-ionized gas, it is in this transition layer that the main absorption of SXR can occur, this requires additional experimental studies.
To test the effectiveness of the micromembrane filter when working in the structure under consideration, experiments were carried out to register radiation pulses in a compact SXR source, for which the experimental sample of the chamber was filled with nitrogen (99.999%) and pumped out with an ESVP 300 (ERSTEVAK Ltd.) spiral pump to a pressure of 0.25 mbar. High-voltage pulse with a rise front of 1 kV/ns and an amplitude of 20 kV entered the cathode of the capillary, the gap between the cathode and the anode was 20 mm, the internal diameter of the capillary was 3 mm. The radiation appearing in the capillary was recorded with a high-speed FDUK-1UVSK (Technoexan Ltd.) silicon photodiode, which has a high sensitivity in a wide spectral range from visible to X-ray radiation. The photodiode was connected to the input of a Tektronix DPO7104 oscilloscope with a 1 GHz bandwidth.

As can be seen from the oscillogram (figure 4), the amplitude of the radiation signal falls by 30% after installing a thin-film aluminum filter (0.8 um thickness), and the duration of the radiation pulse is reduced from 52 ns to 10 ns. Specifically, the long back front disappears, which corresponds to the cut-off of the visible part of the radiation and the passage of SXR lying in the range of 2–15 nm.

Figure 4. Oscillograms of radiation pulses received in a compact SXR source, working gas - nitrogen, pressure 0.24 mbar, amplitude of voltage pulse 20kV: 1 - signal from an open photodiode, 2 - signal from a photodiode closed by a thin-film aluminum filter.

Thus, in the present work, a vacuum system for a compact MRI source is developed that performs two functions at once: 1) vacuum separation between an SXR source operating at low pressures (up to 1 mbar) and a high vacuum system operating at pressures up to \(10^{-6}\) mbar and 2) cut-off of visible radiation and selective emission of radiation in the range of 2–15 nm. The design is easily joined with the subsequent steps of any vacuum systems; this can be used to construct X-ray microscopes for working in a “water window”, compact lithographs and systems for processing the surface of materials with SXR beams, etc.

Acknowledgment
This work was supported by RFBR, project number 18-08-01066 ("Development of photon-based methods for study of the transformation of healthy cells into cancer, including submicroscopy").

References
[1] Wyndham E S, Favre M, Valdivia M P, Valenzuela J C, Chuaqui H and Bhuyan H 2010 Rev. Sci. Instrum. 81(9)
[2] Klosner M A and Silfvast W T 2001 Appl. Opt. 40(27) 4849–51
[3] Wachulak P W et al. 2016 JINST 11 P07002
[4] Brose S et al. 2012 Thin Solid Films 520(15) 5080–5
[5] Hoffman D M 1998 Handbook of vacuum science and technology (Elsevier Inc.)
[6] Timshina V B M, Eliseev S, Kalinin N and Letunovskaya M 2019 J. Appl. Phys. 143302 125
[7] Zakharov S V, Zakharov V S, Novikov V G, Mond M and Choi P 2008 Plasma Sources Sci. Technol. 17(2)