

Conference Paper

Beryllium Materials and Their Application in Energetics of Future

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Abstract

Beryllium materials are widely used in science and technology. They are of importance both for nuclear and thermonuclear energy applications. Additionally, metallic beryllium is transparent to x-ray radiation and used in the manufacture of X-ray windows and refractive x-ray lenses. SC "VNIINM" has developed beryllium materials for various purposes: for coating of the ITER's first wall, for focusing and controlling X-ray radiation. Porous beryllium is of particular interest as it is an upcoming trend as a temper and neutron multiplier in blankets of thermonuclear facilities. A unique combination of X-ray optical properties also makes it possible to use porous beryllium in imaging devices in a synchrotron radiation beam (speckle suppressor).

Keywords: beryllium composite, porosity, beryllium-foam, ITER, X-ray optics

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1. INTRODUCTION

Beryllium has an important function in the area of strategic materials. This is due to its nuclear properties and many other unique features, which make beryllium indispensable in the defense complex, space technologies, as well as in other strategically important areas of technology and science. Seven decades of VNIINM's activities have been marked by many achievements in the technology of beryllium, its compounds and materials based on the same. As part of ITER creation work, it is worth mentioning the global recognition of the beryllium grade (TGP-56PS) developed by VNIINM JSC and applied as one of the materials for covering the first wall of the vacuum chamber, as well its successful certification, qualification and testing in hydrogen and deuterium plasma. As regards the prospects of beryllium technologies developed by VNIINM, a few directions may be pointed out. The development of technologies applying beryllium hydride is of particular interest [1]. This article presents the data about application of perspective beryllium materials in thermonuclear power engineering and in synchrotron investigations, one of the most promising areas of modern material science.

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2. MATERIALS AND METHODS

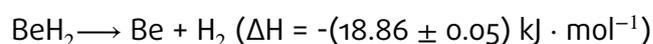
Here we report about getting and studying high-porous nanocrystalline beryllium (beryllium foam) and porous beryllium composite material made from a mixture of metal beryllium powder with beryllium foam. The structure was studied by optical microscopy, electronic microscopy and atomic force microscopy techniques. The studies were made by mercury injection techniques and hydrostatic weighing in decane. Furthermore, the compressive strength σ_p and microhardness of samples were determined. The investigations of optical and X-ray properties of beryllium materials were carried out at the Micro Optics Test Bench on the ID06 beamline of the European Synchrotron Radiation Source (ESRF).

3. RESULTS AND DISCUSSION

3.1. The use of beryllium in fusion energy

VNIINM JSC has developed a technology for producing a unique material of highly porous microcellular beryllium with a nanocrystalline structure (beryllium foam) [2]. The material has a density of 0.2 - 1.8 g / cm³ and high strength. The developed technology makes it possible to produce products of complex shape with specified characteristics, including a density gradient along the thickness of the product.

This technology is based on pyrolysis of metastable beryllium hydride (BeH₂) proceeding in accordance with the following equation:



If pyrolysis is carried out under predetermined conditions, beryllium is formed as a three-dimensional highly porous (70 to 90% porosity) cellular structure with micron pores (cells). The structure of such a material with a density of 0.35 g / cm³ is shown in Fig. 1.

Due to a sharp decrease in viscosity at the glass transition temperature and a relatively low diffusion coefficient of hydrogen, during the thermolysis the hydride particles are forming a microcellular structure. The exuding hydrogen played the role of the foaming agent. As the power of pyrolysis increases in the BeH₂-Be system, the content of metallic beryllium goes up and viscosity of this system rises sharply, and the emerging cellular structure gets solidified in space. A similar process takes place in the formation of foam polymers when using reactive oligomers [3, 4]. At the final stage of pyrolysis, the pore formation process in the walls (partitions) separating the cells takes place. As a result, microcellular beryllium has completely open porosity [5].

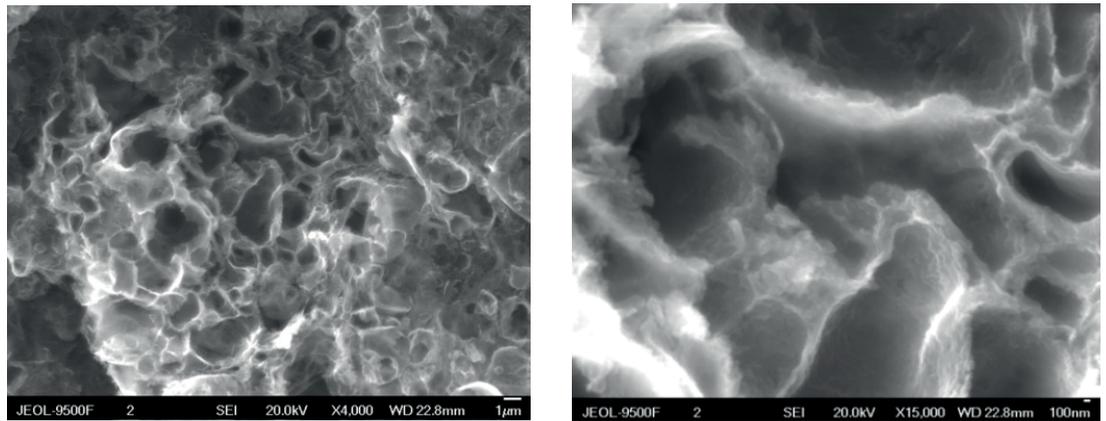


Figure 1: Structure of high-porous beryllium. Scanning electron microscopy.

Investigation of the cell wall structure by atomic force microscopy (Figure 2) reveals that they are formed by 50-100 nm particles and also have a porosity with a pore size of circa 0,1 micron. Investigation of the distribution of pores by mercury porosimetry confirms the presence of porosity in the walls of the cells. The average pore diameter is 0.15 to 0.25 μm . The majority of pores are in the range of 0.1 to 1 μm (Figure 3).

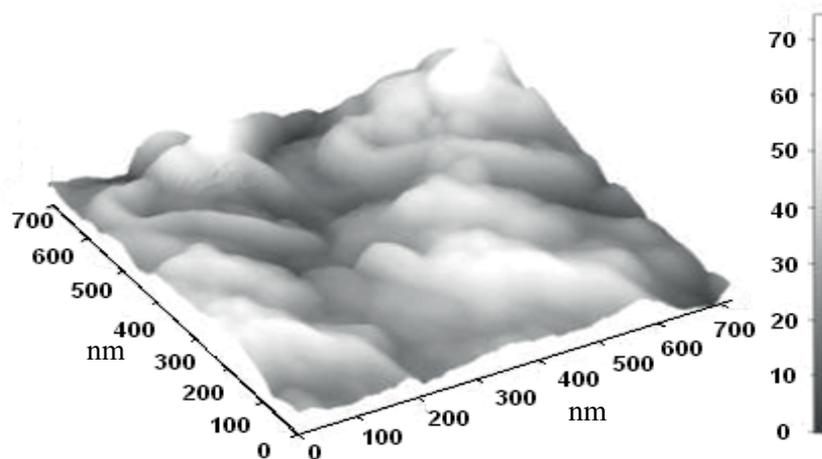


Figure 2: Structure of the cell wall. Atomic force microscopy.

Porous beryllium composite material (20-30% porosity) is offered for use in developed constructions of experimental breeder modules of the DEMO blanket for ITER testing. This material acts as a multiplier and reflector of neutrons enabling to remove radioactive gases due to its open pores structure [6-7].

The composite is produced by heat treatment of a precompressed mixture of metallic beryllium powders and beryllium hydride (BeH_2). The structure is large powder particles of metallic beryllium. The space between these particles is filled with beryllium

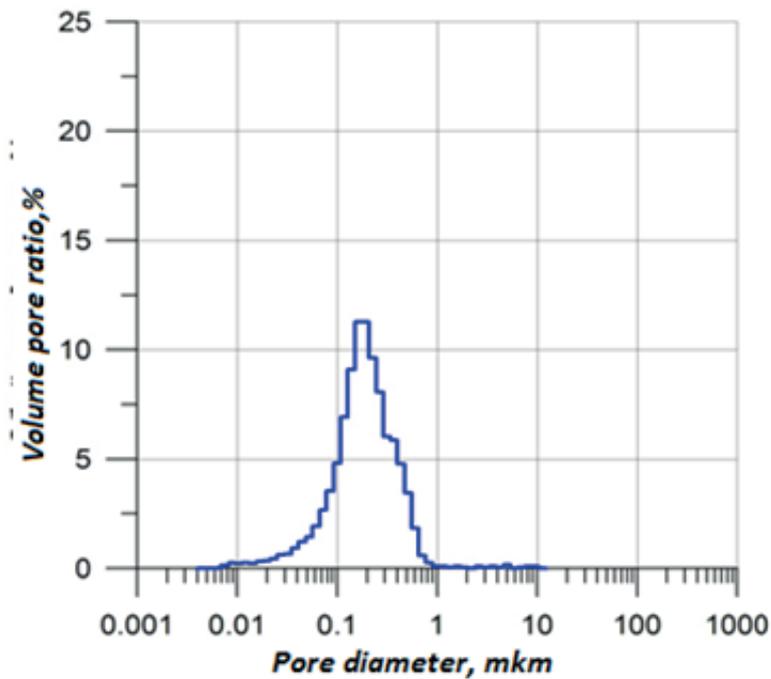


Figure 3: Results of mercury porosimetry.

foam. The evolving hydrogen gives rise to an increasing open porosity [8-10]. The proposed method makes it possible to make products with totally open controlled porosity and with a minimum inhomogeneity [7, 11]. By changing the process parameters (temperature and stress pressure), it may be feasible to control total porosity of the material according to the ratio:

$$P_{tot} = P_{in.} + 0.7124 \cdot X_{BeH_2} \quad (1)$$

where: **P_{tot}** – total porosity; **P_{in.}** – initial porosity of the compression; **X_{BeH₂}** - the volume fraction of beryllium hydride in the mixture.

Metallic powders of PTB-56, PDB-30 beryllium grades and powdery beryllium hydride with a particle size of 1-200 μm were initial materials, which were used in this experiment. Their characteristics are given in Table 1.

The structure and mechanical properties of porous beryllium were studied. The studies were carried out on samples obtained from the four mixtures which composition is shown in Table 2.

Metallographic specimens of beryllium composite with content of beryllium-foam 3.28 % wt and 20.34% porosity were studied by the optical microscopy technique (Figure 4)

TABLE 1: Specification of metall beryllium powders.

Powder index	Beryllium content, % wt.	* Substantial composition of major metal impurity, % wt..	Oxygen content, % wt	Average size of particle, mkm
PTB-56	No less than 98.5	0.26	0.46	35
PDB-30	-	0.12	0.87	18

* Impurities Si, Mn, Fe, Mg, Cr, Ni, Al, Cu.

TABLE 2: Specification of working mixtures.

Sl.No	Beryllium powder index	Beryllium hydride content, % wt.	Beryllium-foam content, % wt
1	PTB-56	4.01	3.28
2	PTB-56	5.88	4.81
3	PTB-56	10.05	8.21
4	PDB-30	9.7	7.93

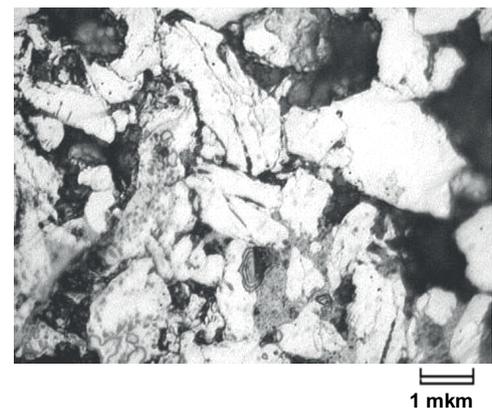
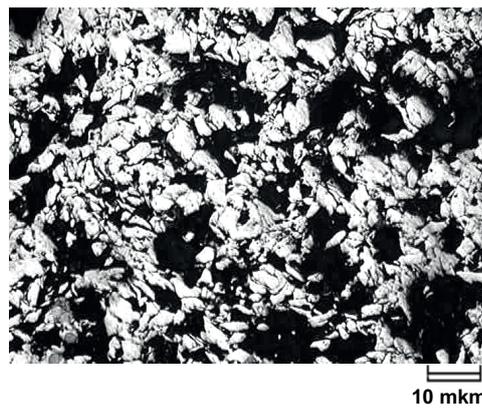


Figure 4: Typical structure of porous beryllium. Optical microscopy technique.

Figure 5 presents the SEM image of a composite sample chip with 31.2% porosity and containing 3.28% wt beryllium foam. Single large particles of metallic beryllium can be clearly seen and the interparticle space between them is filled with a phase of nanocrystalline beryllium, which has a microcellular structure. The size of the micro-cells is $\sim 1\text{-}5\ \mu\text{m}$, and the thickness of the walls is $\sim 0.1\text{-}0.3\ \mu\text{m}$ (100-300 nm).

The images were processed by SIAMS 600 to study pore size distribution. Pore size distributions for two samples with various porosity and contents of the beryllium foam are shown in Figure 6. The presented diagrams are similar, most pores have a radius of less than $10\ \mu\text{m}$. The average radius of such pores is $1.77\ \mu\text{m}$. Also, large pores (50

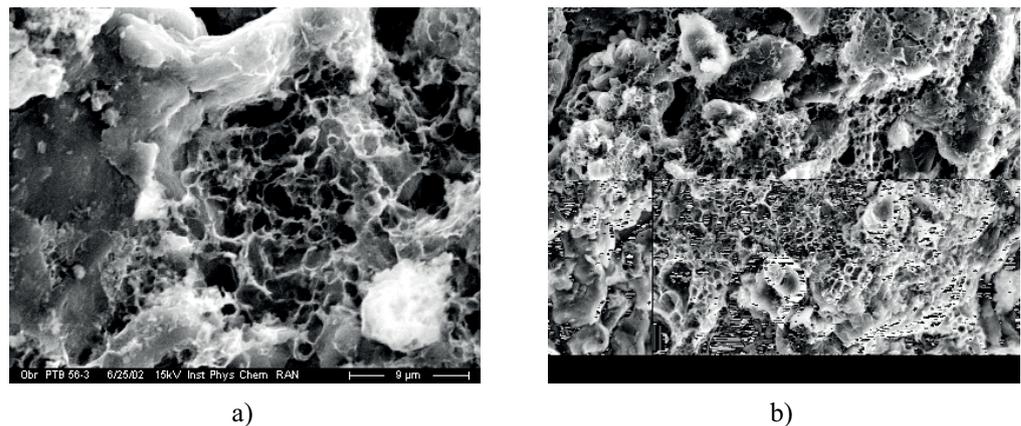


Figure 5: The cleavage structure of porous beryllium composite sample Scanning electron microscopy a) 3000x magnification b) 10000x magnification.

to 100 μm in size) are observed. Their appearance is due to that fact of beryllium foam does not completely fill the space between the larger particles of metallic beryllium.

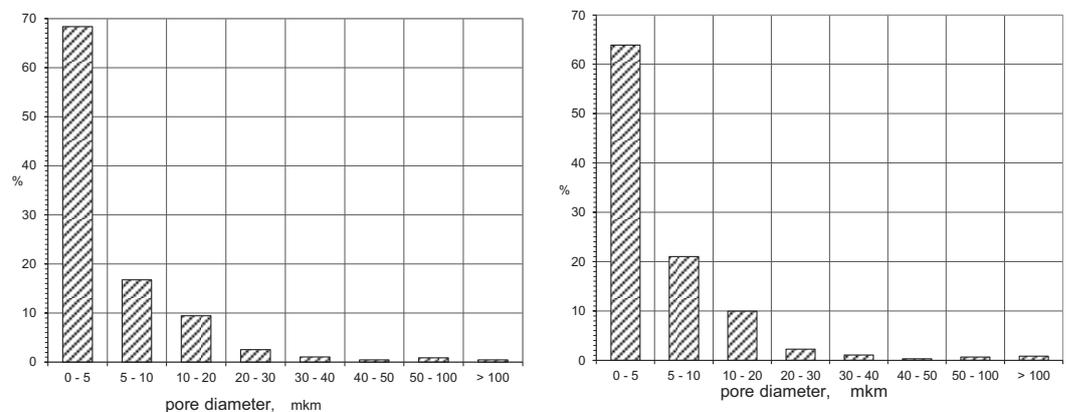


Figure 6: Pore size distribution. a) the sample with 20.34% porosity and 3.28% wt. beryllium foam b) the sample with 24.89 % porosity and 8.21% wt.beryllium foam.

In order to determine the specific surface area and investigate pore size distribution the same sample was researched by mercury porosimetry (Carlo Erba, Italy). The specific surface area is 4.0 m^2/g . The pore size distribution is shown in Fig. 7. It shows that the data obtained by the mercury porosimetry technique correspond to the results of electron and optical microscopy. i.e. most pores have a radius of less than 10 μm . The average radius of pores in the sample is 1.26 μm . The availability of large pores is also confirmed (peaks on the distribution histogram in the region of more than 50 μm).

To effectively remove helium and tritium, which is formed in beryllium as a result of neutron exposure, it is necessary for the composition material to have maximum open porosity. For this reason, the ratio between total and open porosity in samples with various beryllium-foam contents has been studied. Open porosity was determined by

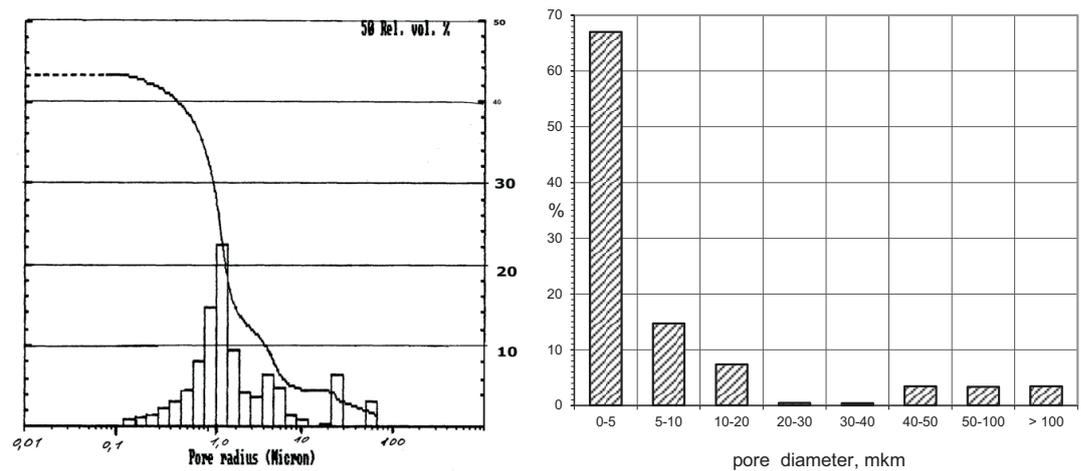


Figure 7: Pore size distribution in the sample of porous beryllium with 31.2% porosity and containing 3.28% wt. beryllium foam. a) histogram from Carlo Erbab) transformed histogram.

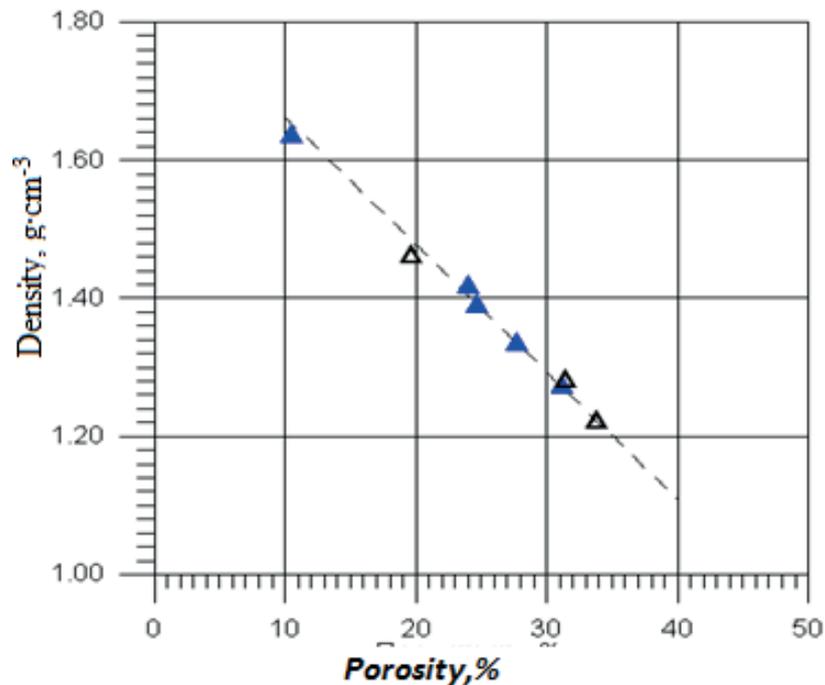
the hydrostatic weighing method in the decane [12]. Total porosity was calculated based on the dimensions and masses of the samples. Samples with the content of 3.28 and 8.21% wt beryllium foam. have been investigated. The results are shown in Figure 8. The direct line is the calculated ratio to density on the total porosity, and the points are experimental values of open porosity. It can be observed that the experimental and calculated values of porosity correlate well. It means that the composite samples have totally open porosity and proportion of open pores does not depend on the beryllium foam content for material in the range of 3.28 to 8.21% wt.

Compressive strength σ_p has been determined on cylindrical samples with 12 mm diameter and height of 15-20 mm. of different porosity. The tests have been carried out at room temperature and a traverse speed of 1 mm / min. Figure 9 shows a typical loading curve. The results of mechanical tests are given in Table 3 and Figure 10.

It is evident that the material has a wholly satisfactory strength - the minimum value is 74.3 MPa for sample with 34 % porosity and the maximum is 379.4 MPa for sample with 10% porosity. It can be observed that strength depends porosity solely (approx. linear dependence), and variations in beryllium foam content in the range of 8.21-3.88 % wt doesn't affect the strength. This fact has practical bearing insofar as a decrease in beryllium foam content reduces the total cost of the material.

Microhardness at various stresses was measured for 20.34 % and 24.89% porous beryllium samples which contained 3.28% wt and 8.21 5wt beryllium foam respectively (Table 4).

The results generated enable to draw a conclusion that even with minimum content of beryllium-foam and maximum porosity, the material has compressive strength of ~



- ▲ - data for samples with 3.38% beryllium-foam;
 △ - data for samples with 8.21% beryllium foam;
 ----- calculated density to total porosity ratio

Figure 8: Results of experimental measurements of open porosity.

75-100 MPa, which is quite sufficient for practical application. Practical research shows that material with such strength characteristics can be machined (grinding, drilling) that increases opportunity of technology [6].

Porous beryllium samples produced by SC VNNINM have been sent to NRG (Petten, Netherlands) for tests on radiation resistance.

3.2. The use of beryllium in synchrotron research

Beryllium has transparency to X-ray radiation that makes it in an irreplaceable material for use in medical and research equipment and in industrial and technological equipment. By applying porous nanoberyllium products, devices for removing X-ray image artifacts in X-ray optics were made [1, 13]. The use of such devices (speckle suppressors) with porous beryllium allows to manage coherence and gets images without speckles that are interference patterns formed by mutual interference of coherent waves having either or both random phase shifts and a random set of intensities and

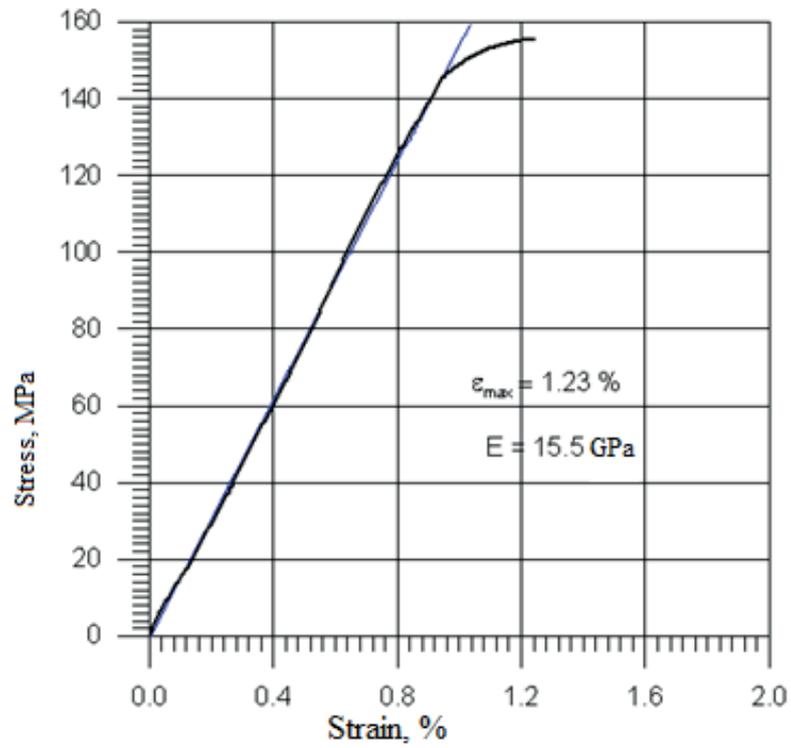


Figure 9: Compression curve of the sample with 31.18% porosity containing 3.28% beryllium foam.

TABLE 3: The results of compression tests.

The composition of Table. 2	Content of beryllium-foam, % wt	Porosity, %	σ_p , MPa
1	3.28	20.34	332.5
		31.18	155.4
		34.00	110.3
2	4.81	23.40	241.9
		31.39	141.2
		379.4	
3	8.21	24.89	222.2
		27.81	188.0
		288.6	
4	7.93	24.13	146.7
		32.20	96.3
		33.93	74.3

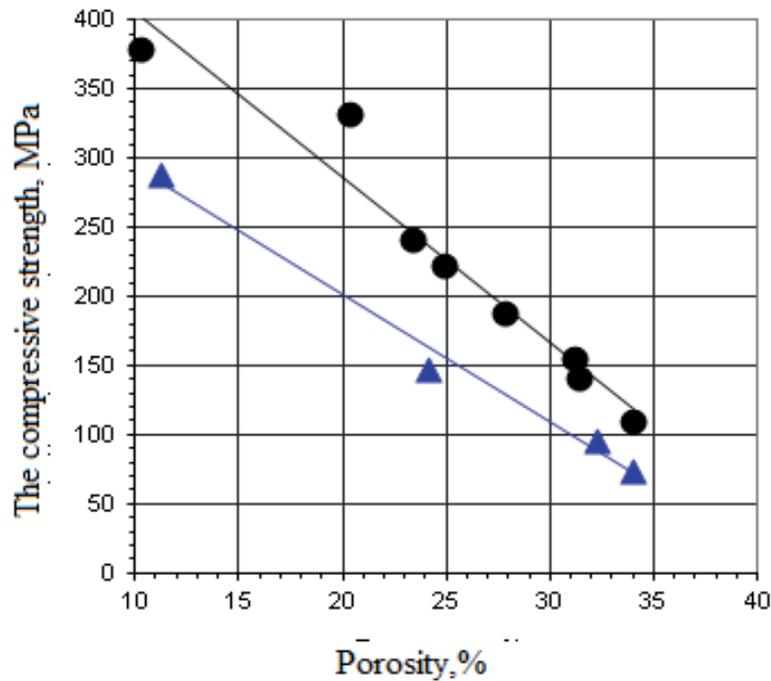


Figure 10: Compression strength in depending for porosity ● - samples from mixtures 1-3 (Table 2) with the content of 3.28-8.21% wt beryllium-foam; ▲ - samples from mixture 4 (Table 2) with the content of 7.93% wt beryllium-foam.

TABLE 4: Microhardness of porous beryllium samples.

Stress	Microhardness, kgf · mm ⁻²	
	20.34 % orosity	24.89 % porosity
50 g	125,5	121,4
100 g	107,1	121,5
150 g	105,0	87,80

reducing the quality of the resulting images. Low X-ray absorption, good mechanical properties and the presence of many small pores make porous beryllium a very attractive material for such devices.

The device containing a 4 mm thick paten of porous nanoberryllium was tested at the Micro Optics Test Bench on the IDo6 beamline of the European Synchrotron Radiation Source (ESRF) with a view to demonstrate the speckle-reduction effect [13].

Setting of the device in front of a boron fiber allows to remove interference fringes registered from the edge of the object in image fixed on the detector (Figure 11).

Figure 12 shows the images of a test object (Siemens Star) before and after setting of the speckle-suppressor based on porous beryllium. The presence of device of speckle suppression in the system significantly affects the image quality almost completely

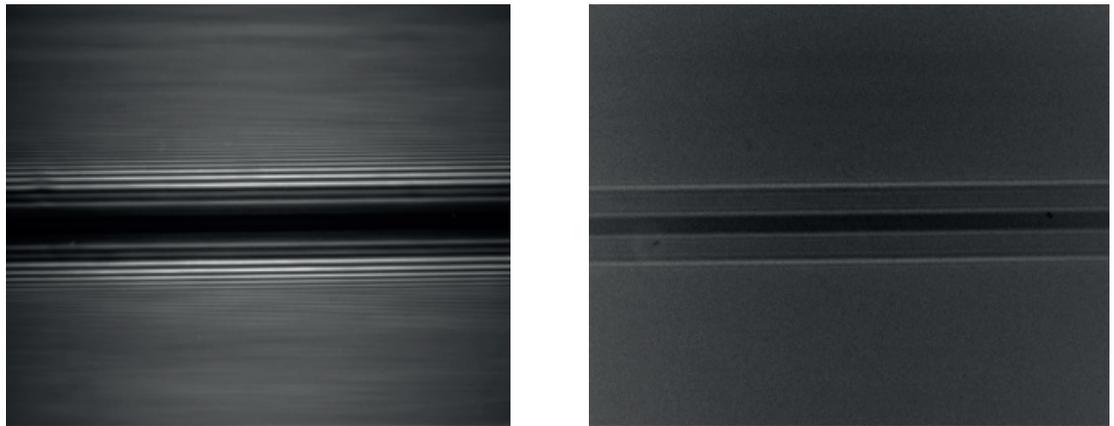


Figure 11: Image of the boron fiber before (a) and after (b) setting of the speckle-suppressor.

eliminating the speckle structures of the image that form due to the granulated structure of beryllium used in the x-ray windows and lenses.

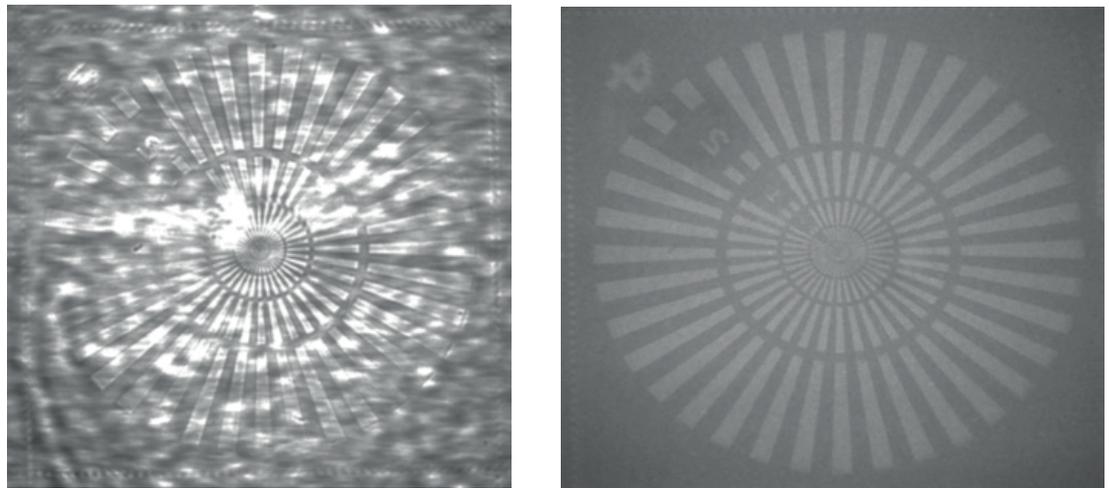


Figure 12: Image of the test object (Siemens Star) before (a) and after (b) setting of speckle-suppressor.

The presented results show that the use of a speckle-suppressor based on porous nanoberyllium in X-ray optics schemes allows to significantly improve the quality of the recorded image and make it clearer. Beryllium is also used as an optical material for X-ray refractive lenses. Such lenses are in demand by synchrotron centers and are manufacturers of X-ray equipment. SC VNIINM in cooperation with Immanuel Kant Baltic Federal University, Shubnikov Institute of Crystallography and National Research Center «Kurchatov Institute», is working on the creation of domestic production of lenses from beryllium [14].

4. CONCLUSIONS

1. A technology of manufacturing of high-porous beryllium with a nanocrystalline structure has been developed. The technology allows to generate complex shape products with specified characteristics, including a density gradient along the thickness of the product.
2. A technology of producing porous beryllium material for use as a part of the breeder modules of the DEMO blanket has been developed. The material has completely open porosity that makes possible to vent radioactive gases. The material with 10.2 - 34.0% porosity has a compressive strength (σ) 74.3 to 379.4 MPa, which is enough for practical use.
3. The possibility of using porous beryllium in devices called "speckle-suppressors" is shown. Such devices are intended to remove artifacts of X-ray images and improve the quality of X-ray images in X-ray optics.

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