

Conference Paper

The Small-angle X-Ray Scattering Investigation of Advanced Beryllium Materials

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Abstract

The small-angle X-ray scattering (SAXS) technique allows to determine size distributions of inhomogeneities in materials and to predict their properties in X-ray optics devices. A number of beryllium materials for the manufacturing of X-ray optics (refractive lenses and speckle suppressors) have been studied by the SAXS. Various composite materials based on porous beryllium have been studied. The effect of adding detonation nanodiamonds to the beryllium matrix on the scattering ability of the material is discussed.

Keywords: porous beryllium, SAXS, detonation nanodiamonds, speckle-suppressor

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1. INTRODUCTION

Small angle X-ray scattering (SAXS) is a non-destructive technique for investigation of distribution of inhomogeneities in materials at resolution from angstroms to hundred nanometers. The technique is based on the analysis of X-ray scattering intensity from fluctuations of electron density at small angles (less 10°). Investigation of features of SAXS images gives one information of distribution and particle (pore) size which allows to predict properties of X-ray optics devices. Recently it was reported [1, 2] that manufacturing technology of metallic beryllium influence SAXS features, and some recommendations of using beryllium materials for fabrication of composite refractive lenses (CRL) were given.

X-ray radiation is typically obtained with X-ray tubes and at synchrotron stations. Synchrotron sources provide beams of high intensity, brightness, degree of polarization and coherence. The use of X-ray optics devices may significantly increase the informative value of X-ray research results. Beryllium is one of the most important

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materials for X-ray optics, in particular, for refractive lenses. It is also promising for manufacturing various X-ray filters, especially speckle-suppressors [3, 4], which are produced from ultradisperse materials that allow to diffuse x-ray radiation and obtain images without speckles. Such ultra disperse materials, e.g. a spinning thin disc or amorphous boron powder, were used earlier as effective x-ray diffusers [4]. Using a rotating random-phase screen allows to reduce the degree of radiation coherence and enhance speckle elimination in transmission images [5]. Speckle-suppressor using porous beryllium was demonstrated efficiency of porous beryllium due to its high level of X-ray scattering. It is known that the solid dense beryllium has a relatively low scattering and absorption of X-rays in the range of low energies. For example the absorption of the 1 mm nanoberyllium plate is about 1% at 12 keV [3]. The use of speckle-suppressor with porous beryllium makes it possible to significantly increase the scattering and thus to destroy the beam coherence and to suppress image speckles. Thus porous beryllium may be considered an advanced media for X-ray optics intended for shading corrections. For the material to be effective, it is necessary to reduce dimensions of structural inhomogeneities which is to increase the scattering ability. This may be achieved by injection of nanosize particles with a different electron density into the porous structure.

2. MATERIALS AND METHODS

2.1. Materials

Detonation nanodiamonds (DND) with characteristic sizes from 4 to 6 nm as a component for the modification of porous beryllium are of a particular interest [6]. Nanodiamonds powder is usually manufactured as an explosion product of carbon-containing explosive [6].

The porous beryllium samples with different contents of nanodiamonds were manufactured and studied (table 1). The relative scattering ability of these samples at CuK α radiation (8.05 keV) was investigated, including pure nanoporous beryllium and nanodiamond powder (Table 1). The samples have completely open porosity about 70-80 %. The porosity (Table 1) was evaluated by hydrostatic weighing in decane using the general method [7].

TABLE 1: Manufactured materials.

S.No	Sample composition				P _{est} %	P _{exp} %
	Beryllium		nanodiamonds			
	% wt	% vol	% wt	% vol		
Be-16/2017	94,56	96,88	5,44	3,12	81,74	80,51
Be-17/2017	76,03	85,00	23,97	15,00	77,71	76,95
Be-18/2017	54,70	68,33	45,30	31,67	79,29	76,82
Be-19/2017	100,00	100,00	0,00	0,00	80,16	79,74
Be-21-2/2017	45,17	59,54	54,83	40,46	71,87	-

2.2. Techniques

The measurements of X-ray scattering intensity were performed on an automatic small-angle x-ray diffractometer "AMUR-K" which is equipped with a linear position-sensitive detector OD3M, at the wavelength λ 1.542 Å (CuK α tube with a pyrolytic graphite monochromator) and Kratky collimation system. The cross section of the X-ray beam at the sample was 0.2 x 8 mm. The range of scattering angles corresponded to the scattering vector modulus $0.1 < s < 1.0 \text{ nm}^{-1}$ ($|s| = \frac{4\pi \cdot \sin \theta}{\lambda}$, 2θ - angle of scattering). The measurements were carried out in a vacuum chamber. The sample-detector distance was 700 mm. The measurement time was 10 minutes per sample. Experimental data were normalized by intensity of incident X-ray beam. After that a correction of collimation distortions were performed following by subtraction of scattering from empty chamber to compensate residual intensity of the primary beam [8]. The measure procedure was carried out in certified techniques that are approved for «AMUR-K» installation [9, 10].

The calculation of size distribution of inhomogeneities was performed using the program POLYMIX which is an advanced version of the program MIXTURE, which, in turn, is included in the package ATSAS [11].

3. EXPERIMENTAL RESULTS AND DISCUSSION

Scattering curves are presented in Fig. 1 and 2. One could observe increasing SAXS intensity in the range from 0.5 to 2.5 nm^{-1} with the growth of nanodiamond concentration. This correlates with the shape of scattering image obtained from the pure nanodiamond powder. (Fig. 1).

It is interesting to note that in the region of very small angles at $s < 0.05 \text{ nm}^{-1}$, the scattering intensity of beryllium composite samples with a nanodiamonds content 45-55%wt significantly exceeds the scattering intensity of each of materials (nanodiamond powder and porous beryllium without nanodiamonds) individually (Fig. 2).

The occurrence of submicron inhomogeneities in the porous Be matrix should increase scattering from beryllium composite in the region close to central beam which, together with the enhancing of wide-angle scattering provided by nanodiamond phase, should increase efficiency of the material as a speckle-suppressor. A further studying of inhomogeneities distribution at sizes greater than 50 - 100 nm will be performed at synchrotron facilities of ultra-small-angle scattering.

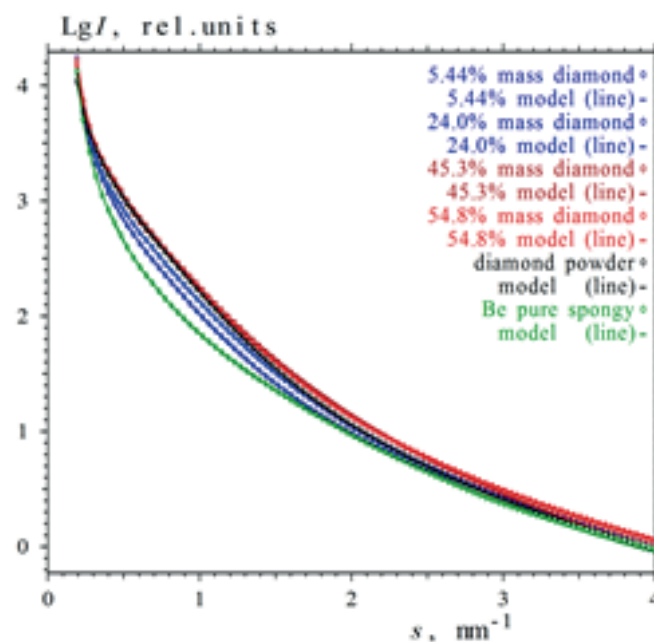


Figure 1: SAXS images from samples of porous beryllium with nanodisperse diamond, the original diamond powder and porous beryllium without add-ons.

Size distributions of scattering inhomogeneities calculated from SAXS data are shown in Fig. 3a. As follows from this picture, increasing the percentage of nanodiamonds in a beryllium matrix results in a growth of a portion of inhomogeneities with an average diameter from 4 to 5 nm, which corresponds to the distribution of particles in the diamond powder.

The peaks in the region of average diameters from 30 to 50 nm are apparently determined by the contribution of the porous structure of beryllium. The difference between shapes of the distributions in this region may be due to computational instability and will be clarified further by the ultra-low angle scattering experiments. An important result of research is that a mixture of nanodiamond powder and porous beryllium

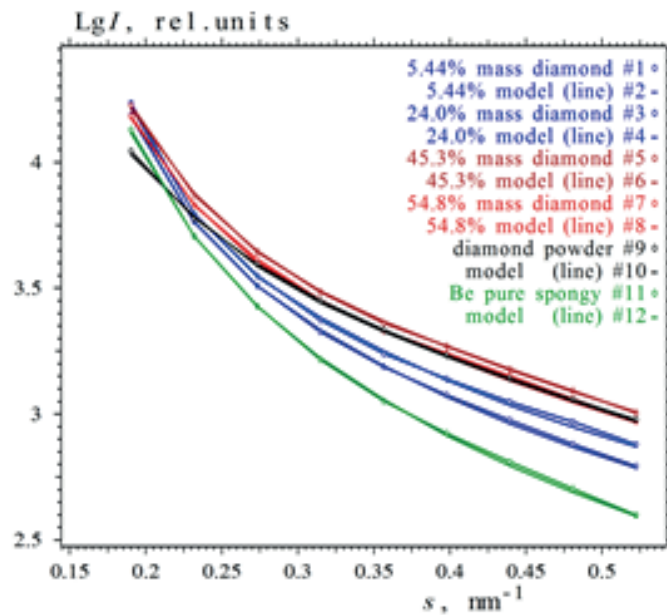


Figure 2: SAXS images Fig. 1 near the primary beam.

demonstrates a higher intensity of SAXS than the original materials. This could be a basis for getting new effective materials for devices of suppression of speckles based on these composites in synchrotron studies.

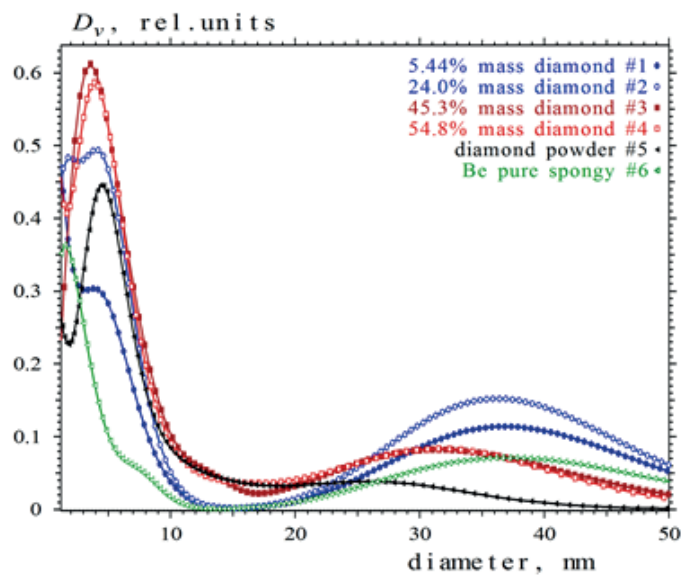


Figure 3: Size distributions of scattering inhomogeneities calculated from SAXS data (Fig 1).

4. CONCLUSIONS

1. The new materials based on porous beryllium containing detonation nanodiamonds were manufactured and investigated.
2. The influence of nanodiamond particles in composite samples of "beryllium-nanodiamonds" on the SAXS ability was established.
3. The distribution of size of inhomogeneities in the volume of the manufactured materials was calculated from SAXS data.
4. It was demonstrated that composites with 44-55 wt% nanodiamonds have a higher intensity of SAXS than porous beryllium and nanodiamonds individually. This can be a basis for getting new effective materials for devices intended for suppression of speckles in transmission images obtained at coherent synchrotron sources.

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