Investigation on the structure of beryllium composite containing nanodiamonds

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Abstract. The article presents the results of research of the structure of advanced beryllium composite materials for use in X-ray and synchrotron technology. The technique of acquiring beryllium composite containing nanodiamonds is described. The investigation on the structure was carried out using the scanning electron microscope microanalyzer Philips XL-30. The spectral analysis of composites was carried out using an energy-dispersive spectrometer with a Sapphire detector.

1. Introduction
The materials applied in X-ray optics, for instance, in speckle suppressors should meet the following requirements: 1) to be radio transparent with minimum absorption 2) to scatter X-ray 3) to have thermal stability of 600 °C or higher. It has been established that the optimal material for making speckle-suppressor devices is a composite consisting of beryllium matrix where the carbon filler is evenly distributed in order to raise the amount of structure inhomogeneities [1]. The composite material has a high scattering ability and allows reducing the degree of radiation coherence [2]. Nevertheless, the main requirement for the composite is a high uniformity of its physical and chemical features. This uniformity could be possible only by means of a uniform distribution of all components throughout the volume.

The need for even distribution of the filler is a hard technical task, as particles of the filler tend to form agglomerate and conglomerate of various shapes [3], resulting in significant negative consequences for end-user products.

2. Materials and Methods
A technique has been proposed for creating a composite material by dispersing mixing of a thermo labile compound (beryllium hydride) and ultrafine detonation nanodiamond powder in fluid with further compaction and thermal treatment. Therefore, during the heat treatment two processes are taking place – decomposition of basic beryllium hydride and establishment of a strong adhesive link between beryllium particles and nanodiamond powder.

The resulting composite has open porosity and advanced mechanical characteristics (at the same porosity) than porous beryllium obtained by traditional ways of powder metallurgy. Furthermore, the structure of the composite consists of nanosized crystallites.

Table 1 presents composition of samples with various content of nanodiamonds.
Table 1. Composition of samples.

| №№ | Sample composition |       |       |       |       |
|-----|--------------------|-------|-------|-------|-------|
|     |                    | wt %  | vol % | wt %  | vol % |
| 1   | Beryllium          | 94.56 | 96.88 | 5.44  | 3.12  |
| 2   | Nanodiamonds       | 76.03 | 85.00 | 23.97 | 15.00 |
| 3   |                   | 54.70 | 68.33 | 45.30 | 31.67 |
| 4   |                   | 45.17 | 59.54 | 54.83 | 40.46 |

The assessment of the composites structure homogeneity has been carried out using the scanning electron microscope microanalyzer Philips XL-30 equipped with an energy-dispersive spectrometer with a Sapphire detector operating under EDAX software.

3. Results and Discussion

It was shown [1] that the maximum x-ray scattering is found in composites with nanodiamonds content from 45 to 55 wt. %. An investigation of the microstructure and elemental analysis has been carried out along the structure cross-section.

Figure 1 presents composite microstructure with filler content at 54.83 wt.% sample 4 from Table 1 with an indication of layers used to assess composition elements.

Table 2 shows the results of composite analysis along the structure cross-section present in Figure 1.

Table 2. Results of the composite elemental analysis along the structure cross-section.

| №№ | C         | O         | Ca        | Fe        | Total     |
|-----|-----------|-----------|-----------|-----------|-----------|
|     | wt %      | at %      | wt %      | at %      | wt %      | at %      | wt %      | at %      |
| 1   | 74.40     | 79.99     | 24.36     | 19.66     | 0.7       | 0.22      | 0.55      | 0.13      | 100.00    | 100.00    |
| 2   | 69.36     | 76.32     | 27.61     | 22.80     | 1.71      | 0.56      | 1.33      | 0.31      | 100.00    | 100.00    |
| 3   | 72.77     | 78.93     | 25.15     | 20.48     | 1.19      | 0.39      | 0.90      | 0.21      | 100.00    | 100.00    |
| 4   | 75.10     | 80.77     | 23.24     | 18.76     | 0.94      | 0.30      | 0.72      | 0.17      | 100.00    | 100.00    |
| 5   | 74.20     | 79.99     | 24.15     | 19.54     | 0.89      | 0.29      | 0.76      | 0.18      | 100.00    | 100.00    |
| 6   | 80.74     | 85.57     | 17.52     | 13.94     | 0.99      | 0.31      | 0.75      | 0.17      | 100.00    | 100.00    |
| 7   | 72.44     | 78.32     | 26.24     | 21.30     | 0.78      | 0.25      | 0.54      | 0.13      | 100.00    | 100.00    |
| 8   | 75.84     | 81.15     | 23.11     | 18.56     | 0.58      | 0.19      | 0.47      | 0.11      | 100.00    | 100.00    |
| 9   | 80.60     | 85.52     | 17.55     | 13.97     | 0.96      | 0.31      | 0.89      | 0.20      | 100.00    | 100.00    |
Basing on the data from Table 2, it can be observed that the content of carbon particles at various layers along the structure cross-section varies from 69 to 81 wt. %, which indicates a relatively uniform distribution of nanodiamond particles in the beryllium matrix. Oxygen occurrence can be ascribed to oxide film formation at the composite surface, whereas impurities of calcium and ferrum stem from the fact that the said impurities were imported when performing the operations of composite making.

![Figure 2. Microstructure and elemental analysis of the first layer composite.](image1)

![Figure 3. Microstructure and elemental analysis of the second layer composite.](image2)

![Figure 4. Microstructure and elemental analysis of the sixth layer composite.](image3)

Figures 2-4 demonstrate SEM of several layers. It can be observed that the composite structure with distributed carbon filler is represented by highly-porous microcellular beryllium matrix as the same structures as described above [4]. Carbon inclusions are spread out in the composite structure particularly as agglomerations distributed at the porous beryllium matrix, whereas the presence in the structure of inhomogeneities with an average diameter of 4-5 nm corresponding to single nanodiamond particles is confirmed by small-angle X-ray scattering [1].
4. Conclusion

A technique has been proposed for creating a composite material by dispersing mixing of a thermo labile compound (beryllium hydride) and ultrafine detonation nanodiamond powder in fluid with further compaction and thermal treatment.

Composites with the content of nanodiamond filler from 5 to 55 wt. % have been produced.

The investigation of elemental analysis of obtained composite demonstrated that carbon particles at various layers along the structure cross-section fluctuate from 69 to 81 wt. %, which indicates a relatively uniform distribution of the filler in the composite.

The composite structure is represented by highly-porous microcellular beryllium matrix with a carbon filler distributed in it. Moreover, carbon inclusions are allocated in the structure mostly as agglomerations distributed at the porous beryllium matrix.

References
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