INVESTIGATION OF PROPERTY OF POLYCRYSTALLINE DIAMOND PURIFIED BY TECHNOLOGY OXIDATIVE ALKALINE MELTS

PhD.[1] Kozub P.A, M.Sc. [1] Chernikov I.A., Dr.Sc. [2] Bagliuk G,A. (1), PhD.[2] Kurovskyi V.Ya. (1), PhD.[1] Kozub S.N.
PF CIT, Ukraine,, Kharkiv [1]
I IPMS of UAS, Ukraine, Kyiv [2]

E-mail: mail@cit-ua.com, , dep36@materials.kiev.ua

Abstract: *Properties of polycrystalline nanodiamond powder after treatment under oxidizing alkali melts of sodium nitrate and sodium hydroxide were investigated. It is established that the dried initial product contains the particles with size up to 100 microns, which are easily broken by ultrasonic treatment to size about 15 micron. A comparative grain-size analysis of samples before and after treatment of oxidative alkaline melts showed that the average grain size is reduced to 6 microns and the particles of less than 1 micron are formed. It is shown that after treatment in the oxidative alkaline melt final material does not contain non-diamond carbon and the dried powder of polycrystalline diamond practically does not aggregate.*

Keywords: POLYCRISTALLINE DIAMOND, DETONATION SYNTHESIS, OXIDATIVE ALCALINE MELTS, DISPERSIVENESS

1. Introduction

Polycrystalline diamond of detonation synthesis is now one of the most perspective materials collecting all the most valuable properties both micro- and nano- dispersed diamond. But in the same time this material has the similar to detonation synthesis problems for its technology of production. And this fact defines high cost of final product.

The most difficult problem for this technology is the removal of graphite from the initial product synthesis. High dispersiveness of nanodiamond particles leads to situation when its reaction ability stays similar with reaction ability of graphite particles even with more size.

As result, increase of oxidation degree of graphite leads to a loss of fine fractions (nanodiamond) and vice versa, decrease of oxidation degree of nanodiamond leads to reducing of efficiency of purification process and to possible presence of large graphite particles in the final product.

On the other hand main feature of product synthesis of polycrystalline nanodiamond powder is probability of inclusion in its grains of particles of graphite. And as result during of cleaning from non-diamond graphite by traditional techniques (chrome acid, nitric acid, alkaline) both full damage and availability of polycrystalline with enhanced concentration of graphite is possible. In any case this is leads to loss of quality and utilization properties of end product.

2. Purification of diamond raw materials by oxidative alkaline melts

Our investigations shows that using of oxidative alkaline melts for process cleaning of diamond containing materials allow to carry out of full elimination of non diamond form of carbon under comparatively soft conditions that ensure maximum yield of nano-diamonds.

Earlier experimental investigations for products of both static and detonation synthesis shown that yield of diamond was not less then 99% and most part of its loss was caused of auxiliary operations – washing of residue, drying, desintegratin [1,2].

Moreover, it was established that better wetting ability of such melts can remove non diamond carbon from the aggregates penetrating in the most inaccessible places of graphite diamond aggregates.

But, theoretically this method also can lead to a negative result such as destruction of the agglomerates to an unacceptably low size particles or reducing of strength characteristics of final product.

3. Experimental research and its results

So to examination of possibility of use of developed technology of treatment of product of detonation synthesis of polycrystalline diamond the samples of raw material which were reacted with melts of hydroxide and nitrate of sodium were analyze on presence of graphite and particle size distribution.

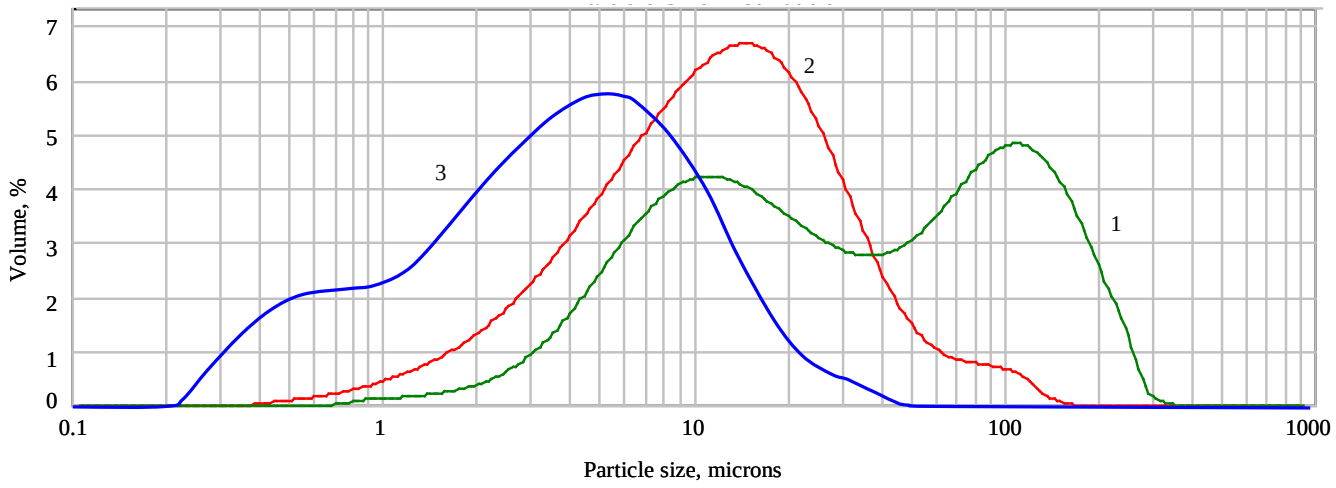

Fig. 1 *Grain-size analysis:*
1- untreated product synthesis; 2 – product synthesis after ultrasonic treatment; 3 – final product after ultrasonic treatment.

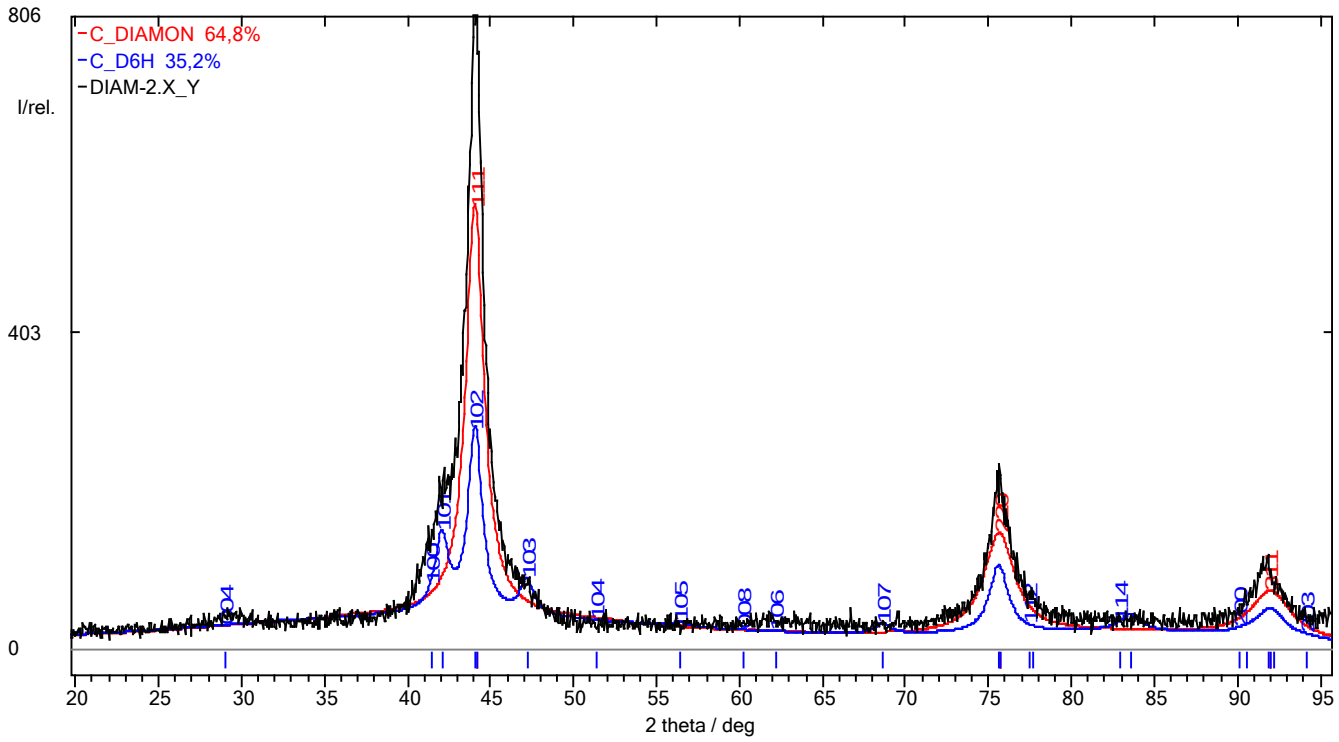

Fig.2 *Data of X-Ray analysis for purified polycrystalline diamond powder*

One lot of raw material was mechanically disintegrated to verify existing in the scientific community and among technologists widespread assumption about obligatory preliminary opening of agglomerates for full graphite removal.

Additional particle size distribution analysis were carried out for samples of end product in the form of slurry suspension, suspension of preliminary dried powder of end product and these suspensions after ultrasonic treatment. It gives the possibility to evaluate the tendency to form aggregates during drying of end product.

As a result, particle-size analysis of product of synthesis shows that main parts of particles have size 12-18 microns and 90-110 microns with roughly comparable amount of these fractions (Fig.1, line 1).

After ultrasonic treatment more than 90% of fraction with size 90-110 microns was destroyed and basic fraction becomes the fraction with prevalent size of particle about 15 microns (Fig.1, line 2).

It means that real size of aggregates which react with oxidation alkaline melt at average size 12-18 microns.

Also it should be noted absence of fraction of less than 2 microns which indicates sufficient mechanical strength of the particles of the starting material before chemical treatment.

For determination of possible impact of the melt on the fine fraction of the diamond-like carbon were treated three different samples. After reaction of this samples with oxidative alkaline melt the amount of diamond-like carbon were 30.4, 30.1 and 30.6 % from initial mass of untreated product of synthesis, product of synthesis after ultrasonic treatment and product of synthesis after mechanic disintegration respectively.

This difference is within the experimental error, and indicates the possibility of purification of polycrystalline diamond powder even without prior grinding.

These conclusions are also supported by almost identical X-ray diffraction patterns of all three samples that do not contain non-diamond carbon reflexes and diamond carbon is represented by two modifications with similar reflexes (see Fig.2).

It should be noted that the distribution of particle fractions indicates the appearance of a small amount (about 5-7%) of fine particles with a size from 0.4 to 0.8 microns (Fig.1, line 3).

It is accompanied by reducing of amount of fractions with a maximum of about 8 microns and this process is observed for both for suspension and for dry powder. But for last sample the process of reducing of particle size is more noticeable and it is suggest that dried powder agglomerates consist from weakly mechanically interconnected diamond grains in an amount of about 10-12 units.

4. Conclusions

Thus, our investigations have shown that the oxidative alkaline melts can be used for treatment of product of detonation synthesis containing all range of diamond materials - nanoscale, polycrystalline, micropowders without prior classification.

Furthermore, even in the presence of large graphite particles in the raw material, the amount of the residual graphite can be reduced to less than 0.05% within 1 hour of treatment.

It is found that the quality of the final material is not changed in the absence of prior mechanical grinding to opening of graphite and the final product does not contain graphite grains even more than 2 microns.

It has been shown that after reaction the average grain size is reduced by about 3-fold to 5 microns but at the same time it is formed particles with size less than 1 micron.

The resulting powder of polycrystalline diamond is resistant to agglomeration and can be easily recovered to the suspension by ultrasonic treatment.

5. References:

1. Patent of Ukraine № 78915 from 25.04.2007

2. Patent of Ukraine for Utility Model № 99893 from 25.06.2015