

Article

Several Aspects of Application of Nanodiamonds as Reinforcements for Metal Matrix Composites

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Abstract: After detonation synthesis, primary nanodiamond particles are around 4–6 nm in size. However, they join into agglomerates with larger parameters and weak bonds between particles. The introduction of agglomerates into a metal matrix can lead to the weakness of composites. This paper demonstrates the possibility of obtaining a non-agglomerated distribution of nanodiamonds inside a metal matrix. The fabrication method was based on mechanical alloying to create additional stresses and deformations by phase transformations during treatment in a planetary mill. According to the findings, the starting temperature of the reaction between the non-agglomerated nanodiamonds and aluminium matrix reduces to 450 °C. Furthermore, the paper shows that existing methods (annealing for the transformation of a diamond structure into graphitic material and cleaning from this graphitic material) cannot reduce the sizes of nanodiamonds in the agglomerated state. Agglomerated nanodiamonds transform into carbon onions (graphitic material) during annealing in a vacuum in the following way: the nanodiamonds located in the surface layers of the agglomerate are the first to undergo the complete transformation followed by the transformation of nanoparticles in its deeper layers. In the intermediate state, the agglomerate has a graphitic surface layer and a core from nanodiamonds: cleaning from graphite cannot reduce nanodiamond particle size.



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1. Introduction

Carbon nanoparticles, including nanodiamonds (NDs), evoke considerable interest among researchers because of their unique properties [1–12]. The size of primary ND particles is generally 4–6 nm. Nevertheless, depending on synthesis conditions, particles of both smaller (up to 2 nm) and larger (up to 10 nm and more) [1] sizes also occur. A peculiarity of ND powders, which is valid for any powdery nanomaterials, is their agglomeration, i.e., their consolidation into larger aggregates. Aggregation is an issue typical for detonation nanodiamonds. ND agglomerates are divided into primary and secondary: (i) primary ND agglomerates (up to 100 nm) are constituted by primary ND particles with the creation of coherent and incoherent boundaries with C–C bonds (basically) between primary nanoparticles (created spontaneously under explosion conditions); (ii) secondary ND agglomerates are formed through the creation of bonds between oxygen-containing surface groups and Van der Waals forces during ND purification stages. The deagglomeration of detonation nanodiamonds was investigated by several groups of researchers [13–15].

It was ascertained shortly after discovering NDs that heating in an oxygen-free environment to temperatures above 1000 °C resulted in NDs transforming into onion-like carbon nanoparticles (carbon onions), substantially being a graphitic material [16–23]. Furthermore, the mechanism leading to the sp^3 to sp^2 transformation at the ND surface was previously reported [22].

The use of carbon nanoparticles as reinforcements in metal matrix composites (MMC) is one of their future applications [24–26]. The presence of ND agglomerates inside the metal matrix can lead to the decomposition of material under complicated loading because

the strength of agglomerates is weaker than the strength of the metal matrix. This is why it is necessary to develop a non-agglomerated state of nanodiamond-reinforcing particles inside the metal matrix. The most effective method for destroying agglomerates inside the metal matrix is mechanical alloying [27], i.e., treating composite components by balls (milling instruments) in a planetary mill. The purpose of the present study is to show a possibility of the complete crushing of ND agglomerates inside a metal matrix (to obtain a non-agglomerated distribution of nanodiamond-reinforcing particles inside the metal matrix in MMC) and check the workability of the developed material. This means that the size of the reinforcements will become a parameter for nanodiamonds after synthesis, namely 4–6 nm.

The following question appears: is it possible to decrease the size of these reinforcing particles? It is well known that during heating by the electron beam in the transmission electron microscope, nanodiamond particles slowly, layer by layer (or small block by small block), transform into carbon onion. Based on this information, the hypothesis has appeared concerning the possibility of decreasing the nanodiamond size by partially annealing and purification from graphitic layers. However, temperature conditions in a microscope and the furnace are different: in the microscope, a particle receives energy from one side and loses it from the other side, but in the furnace, particles receive energy from all sides. In agglomerated state (in which the particles are used for annealing), the energetic state of particles on the surface of agglomerates and in central zones are different (different free surface and surface energy): it is the reason for a different way of transforming nanodiamonds from the surface and from deeper layers of agglomerates. Part of this paper is about the effect of the agglomerated state of NDs on such transformation processes.

2. Materials and Methods

Commercially available detonation nanodiamonds (produced by Elektrokhimpribor, Russia), in which the diamond component was $95\% \pm 2\%$, were used in this research. The average size of ND particles was 4 to 6 nm (Figure 1a–c). Part of the nanoparticles had defects in their crystal structure in the form of various twins (Figure 1b,c). Primary nanodiamond particles were consolidated into agglomerates (Figure 1d); for the present study, the agglomerates' sizes ranged from 20 microns to 200 microns; the average size of the agglomerates was 50 microns.

Commercially available metals for the matrix were aluminium and copper. Metal matrix composites were fabricated by mechanical alloying (ball milling) [27–29] in planetary mill Retch 400. The diameter of the balls was 12 mm. The “weight of balls/weight of treated materials” rate was 10. Mechanical alloying was executed in closed milling jars with a volume of 500 mL in argon atmosphere during 6–10 h (the stops for cooling were not calculated as milling time).

For the annealing of nanodiamonds, the quantity of investigated material for each experiment was 25–30 g. Annealing was done using the crucible in vacuum furnaces at a residual pressure of 10^{-5} millibar. Before evacuating, the furnace was filled with argon, i.e., the residual atmosphere was mainly argon. Then, the annealing temperature was raised at 20 degrees/min to 1200–1300 °C. The holding period of 1 h was followed by a cool-down to 800 °C at a 20 degrees/min rate, and the heating was then turned off. Samples were removed from the furnace after completely cooling.

The materials were studied using the scanning electron microscope Helios Nanolab 600i, transmission electron microscopes JEOL JEM 2100 F/Cs, JEOL JEM 2100, FEI TITAN 80–300 with EELS (electron energy loss spectroscopy), X-ray diffractometer DRON-3, and the differential scanning calorimeter Netzsch DSC 404C.

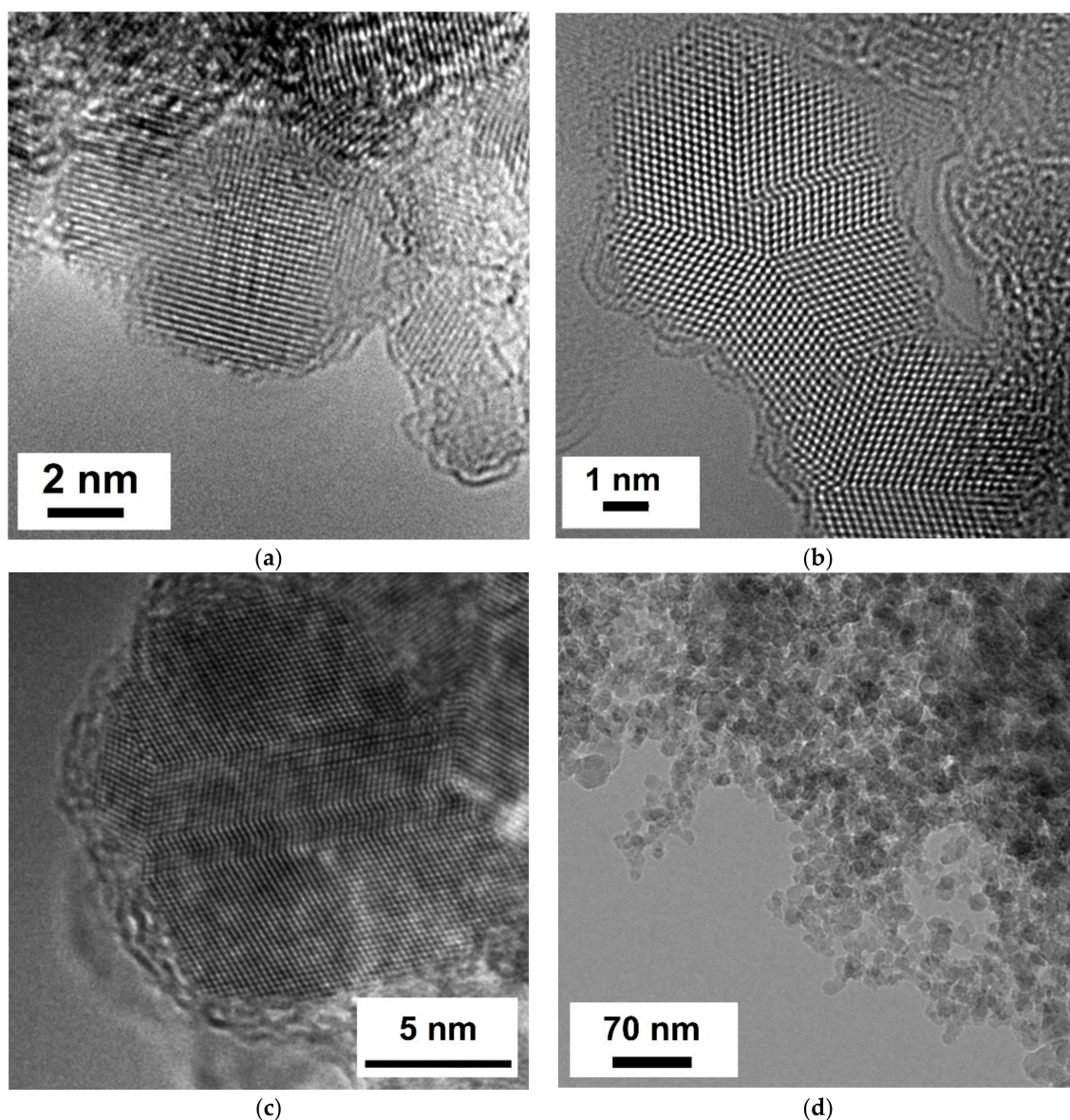


Figure 1. Nanodiamonds of detonation synthesis: (a) primary nanodiamond particle without crystalline defects; (b,c) nanodiamonds with twins; and (d) agglomeration of nanodiamonds.

3. Results and Discussion

Mechanical alloying can lead to the fragmentation of big nanodiamond agglomerates up to single nanoparticles inside the metal matrix after treatment for a long time. However, in a big volume fraction of nano reinforcements, this fragmentation is complicated: it can be executed only up to nanoagglomerates. Figure 2a demonstrates ND nanoagglomerates on a cross-section of “aluminium + 25%vol. nanodiamonds” composite granule (the cross-section was produced by ion beam). It is necessary to create additional stresses and micro deformations around these nanoagglomerates. The addition of a second metal in the matrix leads to the formation of new phases with different densities; this results in changes in material volume and the appearance of additional stresses and deformations. As a result, nanoagglomerates can be destroyed. For a demonstration of this scheme, at first, the composite “Al + 30%vol. ND” was prepared by mechanical alloying for two hours. After, the copper powder was added to the grinding jar, and a new mixture was also treated for two hours. Figure 2b shows an obtained non-agglomerated distribution of ND reinforcements inside a metal matrix. An XRD study determined the appearance of intermetallic phases in the matrix (Figure 3): CuAl_2 (density $D = 4.37 \text{ g/cm}^3$); Cu_4Al_3

($D = 6.86 \text{ g/cm}^3$); Cu_4Al_3 ($D = 5.75 \text{ g/cm}^3$); and Cu_3Al ($D = 7.33 \text{ g/cm}^3$) (Al: $D = 2.7 \text{ g/cm}^3$, Cu: $D = 8.93 \text{ g/cm}^3$).

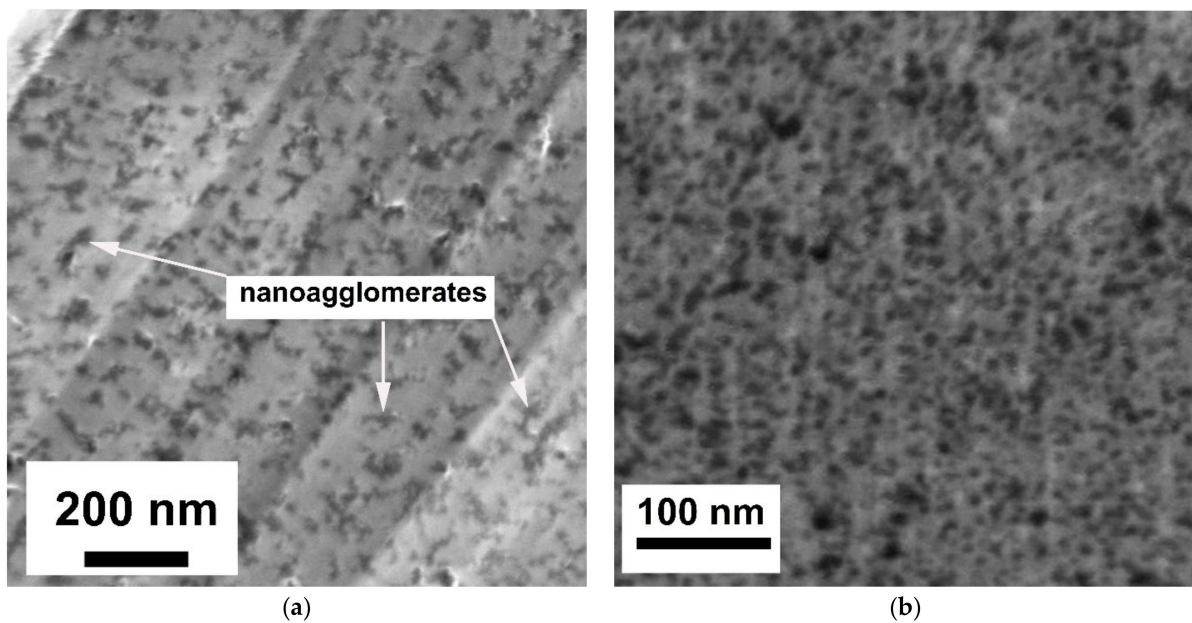


Figure 2. Nanodiamonds inside metal matrix: (a) nanoagglomerates; and (b) non-agglomerated distribution of nanodiamond-reinforcing particles in metal matrix from aluminium and copper.

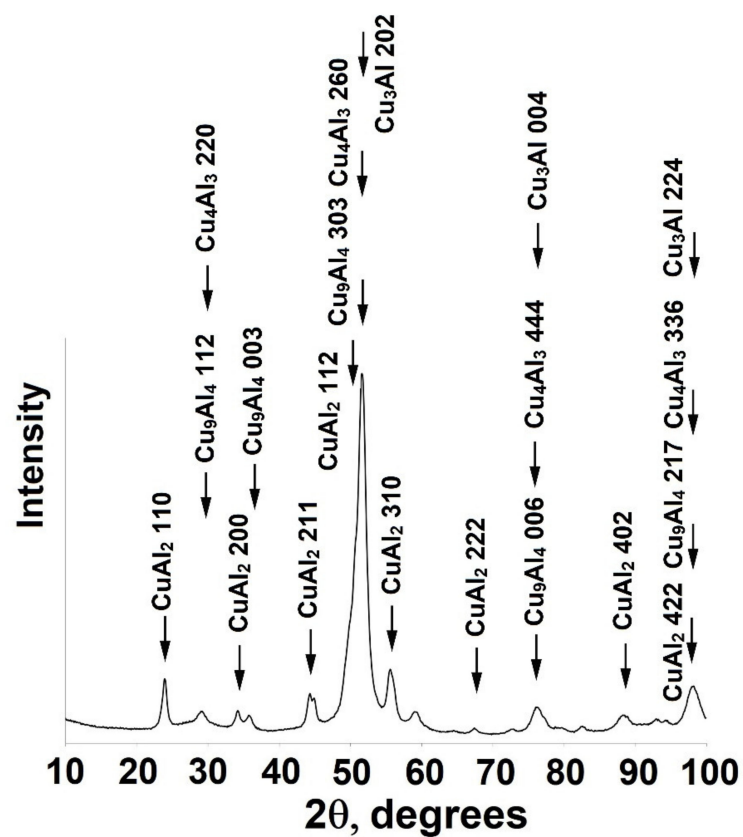


Figure 3. X-ray diffraction patterns from the composite with the matrix from Al and Cu with nanodiamond reinforcements; as shown by the arrows.

The non-agglomerated state of nanodiamond particles leads to a change of properties of the material. A vital change is the cardinally decreasing start temperature of the reaction between the aluminium matrix and diamond nano reinforcements with the formation of aluminium carbide Al_4C_3 : for macro-materials (bulk and big pieces), the start temperature is $1200\text{ }^\circ\text{C}$ [30], for micro-materials (powders) it is $750\text{ }^\circ\text{C}$ [31], but for nanodiamonds, this starting temperature is $450\text{ }^\circ\text{C}$ as it is demonstrated by a DSC study (Figure 4).

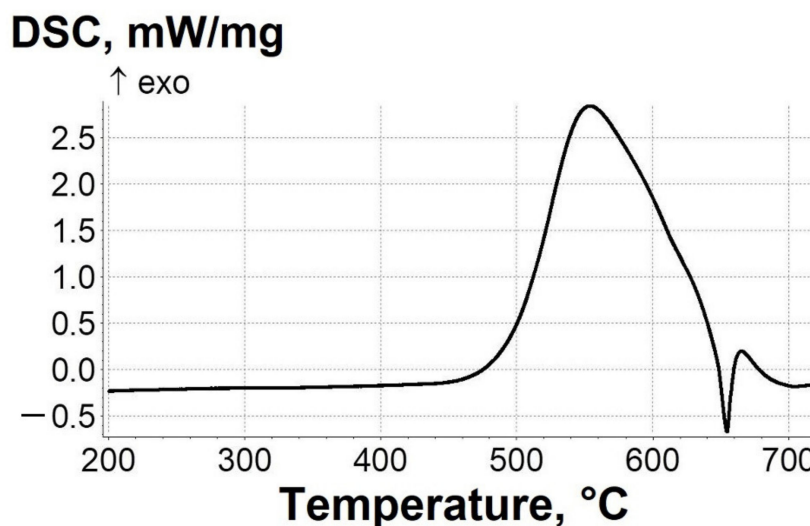


Figure 4. Results of DSC study of nanocomposite “Al + 25% ND”: exo-peak is the result of Al_4C_3 formation; endo-peak is the result of aluminium melting.

The investigation shows a possibility of obtaining the non-agglomerated distribution of nanodiamond reinforcements inside the metal matrix, i.e., the size of nano reinforcements equals approximately 4–6 nm. Furthermore, to determine the possibility of reducing the size of agglomerated nanodiamonds by annealing and cleaning from graphitic material, the process of ND transformation into carbon onions during annealing in a vacuum was studied.

It is necessary to say that there are two hypotheses (or two variants) about the process by which nanodiamonds transform into carbon onions during annealing. According to the first [18], all ND particles simultaneously undergo a transformation, layer by layer. This hypothesis states that each ND particle consists of a diamond core coated with a graphitic material while in an intermediate state. According to the second hypothesis [19], while in the agglomerated state, the nanoparticles in the surface layers of ND agglomerates undergo a complete and rapid transformation first, followed by the transformation of nanoparticles in their deeper layers. While in its intermediate state, each ND agglomerate has a core of diamond nanoparticles and completely transformed carbon onions on the agglomerate’s surface. The principal difference between these hypotheses is explained below. We suppose that the transformation of diamond nanopowders takes place according to the first scheme. In this case, it is possible to reduce the size of the nanoparticles by cleaning intermediately annealed nanodiamond powders of the graphitic substance. On the other hand, if the transformation of nanodiamond nanopowders takes place according to the second scheme, it is necessary to work with the nanopowders obtained via synthesis. Therefore, it is impossible to reduce their size by annealing and cleaning.

It should be noted that a detached ND particle does, in fact, transform layer by layer when exposed to local heating by an electron beam in the electron microscope. Figure 5a–c shows annealing stages for a particle heated with an electron beam in a transmission electron microscope.

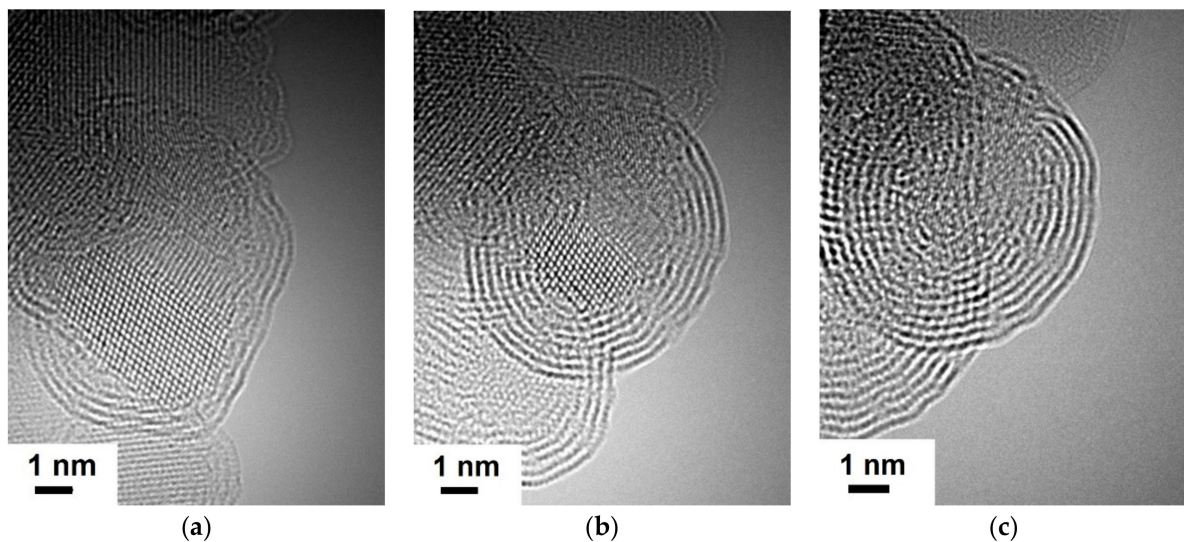


Figure 5. Transformation of a detached nanodiamond particle exposed to local heating by electron beam; images were produced with an interval of 60 s (TEM JEOL JEM 2100 F/Cs, accelerating voltage 200 kV): (a) big diamond core; (b) small diamond core; (c) complete transformation.

However, as investigations show, when massive samples of ND agglomerates are exposed to heat treatment (heating of significant quantities, not local), the transformation process is realized according to the second hypothesis: ND particles from the surface layer of agglomerates transform earlier than nanoparticles located in the central part of the agglomerate. Agglomerated ND powders annealed in vacuum furnaces at 1180 to 1300 °C were studied using the transmission electron microscopy and electron energy loss spectroscopy (EELS). Figure 6a shows the fracture point of the partially transformed ND agglomerate. Parts with particles that have 100% diamond structure and parts with completely transformed particles are clearly visible. This fact attests to the second hypothesis. A separate investigation of the nanoparticles on the surface of partially annealed agglomerates showed that they had undergone complete transformation (Figure 6b).

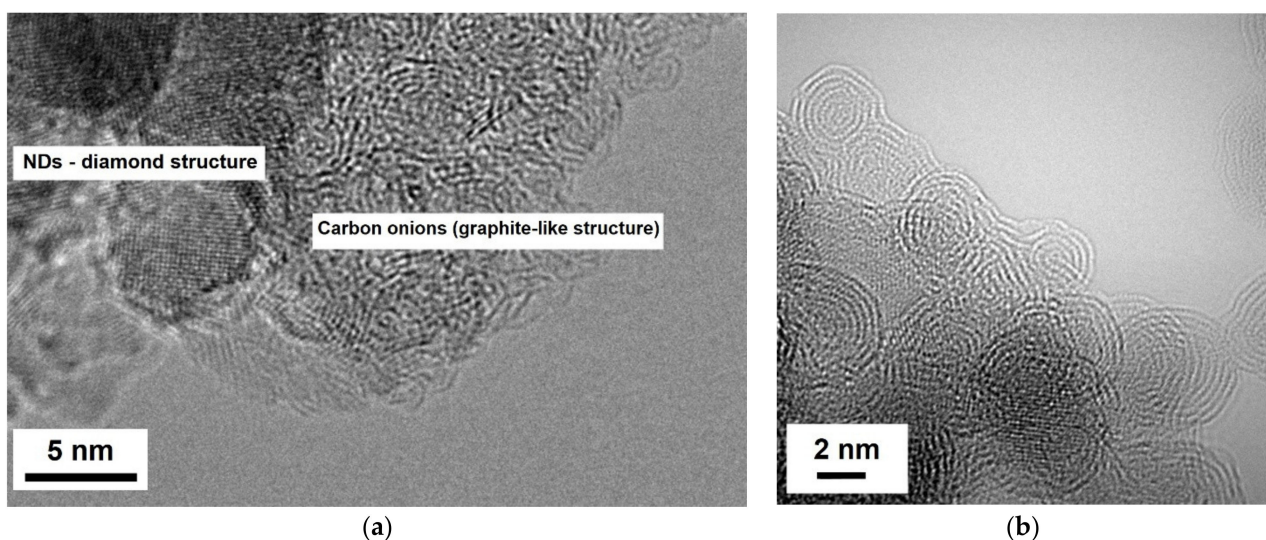


Figure 6. TEM images of nanodiamonds after annealing at 1200 °C: (a) fracture of ND agglomerate after partial transformation; and (b) complete transformation of ND in surface layers of agglomerate.

Figure 7 shows the EEL spectra. The size of the electron beam is within a range of 2–5 nm, and the size of the ND agglomerates is about 50 µm. The experimental method is

shown in Figure 7a. Zone 1 mainly includes carbon onions. The EEL spectrum for zone 1 is shown in Figure 7b: the sp^2 peak (π^* peak) is remarkably high. Zone 2 mainly consists of particles from the central layers of the ND agglomerate and a small number of particles from the surface layers. The EEL spectrum for this zone is shown in Figure 7c: the sp^2 peak (π^* peak) has a much lower intensity than zone 1 (enhanced thickness leads to the noisy image). This result suggests that the ND agglomerate has a core of nanodiamond particles. This core is coated with a layer of graphitic particles (carbon onions), i.e., the second hypothesis is valid for transforming agglomerated ND powders.

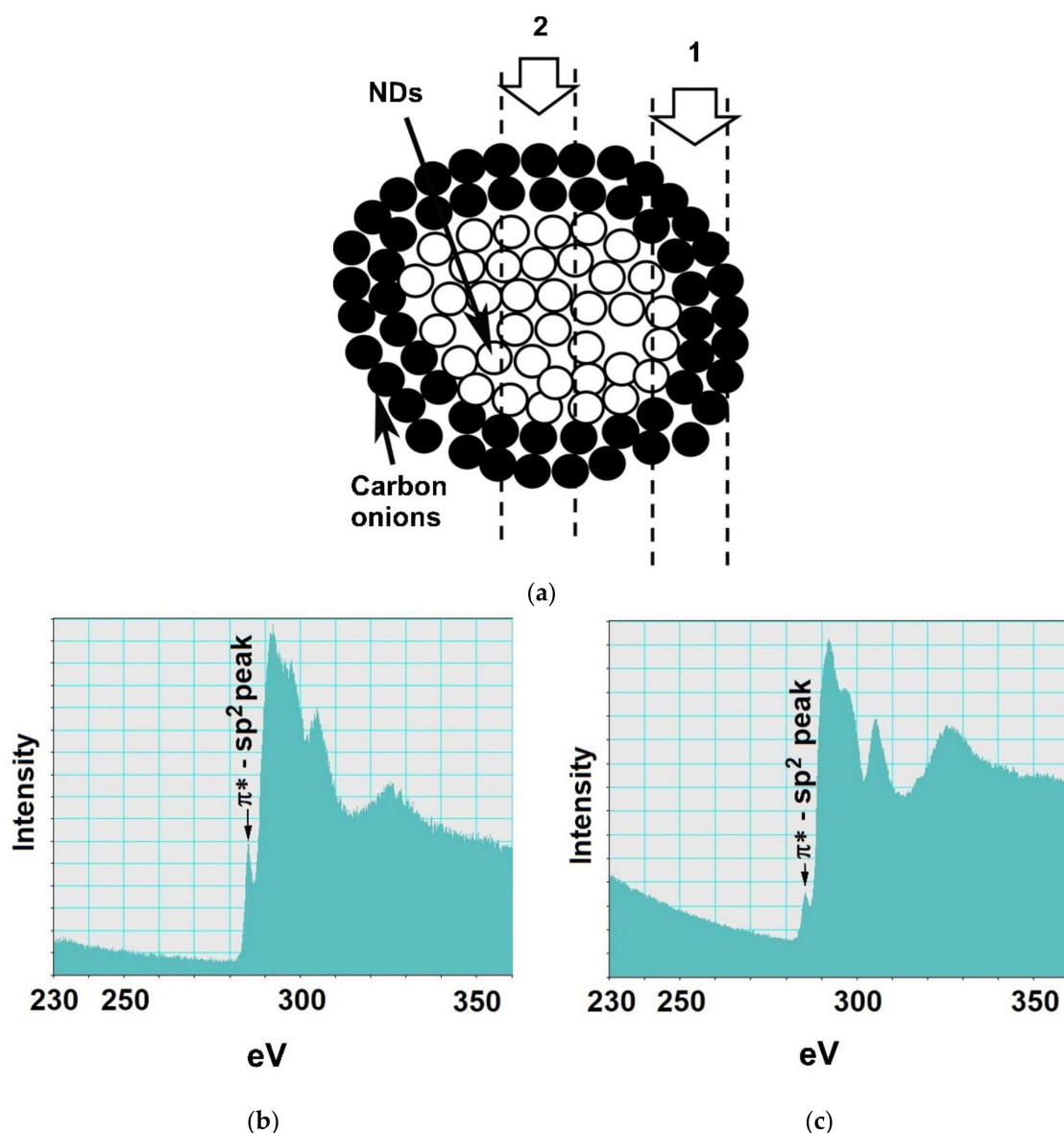


Figure 7. EEL spectra for the surface and central layers of the ND agglomerate: (a) method of data collection; (b) EEL spectrum from zone 1 (particles from surface layers); and (c) EEL spectrum from zone 2 (mainly particles from central layers).

It is probable that several reasons led to this result. In our opinion, the main reason is the energetic state of the nanoparticles involved. It is necessary to note that the energetic state of ND particles located in the surface layer and located in the deeper layers of the agglomerate differs due to several reasons, mainly the difference in the surface energy of the nanoparticles. Nanoparticles on the agglomerate surface have a significant portion of their surface open (free). The free surface of the ND nanoparticles located in deeper layers is much less; i.e., the surface energy is different. This fact leads to the difference

in the energetic state of nanoparticles from the surface and deep layers of agglomerate. In our opinion, exactly this difference in the energetic state leads to the fact that ND particles from the surface layer of the agglomerate transform into carbon onions earlier than the nanoparticles located in the deeper layers of the agglomerate. In the case of removing a graphitic component, the material will consist of initial nanodiamonds with an initial size. This means that the ordinary annealing and purification of agglomerated nanodiamonds cannot reduce particle size: it is necessary to work with the particle size obtained during synthesis.

4. Conclusions

The investigations demonstrated the possibility of obtaining the non-agglomerated distribution of nanodiamond-reinforcing particles inside the metal matrix. The fabrication method is based on mechanical alloying with the creation of additional stresses and deformations.

The starting temperature of aluminium carbide formation in aluminium matrix composites with non-agglomerated nanodiamond-reinforcing particles reduces to 450 °C. However, the composite is workable before this temperature.

The research shows that nanodiamond particles in an agglomerated state transform into onion-like carbon nanoparticles as follows: the nanoparticles located in the surface layers of ND agglomerate transform first (complete transformation, in fact) followed by the transformation of particles in deeper layers. In the intermediate annealing state, the ND agglomerate consists of a diamond core (a core of nanodiamond particles) coated with a layer of carbon onions. This means that the ordinary annealing of agglomerated nanodiamonds cannot reduce their particle size by annealing and purification from the graphitic component. Instead, it is necessary to use the particle size obtained during synthesis.

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