Hindawi Publishing Corporation
Journal of Nanomaterials
Volume 2014, Article ID 271494, 8 pages
http://dx.doi.org/10.1155/2014/271494

Research Article

Nanopolycrystalline Diamond Sintered from Onion-Like Carbon

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Received 17 December 2013; Revised 15 March 2014; Accepted 16 March 2014; Published 24 April 2014

Academic Editor: Jinlong Jiang

The onion-like carbons (OLCs) annealed at 900–1400°C were used as raw materials to synthesize additive-free D-D type nanopolycrystalline diamond (nPCD) compacts in the industrial sintering conditions of 5.5 GPa, 1200°C, and 15 min. The results showed that the OLCs were transformed into additive-free D-D type nPCD compacts in industrial sintering conditions. The nPCD compacts contained a large number of nanotwins. The purities and performances of nPCD compacts were homogeneous in three dimensions. The purity and physical and mechanical performances of the nPCD compact (denoted as $nPCD_{11}$) sintered from the OLCs annealed at 1100°C were the highest. The average Vickers hardness, density, and nanodiamond grain size of $nPCD_{11}$ were 32 GPa, 2.7 g/cm^3, and 12 nm. During sintering process, the graphite layers of OLCs ruptured from inside toward outside forming larger nanodiamond particles. At the same time, the OLCs bonded adjacent to OLCs forming additive-free D-D type nPCD compacts.

1. Introduction

Presently, polycrystalline diamond (PCD) compacts are mainly fabricated using microdiamond as the starting material. Furthermore, catalysts are must be added in the starting material [1]. For example, the forming carbide element catalysts of Si and Ti [2] or catalytic metals of Co and Ni [3, 4] are generally used. In this case, the sintering pressures and temperatures of the PCD compacts usually reach 5.0–6.5 GPa and 1400–1600°C. Recently, some researchers [5, 6] also have successfully fabricated PCD compacts using nonmetal catalysts, such as $FeTiO_3$, Fe_2SiO_4, $Y_3Fe_5O_{12}$, and $CaCO_3$. In this case, the sintering pressure and temperatures of the PCD compacts commonly reach 7.5–8.5 GPa and 1800–2300°C. However, catalysts are easy to remain in PCD compact. And Si and Ti catalysts are easy to form carbide of SiC and TiC [7–10]. The physical and mechanical performances of these catalysts and carbide are not as good as those of diamond, resulting in the existence of weak phase in PCD compacts, which further influences their performances and processing abilities [11, 12].

Synthesis of additive-free diamond-diamond (D-D) type PCD compact is a fundamental way to solve the above problem. Some researchers [13] have tried to fabricate additive-free PCDcompacts using graphite, microdiamond, or nanodiamond as the starting materials. Even though additive-free PCDcompacts have been successfully fabricated, the sintering pressure and temperatures usually reach 12–25 GPa and 1800–2500°C, which are very difficult to be achieved for the current industrial sintering apparatus. Scientists in this field have been working on finding new raw materials for sintering additive-free PCD compact in industrial conditions. The discovery of onion-like carbon (OLC) makes this research possible. OLC is one member of the fullerene family. Its exterior shape is polyhedral and its interior structure is similar to that of onion. Its interlayer distance is usually 0.34 nm, which is close to that of graphite. The innermost diameter of OLC is about 0.7 nm, which is close to that of C_{60} [14]. OLC composes of both sp^3 diamond structure and sp^2 graphite structure, which can be transformed into each other under certain conditions.

In [15], Tomita et al. obtained diamond film by annealing OLC at the temperature of 500°C in the air. The OLC was fabricated by annealing detonation nanodiamond (average grain size was about 5 nm) at 1700°C. This research proves that diamond can be synthesized using OLC as starting material without any catalysts in conditions of very low temperature of 500°C and atmospheric pressure. However, in Tomita's work, only diamond film is prepared because there is no three-dimensional pressure applied during the transformation process of diamond from OLC, that is, there is no pressure for diamond growth in three dimensions. If a three-dimensional pressure is applied during the transformation process, OLC can be transformed into diamond and form diamond compact.

In this work, additive-free D-D type nanopolycrystalline diamond (nPCD) compacts were sintered from onion-like carbons (OLCs) annealed at 900–1400°C and 1 Pa in industrial conditions of 5.5 GPa, 1200°C, and 15 min. The microstructures, purities, and performances of the nPCD compacts were characterized. Based on the results, the formation mechanisms of the additive-free D-D type nPCD compacts sintered from OLCs in industrial conditions were discussed.

2. Experimental Details

The nanodiamond (average grain size was approximated to be 5 nm) used in this work was fabricated by means of detonation [16–18]. OLCs were prepared in volume by annealing the detonation nanodiamond at the temperatures and pressure of 900–1400°C and 1 Pa, which had been reported detailedly in our previous papers [19–23].

The sintering experiments of additive-free D-D type nPCD compacts from OLCs were carried out in a CS-1B type hexahedron anvils press. The nominal pressure of the press was 8000 kN (single-cylinder). Its slave cylinder diameter was 320 mm. Its working pressure reached high pressure of 10 GPa and ultra-high pressure of 100 GPa. Its largest piston path of slave cylinder was 60 mm. Its supercharging ratio was 10.8 : 1. The electric heating power was 30–40 kVA. The main motor power was 11 kN. Its external dimension (width ∗ height ∗ length) was 2150 ∗ 2150 ∗ 2430 mm. Its total weight was 15.5 t.

The main sintering procedures of additive-free nPCD compacts from OLCs were as follows. Firstly, OLCs were put into a graphite canister and prepressed with a 02000048 type press until the OLCs were densely and fully filled in the graphite canister. The external and internal heights of the graphite canister were 7.0 mm and 5.5 mm. The external and internal diameters of the graphite canister were 12.0 mm and 9.0 mm. The jamb wall and bottom thicknesses of the graphite canister were 1.5 mm, respectively. The inner cap diameter and thickness of the graphite canister were 9.0 mm and 1.5 mm. Secondly, the graphite canisters fully filled with OLCs were baked at 120°C for 10 h in a ZT-25-20 type vacuum carbon tube furnace. Thirdly, the sintering samples were assembled as shown in Figure 1. The conductive steel cap, pyrophyllite, and graphite sheet were used. Finally, the samples were put into the hexahedron anvils press for sintering. The sintering pressure, temperature, and time were 5.5 GPa, 1200°C, and 15 min.

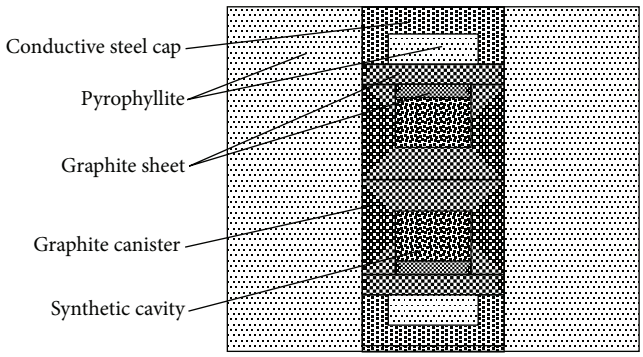


Figure 1: Assembling diagram of sintering sample.

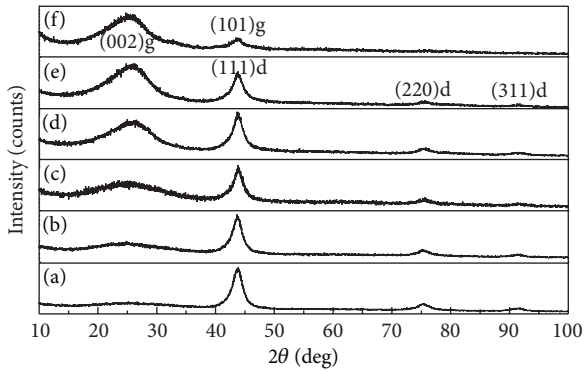


Figure 2: XRD patterns of OLCs annealed at (a) 900°C; (b) 1000°C; (c) 1100°C; (d) 1200°C; (e) 1300°C; (f) 1400°C.

The end surfaces of nPCD compacts were polished with diamond powder (grain size was about 0.5 μm). In order to confirm its purity, microstructure, and density homogenization in three dimensions, various parts of nPCD compacts were cut into slices for characterizing, respectively. The phase analysis of nPCD compacts was carried out using a D-MAX-2500/P type X-ray diffractometer (XRD, Rigaku, Cu Kα, Japan). The microstructures of nPCD compacts were observed using a JEM-2010 type high-resolution transmission electron microscope (HRTEM, Japan). The densities of nPCD compacts were measured using Archimedes principle. A FM-700 type microscopic Vickers hardness tester (Future-Tech, Japan) was used to measure the microharnesses of nPCD compacts. A pyramid diamond indenter was applied. The surface angle and the relative edge angle of the diamond indenter were 136°(± 30′) and 148.7°(± 23′). The load and the loading time were 4.9 N and 10 s. In order to confirm its physical and mechanical homogenization, the microharnesses of nPCD compacts were measured 10 times in different areas.

3. Results and Discussion

Figure 2 shows the XRD patterns of the OLCs annealed at 900°C, 1000°C, 1100°C, 1200°C, 1300°C, and 1400°C. As shown in Figures 2(a)–2(e), the patterns consisted of four

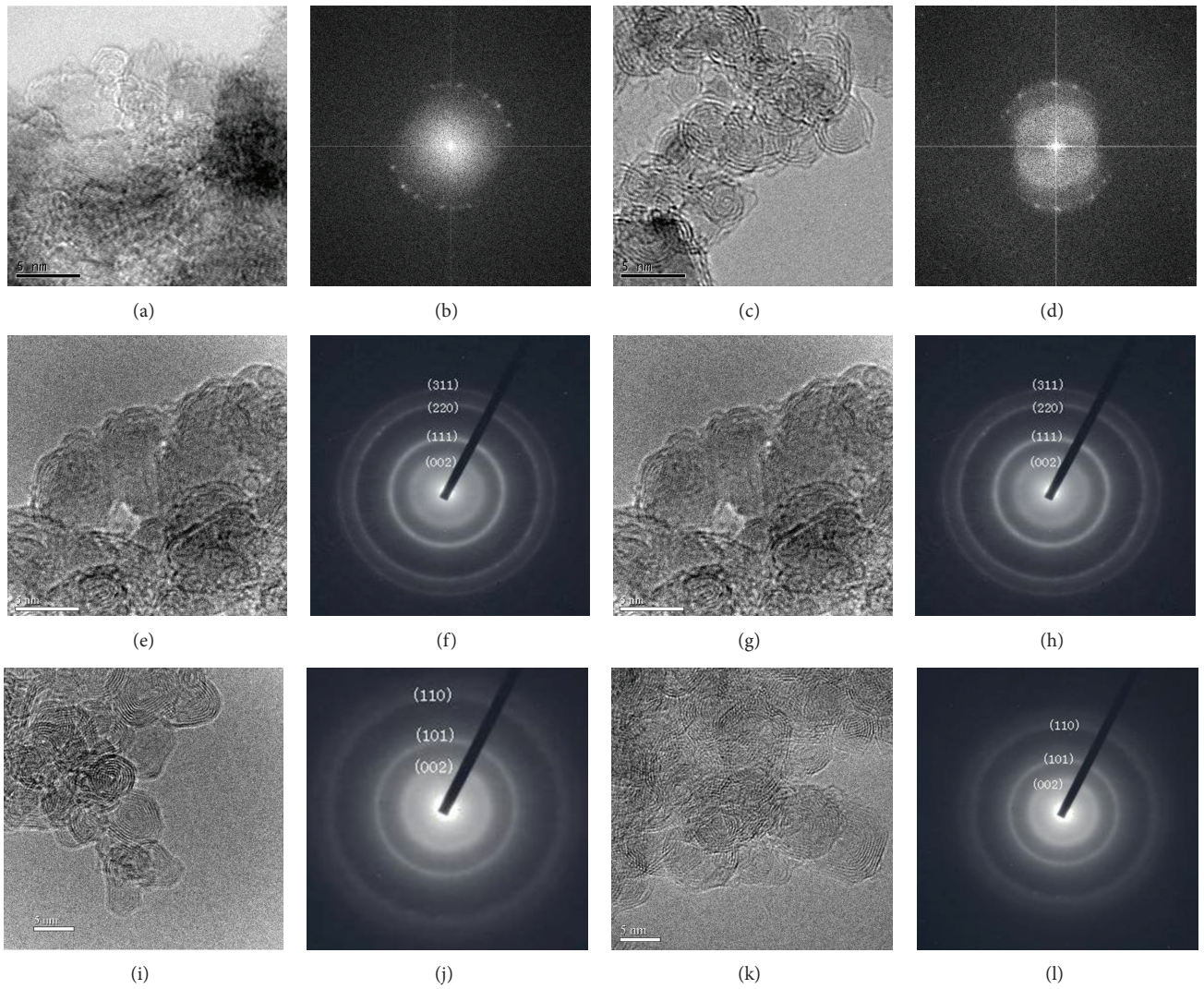


Figure 3: (a), (c), (e), (g), (i), and (k) are HRTEM images and (b), (d), (f), (h), (j), and (l) are SAD of OLCs annealed at 900°C, 1000°C, 1100°C, 1200°C, 1300°C, and 1400°C.

seriously broadened diffraction peaks locating at 44°, 75°, and 91°. The peak locating at 26° corresponded to the diffraction of graphite (002) crystal plane. The seriously broadened diffraction peak of graphite (002) crystal plane came from OLC [24]. The peaks locating at 44°, 75°, and 91° corresponded to the diffractions of diamond (111), (220), and (311) crystal planes. The relative diffraction intensity of graphite (002) crystal plane strengthened gradually with annealing temperature increase, while the relative diffraction intensity of each diamond crystal plane decreased. This illuminated that nanodiamond was transformed gradually into OLC with annealing temperature increase. At the annealing temperature of 1400°C as shown in Figure 2(f), the diffraction peaks locating at 26° and 44° and corresponding to the graphite (002) and (101) crystal planes appeared. The diffraction peaks locating at 75° and 91° and corresponding to the diamond (220) and (311) crystal planes disappeared. These indicated that nanodiamond had been transformed completely into OLC annealed at 1400°C. The XRD patterns in Figure 2 showed strong background, which demonstrated that OLCs contained a certain amount of amorphous carbon. The broadness of diffraction peak might be derived from the stress and strain, nanometer small size, interior lattice distortion, and high density defect of grain.

Table 1 shows the grain sizes of the OLCs annealed at 900–1400°C, which were calculated according to the XRD patterns shown in Figure 2 and Scherrer formula. From the data we could see that the grain sizes of OLCs annealed at 900°C, 1000°C, 1100°C, 1200°C, 1300°C, and 1400°C were 3.4 nm, 3.8 nm, 4.0 nm, 4.2 nm, 4.6 nm, and 6.4 nm. The OLC grain sizes increased gradually with annealing temperature increase.

Figure 3 shows the HRTEM and select-area diffraction (SAD) images of the OLCs annealed at 900°C, 1000°C, 1100°C, 1200°C, 1300°C, and 1400°C. As shown in Figures 3(a) and 3(b), the nanodiamond particle edges had been converted into lamellar graphite when annealed at 900°C. Moreover, the quantity of OLC was very few because the

Table 1: Grain sizes of OLCs annealed at 900–1400°C.

Annealing temperature (°C)	900	1000	1100	1200	1300	1400
Grain size (nm)	3.4	3.8	4.0	4.2	4.6	6.4

temperature of 900°C was too low. As seen from Figures 3(c) and 3(d), the nanodiamond particle edges had been transformed into layer-structural OLC. There was nanodiamond untransformed and coexisted in the center of OLC. The content of OLC increased. From the images shown in Figures 3(e)–3(j), most of OLCs were ellipsoid and a small portion was spherical. The outer of OLC was curved graphite layer. Its core had lattice fringe structure. The interlayer distance of the OLC layers was about 0.344 nm, which was close to that of graphite. The lattice fringe distance of the OLC interior lattice fringe structure was approximate to be 0.21 nm, which corresponded to the diamond (111) crystal planes. These demonstrated that the OLCs annealed at 1100–1300°C were encapsulated untransformed nanodiamond in the core. The OLC axes were parallel to those of diamond (111) crystal planes, showing that the OLC graphitization was initiated from the diamond (111) crystal planes. As shown in the SAD graphs of the OLCs, there was one inner amorphous ring. The outer had three diamond rings. Through calculation, the interlayer distances were about 0.3341 nm, 0.2064 nm, 0.1261 nm, and 0.1074 nm, which corresponded to the graphite (002) and diamond (111), (220), and (311) crystal planes. Demonstrating that, nanodiamond had not been transformed completely into OLC annealed at 1100–1300°C, which was in conformity with the results of XRD patterns and HRTEM image shown in Figures 2(a)–2(e). As seen from the images shown in Figures 3(k) and 3(l), there were no diamond lattice fringe structures existing in the OLC center. Indicating that, nanodiamond had been transformed completely into OLC annealed at 1400°C. The interlayer distance was about 0.345 nm, which was close to that of graphite. The innermost diameter of the OLCs was about 0.69 nm, which was close to that of C_{60}.

The graphite layer number, the maximum, the minimum, and the average particle sizes of the OLCs annealed at 900–1400°C are shown in Table 2. From the data we can see that the OLC particle sizes and the graphite layer number increased gradually with annealing temperature increase. The average particle sizes of OLCs annealed at 900°C, 1000°C, 1100°C, 1200°C, 1300°C, and 1400°C were 5.7 nm, 5.9 nm, 6.1 nm, 6.5 nm, 7.5 nm, and 8 nm, whose average graphite layer numbers were 3, 4, 6, 8, and 12.

Figure 4 shows the XRD patterns of the nPCD compacts sintered for 15 min at 5.5 GPa and 1200°C using the OLCs annealed at 900°C, 1000°C, 1100°C, 1200°C, 1300°C, and 1400°C as the starting materials. There were four seriously broadened diffraction peaks locating at 26°, 44°, 75°, and 91° and corresponding to the graphite (002), diamond (111), (220), and (311) crystal planes. This indicated that the nPCD compacts contained diamond and graphite. The existence of

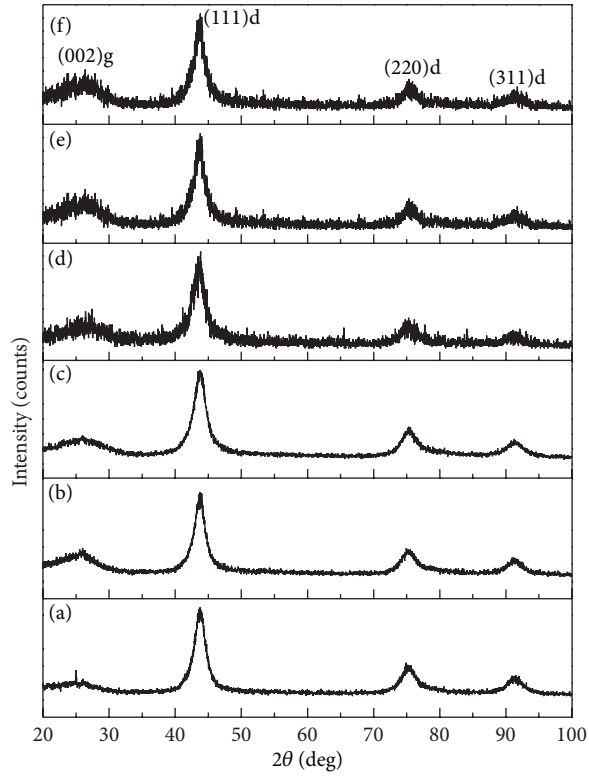


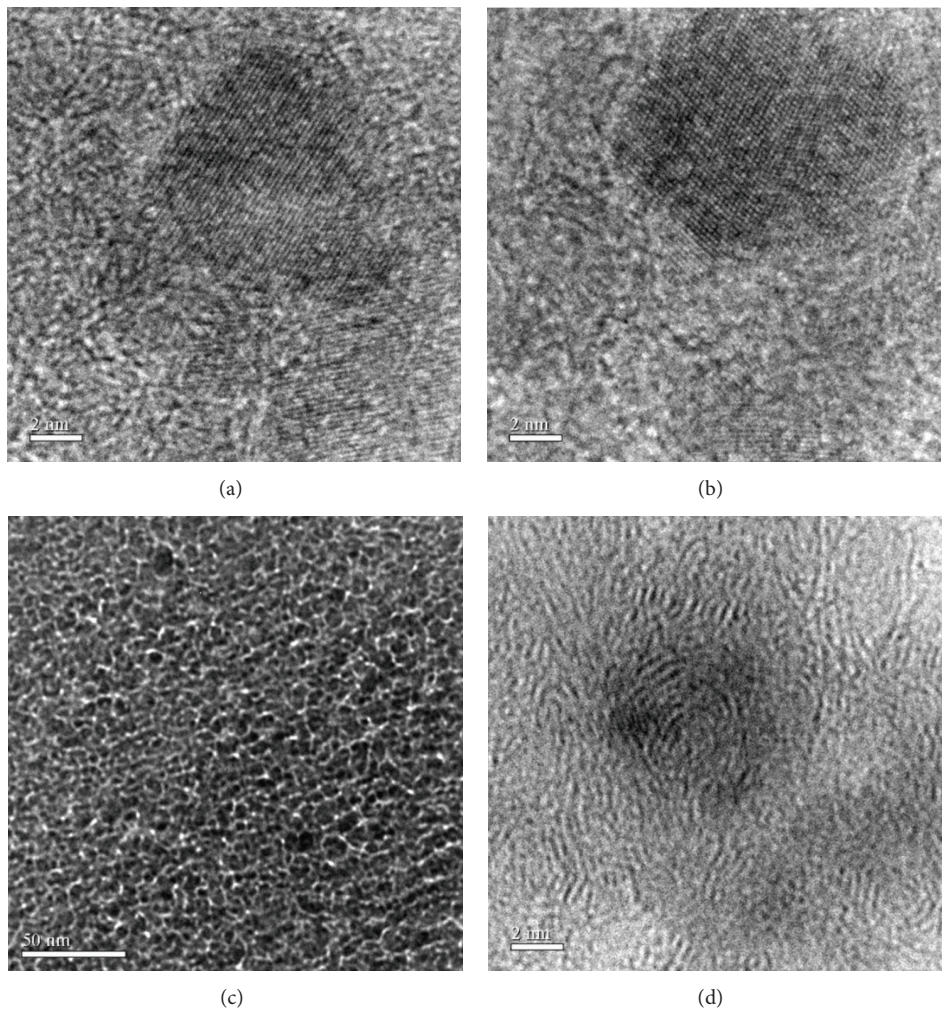
Figure 4: XRD patterns of nPCD compacts sintered for 15 min at 5.5 GPa and 1200°C from OLCs annealed at (a) 900°C; (b) 1000°C; (c) 1100°C; (d) 1200°C; (e) 1300°C; (f) 1400°C.

Table 2: Parameters of OLCs annealed at 1100–1400°C.

Annealing temperature (°C)	Average graphite layer number	Maximum particle size (nm)	Minimum particle size (nm)	Average particle size (nm)
900	Lamellar structure	8.0	5.0	5.7
1000	3	8.3	5.1	5.9
1100	4	8.5	5.3	6.1
1200	6	9	5.5	6.5
1300	8	9.5	5.7	7.5
1400	12	9.8	5.2	8

background showed that nPCD compacts contained a certain amount of amorphous carbon. Compared with each pattern, the nPCD compact sintered from the OLC annealed at 1100°C contained the least graphite and amorphous carbon in the same sintering conditions, showing that the purity of the nPCD compacts sintered from the OLC annealed at 1100°C was the highest.

Figure 5 shows the HRTEM images of the nPCD compact sintered for 15 min at 5.5 GPa and 1200°C using the OLC annealed at 1100°C as the starting material. In Figures 5(a) and 5(b), the lattice fringe structure could be seen clearly. The lattice distance was about 0.2065 nm through Fourier transform, which was very close to 0.2060 nm of diamond (111)



(a) (b)

(c) (d)

Figure 5: HRTEM images of the nPCD compact sintered for 15 min at 5.5 GPa and 1200°C from the OLC annealed at 1100°C: (a), (b), (d) high-resolution, and (c) low-resolution.

crystal plane. Thus this was the diamond (111) crystal plane. Therefore, the particles with lattice fringe structure were diamond. Interestingly, there were numerous nanotwins existing in the nPCD compact. In Figure 5(c), well-defined diamond particles could be observed. The majority of them were irregular. A bit of them were spherical or elliptic. The maximum and the minimum particle sizes of diamond particle in nPCD compact were 14 and 10 nm. The average particle size of diamond was about 12 nm through statistical analysis. As seen in Figure 5(d), there were completely transformed OLCs occurring in the nPCD compact, which composed of 10–12 graphite layers. Descripting that, a small portion OLCs had completely transformed into OLC during the sintering process.

Figure 6 is the SAD of the nPCD compact sintered for 15 min at 5.5 GPa and 1200°C using the OLC annealed at 1100°C as the starting material. There were three polycrystalline diffraction rings and one amorphous ring. Through calculating, the interplanar distances were 0.3445 nm, 0.2064 nm, 0.1261 nm, and 0.1074 nm, corresponding to the graphite (002) and diamond (111), (220), and (311) crystal planes. This showed that the nPCD compact contained diamond, a certain amount of graphite, and amorphous carbon, which conformed to the analysis results of XRD and HRTEM shown in Figures 4 and 5.

Figure 7 shows the fitting lines of Vickers hardness, density, and average diamond grain size of the nPCD compacts sintered for 15 min at 5.5 GPa and 1200°C using the OLCs annealed at 900°C, 1000°C, 1100°C, 1200°C, 1300°C, and 1400°C as the starting material. The Vickers hardness, density, and average diamond grain size (calculated according to the XRD patterns shown in Figure 4 and Scherrer formula) of the nPCD compacts sintered from the OLC annealed at 1100°C were the highest and largest, which were 32 GPa, 2.70 g/cm^3, and 10.7 nm.

Through the above results and analysis, encapsulated nanodiamond (annealed at 900–1300°C) or empty (annealed at 1400°C) OLCs are transformed into additive-free D-D

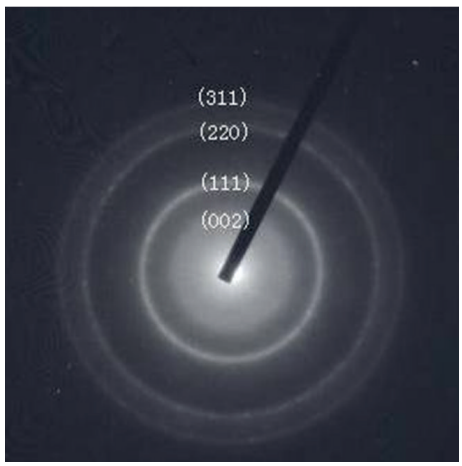


Figure 6: SAD of nPCD compact sintered for 15 min at 5.5 GPa and 1200°C using OLC annealed at 1100°C.

type nPCD compacts in the industrial sintering conditions of 5.5 GPa, 1200°C, and 15 min. The OLC particles are in nanometer size and there are sp^3 structures in it. The sp^3 sites can be the crystal seeds for diamond formation. Thus the transformation of diamond from OLC is easier than that of graphite. So the sintering pressure and temperatures of the nPCD compacts from OLCs are much lower than those of graphite and other raw materials. This work makes it possible for synthesizing additive-free D-D type nPCD compacts in industry conditions. Furthermore because there are no weak phases existing in the nPCD compacts, the performances and processing abilities of them can be improved greatly.

During sintering process, great energies and pressure are generated. The external graphite layers of OLC particles rupture at first under the effects of the pressure and energy, then the graphite layers of OLC rupture from inside towards its outside. In this way, the graphite layers of OLC break gradually, so that the sp^2 graphite structure in OLC is transformed into sp^3 diamond structure forming nanodiamond particles. Furthermore, the nanodiamond particles grow during the transformation process from OLC forming larger nanodiamond particles and bond adjacent nanodiamond particles through dangling bonds forming additive-free D-D type nPCD compacts.

The purity, diamond grain size, Vickers hardness, and density of the additive-free nPCD compacts sintered from the OLCs annealed at 1100°C are the largest and the highest, as shown in Figure 7. The average Vickers hardness and density of the nPCD compacts sintered from the OLCs annealed at 1100°C reach 32 GPa and 2.7 g/cm^3. The average grain size of nanodiamond in the additive-free nPCD compact sintered from the OLC annealed at 1100°C grows up to 10.8 nm, which increases by 170% compared with 4.0 nm (as shown in Figure 2 and Table 1) of the OLC average grain size annealed at 1100°C. There is untransformed nanodiamond encapsulated in the OLC core annealed at 1100°C, which can be used as crystal seed for nanodiamond transformation from OLC. At the same time, the outer graphite layer structure of the OLC annealed at 1100°C is the most suitable for transforming into nanodiamond. Although the OLCs annealed at 900°C, 1000°C, 1200°C, and 1300°C encapsulate untransformed nanodiamonds in the cores, the outer graphite layers of the OLCs have lamellar or unstable structures, which is not suitable for nanodiamond transformation from the OLCs. The OLCs annealed at 1400°C is transformed completely from nanodiamond, there is no untransformed nanodiamond in the core; in other words, there is no crystal seed for nanodiamond transformation from OLC during the sintering process of additive-free D-D type nPCD compacts. Thus the nanodiamond transformation from the OLC annealed at 1100°C forming additive-free D-D type nPCD compacts is the most complete transformation and the average grain size of nanodiamond is the largest. The purity of the nPCD compact sintered from the OLC annealed at 1100°C is the highest, which results in the highest physical and mechanical performances. Meanwhile, the nanotwins existing in the nPCD compacts also contribute to its high performances.

The above results are measured at different points of nPCD compacts. It is found that the purity and physical and mechanical performances of nPCD compacts are homogeneous in three dimensions. The reasons are as follows. Through three-pair hexahedron anvils of CS-1B type hexahedron anvils press, a three-dimensional isostatic pressure can be applied and controlled during the transformation process of diamond from OLCs. For sintering, the conductive steel cap, pyrophyllite, and graphite sheet (as shown in Figure 1) are used, which can make sure that the samples received three-dimensional isostatic pressure and homogeneous current during the nanodiamond transformation process from OLCs. Moreover, the sintering time is adapted 15 min. In such a short time, the pressure and current can be rapidly and evenly passed to every point of the samples, which further ensures that the samples received three-dimensional isostatic pressure and homogeneous current during the sintering process. Thus the purity and physical and mechanical performances of nPCD compacts are homogeneous in three dimensional directions.

4. Conclusions

OLCs were fabricated by annealing detonation nanodiamond at the temperatures of 900–1400°C and the pressure of 1 Pa. The OLCs annealed at 900–1300°C encapsulated untransformed nanodiamond in the core. The OLCs annealed at 1400°C were transformed completely. The grain sizes of OLCs annealed at 900°C, 1000°C, 1100°C, 1200°C, 1300°C, and 1400°C were 3.4 nm, 3.8 nm, 4.0 nm, 4.2 nm, 4.6 nm, and 6.4 nm. The OLC grain size increased gradually with the annealing temperature increase. The average number of OLC graphite layers annealed at 900°C, 1000°C, 1100°C, 1200°C, 1300°C, and 1400°C were 3, 4, 6, 8, and 12.

In the industrial sintering conditions of 5.5 GPa, 1200°C, and 15 min, OLCs annealed at 900–1400°C could be transformed into additive-free D-D type nPCD compacts. The purity and physical and mechanical performances of the nPCD compacts were homogeneous. The purity and physical

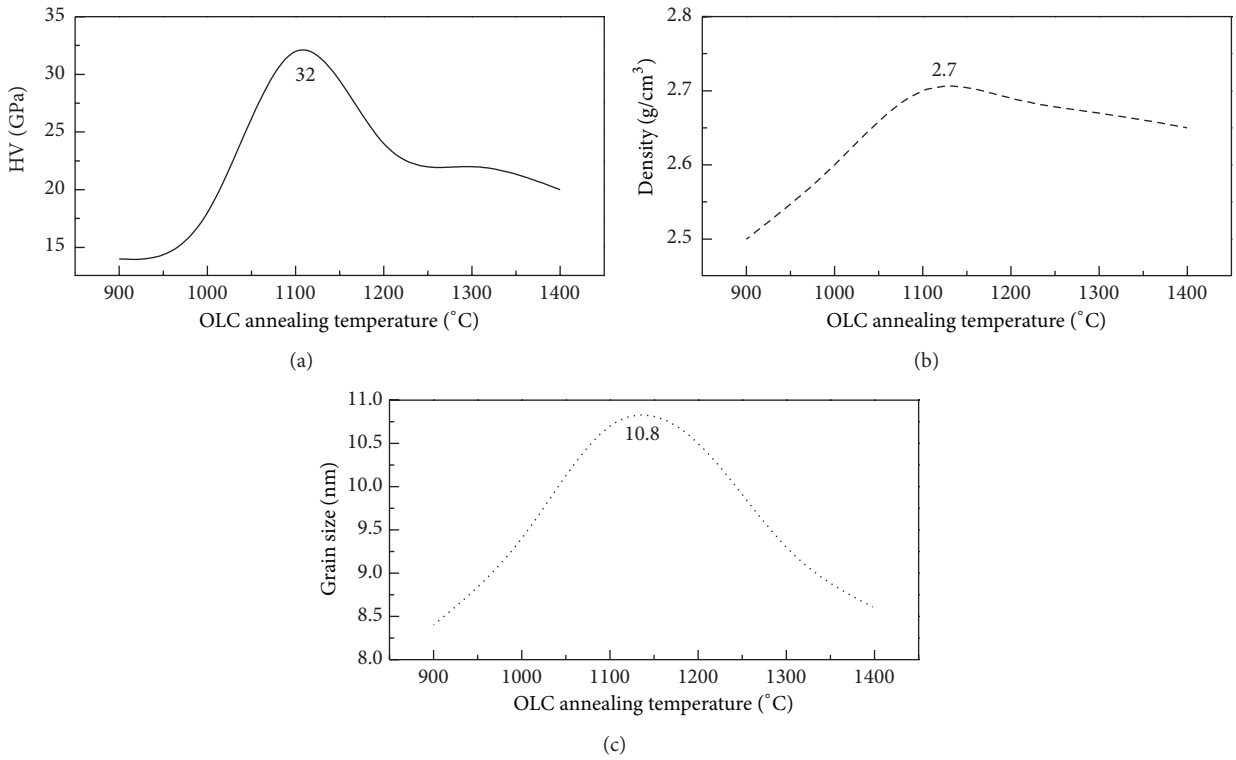


Figure 7: Properties of nPCD sintered from OLCs annealed at 900–1400°C: (a) Vickers hardness; (b) density; (c) grain size.

and mechanical performances of the nPCD compacts sintered from the OLCs annealed at 1100°C were the highest. Moreover, the Vickers hardness, density, and nanodiamond grain size were 32 GPa, 2.7 g/cm^3, and 10.8 nm.

In the sintering process, the OLC graphite layers ruptured from inside towards outside. At the same time, the OLC particles bonded with adjacent OLCs forming larger nanodiamond particles, which further formed additive-free D-D type nPCD compacts. The results of this research show that it is feasible to sinter additive-free D-D type nPCD compacts from OLCs in industrial sintering conditions.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

This work was supported by the National Nature Science Foundation of China (Grant no. 51102205), the Youth Foundation of Hebei Educational Committee (Grant no. QN20131092), and the first batch of young talent support plan in Hebei province.

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