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Shock waves preparing cubic boron nitride nanoparticles

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A B S T R A C T

Cubic boron nitride (c-BN) nanoparticles, whose hardness at sizes below 10 nm is expected to reach a hardness as large as diamonds, have widely potential applications in many areas. However, the related preparation in large amount is very challenging. Here, we report that shock waves are utilized to achieve the productive synthesis of c-BN nanoparticles whose average size is 3 nm, the smallest size so far. This finding may offer an effective strategy for the synthesis of c-BN nanoparticles below 10 nm and expand vital applications in various fields.

1. Introduction

It is known that cubic boron nitride (c-BN) is a typical material possessing fascinating properties such as extreme hardness, outstanding chemical stability, high thermal conductivity, much higher thermal stability than diamonds and wide band gap leading to the optical transparence. According to the Hall-Petch effect, for the c-BN nanoparticles, the hardness is expected to approach that of diamonds when the particle size decreases to several nanometers [1], and they can be widely applied in multiple areas, such as high-precision cutting tools, hard protective coatings, grinding and super-abrasive materials and so on. Owing to the outstanding mechanical and physical properties and the potential applications, much effort has been made to fabricate c-BN nanoparticles with the smallest size possible.

To date, the developed preparation methods could be categorized into three main classes, i.e., phase transformation [2–6] under high temperature and high pressure (HTHP), chemical reaction [7,8] and laser ablation [9,10]. Among them, the sizes of the prepared c-BN nanoparticles by the phase transformation method usually range from 10 to 50 nm [11,12] and the c-BN nanoparticles produced by the chemical reaction have sizes of more than 20 nm [7,8]. Using the ultrafast laser pulse, laser ablation has successfully synthesized the ultrafine c-BN nanoparticles, which first opens the door for the preparation of c-BN nanoparticles below 10 nm [13]. However, effective production is still restricted by laser beam size limitations. Here we report that the shock waves could be employed to accomplish the larger scale production of ultrafine c-BN nanoparticles. The average particle size is 3 nm, which is the smallest size ever reported.

2. Material and methods

Shock waves usually lead to HTHP, and meantime the duration of HTHP state could be tuned accordingly. Considering these unique characteristics, shock waves could be employed to induce the chemical reactions and accomplish the synthesis of some specific materials. For instance, the nano-diamonds could be fabricated from the carbon-contained high-energy explosives such as trinitrotoluene (TNT) and hexogen (RDX), and the average primary particle sizes range from 3 to 6 nm [14–17]. Inspired by the intriguing role of the shock waves in the successful synthesis of ultrafine diamonds, one may conceive that likewise shock waves may also be a promising strategy for the preparation of the c-BN nanoparticles. Herein the solid ammonia borane NH_3BH_3 (AB), identified as one of the most potential hydrogen storage material with efficient hydrogen capabilities 19.6 wt % and the atom ratio B:N = 1:1, is selected as the precursor.

AB usually decomposes in three distinct steps under thermolysis, and the final product is boron nitride (BN) and hydrogen [18], as is shown:

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$$nNH_3BH_3 \xrightarrow{107-117^\circ C} (NH_2BH_2)_n + nH_2 \quad (1)$$

$$n(NH_2BH_2)_n \xrightarrow{150^\circ C} (NHBH)_n + nH_2 \quad (2)$$

$$(NHBH)_n \xrightarrow{1400^\circ C} nBN + nH_2 \quad (3)$$

As anticipated, when subjected to HTHP just caused by shock waves, AB molecules may complete the three decomposition steps at one time and release all hydrogen atoms simultaneously, then the reaction products could nucleate to be the c-BN. The whole process is very fast and the duration may be ~1 us. This is because the rarefaction waves, tightly following the shock waves, always appear and penetrate into the samples, and consequently decrease HTHP dramatically. As a result, there is not enough time for the growth of c-BN nuclei, and they are formed in nano-size. Owing to the chemical reactions producing gases and heat, upon shock, the final HTHP conditions are really difficult to be determined. Base on the impedance-match method [19] and the shocking parameters of AB (the density is 780 kg/m^3, [20] bulk modulus is 9.9 GPa, [21] the calculated sound velocity is 3.6 km/s and linear coefficient between particle velocity and shock velocity is taken to be 1.5 as an estimation), here HTHP conditions are designed to be those needed for the hexagonal-BN to c-BN transformation, i.e. the temperature $T > 1400\,^\circ C$ and the pressure $P > 12$ GPa.

We perform the experiments according to the principle mentioned above and the schematic experimental setup is shown in Fig. 1. The ammonia borane powders were purchased from Zhengzhou Alfachem Co., Ltd with purity 98%. The powders were compressed into a dense slice by means of a mould. Then the slice was put into sample container which was made of steel and can maintain strength at high temperatures, as is shown in Fig. 1. For the experimental setup at room conditions, liquid nitromethane was used as explosive and electrical detonator was employed for firing. Once an electrical current passes through the detonator, it will explodes and subsequently lead to explosion of liquid nitromethane explosive. The flyer plate in front of explosive will be accelerated to a high velocity and impart sample container. So shock waves are created and then penetrate into the ammonia borane samples, inducing the chemical reactions under high temperature and high pressure.

3. Results and discussion

We carried out the shock experiments and the fabricated samples were recovered. The transmission electron microscope (TEM) experiments were performed for the characterizations. The obtained results are shown in Fig. 2. As shown in Fig. 2(a), the nanoparticles are synthesized. Their fast Fourier transformation (FFT) are performed and are shown in Fig. 2(b) and (c), enlarged high resolution TEM (HRTEM) diagram of one nanoparticle is shown in Fig. 2(d) and the selected area electron diffraction pattern is shown in Fig. 2(e). Through analysis on these diffraction patterns and the enlarged TEM diagram of this particle, the plane angles and plane spacing of the nanoparticles can be obtained. It is found that they agree well with the crystalline structure of c-BN, confirming chemical composition c-BN of the nanoparticles and indicating successful preparation of c-BN nanoparticles. This verifies that the preparation principle is rational. Under appropriate HTHP, AB can decompose and release hydrogen totally. For the remnant B and N atoms, due to the long diffusion length and ultrashort time of HTHP, they finally form c-BN nanoparticles. Sizes of the c-BN nanoparticles are summarized and their size distribution is shown in Fig. 3. This figure shows that the prepared c-BN nanoparticles range from 2 nm to 6 nm and the average size is 3 nm, which is the smallest size ever reported.

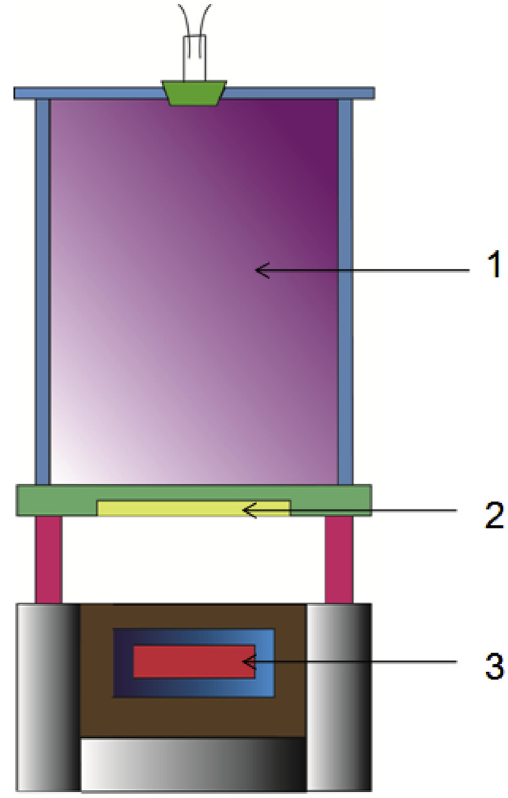

Fig. 1. The schematic experimental setup: 1 (gradual pink zone) is the explosive whose detonation enables the flyer plate 2 (yellow zone) accelerate. 3 is the ammonia borane slice, AB (red zone). When the explosive is detonated, the flyer plate will be accelerated to a high velocity. Then the flyer plate imparts the target and creates the shock waves. The shock waves will penetrate into the sample AB and subsequently results in high temperature and high pressure (HTHP). Under HTHP, AB decomposes and forms nano-c-BN. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

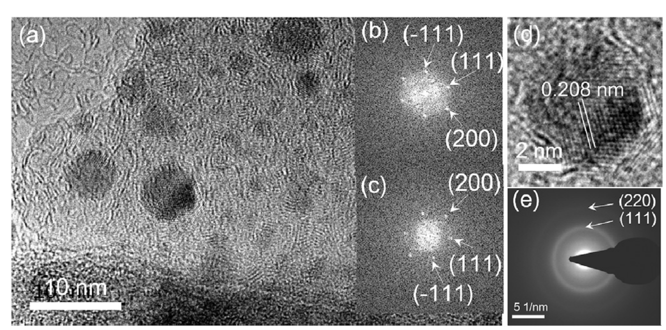

Fig. 2. Summary of Transmission electron microscope (TEM) results: (a) TEM diagrams of nano-c-BN, (b), (c) the related fast Fourier transformation (FFT) patterns and related indices; (d) enlarged HRTEM diagram of one particle; (e) selected area electron diffraction pattern.

At last, let us examine this method again. Comparing with the classical method for preparing nano-c-BN, phase transformation of h-BN under high pressure, the diffusion length of atoms in chemical reaction is longer and the sustaining time of HTHP is much shorter, therefore, using chemical reaction in shock waves can fabricate much smaller nanoparticles. Notably, the shock waves is created by the flyer plate colliding with targets and there is no limitation on

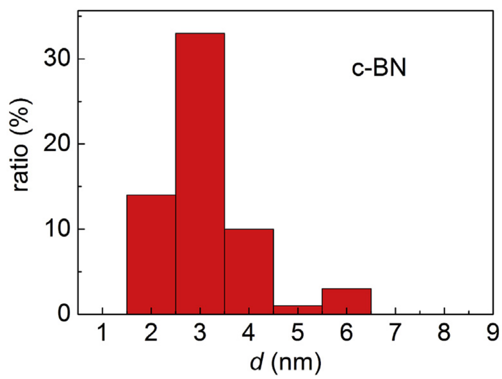

Fig. 3. Size distribution of the c-BN nanoparticles. The sizes of these c-BN nanoparticles are in the range 2–6 nm and their average size is 3 nm.

the size. So, it could produce c-BN in large amount.

4. Conclusion

In summary, using the AB as the precursor, we employed shock waves to synthesize c-BN nanoparticles. Through characterization, c-BN nanoparticles have been successfully synthesized and their average size is 3 nm which is the smallest size ever reported. This preparation method could produce c-BN nanoparticles in large amounts and might be applied in the various areas.

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