

Study of the stability of n-diamond

This article has been downloaded from IOPscience. Please scroll down to see the full text article.

2004 J. Phys.: Condens. Matter 16 2991

(<http://iopscience.iop.org/0953-8984/16/17/026>)

View [the table of contents for this issue](#), or go to the [journal homepage](#) for more

Download details:

IP Address: 129.252.86.83

The article was downloaded on 27/05/2010 at 14:33

Please note that [terms and conditions apply](#).

Study of the stability of n-diamond

**Bin Wen^{1,3}, Tingju Li^{2,3}, Chuang Dong¹, Xingguo Zhang², Shan Yao²,
Zhiqiang Cao², Dehe Wang¹, Shouhua Ji¹ and Junze Jin²**

¹ Department of Materials Engineering, Dalian University of Technology, Dalian 116023, People's Republic of China

² Laboratory of Special Processing of Raw Materials, Dalian University of Technology, Dalian 116023, People's Republic of China

E-mail: wenbin@dlut.edu.cn and tjuli@dlut.edu.cn

Received 15 January 2004

Published 16 April 2004

Online at stacks.iop.org/JPhysCM/16/2991

DOI: 10.1088/0953-8984/16/17/026

Abstract

Powders of n-diamond can be synthesized by pyrogenation of carbon black and nanometre-sized iron catalyst at atmospheric pressure and at a temperature of 1100 °C. The stability of n-diamond was investigated with x-ray diffraction, thermal gravimetric analysis and differential thermal analysis. The results indicated that n-diamond was a metastable phase: it can decompose at room temperature slowly. Thermal decomposition of n-diamond begins at 150 °C and is complete at 400 °C, and the decomposition of n-diamond was an exothermic reaction.

1. Introduction

Diamond, graphite and carbyne are three well-known allotropes of carbon, which are distinguished by the type of electron hybridization [1]. Recently, Konyashin, Jarkov and their co-workers validated a new kind of carbon allotrope, which was a metallic form of carbon with face-centred cubic structure, with a lattice constant of 0.3594 nm [1, 2]. In addition, this new allotrope has been reported in [1, 2, 5–9]. In the literature, this phase was referred to as 'n-diamond'. In this report, we also call this new phase 'n-diamond'.

The n-diamond was synthesized accidentally by various processes such as radiofrequency plasma-aided decomposition of hydrocarbon [3], plasma-assisted chemical vapour deposition using diluted hydrocarbons [4], transformation of graphite under shock compression [5], transformation of C₆₀ films under shock compression [6], transformation of graphite at high pressure and temperature [7], plasma-chemical synthesis with the aid of a carbon plasma jet [2], treatment of a diamond surface in hydrogen plasma [1] and annealing of silica wafers embedded with carbon atoms [8, 9] etc.

³ Authors to whom any correspondence should be addressed.

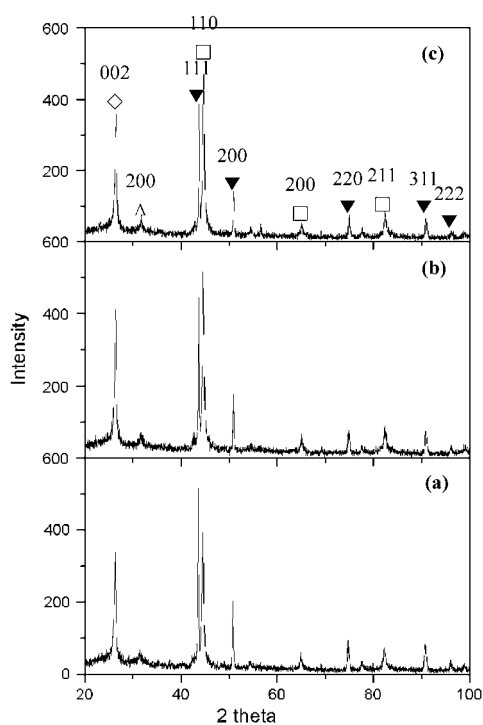


Figure 1. X-ray diffraction pattern of samples. (a) One-day-ageing-treatment sample, (b) 90-days-ageing-treatment sample, (c) 180-days-ageing-treatment sample. ▼, n-diamond; ◇, graphite; □, α -Fe; △, NaCl.

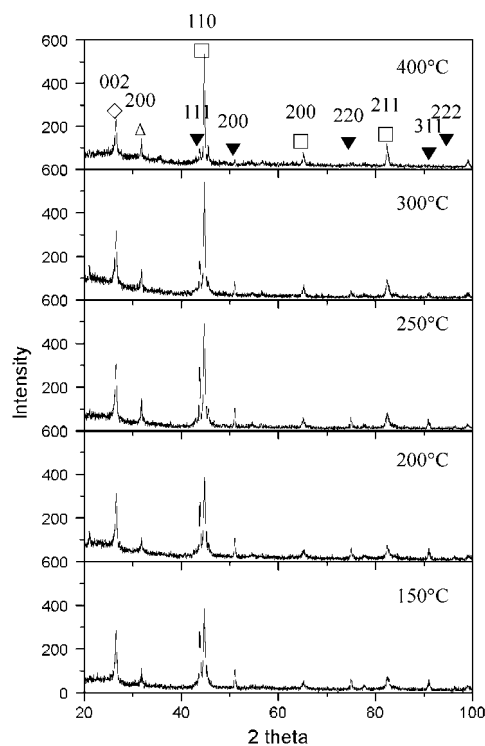


Figure 2. X-ray diffraction pattern of samples heat treated at various temperatures. ▼, n-diamond; ◇, graphite; □, α -Fe; △, NaCl.

Because of the lower output of n-diamond powders, which were synthesized by the above-mentioned processes, in-depth research on the character of n-diamond was very difficult. In 2003, n-diamond powders had been produced largely by the method of catalyzed carbon black in a high magnetic field [10]. Hence research on n-diamond can be put into effective operation. In this report, the stability of n-diamond was studied and the character of n-diamond transformation was proposed.

2. Experiment

An admixture of carbon black N231 powders and colloidal $\text{Fe}(\text{OH})_3$ (from the reaction between FeCl_3 solution and NaOH solution) was compressed in an open stainless steel tank of 100 ml capacity. The mass ratio of carbon to iron in the mixture is 10:1. The tank was maintained at 300 °C for 100 min and sealed in air. In a high magnetic field of 10 T, the tank was maintained at 1100 °C for 100 min and then cooled to room temperature in the furnace. The sealed tank was unsealed ultimately, the reaction products become hot when exposed to air and this would indicate that an exothermic reaction has occurred in the final powders. The products were washed with distilled water. The final powders were dried in an oven at 110 °C and preserved in air.

The final powders were analysed with x-ray diffraction (XRD) after 1-day-ageing-treatment, 90-days-ageing-treatment and 180-days-ageing-treatment.

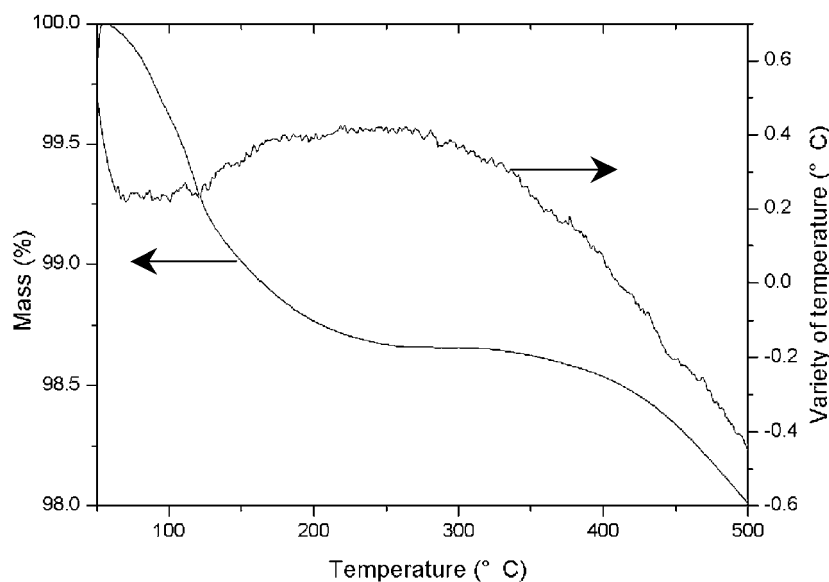


Figure 3. TGA and DTA curves of the final powders.

Then the final powders were heat treated in N_2 at one atmospheric pressure over the temperature range 150–400 °C for 10 min and the phase composition of the heat-treated powders were analysed with XRD. Finally, the thermal properties of n-diamond were investigated by thermal gravimetric analysis (TGA) and differential thermal analysis (DTA) from 50 to 500 °C, at the heating rate of 10 °C min^{-1} in flowing nitrogen.

3. Results and discussion

The XRD patterns were recorded on an XRD-6000 diffractometer with $Cu K\alpha$ radiation (wavelength 0.154 nm). Figure 1(a) shows the XRD patterns of the final powder that had been age-treated for one day in air and that of 90-days-ageing-treatment and 180-days-ageing-treatment are shown respectively in figures 1(b) and (c). The XRD patterns in figure 1 point to the presence of graphite, NaCl, α -Fe and the n-diamond phase in the dried powders. Graphite is one of the reaction products, NaCl is a remnant of the reaction $FeCl_3 + NaOH$ and α -Fe is some remnant catalyst. The presence of n-diamond and amorphous carbon in the final powders has been described in detail in our previous work [10]. As shown in figure 1, with the increase in the ageing-treatment time, the peaks of n-diamond in figure 1 become weaker in intensity and the peaks of graphite in figure 1 increase in intensity, yet the change was very slight. These results indicated that n-diamond in the final powders might decompose slowly on account of the ageing-treatment. To further study the stability of n-diamond, the final samples were heat treated at various temperatures and the heat-treated samples were analysed with XRD.

The XRD patterns of the final samples as a function of heat-treatment temperatures are shown in figure 2. The peaks of the patterns mainly correspond to four phases: n-diamond, graphite, α -Fe and NaCl. As shown in figure 2, the peaks of n-diamond decrease in intensity with increasing temperature of the heat treatment, and when the heat-treatment temperature reaches 400 °C the peaks of n-diamond almost disappeared. This result indicated that n-diamond can decompose completely at 400 °C. This result was also validated by DTA and TGA results.

Thermal analysis was done for the final powders. Figure 3 shows the TGA and DTA curves of the final powders. DTA and TGA show that the thermal decomposition of n-diamond begins at 150 °C and is complete at 400 °C. The DTA curve coincides with the TGA, i.e. the broad exothermic peak (from 150 to 400 °C) was just in the range of significant weight loss. The DTA result from the final sample was consistent with the phase composition change shown in figure 2 and the exotherms between approximately 150 and 400 °C are associated with the decomposition of n-diamond.

4. Conclusions

The stability of n-diamond was studied and the following results were obtained.

- (a) n-diamond was a metastable phase, it can decompose slowly at room temperature;
- (b) thermal decomposition of n-diamond begins at 150 °C and is complete at 400 °C;
- (c) the decomposition of n-diamond was an exothermic reaction.

Acknowledgments

The National Natural Science Foundation of China, project no. 50234020 and the Young Teacher Foundation of Dalian University of Technology, project no. 893202, supported this work.

References

- [1] Konyashin I *et al* 2001 *Diamond Relat. Mater.* **10** 99
- [2] Jarkov S M, Titarenko Ya N and Churilov G N 1998 *Carbon* **36** 595
- [3] Amaratunga G, Putnis A, Clay K and Milne W 1989 *Appl. Phys. Lett.* **55** 634
- [4] Frenklach M, Kematich R, Huang D, Howard W and Spear K E 1989 *J. Appl. Phys.* **66** 395
- [5] Hirai H and Kondo K I 1991 *Science* **253** 772
- [6] Yoo C S, Nellis W J, Sattler M L and Musket R G 1992 *Appl. Phys. Lett.* **61** 273
- [7] Endo S and Idani N 1994 *Phys. Rev. B* **49** 22
- [8] Orwa J O *et al* 2001 *J. Appl. Phys.* **90** 3007
- [9] Peng J L, Bursill L A, Jiang B, Orwa J O and Prawer S 2001 *Phil. Mag. B* **81** 2071
- [10] Wen B *et al* 2003 *J. Phys.: Condens. Matter* **15** 8049