HOT SHOCK CONSOLIDATION OF DIAMOND AND CUBIC BORON NITRIDE POWDERS

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Diamond and cubic boron nitride powders were explosively compacted at high temperature (873K) by using planar impact system at 1.2km/s. Silicon or graphite was added to the mixture to provide enhanced bonding through chemical reaction. Hot consolidated specimens exhibited decreased surface cracks as compared with the specimens consolidated at room temperature. Some of the materials consolidated showed the evidence of melting at the particle surfaces and hot consolidated diamond admixed with graphite powder improved the bonding between particles.

I. INTRODUCTION

Shock consolidation of extremely hard non-oxide ceramics powders has already been attempted by many researchers; diamond [1-5], cubic boron nitride (c-BN) [6] and some other powders [7, 8] have been consolidated, but there still are some problems to be improved. The main problems are remaining cracks, weak interparticle bonding and inhomogeneity of consolidated material.

In this investigation, we tried to shock consolidate diamond and c-BN powders at high temperature to eliminate the problems mentioned above. The technique of hot shock consolidation is expected to eliminate the cracks due to the increase of plastic flow and surface melting of powders and is also expected to improve the bonding between particles by the heating of particle surfaces [10]. The effect of particle size on the consolidated diamond and c-BN powders is examined by comparing the results at room temperature [1-3, 6]. The effect of the addition of graphite or silicon powders is also examined as a means to enhance the bonding with the assistance of reaction [2, 4, 11-13].

II. EXPERIMENTAL PROCEDURES

Three sizes of natural diamond (4-8, 10-15 and 20-25µm) and two sizes of c-BN powders (10-20 and 40-50µm) were used as starting material. The composition of the specimens are shown in TABLE 1. The effect of addition of graphite or silicon powders is examined in these experiments. The powders were pressed to 60% of the theoretical density with the shape of 5mm thick and 12mm in diameter into a stainless steel capsule.

The planar impact system developed by Sawaoka and Akashi [14] was used for the consolidation. The schematic illustration of this set up is shown in FIG.1 (a). Twelve capsules can be compacted simultaneously. Figure 1 (b) shows the set up for hot consolidation developed by Yu and Meyers [15]. The capsules are preheated at 873K and compacted by the flyer plate at 1.2km/s.

The materials consolidated were characterized by optical microscopy, scanning electron microscopy (SEM), X-ray diffraction and Vickers micro-hardness.

III. RESULTS AND DISCUSSION

Figure 2 shows bottom quarter surfaces of diamond and c-BN powders after hot shock consolidation. Shock consolidated materials at room temperature showed many cracks at the bottom surface [5, 6], but some of the specimens hot consolidated showed only a few cracks (FIG.2 (a), (b), (d), (e)). This result is due to heating. High temperature causes the melting of particle surfaces easily and decreases the energy required for consolidation. In this case a flyer plate velocity of 1.2km/s was chosen for the consolidation of diamond and c-BN powders, while 1.8-3.0km/s is required for the consolidation of these powders at room temperature [1-6]. The decrease of shock pressure induces the decrease of surface cracks.

A. Characterization of hot consolidated diamond powders

Figure 3 (a) shows the half of transverse cross section and (b) shows the Vickers microhardness distribution of consolidated 20-25µm diamond powders (specimen # 3). This

specimen shows many cracks in the bottom region, where the micro-hardness values show the higher value. As reported earlier [5, 12] the micro-hardness distribution of shock consolidated material shows the same tendency of the temperature distribution during the passage of shock wave. Higher hardness values are also obtained in the high temperature area in our specimen.

Figure 4 shows the SEM of polished surface of 4-8µm diamond (# 1). No microcracks and voids can be seen in this photograph. Small round dark areas about 1-2µm in diameter are the trace of the diamond particles pulled out. The enlarged SEM in such a dark area generated in 20-25µm diamond (# 3) is shown in FIG.5. This area is composed of small grains about 0.5 - 1µm in diameter. Since the original diamond powder is monocrystalline, the small grains are thought to be generated during cooling of molten surface of the particles. Such small polycrystalline regions in a diamond particle are already found in shock consolidated [3] and shock synthesized diamond powders [16, 17]. The size of these small grains is decreased with the decrease of the size of the original diamond particle. This result depends on the cooling rate of the molten layer [18, 19]. Generation of small amount of graphite is detected by X-ray diffraction in 10-15µm diamond (# 2). Figure 6 shows the cracks generated in one particle consolidated 10-15µm diamond (# 2).

Akashi and Sawaoka [5] reported that 2µm-20µm diamond showed the optimum condition shock consolidated at room temperature. Potta and Ahrens [1] reported the same the effect of particle size. When the original particle size is too small, surface temperature is not sufficient to generate the molten layer to enhance the bonding by the dispersion of shock pressure. When the particle size is too large, many cracks are generated in the particles by the high pressure and a transformation from diamond to graphite is caused by the thick molten layer. In hot consolidated materials, cracks still are formed and the transformation to graphite is generated in 10-15 µm diamond specimens (# 2). The transformation to the graphite is usually generated in larger particle size specimens consolidated at room temperature [5] but this transformation is enhanced in hot consolidated material with smaller particle size by the help of heating. In case of the hot consolidation of pure diamond, smaller particle size is required for the good bonding compared with the consolidation at room temperature. Micro-hardness values in TABLE 1 assure this consideration.

Consolidated diamond with graphite shows the highest hardness value (TABLE 1). Due to the difficulty of the measurements, we only describe the Vickers hardness value more than 55GPa. The improvement of the bonding by the addition of graphite to diamond was already demonstrated for shock consolidation at room temperature by Potta and Ahrens [2]. The explanation provided by them was that the graphite, which has the lower thermal diffusivity, delays thermal equilibrium between molten surface and interior of diamond. This heat enhances the bonding between particles. Deformation of the softer component graphite around the diamond powder also decreases the pressure concentration and fills the pores between diamond particles. On the other hand, degradation of consolidated material appeared

with the addition of silicon powders. We expected the heat generated during reaction between silicon and carbon to enhance the bonding between particles [2, 11-13], but in this case the excess heat generated induced the excess melting of the particle surfaces.

B. Characterization of hot consolidated c-BN powders

Figure 7-9 show SEM of hot consolidated c-BN powders. Figure 7 is a fracture surface of 10-20µm c-BN (# 6) and this fracture surface suggests the melting of particle surfaces. In some areas of this specimen, small voids can be seen caused by the gas generated by the decomposition of c-BN (FIG.8). Consolidated 40-50µm c-BN (# 7) shows high microhardness value (TABLE 1). SEM of fracture surface in FIG.9 shows the evidence of good bonding. Some of the grains in shock consolidated 40-50µm c-BN show the transgranular fracture. As Akashi and Sawaoka [6] reported on the shock consolidated c-BN powders at room temperature the specimen with larger particle size also showed higher hardness value in hot consolidation (TABLE 1). In contrast with the case of diamond, a larger melting layer is thought to be better for consolidation. It is reported that c-BN is more stable than diamond at high temperature in the atmosphere of oxygen [20]. This suggests that the transformation from cubic to hexagonal structure boron nitride is hard to occur even when the cooling rate is slow compared with the case of diamond.

The consolidated c-BN admixed with graphite shows low micro-hardness value compared with the c-BN without graphite (TABLE 1). It is considered that the rapid cooling rate could not be obtained for the generation of diamond because of the thick molten layer. The remaining graphite causes the degradation of consolidated material.

IV. CONCLUSIONS

Diamond and cubic boron nitride powders were explosively consolidated at high temperature by using a planar impact system. Hot shock consolidated materials exhibited a decrease of surface cracks as compared with specimens consolidated at room temperature. The consolidated diamond specimen showed evidence of surface melting of the particles by small crystallized grains generated during cooling of this molten layer. 4-8µm diamond consolidated showed good bonding. Excess cracks and generation of graphite were observed by the increase of particle size. Hot consolidated diamond admixed with graphite showed the highest hardness value, but the improvement of the bonding was not confirmed by the specimen admixed with silicon powders. Hot consolidated 40-50µm c-BN showed good bonding compared with the specimen which has small particles. The improvement of the bonding of c-BN powders by the addition of graphite could not be recognized in the present investigation.

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TABLE 1 Experimental conditions of hot consolidation. Average Vickers hardness values are also listed.

Specimen #	Composition	Average Vickers hardness* / GPa
1	4-8μm natural diamond	29.4
2	10-15µm natural diamond	26.0
3	20-25µm natural diamond	24.4
4	4-8µm natural diamond + graphite (15mass%)	>55
5	4-8µm natural diamond + silicon (7.5mass%)	15.8
6	10-20μm c-BN	37.5
. 7	40-50μm c-BN	53.0
8	40-50μm c-BN + graphite (15mass%)	24.4

^{*;} Load 4.9 or 9.8N for 15sec.

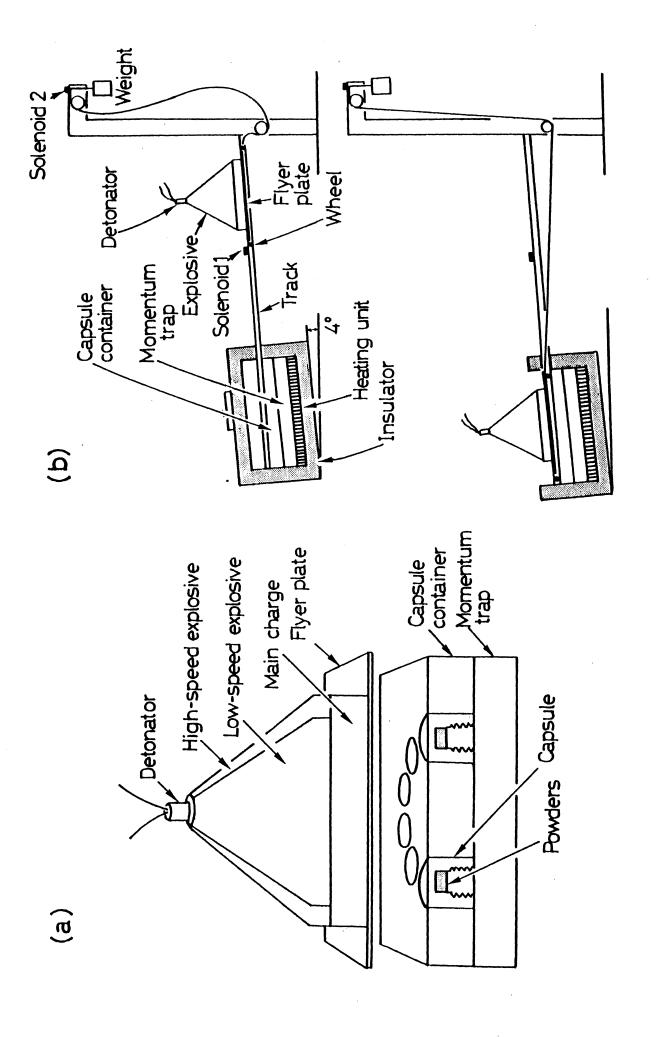


FIG.1 Schematic illustration of planar impact system (a) and set up of hot consolidation (b)

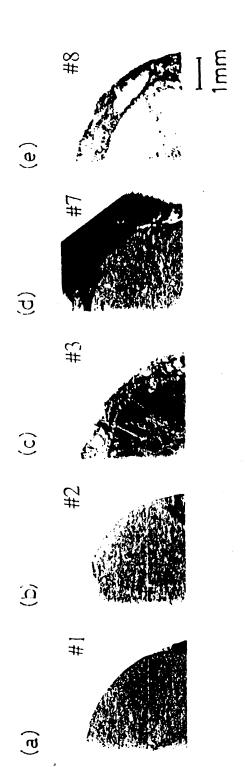


FIG.2 Quarter bottom surfaces of consolidated diamond ((a)+(c)) and c-3N powders ((d),(e)).

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24.2 300 330 45.9 32.4 37.4 12.2 | 18.3 | 13.4 | 10.4 | 33.6 | 11.6 27.1 |20.2 | 22.9 | 16.2 | 14.3

FIG.3 Cross section of consolidated 20-25µm diamond (#3)(a), and its Vickers hardness distribution in GPa.

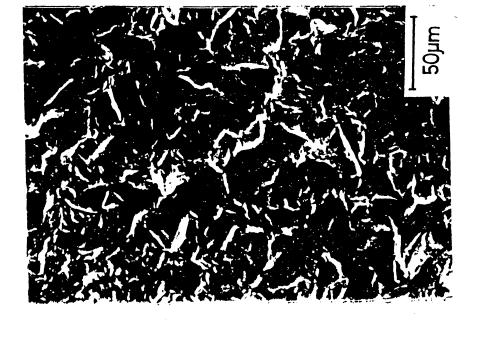


FIG.9 SIM of fracture surface of the common of the common

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FIG.8 Small voids generated in consolidated 10-20µm c-3N (#6).



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FIG.6 Cracks generated in one particle of consolidated

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FIG.5 Enlarged SEM of consolidated 20-25µm

FIG.4 SEM of polished surface of consolidated $4-8\mu m$ diamond (#1).