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Hot explosive compaction of diamond powder using cylindrical geometry

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Abstract Diamond powders were attempted to be consolidated at elevated temperature using an explosive compaction technique employing a cylindrical configuration. A compacted diamond sample recovered from the center of a cylinder, close to the end plug made of stainless steel showed high hardness and tight interparticle bonding between the diamond powders. The sample was characterized on the basis of X-ray diffraction patterns. No graphitization was observed due to heating or compaction, and the hot compacted sample was highly strained by intensive deformation at elevated temperature.

Introduction

Materials processing using explosive energy has been investigated and industrialized due to some excellent properties obtained during the quite short processing time. Explosive welding [1] is widely used for joining dissimilar plates with tight bonding, and shock-synthesized diamond and w-BN are commercially fabricated due to their high hardness [2]. The fabrication of large-sized diamond compacts without binding agents is a long-standing problem. The use of explosive energy has been considered as a possible solution. Some attempts [3–9] have been made for obtaining polycrystalline diamond compacts from diamond powders, and positive results have been reported. Polycrystalline diamond is ideal for its isotropic mechanical properties, which may show improved fracture toughness.

As a purpose of consolidating extremely hard powders, it is necessary to apply high pressure to the powder part [10]. According to investigations performed with different powders, the pressure required for full compaction is proportional to the hardness of the powder particles; the predicted pressure for the compaction of diamond powders is about 90 GPa [10]. An alternative approach is to elevate the temperature of the powders to decrease the hardness, and by this process, enhance surface melting to join each particle [8, 9]. Using a converging underwater shock wave assembly above 1,100 °C, one of the authors has reported the recovery of a diamond compact with high hardness [8, 9]. In that case, PBX, a special, quite expensive explosive with a high detonation velocity above 8 km/s, was employed. The present investigation tries to use a cylindrical assembly to improve the condition of consolidation. Using the cylindrical assembly, a high pressure at the center position is induced under a moderate condition due to the formation of the Mach stem [11]. By using an explosive whose detonation velocity is about 7 km/s, which is commercially available at relatively low cost, it would be beneficial to obtain a compact of high hardness with strong interparticle bonding using this technique. An

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improved method using the cylindrical geometry is proposed, and some experimental results are discussed.

Experimental

The device for the hot explosive compaction is shown in Fig. 1. The basic design is the same as the assembly, which was developed by Pruemmer [12]. Some modifications were made to obtain higher temperature up to 1,200 °C and to set the device at an explosion pit in the Shock Wave and Condensed Matter Research Center, Kumamoto University. The device is separated into two parts. The upper heating part and the lower explosive part are connected with ceramic and stainless steel tubes of the same inner diameter. Once the capsule was heated to the desired temperature, the capsule was dropped to the explosive part. When the capsule was caught by the gypsum plate at the bottom, the explosive was ignited by an electric detonator connected with four detonation fuses of the same length. On recovery, the captured sample underwent a uniform axisymmetrical compression. The mass of explosive used was 0.38 kg, and the explosive was placed along a length 110 mm around a stainless steel tube of 15 mm outer and 12 mm inner diameters. The explosive used was SEP produced by Asahi-Kasei Chemicals Corp. with the detonation velocity of about 7 km/s and the density about 1,300 kg/m^3. The upper and the lower parts were separated by about 1 m, and were connected by a ceramic tube as a guide for capsule lowering. A steel block separating the upper and lower part is important to protect the upper furnace from fragments and vibrations caused by the explosion.

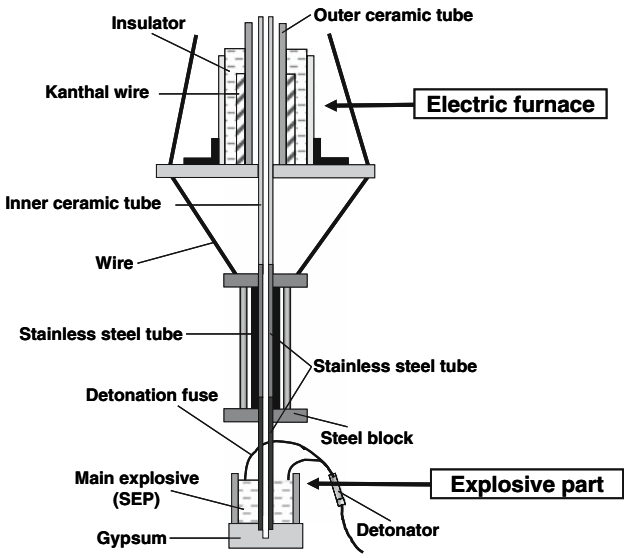

Fig. 1 Cross-section of set-up for hot explosive compaction

The dimensions of the capsule part are illustrated in Fig. 2. The outer and the inner diameters of the capsule were 10 and 7 mm, respectively. TiB_2 powders (7 μm average grain size) were mainly put in a capsule but mixed diamond (60 vol.%), TiB_2 (40 vol.%) and diamond powders were also put close to the end plugs, as illustrated in Fig. 2. The grain size of the diamond powders was 4–8 μm. The diamond powders were single crystalline diamond, commercially provided by Nilaco, Japan. The packing density of the powders was fixed at 50% of theoretical density. TiB_2 powders were selected due to its high hardness next to diamond and c-BN also possessed a similar structure of diamond. The manufacturing of TiB_2 compact has been recognized as a difficult process through conventional sintering technique due to its high melting point. The experimental temperature was estimated on the basis of calibration curve after the sample was dropped from the heated part and the accuracy of the sample temperature was considered as ±10 °C. The deviation of the temperature along the sample axis is considered negligible, as the process employs a long furnace.

Results and discussion

After the hot explosive compaction performed at 1,100 °C in Ar atmosphere, the sample was successfully recovered. The cross-section is shown in Fig. 3. The TiB_2 part on the left hand side shows a central hole, which was caused by the formation of Mach stem due to the convergence of shock pressure toward the center. Such results have often been found while using the cylindrical configuration [11]. The diameter of the Mach stem is considered to be about 2–3 mm with a higher propagating velocity and higher pressurizing condition of the shock wave induced at the center position. The diamond powders, and the diamond and TiB_2 mixed powders placed close to the end plugs seem to have a good condition of consolidation due to their highly converged shock pressure. The pressure close to the end plug with a higher density may also increase the pressure in this region. Unfortunately, the intermediate end plug for the mixed diamond and TiB_2 powder part was broken due to stretching the capsule, but the diamond

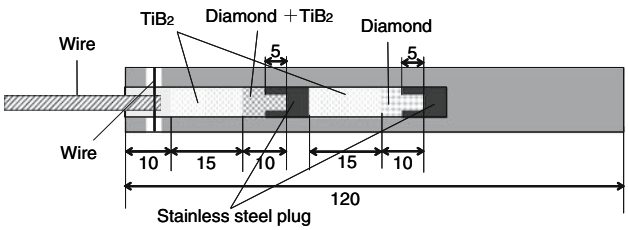

Fig. 2 Dimensions of capsule part

powder part at the end of the capsule was successfully recovered. The diamond area was separated due to some cracks induced in the compacted area. The pressurizing condition in the diamond part requires further clarification, but it is difficult at this moment to perform numerical simulation due to the lack of relevant fundamental data required to analyze the condition of the powder and other parts at elevated temperature.

The optical micrographs of the polished surface for the TiB_2, TiB_2 (40 vol.%) + diamond (60 vol.%), and diamond powder parts are shown in Fig. 4a–c. The micro-Vickers hardness was measured under a load of 9.8 N. The TiB_2 powder part in Fig. 4a was well polished with tight bonding between the particles, and a low porosity, and exhibited a high hardness of 2,500 HV (average). The mixed TiB_2 + diamond part (Fig. 4b) shows a poorly polished surface due to poor interparticle bonding because of the fracture of the capsule due to release waves. The hardness of the areas was not measured because of so many pulled out powder particles. Figure 4c is taken from a fragment of diamond part at the center position close to the end plug and the size is about $2 \times 2.5 \times 3$ mm^3. The photograph in Fig. 4c shows the trace of diamond penetrator under a load of 98 N using macro-Vickers hardness test machine. The interfacial bonding is not clear in this photograph because the polishing was very difficult, but no large pores or cracks are observed in the microstructure. The micro-Vickers hardness of the area was also measured carefully and the average hardness was 6,700 HV under a load of 9.8 N, which corresponds to diamond with a hardness of about 7,000–10,000 HV. High hardness above 12,000 HV has been measured for nano-sized polycrystalline diamond compact synthesized from graphite using high-temperature press machine [13], which is superior to the results reported here. High fracture toughness is expected for the polycrystalline diamond sample as mentioned in the introduction, and actually, we found no crack at the edge of the penetrator even at a loading 98 N in Fig. 4c. Fracture toughness of 5.3 MPa m$^{-1/2}$ for a polycrystalline diamond obtained through Chemical Vapor Deposition (CVD) process has been reported on the basis

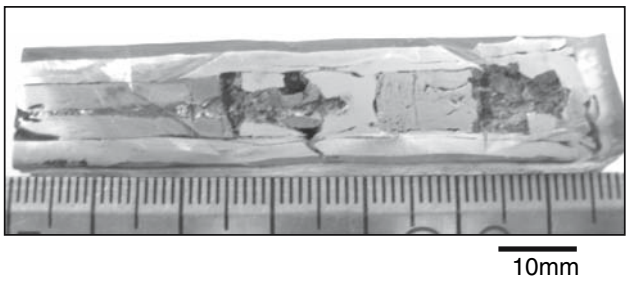

Fig. 3 Longitudinal cross-section of compacted sample

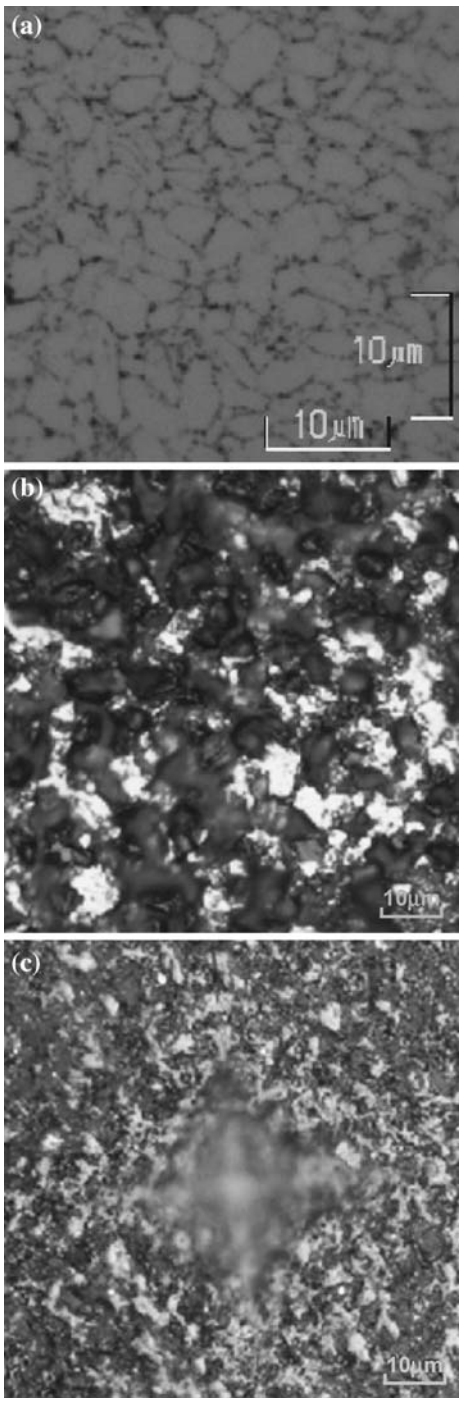

Fig. 4 Optical micrograph of polished surface of TiB_2 (**a**), TiB_2 + diamond (**b**) and diamond (**c**) parts

of same procedure [14]. The present observation of no generated cracks from the edge of the penetrator may suggest the potential of the diamond compact obtained from the present method to have higher fracture toughness than the CVD diamond.

In previous reports for the hot explosive compaction of diamond powders using converging underwater shock

wave, a compact showing high hardness was measured after the same preheating temperature at 1,100 °C [8, 9]. As pointed out in the former investigations [9], softening or plastic deformation of the diamond is expected at this temperature [15], and the condition of consolidation improves remarkably. Since no successful result has been confirmed from the experiments at higher temperature experiments, the temperature should be the optimum condition for hot explosive compaction of diamond powders. Under the higher temperature condition, graphitization or other undesirable phenomena are expected. The previous reports employed PBX explosive with high detonation velocity of more than 8 km/s, but it must be emphasized that we could obtain a compact with high hardness using the explosive SEP, whose detonation velocity is similar to the industrial explosives, by controlling the pressurizing condition.

A Scanning Electron Microscope (SEM) image of the fractured surface of the diamond compact is shown in Fig. 5. From the fractured surface, the compact was well densified, and the surface showed a roughness in the order of the size of the original diamond powders of 4–8 μm. Interparticle bonding between the diamond particles should be quite good, because no separation or pulled-out grains were observed. The measurement of density was not made due to the small size of the recovered sample. Considering the measured high hardness and the expected intensive plastic deformation at elevated temperature above 1,000 °C, a high theoretical density of more than 90% is expected similar to the results reported earlier [8, 9].

Figure 6 shows X-ray diffraction patterns of diamond for as-received powders (a), as heat treated powders at 1,100 °C for 1 h in Ar atmosphere (b) and after explosive compaction (c). It is clear that no graphitization is found after the heat treatment for a limited time of 1 h. Furthermore, no weight loss due to the oxidation was observed. All three patterns show only diamond peaks; the width is slightly decreased after the heat treatment, and is broadened after the shock compaction. After compaction, no separation of $K\alpha_1$ and $K\alpha_2$, as-seen in the as received and 1,100 °C annealed condition, can be observed any more. No transformation to the graphite phase of carbon occurred.

It is well known that the change in the width of the half-height peaks at different diffraction angles can suggest the condition of its crystalline nature, and the method is known as Hall–Williamson plot [16]. Figure 7 shows the plots for the three cases as shown in Fig. 6, where β is the half-height width of the peaks and λ is the wavelength of the X-ray. Since the slope of the plots is proportional to the lattice strain, it is clear that the strain was released during the heat treatment, and a large amount of distortion is induced in the compacted bulk sample. It has been reported that diamond can exhibit dislocation sliding, in other words, can show plastic deformation under such a high temperature [15, 17]. The measured lattice distortion in the bulk sample is 0.44%. This value is the highest one ever observed in shock compacted diamond powder. For room temperature experiments done by Akashi and Sawaoka [6], lattice distortion was ranging up to 0.3%. This large strain proves that an extensive plastic deformation is occurring during hot explosive compaction of diamond powder at 1,100 °C. However, a deviation from linearity was observed in the Hall–Williamson plot. The reason for this deviation could be a mechanism of plastic deformation leading to preferred orientation. Further research is necessary to clarify this phenomenon.

The mechanism of interparticle bonding is mainly considered through the surface melting of each particle followed by an intensive deformation [18], but the X-ray diffraction analysis suggests the straining of particle. For further characterization of the crystalline substructure in

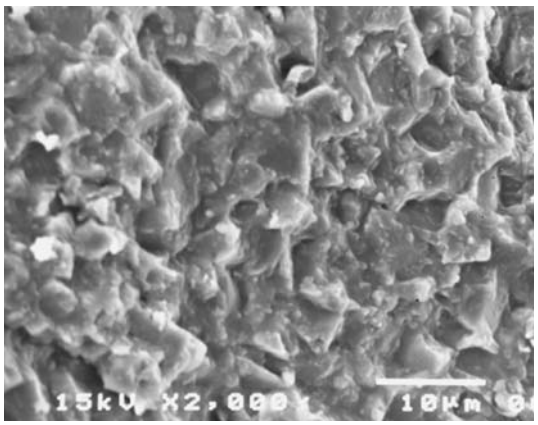

Fig. 5 SEM of fractured surface of diamond sample

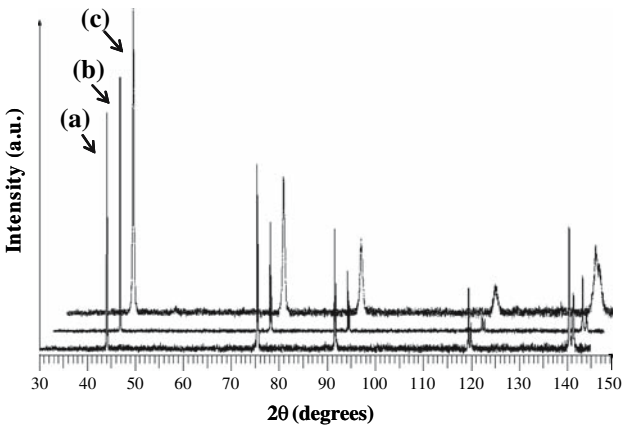

Fig. 6 X-ray diffraction patterns (Cu–Kα) of diamond for as-received powders (**a**), as heat treated powders at 1,100 °C for 1 h (**b**) and after explosive compaction (**c**)

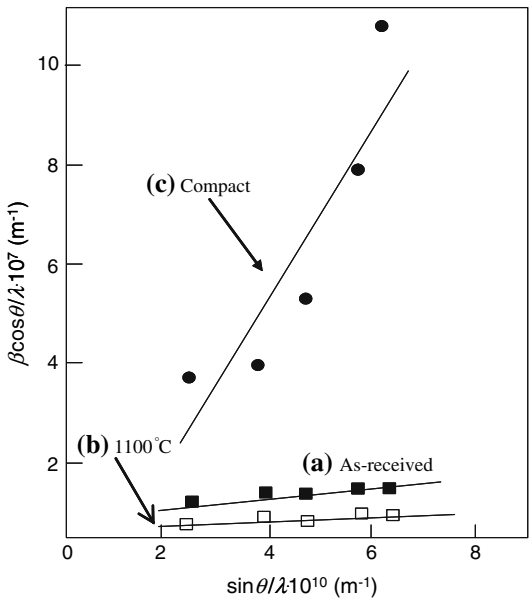

Fig. 7 Hall–Williamson plot for as-received diamond powders (**a**), as heat treated powders at 1,100 °C for 1 h (**b**) and compacted sample (**c**)

detail, the analysis using Transmission Electron Microscope (TEM) is being considered and prepared.

Conclusions

The present article shows the possible preparation of polycrystalline diamond without a binding agent by hot explosive compaction. Using a cylindrical geometry with the diamond powders at the center and end position enables a higher pressurizing condition. A diamond compact was successfully recovered showing high hardness, 6,700 HV, without showing any graphitization of the compact. X-ray diffraction analysis showed that the diamond compact was highly distorted by an intensive plastic deformation at high pressure and high temperature.

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