High temperature shock consolidation of hard ceramic powders

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Abstract

High-temperature shock consolidation of hard ceramic powders was used as a means to improve bonding between powders and to decrease the number of cracks generated in the consolidated sample. A converging underwater shock-wave assembly was used for the compaction, and TiB\textsubscript{2}, c-BN and their mixed powders were consolidated at various conditions up to 850°C. The positive effects by heating the powders were confirmed by the experiments conducted.

Keywords: High pressure; High temperature; Shock waves; Shock consolidation of powders

1. Introduction

Shock consolidation has been studied for densifying hard ceramic powders which are very difficult to sinter by conventional techniques. The most difficult problem in the use of this technique is cracking in the recovered sample due to the propagation of strong shock waves. The use of high temperature is expected to enhance plastic deformation and surface melting of the powders, and to enable the reduction of the shock pressure required for the consolidation. Some experiments have been tried [1–5] using the flyer impact method.

In the present investigation, high-temperature shock consolidation using a converging underwater shock wave assembly [6] was conducted up to an experimental temperature of 850°C, and attempts were made to consolidate some ceramic powders under various conditions.

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2. Experimental apparatus and conditions

Fig. 1 shows the set-up for high-temperature shock consolidation. After heating the powders at the condition shown in Fig. 1(a), the furnace cover was removed, and the upper part including explosive and converging underwater shock-wave assembly was lowered and set just above the heated powder part as shown in Fig. 1(b). The explosive was detonated within 3 s after the final set, and the system was designed to push up the upper explosive part again in case of emergency.

Detail of the underwater shock-wave assembly is shown in Fig. 2. Converging angle was decided at 20° based on analytical [7] and experimental results conducted at room temperature [6]. The experimental conditions are listed in Table 1. In the present investigation, TiB\textsubscript{2}, c-BN and their mixed powders were tried to be consolidated at various conditions. Initial thickness of powders $D_p$ was fixed at 10 mm and their packing density was about...
60 vol%. Two types of explosive, SEP and PBX, were used, and detonation velocities were 7.0 and 8.4 km/s, respectively.

3. Results and discussion

The pressure distribution just above the powder part were calculated by finite-difference method [7] as shown in Fig. 3(a) and (b) for explosive SEP. The calculated results showed very uniform pressure distribution when the converging angle was 20° in contrast to the result when the angle was 30°. The maximum pressure 17.1 GPa for 20° was increased due to a converging effect from 11.0 GPa for initial underwater shock pressure applied from detonation of the explosive used. The maximum water pressure under a converging angle 20° for PBX was estimated at 26.0 GPa.

Fig. 4 shows an example of cross-sectional area and microstructure obtained by the high temperature experiment for TiB$_2$/c-BN mixed composite. Few cracks were observed from Fig. 4(a), and the microstructure shown in Fig. 4(b) suggested that the softer component TiB$_2$ powders was well deformed around the harder component c-BN powders. The c-BN powders were not well deformed, keeping their rectangular structure without generating cracks. The hardness values measured
### Experimental conditions

<table>
<thead>
<tr>
<th>Number</th>
<th>Powders (size)</th>
<th>Temperature</th>
<th>Explosive</th>
</tr>
</thead>
<tbody>
<tr>
<td>#RC1</td>
<td>c-BN 70 wt% (12–22 μm)</td>
<td>RT</td>
<td>SEP</td>
</tr>
<tr>
<td></td>
<td>TiB₂ 30 wt% (1–2 μm)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>#HC1</td>
<td>TiB₂ 30 wt% (1–2 μm)</td>
<td>973 K</td>
<td>SEP</td>
</tr>
<tr>
<td>#HC2</td>
<td>TiB₂ 30 wt% (36–54 μm)</td>
<td>1023 K</td>
<td>SEP</td>
</tr>
<tr>
<td>#HC3</td>
<td>TiB₂ 30 wt% (1–2 μm)</td>
<td>1123 K</td>
<td>PBX</td>
</tr>
<tr>
<td>#HC4</td>
<td>c-BN 60 wt% (36–54 μm)</td>
<td>1123 K</td>
<td>PBX</td>
</tr>
<tr>
<td>#HC5</td>
<td>TiB₂ (1–2 μm)</td>
<td>1023 K</td>
<td>SEP</td>
</tr>
<tr>
<td>#HC6</td>
<td>c-BN 80 wt% (36–54 μm)</td>
<td>1123 K</td>
<td>PBX</td>
</tr>
<tr>
<td></td>
<td>c-BN 20 wt% (0–2 μm)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Fig. 3. Calculated water pressure distribution just above powders (converging angle = 30° (a) and = 20° (b)), and maximum pressure distribution in powder part (c).

are listed in Table 2; the hardness showed very high values from 30 to 40 GPa for sample #HC3 which is shown in Fig. 4. The effects of temperature is clear; the low-temperature experiments #RC1 showed very low hardness values from 2 to 3 GPa.

The hardness normally decreased from the upper to the lower side, as shown in Table 2, due to
Fig. 4. Cross-sectional area (a) and microstructure (b) of TiB$_2$/c-BN composite (#HC3).

Table 2
Measured Vickers hardness. Hv-U, Hv-M and Hv-L are Vickers hardness for upper, middle and lower areas, respectively.

<table>
<thead>
<tr>
<th>Number</th>
<th>Hv-U/GPa</th>
<th>Hv-M/GPa</th>
<th>Hv-L/GPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>#RC1</td>
<td>2.8</td>
<td>2.7</td>
<td>2.3</td>
</tr>
<tr>
<td>#HC1</td>
<td>18.3</td>
<td>12.0</td>
<td>14.9</td>
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<tr>
<td>#HC2</td>
<td>26.1</td>
<td>20.1</td>
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</tr>
<tr>
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<td>39.7</td>
<td>30.4</td>
<td>31.0</td>
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<tr>
<td>#HC4</td>
<td>28.8</td>
<td>35.5</td>
<td>30.5</td>
</tr>
<tr>
<td>#HC5</td>
<td>22.5</td>
<td>15.8</td>
<td>14.3</td>
</tr>
<tr>
<td>#HC6</td>
<td>34.0</td>
<td>40.5</td>
<td>30.5</td>
</tr>
</tbody>
</table>

a decrease in shock pressure towards the lower side. Maximum pressure distribution in the powder is shown in Fig. 3(c). In this figure, the water pressure at room temperature was calculated instead of the case for powders because high-temperature Hugoniot of powders are unknown. The calculated pressure decreases toward the lower side due to dissipation of shock energy which corresponds with the measured hardness value.

The experimental results showed that the number of cracks was significantly decreased by the use of high temperature in comparison with the experiments conducted at room temperature. It is suggested that this is mainly due to (a) softening of the powders, and (b) the difference in the temperature between undeformed interior and deformed surface areas as Kondo reported [8], and is schematically illustrated in Fig. 5. It was reported that the deformed powder surface was changed to melting layer which would enhance bonding between powders. The difference in initial and the melting temperatures is smaller in case of the high-temperature experiment, which reduces thermal residual stress which results in cracks through the powders.

TiB$_2$ and c-BN powders were also consolidated, and their hardness obtained under moderate

Fig. 5. Schematic illustration for formation of melting layer for room (a) and high-temperature (b) shock consolidation.
conditions also showed very high values as shown in Table 2, which almost correspond with their monolithic hardness.

In case of the use of small powders admixed with the larger powders as shown for c-BN powders (# HC6), the bonding between powders was significantly improved. Concentration of shock energy to the small powders having large particle surface area enables to improve the condition of the bonding. This is because the energy given to the powders is dissipated at the surface areas which mainly deforms.

The higher temperature experiments are preferable to the use of high shock pressure in obtaining a material having better mechanical property. The conditions should be chosen considering the softening of the powders used. For example, high-temperature hardness of c-BN bulk material was measured for different temperature [9], and the dynamic hardness was decreased from about 40 GPa at room temperature to about 20 GPa at 800–900°C. Since the pressure required for the consolidation almost corresponded with their Vickers hardness [10], it is easily expected that the shock pressure required at above 800°C decreases to almost half of that for consolidating the powders at room temperature.

4. Conclusions

Shock consolidation of powders at elevated temperature was performed using a converging underwater shock-wave assembly. Experiments were conducted for TiB₂, c-BN and their mixed powders at different temperatures up to 850°C. The samples successfully recovered under moderate conditions showed few cracks due to softening of the powders at high temperature, and due to the effect of decrease in their thermal difference between molten surface and interior of the powders.

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References