A novel assembly used for hot-shock consolidation

P Chen¹ and Q Zhou
State Key Laboratory of Explosion Science and Technology, Beijing Institute of Technology, Beijing 100081, China

E-mail: pwchen@bit.edu.cn

Abstract. A novel assembly was developed for hot-shock consolidations of powders. The underwater shock wave and the high-temperature preheating, which are considered as two effective ways to eliminate cracks, were combined in the system. In this work, a SHS (self-propagating high-temperature synthesis) reaction mixture was used as chemical furnace to preheat the precursor powder, and the water column as well as the explosive attached to it was detached from the furnace by a solenoid valve fixed on the slide guide. When the precursor powders were preheated to the designed temperature, the solenoid valve was switched on, and then the water column and the explosive slid down along the slide guide by gravity. At the moment the water container contacted with the lower part, the explosive was initiated, and the generated shock wave propagated through the water column to compact the powders. So the explosive and water column can be kept cool during the preheating process. The intensity of shock wave loading can be adjusted by changing the heights of water column. And the preheating temperature is controlled in the range of 700~1300 °C by changing the mass of the SHS mixture. In this work, pure tungsten powders and tungsten-copper mixture were separately compacted using this new assembly. The pure tungsten powder with a grain size of 2 μm were compacted to high density (96 %T.D.) at 1300 °C, and the 90W-10Cu (wt pct) mixtures were compacted to 95.3 %T.D. at 970 °C. The results showed that both samples were free of cracks. The consolidated specimens were then characterized using SEM analysis and micro-hardness testing.

1. Introduction
Shock consolidation is considered as a promising method for fabricating hard-to-consolidate materials, such as refractory alloys and ceramics, because the shock wave generates extremely high pressure and high temperature at the surface of particles. However, cracks generated by tensile stresses and thermal residual stresses under the rapid process limit the industrialization of this technique. In order to eliminate cracks, preheating and low shock pressures are desirable [1]. The preheating of powders is expected to enhance plastic deformation and surface melting of powders, and to enable the reduction of the shock pressure required [2]. Hokamoto et al. show the cracks being eliminated by preheating in the shock consolidation of high-speed steel powders [3]; and Kecskes et al. fabricated W-Ti alloys without cracks using hot-shock consolidation [4]. A prolonged duration and uniform distribution of shock pressure, which can be achieved by using the underwater shock wave, are also beneficial for the consolidation of crack-free samples. Recently explosive compactions using underwater shock wave

¹ To whom any correspondence should be addressed.
have been reported; and the authors claimed that samples without cracks had been successfully obtained [5-7].

In this work, a new assembly combining the underwater shock wave and chemical furnace is modified from the assembly developed at Beijing Institute of Technology [8-10]. In these assemblies, the sample powders are preheated by a large amount of heat released via a self-propagating high-temperature synthesis (SHS) reaction. The reaction mixture is placed around the sample to form a chemical furnace, so the powder sample can be heated and consolidated in the same fixture. Pure tungsten and tungsten-copper composites are successfully consolidated and the performance and advantages of this assembly are discussed.

2. Experimental Procedure

Figure 1 shows the schematic illustrations of the new assembly, which consists of two main components: a loading part including the explosive and a water column, and a recovery part including the sample powder, the SHS compact and the SHS reaction vessel. The intensity of shock wave can be adjusted by altering the thickness of water column which is sealed in a mild steel container. The reaction vessel is not only used to contain the chemical furnace but also used for the recovery of the samples. The SHS compact is made of SHS mixture via uniaxially pressing, as an annulated column with a cavity in its center for the sample capsule. The powders are pressed into a 304 stainless steel capsule with two pistons on both sides to propagate the loading wave. The water column as well as the explosive attached to it was detached from the reaction vessel by a solenoid valve fixed on the slide guide, as shown in figure 1. After the SHS compact was ignited by the electric wire coil, the heat generated by the reaction of the SHS began to preheat the powders. So the explosive and water column was kept cool during the preheating process. As soon as the powders bed reached the fixed temperature, the solenoid valve was switched on, and then the water column and the explosive slid down along the slide guide by gravity. At the moment the water container contacted with the reactive vessel, the explosive was initiated, and the generated shock wave propagated through the water column to compact the powders.

In our experiment, the explosive used to generate shock wave is nitro methane, with a detonation velocity of 6.3 km/s and a detonation pressure of 11.9 GPa. The TiO2-C-Al-Fe2O3 powders were blended in a certain mass ratio and dry-mixed as the SHS reaction mixture. This mixture is a highly exothermic reactive system, when ignited, will generate a high temperature higher than 2000 K. W powder with average particle size of 2 μm and purity of >99 % and Cu powder with particle size of 2 μm and purity of >99 % were used. The W and Cu powders were mixed in a weight ratio of 9:1 using a planetary ball-mill at a speed of 300 rpm for 2 h.

The temperature measurements were carried out before the shock loading, to determine the ignition time for explosive and to evaluate the temperature distribution in the powder. As shown in figure 2, at 175 to 180 seconds, the powder bed reaches the highest temperature and becomes isothermal at the same time. The density of the recovered sample was measured by Archimedes’ method. The microstructure was investigated using light microscopy and scanning electron microscopy (SEM). The hardness as well as the modulus was also tested in the nano-indentation experiments. The pressure applied to powders was predicted by numerical simulation using AUTODYN-2D. The experimental conditions and results were listed in table 1 including the conditions for the consolidation of pure tungsten and W-Cu powder at various temperatures.
Table 1. Experimental conditions and results of as recovered samples.

| Sample | Preheating Temperature [°C] | Pressure [GPa] | Modulus [GPa] | Hardness [GPa] | Density [%T.D.] |
|--------|-----------------------------|----------------|---------------|---------------|----------------|
| W-1    | 1300                        | 3.1            | 247           | 3.4           | 96.7           |
| W-2    | 1100                        | 3.1            | 121           | 2.4           | 84.5           |
| W-5    | 1300                        | 2.3            | 189           | 2.6           | 88.5           |
| W-Cu-8 | 970                         | 3.1            | 269           | 5.9           | 95.3           |
| W-Cu-9 | 770                         | 4.3            | 189           | 3.7           | 90.0           |
| W-Cu-10| 1028                        | 3.1            | 243           | 5.1           | 92.0           |

Figure 1. Schematic diagram of the new assembly: 1-detonator; 2-explosive; 3-solenoid valve; 4-water column; 5-water container; 6-piston; 7-powder; 8-SHS compact; 9-SHS reaction vessel; 10-spall plates.

Figure 2. Typical result of temperature measurement during preheating phase.

3. Results and Discussion
Figure 3 shows the representative picture of W-1 sample after being machined out from the capsule. Few cracks can be observed for both samples, as shown in figure 3. Figure 4 shows the surface morphology of the pure tungsten samples. It can be seen that for all the pure W samples, the mean grain size is still nearly the same as the initial powder size. That owes to the short preheating period of 2 mins before consolidation and the extremely high cooling rates during shock consolidation. It is also observed that some particles were crushed into smaller pieces by the ultra-high strain rate, which is common during the void collapse of brittle particles under shock consolidation. The dark areas observed in figure 4(b) and figure 4(c) were the result of weak bonding, most of which were generated as the particle debonds from adjacent particles. Some voids can be observed in W-2, as shown in figure 4(b). The explanation is that the plastic deformation of powders was not enough to fill the voids between the particles due to the low preheating temperature and pressure as the W-2 was subjected to.

Figure 3. The recovered W samples: W-1.

Figure 4. The optical micrographs of the W samples: (a) W-1; (b) W-2; (c) W-5.

Figure 5. Fracture morphologies of the pure W-1 sample (a) and magnified view of W-1 (b).
The fracture morphology in figure 5a shows the nature of brittle inter-granular of the pure tungsten sample. The void collapse and surface melting are the sintering mechanisms of pure W by HSC. The former one contributes to the densification behaviour of powders, and the latter one is responsible for the bonding between the particles. As shown in figure 5a, the voids collapse due to the plastic deformation of tungsten is the dominant mechanism of the densification. However, the localized melting zone can also be clearly distinguished in figure 5b, indicated by the arrow.

The W-Cu composites were also consolidated using the new assembly. No cracks were observed on the recovered sample, as shown in figure 6. The experimental results and the optical micrographs of the W-Cu samples are shown in table 1 and figure 7, respectively. Figure 7 shows that the distribution of Cu becomes more uniform as the preheating temperature increasing. Further investigation of hot shock consolidation of W-Cu composites using this novel assembly is still underway.

4. Conclusion
A novel assembly was developed for hot-shock consolidations of powders. The underwater shock wave and the high-temperature preheating caused by a SHS reaction, which are considered as two effective ways to eliminate cracks, were combined in the system.

Pure tungsten powders and tungsten-copper mixture were separately compacted using this new assembly. The pure tungsten powder with a grain size of 2 μm was compacted to a high density (96 %T.D.) at 1300 ºC, and the 90W-10Cu (wt pct) mixtures were compacted to 95.3 % at 970 ºC. The recovered samples showed that both samples were free of macro-cracks, which indicate the validity of this new assembly in minimizing cracking.

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