HP-SPS at very high pressure (6 GPa) for hard and super hard materials

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Highlights

- Innovative pulsed electric current process under very high pressure by (HP-SPS) equipment
- Belt type apparatus with large volume chamber permits to manufacture: 6 GPa-1800°C.
- Pulsed electric current based on Fuji electric generator comparable to conventional.
- Sintering of hard materials with low chemical reactivity such as carbide, nitride and boride.

1. Introduction

Electric current assisted/activated sintering (ECAS) provides wide options for fabrication of nanostructured ceramics. Spark plasma sintering (SPS) is one of the well- known ECAS technique. SPS provides options to synthetize materials with controlled properties either with powders or with pre-compacted samples. When the sample used for sintering is conductive the heat is generated by Joule heating and while the sample is insulator the heat is transmitted from the conductive mold to the sample. In conventional SPS processes different operating parameters such as pulse duration, heating/cooling rate, dwell time, temperature or pressure can be varied to obtain the desired results. There are numerous studies highlight the influence of these different parameters. By controlling the aforesaid parameters it is possible to modulate the sample dependent parameters such as the particle size, crystal structure and chemical composition of the sintered compact. Influence of pressure on the mechanical properties of hard and super-hard materials are well known. The thermo-mechanical properties of classical SPS mold allows to reach pressure up to 120 MPa. This pressure limit can be further extended when special type die systems are used. Double stage mold which combine SiC and graphite can allow to reach high pressures up to 1 GPa. Recently there are few reports on the possibility to combine pulsed power source with high pressure toroidal type apparatus reaching pressure up to 7.8 GPa and temperature of 1950°C [1]. In the present work, we will describe about the in-house built high pressure SPS equipment that enables to reach maximum pressure of 6 GPa and its versatility for syntheses and sintering of hard and super-hard materials.

2. Methods

This work presents the influence of pressure activated by pulsed electric current in the case of conventional spark plasma sintering (SPS) system from Fuji Electronic Industrial Co., Ltd., Japan (Dr. Sintering Series SPS-515S) compared to very high pressure spark plasma sintering (HP-SPS) (Figure 1) system based on the same electric generator manufacturer system from Fuji Electronic Industrial (SCM-3000 model). SCM-3000 model electric generator is capable to reach 3000 A for 10 V with unit pulse has a of 3.3 ms duration similar to a conventional SPS from Fuji. A unique HP-SPS equipment [2] with Belt type apparatus used for this work will be described as a technological advance in ceramic processing. Technical efforts have pushed the conventional SPS pressure up to 1 GPa with temperature limited to 900°C [3]. HP-SPS-Belt conditions could be pushed to 6 GPa and 1800°C with this innovative equipment.

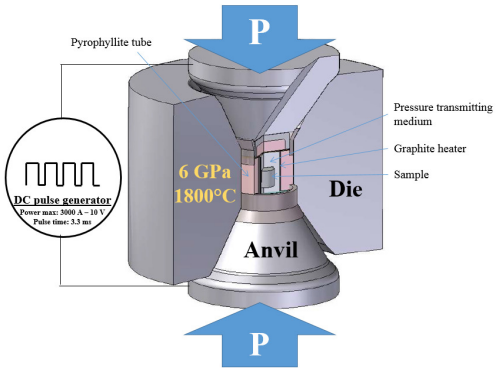

Figure 1: Scheme of the HP-SPS system

3. Results and discussion

The Belt apparatus is one of the most used high pressure generation system. We use Belt apparatus to convert the system to be reach very high pressure. In Belt apparatus, great care should be taken during the design of the die assembly in order to get an optimized tangential stress-strain response on the inside of the die during the load application. Further Belt is an eye-blind apparatus and pressure and temperature were calibrated before the experiments. The design of our Belt apparatus allows to reach pressure until 6 GPa with 1000 tons load of the hydraulic press. The application of very high pressure, where pressure is a driving force like temperature can help to sinter hard materials which presents very low reactivity such as oxide, carbide, boride, nitride or presents thermal instability for materials which exist only under pressure such as cubic phase of C (diamond), SiC (moissanite), BN, diamond. The pressure effect also help avoiding thermal decomposition of materials and reaching extreme densities of ceramics. This work focus the interest of HP-SPS on hard, super-hard and ultra-hard materials; materials such as oxide like Al_2O_3 in comparison with conventional HP-HT [4] and as well as carbide (WC, SiC), boride (TiB_2, BN), nitride (Si_3N_4) and diamond will be presented.

Conclusions

We have designed and performed a unique HP-SPS-Belt equipment either for classical large volume purpose to function with classical SPS functionalities needing high pressure conditions. We will present the instrumentation and versatility of the designed HP-SPS-Belt equipment. Various hard and super hard materials sintered by this novel HP-SPS-Belt equipment will be presented and discussed in detail.

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Keywords
SPS; High pressure; Hard materials