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Preparation of Boron Carbide Ceramics by Sintering: A Literature Review

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Abstract

Boron carbide (B_4C) has a series of excellent properties such as high hardness, high melting point, small bulk density, acid and alkali corrosion resistance, and has been widely used in machinery, chemical industry, military, aerospace and other fields. The research progress and application of boron carbide materials at home and abroad in recent years are reviewed. The effects of sintering processes such as non-pressing sintering, hot pressing sintering, hot isostatic sintering, discharge plasma sintering and microwave sintering and sintering additives (elemental, oxide, carbide) on densification of boron carbide materials are analyzed, and the development of boron carbide materials is prospected.

Keywords

Boron carbide ceramics, Sintering, Grain size.

1. INTRODUCTION

By describing the sintering mechanism and process of boron carbide ceramics, it is concluded that different sintering conditions have different effects on the products during the whole sintering process. According to the requirements of the main performance of B4C ceramics, several main factors affecting sintering were obtained from the mechanism and experimental conclusions. In order to improve the mechanical properties of B4C ceramic materials, the formation of B4C grains and the increase of density in the sintering process can be better controlled.

2. SINTERING METHOD

2.1. Non-pressure sintering

Non-pressure sintering is a conventional sintering method, it refers to the state of atmospheric pressure, the forming body placed in the atmospheric sintering furnace, according to the design of the sintering temperature and certain atmosphere conditions, the body through physical and chemical changes, become stable volume, with certain performance of the compact block, this is the most commonly used, is also the most simple way of sintering. In order to promote sintering, it is usually necessary to add sintering additives, such as metal elemental, oxide, boride, etc., which can remove the oxide layer on the surface of boron carbide particles, reduce sintering temperature, inhibit grain growth, improve mechanical properties, etc.

But the non-pressure sintered boron carbide ceramics because of the simple process, low cost, sintering conditions are not too much requirements, widely used in large-scale industrial production, but because of the high sintering temperature, the grain is easy to overgrow, so that the sintering process is difficult to control, the product performance is unstable. Boron carbide

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micro-powder and silicon carbide micro-powder can be used as the main raw materials [1], and a certain proportion of carbon black can be added. The results show that with the increase of carbon black, the relative density, compressive strength and Vickers hardness of the sample increase first and then decrease, and the standard deviation of volume density basically decreases first and then increases. The properties of boron carbide ceramics produced according to the optimal formula are obviously improved.

2.2. Hot pressing sintering

Hot pressing sintering is to load evenly mixed boron carbide powder into a high strength graphite mold, placed in the hot pressing sintering furnace, in the sample heating at the same time for pressure, so as to form dense and uniform ceramic products, is a sintering method combining the forming and sintering process. However, the hot pressing sintering of boron carbide ceramics has excellent microstructure, high densification degree and excellent mechanical properties, but it still has some defects such as complex process, small production scale and low efficiency.

Using B4C-5%-10%Calcium-Silicon as raw material, Tkachenko G[2] et al. used the hot pressing technology (the hot pressing temperature ranges from 1950 to 2100℃, With the pressure of 30MPa and the holding time of 5-10min), the B4C composites with the flexural strength of up to 300MPa and the cracking strength of up to 3MP×$m^{1/2}$ were prepared. Mei Z A[3] et al sintered boron carbide ceramics by hot pressing. The research results showed that when the sintering temperature was higher than the critical temperature of grain growth (2100℃), the increase of pressure would lead to obvious grain size anomaly, and found that normal grain growth was controlled by the grain boundary diffusion mechanism, while abnormal grain growth followed the Bose Einstein grain growth law.

Boron carbide ceramics can also be prepared by oscillating hot pressing sintering [4]. The relative density of boron carbide ceramics increases gradually with the increasing of oscillating pressure. When the oscillating pressure is 50±10 MPa, it is close to complete density, and the hardness and fracture toughness of the ceramics also increase gradually.

2.3. High temperature isostatic pressing sintering

High temperature isostatic pressing sintering (HIP) is a sintering method in which the ceramic powder, billet or prefired body is subjected to uniform force in various methods and densification is achieved by applying pressure to the sample in a gaseous medium at high temperature. The usual gaseous medium is argon or nitrogen, and the pressure is usually tens to hundreds of mpa. This sintering method can produce ceramic parts with high strength, high density, complex shape and precise size at low temperature.

In the process of industrial production, the matrix with certain residual pores is generally obtained by the preparation process of non-pressure sintering, and then the ceramic material with the highest degree of densification is obtained by the hot isostatic sintering process to eliminate the residual pores [5]. Cho[6] et al. obtained completely dense boron carbide ceramics after hot isostatic pressing treatment of boron carbide ceramics with a density of 93.0% obtained by sintering without pressure. Larsson[7] et al. prepared boron carbide ceramics by hot isostatic pressing sintering technology, and studied the effect of adding boron elemental on boron carbide sintering. The results show that adding boron can effectively increase the density of the product, control the grain size and increase the density.

2.4. Spark Plasma Sintering

Spark plasma sintering (SPS) is a rapid sintering method that applies specific sintering power source and pressing pressure to sintered powder, which integrates activation, hot pressing and heating to produce high performance ceramics. It has the characteristics of fast heating rate,

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short sintering time, uniform structure, fine grain and so on. In addition, SPS can make particles produce local high temperature and melt the surface through effective discharge, which can remove the oxide film on the surface of particles.

The optimal sintering temperature, applied pressure, holding time and heating rate of dense boron carbide ceramics prepared by discharge plasma sintering are 1800℃, 80 MPa, 12 min and 100℃/min, respectively. Sintering temperature and applied pressure are the main factors affecting the densification of boron carbide ceramics. When the sintering temperature is lower than 1800℃, increasing the sintering temperature can obviously improve the density of boron carbide ceramics. Once the sintering temperature reaches 1850℃ or above, boron carbide sample will oversintered due to the presence of sintering additives, resulting in the reduction of boron carbide density. The relative density of boron carbide can be significantly increased by applying pressure, but the effect of pressure on the density of boron carbide is not obvious when the density of boron carbide reaches a certain level. The holding time and heating rate are also factors affecting the densification of boron carbide ceramics. With the increase of the heating rate, the pulse current increases and the heat generated by the Joule effect of the sample increases, which is conducive to the densification of boron carbide ceramics. Prolonging the holding time is conducive to discharging the pores in the sample, thus forming dense areas in some regions prefertively and improving the density of boron carbide ceramics.

Wang Ling et al. [8] used the discharge plasma sintering process to prepare boron carbide powder rich in boron as raw material. Under the conditions of 2 200 ℃ and 5 min sintering time, the sample with good mechanical properties was prepared with the density of 99.6%, the bending strength of 550.1 MPa and the hardness of 39.52 GPa. The experimental results, combined with the conspeculations of Wang Xiu-fen et al. [9], show that during the SPS sintering process, due to the spontaneous heat inside the powder, the growth of the grain is greatly inhibited. When the discharge in the grain gap, the local high temperature of several thousand degrees will be generated, causing the evaporation and melting of the grain surface, thus promoting the sintering of the material This is why "the measured temperature is much lower than the actual temperature, so SPS can achieve low temperature and rapid sintering".

2.5. Microwave sintering

Microwave sintering is a new sintering process, which has many advantages such as fast temperature rise, uniform temperature field, energy saving and environmental protection. Its main working principle is the sintering process that uses the interaction between microwave and material to heat the material itself through dielectric loss, which is a feasible way to rapidly sintering high density boron carbide ceramics [10].

Boron carbide ceramics were prepared from ultrafine boron carbide powders by microwave sintering. The results show that boron carbide ceramics with an average particle size of about 20 μm and a density of 95% were prepared under the condition of being heated by 2.45GHz microwave radiation to 2000 ℃ for 12 min. There were twins and microcracks in the microstructure, which played a toughening effect, and the energy consumption of microwave sintering was 18% lower than that of induction hot pressing [11].

The sintering temperature of microwave sintering is low, usually below 1500 ℃, the heating rate is fast, the temperature field is uniform, and the microwave sintering energy efficiency is high, which is a kind of green sintering method. However, microwave sintering equipment is expensive, and has high requirements on the type of materials and the uniformity of samples. Therefore, microwave parameters need to be specially designed according to the properties of samples, and the operation is complicated, so it is difficult to apply in a large scale at present.

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3. CONCLUSION

Regarding the future development direction of sintering densification of boron carbide materials, there are mainly: mixed application of various sintering processes, make full use of the advantages and disadvantages of various sintering processes, learn from each other, combine a variety of sintering processes, reduce sintering densification temperature, reduce sintering costs, improve the density of sintering products, so as to expand the application of boron carbide materials; Using composite sintering additives, different kinds of sintering additives have their special properties of promoting sintering and performance improvement, by adding composite sintering additives, comprehensive use of the advantages of different kinds of sintering additives, in the case of maximum density, without damaging the excellent mechanical properties of boron carbide, and improve the bending strength, fracture toughness and other properties of boron carbide ceramics.

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