Research Progress of Silicon carbide Powder Shaping Method

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Abstract. In order to meet the high-end silicon carbon powder materials requirements of its morphology and size, etc, the shaping process of the powder particle is becoming more and more attention. In this paper, the advance in research of silicon carbon powder of plastic methods is briefly reviewed, and a new shaping method of molten salt method is proposed for SiC powder particles.

1. Introduction

The silicon carbide materials are applied widely with chemical stability, good wear resistance, high thermal conductivity and other properties [1], especially in the field of high-end applications such as energy, aerospace and automotive industries, marine engineering [2]. But the shape of silicon carbide powder particle used in the industry have many sharp edges, uneven surface, spherical degree is poor, the performance level of silicon carbide materials prepared is not neat, severely limits the high-end application of silicon carbide. So, it is very important for the high end application of silicon carbide powder particles to shape to meet its high performance in a more demanding environment.

The shaping methods of SiC powder include mechanical method and physical chemical method.Mechanical method change the shape of the particles by an external grinding force on particles, low cost, relatively simple process, high yield, however, its essence is grinding method, with severe pollution, loud noise, and the purity of the powder obtained is not high; physical-chemical method prepares powder by chemical reaction or phase transformation, by the ions, atoms, molecules after nucleation and crystals grown,it has complex production process, high cost and low yield. SiC powder plastic commonly used mechanical methods having milling method [3], stirring ball milling, jet mill method, vibration mill grinding, mechanical impact grinding method and attrition milling ; physical-chemical methods include oxidation corrosion combined milling process , sodium hydroxide etching method.

2. Mechanical shaping method

2.1 Milling shaping method

High energy ball milling is generally used in ball milling methods. Wang Hongtao, Chen Xiao, etal [4] studied the effect of the milling time, ball material ratio, rotational the comprehensive analysis shows that the optimal parameters for milling time 40h , ball ratio 10, the speed of 200r/min, on this basis, use the powder metallurgy technique for preparing bulk materials or preparing metal matrix composites materials as a ceramic reinforced phase [5]. In addition, in milling of silicon and graphite mixing process [6], by the use of high-resolution electron microscopy (HREM), in some areas C atoms diffuse into the silicon crystal, and forming SiC particles by local self-excited reaction in other areas, compared to direct milling of silicon carbide particles, the particle size of SiC obtained by this method is not uniform, thus further study is needed to improve particle agglomeration.

2.2 Stirring mill shaping method

From the point of reducing the particle size, wet grinding is superior to dry grinding. Shaping the

SiC powder particles, the Qi Limin [7] used the wet grinding and shaping of coarse particles of silicon carbide, 85% to 95% of the primary particles are ground to a particle size smaller than 1μm and not require re-classification, but it can form particles with certain strength, that the powder tapers to a certain extent, the specific surface area and specific surface energy also increase to a certain extent, the emergence of a "reverse grinding point",when the powder is blocked thinner tapering to a certain extent, even particles become the larger agglomeration, to overcome this shortcoming, the preliminary solution is to add appropriate dispersant [8], reducing the surface energy of the ultra-fine powder, to improve dispersion of ultrafine powder . The powder product has stable performance, good consistency, and the level of similar foreign products. It can also solve the problem of aggregation by shaping process itself.

2.3 Attrition milling shaping method

The sanding machine uses sand media with the same composition as powder and polymer coating with the same powder body, without introducing impurities, and without pickling, and good grinding effect, and high efficiency. Therefore attrition milling method has certain advantages over other methods [9].

Chen Haya [10] using a sand grinder for shaping silicon carbide particles, obtained the fine shaping effect for no more than 100μm particles is obvious, in addition,the tap density of 100μm plastic powder can reach 1.96 g/cm^3, compared with the original powder tap density were respectively increased by 14.62%, an oxygen content of 0.73% (mass fraction). Huang Zhengren etal [11] also adopts attrition milling method to prepare the median diameter of 0.47μm powders with narrow size distribution and oxygen content of less than 1.5% (mass fraction) of ultrafine SiC powder,from 7.3μm high-purity SiC after 18h grinding, grain refinement is obvious in attrition grinding method has improved.

2.4 Fluidized bed impinging air-flow crushing shaping method

Guo Xingzhong etal [12] using a fluidized bed opposed jet milling method of SiC powder superfine treatment found that the higher the working pressure, the more obvious shaping effect, when the working pressure is 0.6MPa, the average particle size of SiC powder reduced from 3.01μm to 0.75μm, the proportion of particle size less than 2.5μm rose from 82.86% to 99.85%, the particle size distribution mainly changes from 0.4~1.5μm and 8~23μm of bimodal distribution into the 0.4~1.2μm unimodal distribution, distribution width decreased from 25.125 to 0.833; mainly flaky, small block particles exist after shaping, with uniform distribution, bulk particle before plastic existed prior to the disappearance, agglomeration significantly reduced compared with that before shaping.

In summary, the shaping of the SiC particles by using ball milling method, particle size can meet the requirement, but the introduction of impurities Fe and excess oxygen, after milling it generally requires acid or alkali washing treatment, increasing environmental pressures; attrition milling can reduce the introduction of impurities volume, the particle size is uniform, but the processing efficiency is low, the grain size still can not fully meet the requirements of the properties and structure of ceramic materials, and these two kinds of ball milling methods are difficult to solve the problem of powder agglomeration; jet mill method can solve the agglomeration.

3. Physical -chemical shaping method

3.1Oxidation corrosion combined milling technology shaping method

The principle of oxidation corrosion combined milling process [13] is firstly the oxidation treatment [14] for irregular local place of SiC particles, the silicon carbide particle edges are completely oxidized, producing silicon dioxide, then through ball milling shaping of silicon carbide, the oxide edges of silicon carbide particles after oxidation is grinded to remove. In the control of the shape of unshaped refractory additives silicon carbide powder, Shi Ce etal [15] using this method of processing silicon carbide powder shape,test through oxidation techniques at different temperatures and holding time,combined with the optimal milling parameters, test results show that the silicon carbide powder particle angularity at 1150℃was oxidized for 5h, and then use the difference in hardness of SiO_2 and SiC under optimum milling process parameters to mill, obtaining silicon

carbide powder of the optimal length and sphericity. By combining the optimum milling process and the oxidation technology, the silicon carbide powder with the required shape can be obtained.

Based on the study oxidative corrosion combined milling process, Shi Ce [16] also proposed a new method, namely sodium hydroxide etch process method [17], the reaction equations of the process involved are as follows:

$$\text{Oxidation reaction}\begin{cases}2SiC(s)+3O_2\rightarrow 2SiO_2(s)+2CO(g) & (1)\\ SiC(s)+2O_2\rightarrow SiO_2(s)+CO_2(g) & (2)\end{cases}$$

$$\text{Corrosion reaction}\quad 2NaOH+SiO_2\rightarrow Na_2SiO_3+H_2O \quad (3)$$

Combined with sodium hydroxide etching process and oxidation combustion process , the optimal boiling temperature of 100-120℃ and the best boiling time is 5h, but this method has many errors in the course of the experiment, the shaping effect has many variables, and the resulting product purity is low, so hope the researchers to do more relevant research in this field, make the research more controllable, maneuverability and practical significance.

4. New method of molten salt shaping silicon carbide powder

With the increasing requirements of the silicon carbide material, the renewal cycle of plastic technology is getting shorter, how to improve the effect of plastic surgery and reduce energy consumption has become an important issue in the current research. Both mechanical method and physical-chemistry method of silicon carbide powder particles, have their own advantages and disadvantages, but also generally exist adhesion, cohesion, excessive wear and tear, easy to burst, reunion, electrostatic and other difficult problems. Molten salt method using one or more molten salt as a medium has a lower viscosity and higher heat capacity, in which the particles migrate and diffuse quickly, morphology of the powder can achieve maximum degree of control. the application of molten salt method currently used in the manufacture of inorganic powder is more than [18], but the research on silicon carbide particle shaping is seldom used. Based on this, this paper presents a new method of molten salt method of silicon carbide particle shaping, previously much of feasibility analysis and study has been carried out, initially identified the shaping process and the corresponding process parameters, and achieved good shaping effect. The author by the molten salt method of β-SiC particle [19,20] with D50 14μm and the bulk density 1.2g/cm^3 conducted particles shaping research, mainly studied the effect of molten salt system, ratio, temperature and time parameters on the shaping effect, the results show that the complex salt shaping effect is obvious; some molten salt at 800℃ for 16h can get the β-SiC powder with D50 of 50μm and bulk density of 1.98g / cm^3; and some melt salt at 750℃ for 24 hours can obtain powder with D50 of 1μm and a bulk density of 1.56g / cm^3. From the experimental results, molten salt method is easy to control the shape and size of the powder particles, structure of powder obtained is homogeneous, less agglomeration, the method is simple and easy to operate, can greatly improve the bulk density of the powder particles, it is expected to become a new technology for β-SiC powder particles shaping.

5. Summary

To meet the demands of aerospace, military and other high-tech fields for high-performance silicon carbide materials, we must first prepare silicon carbide powder with good performance, mechanical and physical-chemical method is difficult to prepare micro-nano SiC powder with good degree of sphericity, molten salt method is easy to obtain powder particles of a narrow particle size distribution,good sphericity, a new way required for the preparation of high performance silicon carbide powder material has opens up.

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