Notice

DESCRIPTION CN120681759A

A rapid preparation method for silicon carbide particles based on flash Joule heating process

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一种基于闪光焦耳热工艺的碳化硅颗粒快速制备方法

[0001]

Technical Field

技术领域

[n0001]

This invention relates to the field of silicon carbide preparation, and specifically to a rapid method for preparing silicon carbide particles based on flash Joule heating.

本发明涉及碳化硅制备领域，具体涉及一种基于闪光焦耳热工艺的碳化硅颗粒快速制备方法。

[0003]

Background Technology

背景技术

[n0002]

As one of the strategic materials of the new era, silicon carbide has a series of excellent physical and chemical properties, such as excellent mechanical properties, high thermal conductivity, small coefficient of thermal expansion, stable chemical inertness, low oxidation rate, high breakdown field strength, and can be used as a reinforcing material. Silicon carbide has great application prospects in fields such as structural materials, coatings, catalysts, and high-power devices.

作为新时代战略材料之一，碳化硅具有一系列优异的物化属性，如力学性能出色、热导率高、热膨胀系数小、具有稳定的化学惰性、氧化速度低、击穿场强高且可以作为增强材料使用，碳化硅在诸如结构材料、覆膜涂层、催化剂、高功率器件等领域具有巨大的应用前景。

Numerous methods exist for preparing silicon carbide particles, but many of these methods, such as carbothermal reduction and chemical vapor deposition, are often limited in practical applications due to issues such as harsh reaction conditions, complex processes, high costs, or long preparation cycles. They cannot meet the requirements of modern society for silicon carbide material preparation.

碳化硅颗粒的制备方法层出不穷，但如碳热还原法、化学气相沉积法等诸多方法往往伴随如反应条件苛刻、工艺复杂、成本高或是制备周期长等问题限制了在实际制备方面的应用，无法满足现代社会对碳化硅材料制备领域的要求。

Therefore, a simple method is needed to prepare silicon carbide materials that requires no complicated conditions, has a shorter cycle, and is economical and practical.

因此需要采用一种无需复杂条件、周期更短、经济实用的简易方法制备出碳化硅材料。

[n0003]

The flash Joule heating method is an emerging material preparation technique with advantages such as simple process, short cycle and low cost. It is currently mainly used in the

field of high-efficiency graphene preparation. By using the voltage stored in the device capacitor, preparation can be completed in less than one second, which is a high efficiency that no other silicon carbide preparation method has. The method has a wide range of raw materials to choose from, low material cost and equipment cost, and has good flexibility and economy.

闪光焦耳热法是一种新兴的材料制备手段，具有工艺简单、周期短、成本低的优势，目前主要应用在高效制备石墨烯领域，通过将设备电容中储存的电压可在不到一秒的时间内完成制备，这是所有其他碳化硅制备方法所不具备的高效性；方法可选原料多、材料成本及设备成本低，具有很好的灵活性与经济性。

[0006]

Summary of the Invention

发明内容

[n0004]

To address the aforementioned problems, this invention provides a rapid preparation method for silicon carbide particles based on flash Joule heating. By simply mixing raw materials in a solid phase and loading them into a custom-made quartz tube, which is then clamped onto a

device base, silicon carbide particles can be prepared in less than one second. This method features a short preparation cycle, low process cost, and simple process steps, effectively addressing the shortcomings of many current silicon carbide preparation technologies.

针对上述问题，本发明提供了一种基于闪光焦耳热工艺的碳化硅颗粒快速制备方法，通过将原材料简单固相混合装载至定制石英管中，夹放置于设备底座上，在不到一秒的时间内制备得到碳化硅颗粒；该方法具有制备周期短、工艺成本低、工艺步骤简单等特点，可以很好的补足现在许多碳化硅制备技术所欠缺的短板。

[n0005]

To achieve the above-mentioned objectives, the technical solution adopted by this invention is as follows:

为了达到上述发明目的，本发明采用的技术方案如下：

[0009]

This solution provides a method for preparing silicon carbide particles based on flash Joule heating technology, including the following steps:

本方案提供一种基于闪光焦耳热工艺的碳化硅颗粒制备方法，包括以下步骤：

[0010]

Step (1): Weigh the raw materials (which can be Si powder and conductive carbon black) at a mass ratio of 1:1 (with excess carbon component), and then mix and grind the raw materials evenly in a mortar or a ball mill.

步骤（1）：将原料（可为Si粉末与导电炭黑）按照质量比1:1（碳组分过量）称量后，可通过研钵将原料混合研磨均匀或通过球磨机将原料充分混合；

[0011]

Step (2): Fill the mixed raw materials into a custom quartz tube (6mm or 12mm in diameter) and place graphite/copper electrodes of the same diameter at both ends to complete the filling;

步骤（2）：将混合原料装填入定制石英管内（直径为6mm或12mm）并在两端放入相同直径的石墨电极/铜电极完成装填；

[0012]

Step (3): Place the quartz tube containing the raw material on the equipment base, adjust the sample resistance to 1-3Ω by controlling the electrode pressure on both sides of the quartz tube (the resistance may vary depending on the carbon source), place it in the reaction vessel, connect the positive and negative electrodes, and evacuate the reaction vessel.

步骤（3）：将装有原料的石英管放置在设备底座上，通过控制石英管两侧电极压力调节样品电阻至1-3Ω（根据不同碳源电阻可有所不同）放置在反应釜中连接正负电极，并将反应釜抽真空；

[0013]

Step (4): Charge the flash Joule heating device to the set voltage of 150V, set the voltage release time to 400ms, and perform flash discharge operation on the sample to complete the reaction;

步骤（4）：对闪光焦耳热设备进行充电至设定电压150V，设置电压施放时间为400ms，对样品进行闪热放电操作，完成反应；

[0014]

Step (5): After waiting for the discharge in step (4) to be completed and cooled, the product in the quartz tube is taken out. At this time, the main components of the product are silicon carbide and graphene. Pure phase silicon carbide is obtained by calcining the product in air at 800°C to remove the graphene in the product.

步骤（5）：在等待步骤（4）放电完毕并冷却后，取出石英管中的产物；此时产物中主要成分为碳化硅与石墨烯，通过对产物在空气中以800°C煅烧以去除产物中石墨烯得到纯相碳化硅。

[n0006]

Furthermore, the silicon-containing raw materials used in step (1) of this method include, but are not limited to, Si powder, SiO₂ powder, SiO powder, silicon-containing industrial waste, etc.; the carbon-containing raw materials include, but are not limited to, carbon black, graphite, porous carbon biochar, industrial carbon-containing waste, etc.

进一步地，本方法中步骤（1）中使用的含硅原材料包括但不限于Si粉、SiO₂粉、SiO粉、含硅工业废料等；含碳原料包括但不限于炭黑、石墨、多孔碳生物炭、工业含碳废料等。

[n0007]

Furthermore, the customized reaction quartz tube size in step (2) of this method includes, but is not limited to, 6mm and 12mm, which can be selected according to the volume of the raw materials.

进一步地，本方法中步骤（2）的定制反应石英管尺寸包括但不限于6mm、12mm，可根据原料体积选择。

[n0008]

Furthermore, in step (4) of this method, the discharge voltage parameter can be 100~180V, preferably 150V, and the discharge time can be 300~1000ms, preferably 400ms.

进一步地，本方法中步骤（4）中放电电压参数可以为100~180V，优选为150V，放电时间可选300~1000ms，优选为400ms。

[n0009]

Furthermore, in step (5) of this method, the purification and calcination parameters are 800°C for 30 min.

进一步地，本方法中步骤（5）中纯化煅烧参数为800°C，30min。

[n0010]

In summary, the present invention has the following advantages:

综上所述，本发明具有以下优点：

[0020]

This invention provides a rapid preparation method for silicon carbide particles based on flash Joule heating. Using low-cost flash Joule heating equipment, silicon carbide particles can be prepared in less than one second without pretreatment, catalyst, template, or cooling time, and the process steps are very simple.

本发明提供了一种基于闪光焦耳热工艺的碳化硅颗粒快速制备方法，采用成本低廉的闪光焦耳热设备，在无需预处理、无需催化剂、无需模板、无需冷却时间的情况下，在不到一秒内即可制备得到碳化硅颗粒，并且工艺步骤十分简单。

[n0011]

The flash Joule heating process used in this invention has strong scalability. Industrial-scale production can be achieved by modifying the sample introduction method or increasing the size of the quartz tube. At the same time, there are various raw material options, and

inexpensive industrial waste can be used for preparation, which greatly reduces costs and is environmentally friendly.

本发明所使用的闪光焦耳热工艺有很强的扩展性，通过修改进样方式或扩大石英管尺寸可实现产业化规模生产，同时原材料的选择多样，可以使用廉价的工业废料进行制备，大大减少了成本的同时具有环保意义。

[0022]

Attached Figure Description

附图说明

[n0012]

Figure 1 is a simplified flowchart of a rapid preparation method for silicon carbide particles based on flash Joule heating process;

图1为一种基于闪光焦耳热工艺的碳化硅颗粒快速制备方法的简单流程图；

[0024]

Figure 2 is a circuit diagram of the flash heating device;

图2为闪热设备电路原理图；

[0025]

Figure 3 is a TEM image of the silicon carbide particles obtained in Example 1.

图3为实施例1最后所得碳化硅颗粒TEM图；

[0026]

Figure 4 shows an HRTEM image of the silicon carbide particles obtained in Example 1 (SiC as determined by interplanar spacing measurement).

图4为实施例1所得碳化硅颗粒HRTEM图(经过晶面间距测量为SiC)

[0027]

Detailed Implementation

具体实施方式

[n0013]

To make the objectives, technical solutions, and advantages of this invention clearer, the invention will be further described in detail below with reference to embodiments.

为了使本发明的目的、技术方案及优点更加清楚明白，以下结合实施例，对本发明进行进一步详细说明。

It should be understood that the specific embodiments described herein are only for explaining the present invention and are not intended to limit the present invention. That is, the described embodiments are only some embodiments of the present invention, and not all embodiments.

应当理解，此处所描述的具体实施例仅用以解释本发明，并不用于限定本发明，即所描述的实施例仅仅是本发明一部分实施例，而不是全部的实施例。

[n0014]

Therefore, the following detailed description of the embodiments of the present invention is not intended to limit the scope of the claimed invention, but merely to illustrate selected embodiments of the invention.

因此，以下对提供的本发明的实施例的详细描述并非旨在限制要求保护的本发明的范围，而是仅仅表示本发明的选定实施例。

Based on the embodiments of the present invention, all other embodiments obtained by those skilled in the art without inventive effort are within the scope of protection of the present invention.

基于本发明的实施例，本领域技术人员在没有做出创造性劳动的前提下所获得的所有其他实施例，都属于本发明保护的范围。

[0030]

Example

实施例

[n0015]

This example provides a rapid preparation method for silicon carbide particles based on flash Joule heating, as shown in Figure 1, including the following steps:

本例提供一种基于闪光焦耳热工艺的碳化硅颗粒快速制备方法，如图1所示，包括以下步骤：

[0032]

Step (1): Weigh 20mg of each of the raw materials (Si powder and conductive carbon black) at a mass ratio of 1:1 (excess carbon component), and then mix and grind the raw materials evenly and thoroughly using a mortar and pestle.

步骤（1）：将原料（Si粉末与导电炭黑）按照质量比1:1各称量20mg（碳组分过量）后，通过研钵将原料混合研磨均匀充分混合；

[0033]

Step (2): Fill the mixed raw materials into a custom quartz tube (6 mm in diameter) and place copper electrodes of the same diameter at both ends to complete the filling;

步骤（2）：将混合原料装填入定制石英管内（直径为6mm）并在两端放入相同直径的铜电极完成装填；

[0034]

Step (3): Place the quartz tube containing the raw material on the equipment base, adjust the sample resistance to 1-3Ω by controlling the electrode pressure on both sides of the quartz tube, place it in the reaction vessel, connect the positive and negative electrodes, and evacuate the reaction vessel.

步骤（3）：将装有原料的石英管放置在设备底座上，通过控制石英管两侧电极压力调节样品电阻至1-3Ω,放置在反应釜中连接正负电极，并将反应釜抽真空；

[0035]

Step (4): Charge the flash Joule heating device to the set voltage of 150V, set the voltage release time to 400ms, and perform flash discharge operation on the sample to complete the reaction;

步骤（4）：对闪光焦耳热设备进行充电至设定电压150V，设置电压施放时间为400ms，对样品进行闪热放电操作，完成反应；

[0036]

Step (5): After waiting for the discharge in step (4) to be completed and cooled, the product in the quartz tube is taken out. At this time, the main components of the product are silicon carbide and graphene. Pure phase silicon carbide is obtained by calcining the product in air at 800°C to remove the graphene in the product.

步骤（5）：在等待步骤（4）放电完毕并冷却后，取出石英管中的产物；此时产物中主要成分为碳化硅与石墨烯，通过对产物在空气中以800°C煅烧以去除产物中石墨烯得到纯相碳化硅。

[0037]

Example

实施例

[n0016]

This example provides a rapid preparation method for silicon carbide particles based on flash Joule heating, as shown in Figure 1, including the following steps:

本例提供一种基于闪光焦耳热工艺的碳化硅颗粒快速制备方法，如图1所示，包括以下步骤：

[0039]

Step (1): Weigh 20mg of each of the raw materials (SiO₂ powder and conductive carbon black) at a mass ratio of 1:1 (excess carbon component), and then mix and grind the raw materials evenly and thoroughly using a mortar and pestle.

步骤（1）：将原料（SiO₂粉末与导电炭黑）按照质量比1:1各称量20mg（碳组分过量）后，通过研钵将原料混合研磨均匀充分混合；

[0040]

Step (2): Fill the mixed raw materials into a custom quartz tube (6 mm in diameter) and place copper electrodes of the same diameter at both ends to complete the filling;

步骤（2）：将混合原料装填入定制石英管内（直径为6mm）并在两端放入相同直径的铜电极完成装填；

[0041]

Step (3): Place the quartz tube containing the raw material on the equipment base, adjust the sample resistance to 1-3Ω by controlling the electrode pressure on both sides of the quartz tube, place it in the reaction vessel, connect the positive and negative electrodes, and evacuate the reaction vessel.

步骤（3）：将装有原料的石英管放置在设备底座上，通过控制石英管两侧电极压力调节样品电阻至1-3Ω,放置在反应釜中连接正负电极，并将反应釜抽真空；

[0042]

Step (4): Charge the flash Joule heating device to the set voltage of 150V, set the voltage release time to 400ms, and perform flash discharge operation on the sample to complete the reaction;

步骤（4）：对闪光焦耳热设备进行充电至设定电压150V，设置电压施放时间为400ms，对样品进行闪热放电操作，完成反应；

[0043]

Step (5): After waiting for the discharge in step (4) to be completed and cooled, the product in the quartz tube is taken out. At this time, the main components of the product are silicon carbide and graphene. Pure phase silicon carbide is obtained by calcining the product in air at 800°C to remove the graphene in the product.

步骤（5）：在等待步骤（4）放电完毕并冷却后，取出石英管中的产物；此时产物中主要成分为碳化硅与石墨烯，通过对产物在空气中以800°C煅烧以去除产物中石墨烯得到纯相碳化硅。