Notice

DESCRIPTION CN118359200A

A method for purifying quartz sand using flash Joule heat treatment

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一种利用闪速焦耳热处理提纯石英砂的方法

[0001]

Technical Field

技术领域

[n0001]

This invention relates to the field of quartz sand purification, and specifically to a method for purifying quartz sand using flash Joule heat treatment.

本发明涉及石英砂提纯领域，具体涉及一种利用闪速焦耳热处理提纯石英砂的方法。

[0003]

Background Technology

背景技术

[n0002]

Flash Joule heating (FJH) technology has become a hot topic in the field of new material preparation in recent years. Its characteristic is that it can heat and cool materials to thousands of degrees in a very short time, such as milliseconds.

闪速焦耳加热技术(FJH)近年来成为新型材料制备领域的热点技术，其特点是能在毫秒极的时间内对材料进行上千度的升降温处理。

This technology has great potential in the preparation of high-entropy materials, the production of high-quality graphene, the recovery of battery electrode materials, and the synthesis of special catalysts.

该技术在高熵材料的制备、优质石墨烯的生产、电池电极材料的回收、特殊催化剂的合成等方面大展拳脚。

In addition, Hu Liangbing and others improved the technology and successfully applied it to the sintering of special ceramics, realizing the ultra-fast heating and cooling treatment of ceramic materials using this technology.

另外，胡良兵等人改进该技术后成功应用于特种陶瓷的烧结，实现了该技术对陶瓷材料的超快速升降温处理。

[n0003]

Currently, the purification process of quartz sand in my country usually includes a combination of techniques such as water quenching, magnetic separation, flotation, acid leaching, and complexation.

目前，我国提纯石英砂工艺通常包括水淬、磁选、浮选、酸浸、络合等技术手段的组合。

Existing processes have very limited effectiveness in removing inclusions and isomorphic impurities from the interior of quartz sand materials.

现有的工艺对石英砂材料内部的包裹体杂质和类质同象杂质去除效果十分有限。

To remove internal impurities from quartz sand, a feasible method is to use extreme cooling and heating techniques to cause the inclusions in the material to burst, thereby creating more cracks. Patents CN1562743A, CN102180584A, and CN109081352A respectively employ "cold bursting," "microwave thermal bursting," and "cold-hot collision bursting" methods to attempt to improve the purification effect by creating cracks on the surface of quartz sand. However, these methods have not completely solved the problems of insufficient purification purity and energy consumption caused by long-term extreme heat and deep cryogenic technology.

为了去除石英砂的内部杂质，通过极冷极热技术来使材料中的包裹体发生爆裂而产生更多裂纹是一个可行的方法。 专利CN1562743A、CN102180584A以及CN109081352A中分别采用了“冷爆裂”、“微波热爆裂”和“冷热对撞爆裂”的方式试图通过在石英砂表面创造裂缝提升提纯效果，但仍未完全解决提纯的纯度不足以及长时间极热深冷技术带来的能耗问题。

[0006]

Summary of the Invention

发明内容

[n0004]

The purpose of this invention is to provide a method for purifying quartz sand using flash Joule heat treatment, which is time-saving, energy-efficient, and yields high-purity quartz sand.

本发明的目的是提供一种利用闪速焦耳热处理提纯石英砂的方法，该方法耗时短，能耗低，获得的石英砂纯度高。

[n0005]

To achieve the above objectives, the present invention employs the following technical solution:

为达到上述目的，本发明采用以下技术方案予以实现：

[n0006]

A method for purifying quartz sand using flash Joule heat treatment includes the following steps:

一种利用闪速焦耳热处理提纯石英砂的方法，包括以下步骤，

[n0007]

(1) The quartz sand raw material is ball-milled and sieved to obtain quartz sand A with a particle size of less than 100 mesh;

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(1)将石英砂原料进行球磨，筛分，获得粒径小于100目的石英砂A；

[n0008]

(2) Place quartz sand A on a conductive substrate of a Joule furnace, heat it with a voltage of 15-30V and a current of 50-250A for 200ms-1min, and then cool it for no more than 10s to perform flash Joule heat treatment. After completion, quartz sand B is obtained.

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(2)将石英砂A置于焦耳炉的导电基底上，采用15～30V的电压50～250A的电流加热200ms～1min后冷却不超过10s，进行闪速焦耳热处理，完成后得到石英砂B；

[n0009]

(3) Quartz sand B is put into an acid pickling tank and acid leached at 65-85°C with inorganic mixed acid with a concentration of not less than 4mol/L. After completion, material C is obtained.

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(3)将石英砂B投入酸洗罐中使用浓度不低于4mol/L的无机混酸在65-85°C下酸浸，完成后得到物料C；

[n0010]

(4) Wash and dry material C to obtain quartz sand D;

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(4)洗涤物料C并干燥，得到石英砂D；

[n0011]

(5) After mixing the quartz sand D with the chlorinating agent, add it to the chlorination roasting furnace and chlorinate and roast at 1400-1600°C. After completion, cool to obtain high-purity quartz sand.

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(5)将石英砂D与氯化剂混合后加入氯化焙烧炉中，在1400～1600°C下氯化焙烧，完成后冷却得到高纯石英砂。

[n0012]

Furthermore, in step (1), the silica content in the quartz sand raw material is not less than 98%.

进一步地，所述步骤(1)中石英砂原料中二氧化硅含量不低于98%。

[n0013]

Furthermore, in step (2), the conductive substrate material is one of carbon cloth, carbon paper, graphite paper, or carbon fiber cloth.

进一步地，所述步骤(2)中导电基底材料为碳布、碳纸、石墨纸或碳纤维布中的一种。

[n0014]

Furthermore, in step (2), there is a conductive substrate on the top and bottom of the Joule furnace, and the quartz sand A is located between the two substrates.

进一步地，所述步骤(2)中焦耳炉上下各有一块导电基底，石英砂A处于两基底之间。

[n0015]

Furthermore, in step (2), the flash Joule heat treatment is carried out in a vacuum environment, and the treatment is performed 1 to 6 times.

进一步地，所述步骤(2)中闪速焦耳热处理在真空环境中进行，处理次数为1～6次。

[n0016]

Furthermore, in step (3), the inorganic mixed acid is composed of hydrochloric acid, nitric acid and hydrofluoric acid, wherein the concentration of hydrochloric acid is not less than 3 mol/L, the concentration of nitric acid is not less than 0.5 mol/L and the concentration of hydrofluoric acid is not less than 0.5 mol/L.

进一步地，所述步骤(3)中无机混酸由盐酸、硝酸、氢氟酸组成，其中盐酸浓度不低于3mol/L、硝酸浓度不低于0.5mol/L、氢氟酸浓度不低于0.5mol/L。

[n0017]

Furthermore, the acid leaching time in step (3) shall not be less than 4 hours.

进一步地，所述步骤(3)中酸浸时间不少于4h。

[n0018]

Furthermore, the acid leaching process in step (3) is assisted by ultrasonic treatment at low frequency (20kHz) and high frequency (40kHz) and pressure treatment at 0.2 to 0.5MPa.

进一步地，所述步骤(3)中酸浸过程有中酸浸过程有低频20kHz、高频40kHz的超声处理辅助与0.2到0.5MPa的加压处理辅助。

[n0019]

Furthermore, the chlorinating agent used in step (5) is either a gaseous chlorinating agent or a solid chlorinating agent. The gaseous chlorinating agent is one or two of HCl and Cl₂, and the solid chlorinating agent is one or more of NaCl, KCl, and NH₄Cl. When using a gaseous chlorinating agent, the quartz sand should be chlorinated and roasted in an atmosphere where the volume ratio of chlorinating agent to nitrogen is 1:1 to 1:4. When using a solid chlorinating agent, 1.5% to 4% of the mass fraction of chlorinating agent is mixed with the quartz sand and then chlorinated and roasted.

进一步地，所述步骤(5)采用的氯化剂选用气体氯化剂或固体氯化剂，气体氯化剂为HCl、Cl₂中的一种或两种，固体氯化剂为NaCl、KCl、NH₄Cl中的一种或多种，使用气体氯化剂应当控制石英砂处于氯化剂与氮气体积比为1:1～1:4的气氛中进行氯化焙烧，使用固体氯化剂时将石英砂质量分数1.5%4%的氯化剂与石英砂混合后进行氯化焙烧。

[n0020]

Furthermore, in step (5), the chlorination roasting is carried out using microwave heating, and the holding time is 2 to 5 hours.

进一步地，所述步骤(5)中氯化焙烧采用微波加热，保温时间为2～5h。

[n0021]

Compared with the prior art, the present invention has the following beneficial effects:

相对于现有技术而言，本发明具有以下有益效果：

[n0022]

1.

1.

Based on the weak electrical conductivity of quartz sand, this invention selects a conductive substrate as the support for Joule heating. Considering the poor heat transfer performance

and high melting point of quartz sand, a parameter setting strategy of higher voltage and current and longer processing time is adopted to reasonably configure the parameters of the Joule furnace.

本发明依据石英砂弱导电的材料特性，选择了导电基底承载的方式进行焦耳加热，依据石英砂材料传热性能较差、熔点高的特点采用了较高电压电流、较长处理时间的参数设置策略，对焦耳炉进行了合理的参数配置。

[n0023]

2.

2.

This invention utilizes the extremely rapid heating and cooling speed of flash Joule heat treatment technology to perform extremely hot and cold treatment on quartz sand in a very short time, greatly shortening the processing time and effectively saving the huge energy consumption caused by the long heating and cooling process required by traditional extreme heating and cooling.

本发明利用闪速焦耳热处理技术升降温速度极快的特点，在极短的时间内对石英砂进行极热极冷处理，处理时间大幅缩短，从而有效节省了传统极热极冷需长时间升降温处理带来的巨大能耗。

[n0024]

3.

3.

The rapid heating and cooling rates of flash Joule heating technology far exceed those of traditional cold and heat treatment processes. The extremely fast heating and cooling rates can better promote the bursting of quartz sand inclusions and the formation of surface cracks, thereby effectively exposing internal impurities and facilitating the thoroughness of subsequent impurity removal. Compared with traditional purification processes, this improves the purification effect of quartz sand.

闪速焦耳热技术的快速升降温速度远远大于传统冷热处理工艺，极快的升降温速度，能更好促进石英砂包裹体的爆裂以及表面裂纹的生成，进而能有效暴露其内部的杂质，便于后续除杂的彻底性，相较传统提纯工艺改善了石英砂的提纯效果。

[n0025]

4.

4.

Compared with existing technologies that purify quartz sand through hot and cold collision blasting, the SiO₂ content in the purified quartz sand is about 99.9950%, while the quartz sand prepared by this invention has a purity of 99.998%, reaching a high-end level. Moreover, the operation is simple and easy to implement, the process is streamlined, it is easy to promote, and it has obvious economic and practical value.

相较现有技术通过冷热对撞爆裂法提纯石英砂，其提纯后的石英砂中SiO₂含量在99.9950%左右，而本发明制备的石英砂纯度在99.998%，达到了高端水平，且操作简单易行，工序精简，易于推广，具有明显的经济实用价值。

[n0026]

Furthermore, this invention employs the feature of heating surrounded by a dual conductive substrate, which ensures that the quartz sand is heated evenly and rapidly. It also uses a vacuum environment to avoid minor reactions between the protective gas and the material at excessively high temperatures.

进一步地，本发明采用双导电基底包围加热的特点，使石英砂受热均匀、快速，并选用真空环境，避免过高温度下保护气体与物料的少量反应。

[n0027]

Furthermore, the present invention includes a hot-press acid leaching step, in which heating and pressurization are combined with ultrasonic treatment to further expand the cracks and defects formed on the quartz sand by Joule heating, thereby increasing the specific surface area of the material and effectively improving the leaching effect of impurities.

进一步地，本发明设置热压酸浸步骤，加热加压同时辅以超声处理进一步扩大焦耳加热处理在石英砂上形成的裂纹与缺陷，提升物料比表面积，有效提升杂质浸出效果。

[0031]

Attached Figure Description

附图说明

[n0028]

Figure 1 is a schematic diagram of the process flow of the present invention.

图1为本发明的工艺流程示意图。

[0033]

Detailed Implementation

具体实施方式

[n0029]

The present invention will be further described in detail below with reference to specific embodiments. These descriptions are for explanation purposes only and are not intended to limit the scope of the invention.

下面结合具体的实施例对本发明做进一步的详细说明，所述是对本发明的解释而不是限定。

[n0030]

Referring to Figure 1, the present invention uses coarsely treated quartz sand as raw material. Taking quartz sand ore produced in a certain place in Shaanxi Province as an example, it is subjected to one or more of the following methods: crushing, water quenching, magnetic

separation, flotation, acid leaching, complexation, etc. to remove surface impurities and perform coarse purification. The silica content in the coarsely purified quartz sand is not less than 98%.

参见图1，本发明采用经过粗处理后的石英砂作为原料，以陕西省内某地所产的石英砂矿为例，将其经过破碎、水淬、磁选、浮选、酸浸、络合等方式中的一种或者多种组合处理掉表面附着的杂质进行粗提纯，粗提纯后的石英砂中二氧化硅含量不低于98%。

[n0031]

The method for purifying the coarsely treated quartz sand using flash Joule heat treatment includes the following steps:

利用闪速焦耳热处理提纯上述粗处理后的石英砂方法包括以下步骤：

[n0032]

(1) The coarsely treated quartz is ball-milled and sieved to obtain quartz sand A with a particle size of less than 100 mesh.

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(1)将粗处理后的石英进行球磨，筛分，获得粒径小于100目的石英砂A；

[n0033]

(2) Place the quartz sand A obtained in step (1) on a conductive substrate of a Joule furnace for flash Joule heat treatment. Specifically, use a voltage of 15-30V and a current of 50-250A to heat for 200ms-1min and then cool for no more than 10s. Quartz sand A is treated once by heating and cooling down to below 50°C. Repeat the treatment 1-6 times. The ultra-fast heating and cooling treatment promotes the rupture of gas-liquid inclusions in the quartz sand, causing more cracks to appear on its surface. After completion, quartz sand B is obtained.

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(2)将步骤(1)所得石英砂A置于焦耳炉的导电基底上进行闪速焦耳热处理，具体采用15～30V的电压、50～250A的电流加热200ms～1min后冷却不超过10s，石英砂A经过加热升温后冷却降温至低于50°C为一次处理，重复处理1-6次，通过超快速的升降温处理促进石英砂内气液包裹体破裂，使其表面产生更多裂纹，完成后得到石英砂B；

[n0034]

(3) Quartz sand B is put into an acid pickling tank and acid-leached at 65-85°C for at least 4 hours using an inorganic mixed acid composed of hydrochloric acid, nitric acid and hydrofluoric acid. During acid leaching, ultrasonic treatment at low frequency 20kHz and high frequency 40kHz and pressure treatment at 0.2-0.5MPa are used to remove the internal impurities exposed by the quartz sand after flash Joule heat treatment. The concentration of

hydrochloric acid in the inorganic mixed acid is not less than 3mol/L, the concentration of nitric acid is not less than 0.5mol/L and the concentration of hydrofluoric acid is not less than 0.5mol/L. After completion, material C is obtained.

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(3)将石英砂B投入酸洗罐中，使用由盐酸、硝酸和氢氟酸组成的无机混酸在65-85°C下酸浸至少4h，酸浸时配合低频20kHz、高频40kHz的超声处理辅助与0.2～0.5MPa的加压处理辅助，去除石英砂在闪速焦耳热处理后暴露出的内部杂质，所使用的无机混酸中盐酸浓度不低于3mol/L、硝酸浓度不低于0.5mol/L、氢氟酸浓度不低于0.5mol/L，完成后得到物料C；

[n0035]

(4) Wash and dry material C to obtain quartz sand D;

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(4)洗涤物料C并干燥，得到石英砂D；

[n0036]

(5) After mixing the quartz sand D with the chlorinating agent, add it to the chlorination roasting furnace and perform high-temperature chlorination roasting. The chlorination roasting temperature is 1400-1600°C and the holding time is 2-5h. During the roasting process, the quartz sand undergoes multiple phase transformations, which can further enrich the microcracks in the quartz sand. At the same time, it activates isomorphous impurities and

accelerates their diffusion to the surface. It reacts with the chlorinating agent to generate easily volatile substances that overflow from the roasting furnace. After completion, cool to obtain high-purity quartz sand.

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(5)将石英砂D与氯化剂混合后加入氯化焙烧炉中，高温氯化焙烧，氯化焙烧温度为1400～1600℃，保温时间为2～5h，焙烧过程中石英砂经历多次相变，能进一步丰富石英砂中的微裂纹，同时活化类质同象杂质并加速其向表面的扩散，与氯化剂反应生成易于挥发的物质溢出焙烧炉，完成后冷却得到高纯石英砂。

[n0037]

Example 1:

实施例1：

[n0038]

After coarse purification, the silica content of the quartz sand raw material is 98%, and the main impurities are Fe: 80ppm and Al: 50ppm.

石英砂原料经过粗提纯后：二氧化硅含量为98%，主要杂质含量为Fe：80ppm，Al：50ppm。

[n0039]

(1) The coarsely purified quartz sand raw material is ball-milled and sieved to obtain quartz sand A with a particle size of less than 100 mesh.

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(1)将粗提纯后的石英砂原料进行球磨，筛分，获得粒径小于100目的石英砂A；

[n0040]

(2) Quartz sand A was placed on a carbon cloth conductive substrate in a Joule furnace and subjected to flash Joule heat treatment twice. The Joule furnace parameters were set as follows: voltage 25V, current 200A, heating time 30s, cooling time 10s. After completion, quartz sand B was obtained.

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(2)将石英砂A置于焦耳炉的碳布导电基底上进行闪速焦耳热处理2次，设置焦耳炉参数为：电压25V、电流200A、加热时间30s，冷却10s，完成后得到石英砂B；

[n0041]

(3) Quartz sand B is put into an acid pickling tank and acid-leached at 65°C for 6 hours with inorganic mixed acid. The concentration of hydrochloric acid in the inorganic mixed acid is not less than 3 mol/L, the concentration of nitric acid is not less than 0.5 mol/L, and the

concentration of hydrofluoric acid is not less than 1 mol/L. During acid leaching, ultrasonic treatment at low frequency of 20 kHz and high frequency of 40 kHz and pressure treatment at 0.2 MPa are used as auxiliary treatments. After completion, material C is obtained.

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(3)将石英砂B投入酸洗罐中用无机混酸在65°C酸浸6h，无机混酸中盐酸浓度不低于3mol/L、硝酸浓度不低于0.5mol/L、氢氟酸浓度不低于1mol/L，酸浸时配合低频20kHz、高频40kHz的超声处理辅助与0.2MPa的加压处理辅助，完成后得到物料C；

[n0042]

(4) Wash and dry material C to obtain quartz sand D;

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(4)洗涤物料C并干燥，得到石英砂D；

[n0043]

(5) After mixing quartz sand D with 2.5% ammonium chloride by mass, put it into a microwave chlorination roasting furnace and perform high-temperature chlorination roasting at 1500°C for 5 hours. After the material is cooled, high-purity quartz sand product is obtained.

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(5)将石英砂D与其质量分数2.5%的氯化铵混合后投入微波氯化焙烧炉，在1500°C进行高温氯化焙烧5h，待物料冷却即得高纯石英砂产品。

[n0044]

The obtained product was tested and found to have a purity of 99.998%, with the main impurities being Fe: 0.7 ppm and Al: 11 ppm.

对所得产品检测：其纯度为99.998%，主要杂质含量Fe：0.7ppm、Al：11ppm。

[n0045]

Example 2:

实施例2：

[n0046]

(1) The same quartz sand raw material as in Example 1 was ball-milled and sieved to obtain quartz sand A with a particle size of less than 100 mesh;

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(1)将同实施例1一样的石英砂原料进行球磨，筛分，获得粒径小于100目的石英砂A；

[n0047]

(2) Quartz sand A was placed on a carbon cloth conductive substrate in a Joule furnace and subjected to flash Joule heat treatment twice. The Joule furnace parameters were set as follows: voltage 15V, current 120A, heating time 30 seconds, cooling time 5s. After completion, quartz sand B was obtained.

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(2)将石英砂A置于焦耳炉的碳布导电基底上进行闪速焦耳热处理2次，设置焦耳炉参数为：电压15V、电流120A、加热时间30秒，冷却5s，完成后得到石英砂B；

[n0048]

(3) Quartz sand B was put into an acid pickling tank and acid-leached at 65°C for 6 hours with inorganic mixed acid. The inorganic mixed acid contained hydrochloric acid with a concentration of 3 mol/L, nitric acid with a concentration of 0.5 mol/L, and hydrofluoric acid with a concentration of 1 mol/L. During acid leaching, ultrasonic treatment with a low frequency of 20 kHz and a high frequency of 40 kHz and pressure treatment with 0.4 MPa were used as auxiliary treatments. After completion, material C was obtained.

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(3)将石英砂B投入酸洗罐中使用无机混酸在65°C酸浸6h，无机混酸中盐酸浓度3mol/L、硝酸浓度0.5 mol/L、氢氟酸1mol/L，酸浸时配合低频20kHz、高频40kHz的超声处理辅助与0.4MPa的加压处理辅助，完成后得到物料C；

[n0049]

(4) Wash and dry material C to obtain quartz sand D;

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(4)洗涤物料C并干燥，得到石英砂D；

[n0050]

(5) After mixing quartz sand D with 2.5% ammonium chloride by mass, put it into a microwave chlorination roasting furnace and perform high-temperature chlorination roasting at 1500°C for 5 hours. After the material is cooled, high-purity quartz sand product is obtained.

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(5)将石英砂D与其质量分数2.5%的氯化铵混合后投入微波氯化焙烧炉，在1500°C进行高温氯化焙烧5h，待物料冷却即得高纯石英砂产品。

[n0051]

The obtained product was tested and found to have a purity of 99.998%, with the main impurities being Fe: 0.9 ppm and Al: 16 ppm.

对所得产品检测：其纯度为99.998%，主要杂质含量Fe：0.9ppm、Al：16ppm。

[n0052]

Example 3:

实施例3：

[n0053]

(1) The same quartz sand raw material as in Example 1 was ball-milled and sieved to obtain quartz sand A with a particle size of less than 100 mesh;

---

(1)将同实施例1一样的石英砂原料进行球磨，筛分，获得粒径小于100目的石英砂A；

[n0054]

(2) Quartz sand A was placed on a carbon cloth conductive substrate in a Joule furnace and subjected to flash Joule heat treatment once. The Joule furnace parameters were set as follows: voltage 25V, current 200A, heating time 30s, cooling time 3s. After completion, quartz sand B was obtained.

---

(2)将石英砂A置于焦耳炉的碳布导电基底上进行闪速焦耳热处理1次，设置焦耳炉参数为：电压25V、电流200A、加热时间30s，冷却3s，完成后得到石英砂B；

[n0055]

(3) Quartz sand B was put into an acid pickling tank and acid-leached at 65°C for 6 hours with inorganic mixed acid. The inorganic mixed acid contained hydrochloric acid with a concentration of 3 mol/L, nitric acid with a concentration of 0.5 mol/L, and hydrofluoric acid with a concentration of 1 mol/L. During acid leaching, ultrasonic treatment with a low frequency of 20 kHz and a high frequency of 40 kHz and pressure treatment with 0.3 MPa were used as auxiliary treatments. After completion, material C was obtained.

---

(3)将石英砂B投入酸洗罐中使用无机混酸在65°C酸浸6h，无机混酸中盐酸浓度3mol/L、硝酸浓度0.5mol/L、氢氟酸1mol/L，，酸浸时配合低频20kHz、高频40kHz的超声处理辅助与0.3MPa的加压处理辅助，完成后得到物料C；

[n0056]

(4) Wash and dry material C to obtain quartz sand D;

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(4)洗涤物料C并干燥，得到石英砂D；

[n0057]

(5) After mixing quartz sand D with 2.5% ammonium chloride by mass, put it into a microwave chlorination roasting furnace and perform high-temperature chlorination roasting at 1500°C for 5 hours. After the material is cooled, high-purity quartz sand product is obtained.

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(5)将石英砂D与其质量分数2.5%的氯化铵混合后投入微波氯化焙烧炉，在1500°C进行高温氯化焙烧5h，待物料冷却即得高纯石英砂产品。

[n0058]

The obtained product was tested and found to have a purity of 99.997%, with the main impurities being Fe: 0.9 ppm and Al: 19 ppm.

对所得产品检测：其纯度为99.997%，主要杂质含量Fe：0.9ppm、Al：19ppm。

[n0059]

Example 4:

实施例4：

[n0060]

(1) The same quartz sand raw material as in Example 1 was ball-milled and sieved to obtain quartz sand A with a particle size of less than 100 mesh;

---

(1)将同实施例1一样的石英砂原料进行球磨，筛分，获得粒径小于100目的石英砂A；

[n0061]

(2) Quartz sand A was placed on a carbon cloth conductive substrate in a Joule furnace and subjected to flash Joule heat treatment three times. The Joule furnace parameters were set as follows: voltage 25V, current 200A, heating time 40s, cooling time 7s. After completion, quartz sand B was obtained.

---

(2)将石英砂A置于焦耳炉的碳布导电基底上进行闪速焦耳热处理3次，设置焦耳炉参数为：电压25V、电流200A、加热时间40s，冷却7s，完成后得到石英砂B；

[n0062]

(3) Quartz sand B was put into an acid pickling tank and acid-leached at 65°C for 6 hours with inorganic mixed acid. The inorganic mixed acid contained hydrochloric acid with a concentration of 3 mol/L, nitric acid with a concentration of 0.5 mol/L, and hydrofluoric acid

with a concentration of 1 mol/L. During acid leaching, ultrasonic treatment with a low frequency of 20 kHz and a high frequency of 40 kHz and pressure treatment with 0.2 MPa were used as auxiliary treatments. After completion, material C was obtained.

---

(3)将石英砂B投入酸洗罐中使用无机混酸在65°C酸浸6h，无机混酸中盐酸浓度3mol/L、硝酸浓度0.5 mol/L、氢氟酸1mol/L，酸浸时配合低频20kHz、高频40kHz的超声处理辅助与0.2MPa的加压处理辅助，完成后得到物料C；

[n0063]

(4) Wash and dry material C to obtain quartz sand D;

---

(4)洗涤物料C并干燥，得到石英砂D；

[n0064]

(5) After mixing quartz sand D with 2.5% ammonium chloride by mass, put it into a microwave chlorination roasting furnace and perform high-temperature chlorination roasting at 1500°C for 5 hours. After the material is cooled, high-purity quartz sand product is obtained.

---

(5)将石英砂D与其质量分数2.5%的氯化铵混合后投入微波氯化焙烧炉，在1500°C进行高温氯化焙烧5h，待物料冷却即得高纯石英砂产品。

[n0065]

The obtained product was tested and found to have a purity of 99.998%, with the main impurities being Fe: 0.6 ppm and Al: 10 ppm.

对所得产品检测：其纯度为99.998%，主要杂质含量Fe：0.6ppm、Al：10ppm。

[n0066]

Example 5:

实施例5：

[n0067]

(1) The same quartz sand raw material as in Example 1 was ball-milled and sieved to obtain quartz sand A with a particle size of less than 100 mesh;

---

(1)将同实施例1一样的石英砂原料进行球磨，筛分，获得粒径小于100目的石英砂A；

[n0068]

(2) Quartz sand A was placed on a carbon paper conductive substrate in a Joule furnace and subjected to flash Joule heat treatment 4 times under vacuum conditions. The Joule furnace parameters were set as follows: voltage 30V, current 50A, heating time 200ms, cooling time 1s. After completion, quartz sand B was obtained.

---

(2)将石英砂A置于焦耳炉的碳纸导电基底上，在真空条件下进行闪速焦耳热处理4次，设置焦耳炉参数为：电压30V、电流50A、加热时间200ms，冷却1s，完成后得到石英砂B；

[n0069]

(3) Quartz sand B was put into an acid pickling tank and acid-leached at 85°C for 4 hours with inorganic mixed acid. The inorganic mixed acid contained hydrochloric acid with a concentration of 4 mol/L, nitric acid with a concentration of 1 mol/L, and hydrofluoric acid with a concentration of 0.5 mol/L. During acid leaching, ultrasonic treatment with a low frequency of 20 kHz and a high frequency of 40 kHz and pressure treatment with a pressure of 0.4 MPa were used as auxiliary treatments. After completion, material C was obtained.

---

(3)将石英砂B投入酸洗罐中使用无机混酸在85°C酸浸4h，无机混酸中盐酸浓度4mol/L、硝酸浓度1mol/L、氢氟酸0.5mol/L，酸浸时配合低频20kHz、高频40kHz的超声处理辅助与0.4MPa的加压处理辅助，完成后得到物料C；

[n0070]

(4) Wash and dry material C to obtain quartz sand D;

---

(4)洗涤物料C并干燥，得到石英砂D；

[n0071]

(5) After mixing quartz sand D with 1.5% potassium chloride by mass, put it into a microwave chlorination roasting furnace and chlorinate at 1600°C for 3 hours. After the material is cooled, the high-purity quartz sand product is obtained.

---

(5)将石英砂D与其质量分数1.5%的氯化钾混合后投入微波氯化焙烧炉，在1600°C进行高温氯化焙烧3h，待物料冷却即得高纯石英砂产品。

[n0072]

The obtained product was tested and found to have a purity of 99.998%, with the main impurities being Fe: 0.9 ppm and Al: 13 ppm.

对所得产品检测：其纯度为99.998%，主要杂质含量Fe：0.9ppm、Al：13ppm。

[n0073]

Example 6:

实施例6：

[n0074]

(1) The same quartz sand raw material as in Example 1 was ball-milled and sieved to obtain quartz sand A with a particle size of less than 100 mesh;

---

(1)将同实施例1一样的石英砂原料进行球磨，筛分，获得粒径小于100目的石英砂A；

[n0075]

(2) Quartz sand A was placed on a graphite paper conductive substrate in a Joule furnace and subjected to flash Joule heat treatment 6 times under vacuum conditions. The Joule furnace parameters were set as follows: voltage 20V, current 100A, heating time 1min, cooling time 5s. After completion, quartz sand B was obtained.

---

(2)将石英砂A置于焦耳炉的石墨纸导电基底上，在真空条件下进行闪速焦耳热处理6次，设置焦耳炉参数为：电压20V、电流100A、加热时间1min，冷却5s，完成后得到石英砂B；

[n0076]

(3) Quartz sand B was put into an acid pickling tank and acid-leached at 70°C for 5 hours with inorganic mixed acid. The inorganic mixed acid contained hydrochloric acid with a concentration of 3 mol/L, nitric acid with a concentration of 2 mol/L, and hydrofluoric acid with a concentration of 2 mol/L. During acid leaching, ultrasonic treatment with a low frequency of 20 kHz and a high frequency of 40 kHz was used as an auxiliary treatment, and pressure treatment with 0.3 MPa was used as an auxiliary treatment. After completion, material C was obtained.

---

(3)将石英砂B投入酸洗罐中使用无机混酸在70°C酸浸5h，无机混酸中盐酸浓度3mol/L、硝酸浓度2mol/L、氢氟酸2mol/L，酸浸时配合低频20kHz、高频40kHz的超声处理辅助与0.3MPa的加压处理辅助，完成后得到物料C；

[n0077]

(4) Wash and dry material C to obtain quartz sand D;

---

(4)洗涤物料C并干燥，得到石英砂D；

[n0078]

(5) After mixing quartz sand D with sodium chloride and ammonium chloride at a mass fraction of 2%, put it into a microwave chlorination roasting furnace and perform high-temperature chlorination roasting at 1400°C for 3 hours. After the material is cooled, high-purity quartz sand product is obtained.

---

(5)将石英砂D与其质量分数2%的氯化钠和氯化铵混合后投入微波氯化焙烧炉，在1400°C进行高温氯化焙烧3h，待物料冷却即得高纯石英砂产品。

[n0079]

The obtained product was tested and found to have a purity of 99.998%, with the main impurities being Fe: 0.9 ppm and Al: 11 ppm.

对所得产品检测：其纯度为99.998%，主要杂质含量Fe：0.9ppm、Al：11ppm。

[n0080]

Example 7:

实施例7：

[n0081]

(1) The same quartz sand raw material as in Example 1 was ball-milled and sieved to obtain quartz sand A with a particle size of less than 100 mesh;

---

(1)将同实施例1一样的石英砂原料进行球磨，筛分，获得粒径小于100目的石英砂A；

[n0082]

(2) Quartz sand A was placed on the carbon fiber cloth conductive substrate of the Joule furnace and subjected to flash Joule heat treatment 5 times under vacuum conditions. The Joule furnace parameters were set as follows: voltage 15V, current 250A, heating time 1s, cooling time 10s. After completion, quartz sand B was obtained.

---

(2)将石英砂A置于焦耳炉的碳纤维布导电基底上，在真空条件下进行闪速焦耳热处理5次，设置焦耳炉参数为：电压15V、电流250A、加热时间1s，冷却10s，完成后得到石英砂B；

[n0083]

(3) Quartz sand B was put into an acid pickling tank and acid-leached at 80°C for 4 hours with inorganic mixed acid. The inorganic mixed acid contained hydrochloric acid with a

concentration of 3 mol/L, nitric acid with a concentration of 1 mol/L, and hydrofluoric acid with a concentration of 0.5 mol/L. During acid leaching, ultrasonic treatment with a low frequency of 20 kHz and a high frequency of 40 kHz and pressure treatment with a pressure of 0.5 MPa were used as auxiliary treatments. After completion, material C was obtained.

---

(3)将石英砂B投入酸洗罐中使用无机混酸在80°C酸浸4h，无机混酸中盐酸浓度3mol/L、硝酸浓度1mol/L、氢氟酸0.5mol/L，酸浸时配合低频20kHz、高频40kHz的超声处理辅助与0.5MPa的加压处理辅助，完成后得到物料C；

[n0084]

(4) Wash and dry material C to obtain quartz sand D;

---

(4)洗涤物料C并干燥，得到石英砂D；

[n0085]

(5) Place the quartz sand D in a chlorine and nitrogen mixed atmosphere with a volume ratio of 1:1, and use a microwave chlorination roasting furnace to perform high-temperature chlorination roasting at 1400°C for 4 hours. After the material is cooled, the high-purity quartz sand product is obtained.

---

(5)将石英砂D置于体积比为1:1的氯气与氮气混合气氛中，采用微波氯化焙烧炉，在1400°C进行高温氯化焙烧4h，待物料冷却即得高纯石英砂产品。

[n0086]

The obtained product was tested and found to have a purity of 99.998%, with the main impurities being Fe: 0.9 ppm and Al: 11 ppm.

对所得产品检测：其纯度为99.998%，主要杂质含量Fe：0.9ppm、Al：11ppm。

[n0087]

Example 8:

实施例8：

[n0088]

(1) The same quartz sand raw material as in Example 1 was ball-milled and sieved to obtain quartz sand A with a particle size of less than 100 mesh;

---

(1)将同实施例1一样的石英砂原料进行球磨，筛分，获得粒径小于100目的石英砂A；

[n0089]

(2) Quartz sand A was placed between two carbon cloth conductive substrates in a Joule furnace and flash Joule heat treatment was performed twice under vacuum conditions. The Joule furnace parameters were set as follows: voltage 15V, current 120A, heating time 50s, cooling time 8s. After completion, quartz sand B was obtained.

---

(2)将石英砂A置于焦耳炉的两块碳布导电基底之间，在真空条件下进行闪速焦耳热处理2次，设置焦耳炉参数为：电压15V、电流120A、加热时间50s，冷却8s，完成后得到石英砂B；

[n0090]

(3) Quartz sand B was put into an acid pickling tank and leached at 65°C for 4 hours with inorganic mixed acid. The mixed acid contained hydrochloric acid with a concentration of 3 mol/L, nitric acid with a concentration of 0.5 mol/L, and hydrofluoric acid with a concentration of 0.5 mol/L. During the acid pickling, ultrasonic treatment with a low frequency of 20 kHz and a high frequency of 40 kHz and pressure treatment with 0.2 MPa were used as auxiliary treatments. After the process was completed, material C was obtained.

---

(3)将石英砂B投入酸洗罐中使用无机混酸在65°C酸浸4h，混酸中盐酸浓度3mol/L、硝酸浓度0.5mol/L、氢氟酸0.5mol/L，酸浸时配合低频20kHz、高频40kHz的超声处理辅助与0.2MPa的加压处理辅助，完成后得到物料C；

[n0091]

(4) Wash and dry material C to obtain quartz sand D;

---

(4)洗涤物料C并干燥，得到石英砂D；

[n0092]

(5) Place the quartz sand D in a mixed atmosphere of hydrogen chloride and nitrogen with a volume ratio of 1:3, and use a microwave chlorination roasting furnace to perform high-temperature chlorination roasting at 1600°C for 2 hours. After the material is cooled, the high-purity quartz sand product is obtained.

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(5)将石英砂D置于体积比为1:3的氯化氢与氮气混合气氛中，采用微波氯化焙烧炉，在1600°C进行高温氯化焙烧2h，待物料冷却即得高纯石英砂产品。

[n0093]

The obtained product was tested and found to have a purity of 99.998%, with the main impurities being Fe: 0.9 ppm and Al: 12 ppm.

对所得产品检测：其纯度为99.998%，主要杂质含量Fe：0.9ppm、Al：12ppm。

[n0094]

This invention employs flash Joule heat treatment technology. Utilizing the extremely rapid heating and cooling rates of this technology, it can better promote the bursting of inclusions in quartz sand and the formation of surface cracks, exposing more internal impurities and facilitating thorough subsequent impurity removal. Compared to existing technologies that purify quartz sand through hot-cold collision bursting, which result in quartz sand with a SiONER content greater than 99.9950%, the quartz sand prepared by this invention achieves a purity of 99.998%, reaching a high-end level. Furthermore, flash Joule heat treatment technology has low energy consumption and short processing time, demonstrating significant economic and practical value.

本发明采用闪速焦耳热处理技术，利用该技术极快的升降温速度，能更好促进石英砂包裹体的爆裂以及表面裂纹的生成，暴露出更多的内部杂质，便于后续除杂的彻底性，相较现有技术通过冷热对撞爆

裂法提纯石英砂，其提纯后的石英砂中SiO₂含量大于99.9950%，而本发明制备的石英砂纯度在99.998%，达到了高端水平，同时闪速焦耳热处理技术的能耗低，处理时间短，具有明显的经济实用价值。