Notice

DESCRIPTION CN119591096A

An integrated apparatus and method for simultaneously preparing graphene and silicon carbide nanowires via flash Joule heating.

一种闪蒸焦耳热法同时制备石墨烯及碳化硅纳米线的一体化设备及方法

[0001]

Technical Field

技术领域

[n0001]

This invention belongs to the field of composite material manufacturing, and specifically relates to an integrated device and method for simultaneously preparing graphene and silicon carbide nanowires by flash Joule heating.

本发明属于复合材料制造领域，特别涉及一种闪蒸焦耳热法同时制备石墨烯及碳化硅纳米线的一体化设备及方法。

[0003]

Background Technology

背景技术

[n0002]

Graphene is a material with a two-dimensional honeycomb lattice structure composed of carbon atoms with sp2 hybrid orbitals. It has excellent electrical conductivity, thermal conductivity, piezoelectric properties and optical properties.

石墨烯是一种由碳原子以sp2杂化轨道组成的二维蜂窝状晶格结构的材料，具有优异的导电性能、热导率、压电性能以及光学性能。

Common methods for preparing graphene include mechanical exfoliation, chemical vapor deposition, and chemical reduction.

常见的制备石墨烯的方法主要有机械剥离法、化学气相沉积法、化学还原法等。

Among them, mechanical exfoliation has low yield, is difficult to prepare large-area graphene, and the exfoliated graphite lacks uniformity; chemical vapor deposition has high equipment costs and high operational requirements, and is not suitable for large-scale production; chemical reduction usually has many structural defects, and also has the problem of residual reducing agent contaminating the material.

其中，机械剥离法产量低，难以制备大面积石墨烯，且剥离的石墨缺乏均匀性；化学气相沉积法对设备成本和操作要求高，不适合大规模生产；化学还原法通常存在较多的结构缺陷，同时存在还原剂残留污染材料的问题。

[n0003]

Silicon carbide is a wide-bandgap semiconductor composed of silicon and carbon. It has high covalent bond energy and a stable structure. One-dimensional silicon carbide nanomaterials

have unique physicochemical properties, excellent mechanical properties and high electrical conductivity.

碳化硅是由硅和碳组成的宽带隙半导体，具有较高的共价键能和稳定的结构，一维碳化硅纳米材料具有独特的物理化学性质、优异的机械性能和高电导率。

Common methods for preparing silicon carbide nanowires include carbothermal reduction, chemical vapor deposition, electrospinning, and sol-gel methods.

常见的制备碳化硅纳米线的方法主要有碳热还原法、化学气相沉积法、静电纺丝法、溶胶凝胶法等。

Among them, the carbothermal reduction process involves multiple reactions, making the production process complex and unable to be mass-produced; the chemical vapor deposition method requires precise control of temperature and pressure, the required equipment is expensive, and the deposition rate is slow; the electrospinning method has low production efficiency and complex equipment; the sol-gel method requires relatively expensive precursors and catalysts, as well as complex post-processing, resulting in high costs and significant environmental impact.

其中，碳热还原法过程涉及到多个反应，生产过程复杂，无法大规模生产；化学气相沉积法需要精确控制温度和压力，所需设备昂贵，沉积速度慢；静电纺丝法生产效率低，装置复杂；溶胶凝胶法需要较为昂贵的前驱体和催化剂，同时需要复杂的后处理，成本较高，对环境影响大。

[n0004]

Currently, most methods for preparing graphene and silicon carbide nanowires are still in the laboratory stage, and they are inefficient and costly. Achieving industrial production of graphene and silicon carbide nanowires still faces many challenges.

目前，制备石墨烯和碳化硅纳米线的方法大多处于实验室阶段，且效率低、成本高，实现石墨烯和碳化硅纳米线工业化生产仍然面临许多挑战。

Flash Joule heating is a novel material preparation technology that can instantly heat materials to extremely high temperatures (up to 3000K or more), enabling the preparation of high-quality, low-defect graphene. At the same time, this rapid heating and cooling process also helps to form silicon carbide nanowires. Compared to traditional heating methods, flash Joule heating technology requires simpler equipment and has lower maintenance costs; the heating process can be precisely controlled by adjusting the current, making it easy to automate and become intelligent; and it does not produce exhaust gas or other harmful emissions, making it more environmentally friendly. However, existing flash Joule heating technology only focuses on the preparation of a single product. In practice, a large amount of

energy is dissipated through thermal radiation, and there are certain requirements for the conductivity of the material. Non-highly conductive materials need to introduce conductive additives or substrates, which leads to impurities and makes it difficult to obtain independent products. Currently, the flash evaporation Joule heating method for preparing nanomaterials such as graphene suffers from low energy utilization, and it is difficult to meet specific conductivity requirements when preparing nanomaterials with non-highly conductive precursors such as silicon carbide nanowires. Therefore, it is of great significance to study a synthesis process for nanomaterials with high energy utilization, high production efficiency and adaptability to various non-highly conductive precursors.

闪蒸焦耳热技术是一种新型的材料制备技术，它能在瞬间将材料加热至极高温度(可达3000K以上)，能够制备高质量、低缺陷的石墨烯。 同时，这种快速的升温和降温过程也有助于形成碳化硅纳米线。 相较于传统加热方式，闪蒸焦耳热技术所需设备较为简单，维护成本较低；加热过程可以通过电流调节进行精确控制，便于实现自动化和智能化；不会产生废气或其他有害排放，更加符合环保要求。 然而，现有闪蒸焦耳热技术只聚焦于单一产物制备，在实际过程中大量能量通过热辐射的方式耗散，且对材料导电性有一定的要求，非高导电材料需要引入导电添加剂或基底，这导致杂质和难以获得独立产物。 目前，闪蒸焦耳加热法制备石墨烯等纳米材料存在能量利用率不高的问题，且制备碳化硅纳米线等非高导电前驱体的纳米材料难以满足特定的电导率要求。 因此，研究一种能量利用率高、生产效率高和适应多种非高导电前驱体的纳米材料的合成工艺具有重要意义。

[0007]

Summary of the Invention

发明内容

[n0005]

To address the problems existing in the prior art, this invention proposes an integrated equipment and method for simultaneously preparing graphene and silicon carbide nanowires via flash Joule heating. This method is adaptable to the processes of various non-highly conductive precursor nanomaterials and can simultaneously prepare high-quality, low-defect graphene and silicon carbide nanowires without requiring prolonged high-temperature heating, thus meeting the needs for high-efficiency, low-cost, and easily scalable production.

针对现有技术存在的问题，本发明提出一种闪蒸焦耳热法同时制备石墨烯及碳化硅纳米线的一体化设备及方法，可适应多种非高导电前驱体的纳米材料的工艺，能够在不需要长时间高温加热的情况下，同时制备高质量、低缺陷的石墨烯和碳化硅纳米线，满足高效率、低成本、易规模化的生产需求。

[n0006]

This invention is achieved through the following technical solution:

本发明通过下述技术方案实现：

[n0007]

An integrated device and method for simultaneously preparing graphene and silicon carbide nanowires by flash Joule heating includes a vacuum system, a detection system, a central control system, a discharge system, a heating tube, a reaction tube, and a deposition tube.

一种闪蒸焦耳热法同时制备石墨烯及碳化硅纳米线的一体化设备及方法，包括真空系统、检测系统、中央控制系统、放电系统、加热管、反应管、沉积管；

[n0008]

The vacuum system includes a fixing device, a vacuum chamber, and a vacuum pump. The fixing device is used to fix the heating tube, reaction tube, deposition tube, and electrodes inside the vacuum chamber. The vacuum chamber is connected to the vacuum pump, which extracts air from the vacuum chamber.

真空系统包括固定装置、真空箱、真空泵；固定装置用于固定加热管、反应管、沉积管和电极放置于真空箱内，真空箱连接真空泵，通过真空泵抽取真空箱内空气；

[n0009]

The detection system includes a vacuum pressure gauge, an infrared thermometer, and a circuit monitoring device. The vacuum pressure gauge is connected to the vacuum chamber to measure the air pressure inside the chamber. The infrared thermometer is connected to the vacuum chamber to measure the temperature inside the heating tube, reaction tube, and deposition tube. The circuit monitoring device measures the voltage, current, and resistance across the conductive carbon source inside the heating tube. The detection system monitors the reaction data and transmits it to the central control system via electrical signals.

检测系统包括真空压力表、红外测温仪、电路监测装置；真空压力表连接真空箱测量真空箱内气压值，红外测温仪连接真空箱测量加热管、反应管和沉积管内温度，电路监测装置测量加热管内导电碳源两端的电压、电流和电阻，检测系统监测反应数据，通过电信号传输至中央控制系统；

[n0010]

The central control system plays the role of command transmission and data processing in the entire equipment, and connects the detection system and the discharge system. The central control system selects appropriate discharge parameters based on the resistance data transmitted by the receiving circuit monitoring device, and transmits charge and discharge commands to the discharge system. When the discharge system discharges, the central control system receives the temperature data from the infrared thermometer and the real-

time voltage and current data from the circuit monitoring device, and makes corresponding feedback to adjust the new discharge parameters and transmit charge and discharge commands in a timely manner. After the discharge is completed, the central control system records and organizes the reaction data of the detection system.

中央控制系统在整个设备中起到命令传递、数据处理的作用，连接检测系统、放电系统；中央控制系统根据接收电路监测装置传输的电阻数据选择合适的放电参数，向放电系统传输充放电命令，放电系统放电时，中央控制系统接收红外测温仪的温度数据和电路监测装置的实时电压、电流数据，并及时做出相应反馈调节新的放电参数并传递充放电命令，放电结束后记录并整理检测系统反应数据；

[n0011]

The discharge system includes an inductor, a charging device, and electrodes. The inductor receives instructions from the central control system and controls the rapid closing of the control circuit switch to control the charging and discharging time and voltage of the charging device. The charging device consists of capacitors used to store and release electrical energy. The charging device is connected to electrodes made of pure copper with threads, which can be fixed to the fixing device by nuts. At the same time, it clamps the conductive carbon source inside the heating tube, and the resistance is controlled by the degree of clamping.

放电系统包括电感装置、充电装置、电极；电感装置接收中央控制系统指令，控制电路开关的迅速闭合实现控制充电装置的充放电时间和电压，充电装置由电容组成，用于存储和释放电能，充电装置连接电极，电极由纯铜制成，带有螺纹，可通过螺母固定于固定装置，同时夹紧加热管内的导电碳源，通过夹紧程度控制电阻；

[n0012]

The heating tube is made of quartz and contains a conductive carbon source. When the discharge system discharges, the current passes through the conductive carbon source to generate Joule heat, and the heat is transferred to the reaction tube and the deposition tube.

加热管由石英制成，管内装有导电碳源，放电系统放电时，电流通过导电碳源产生焦耳热，热量通过热传递至反应管和沉积管内；

[n0013]

The reaction tube is made of quartz, and conductive plugs are installed on both sides of the tube opening. The conductive plugs are made of pure copper and are connected to electrodes. They have an auxiliary Joule heating effect on the mixed carbon source and silicon source inside the tube. A row of several frusto-shaped holes is opened at the top of the tube wall. The reactants inside the tube react with heat and sublimate into a gaseous state, which enters the deposition tube through the small holes for deposition.

反应管由石英制成，管口两侧装有导电堵头，导电堵头由纯铜制成，连接电极，对管内混合碳源和硅源具有辅助焦耳加热作用，管壁正上方开有一排若干圆台形小孔，管内反应物受热反应并升华成气态，通过小孔进入沉积管进行沉积；

[n0014]

The deposition tube is made of quartz, and insulating plugs made of glass fiber are installed on both sides of the tube opening. These insulating plugs are used to fix the reaction tube and the deposition tube, and at the same time prevent the reaction gas from escaping. The inside of the tube wall is coated with a catalyst. After the reaction product gas enters the deposition tube, it adheres to the inside of the tube wall and deposits to form the product.

沉积管由石英制成，管口两侧装有绝缘堵头，绝缘堵头由玻璃纤维制成，用于固定反应管和沉积管，同时防止反应气体逃逸，管壁内涂有催化剂，反应生成物气体进入沉积管后附着在管壁内沉积生成产物。

[n0015]

An integrated apparatus and method for simultaneously preparing graphene and silicon carbide nanowires via flash Joule heating includes the following steps:

一种闪蒸焦耳热法同时制备石墨烯及碳化硅纳米线的一体化设备及方法，包括以下步骤：

[n0016]

Step 1: Select a suitable conductive carbon source, pre-treat the conductive carbon source to make it into powder, fill it into the heating tube, and press it with electrodes;

步骤一：选择合适的导电碳源，对导电碳源进行前处理，制成粉末状填充至加热管内，通过电极压紧；

[n0017]

Step 2: Select suitable carbon and silicon sources, pre-treat them to make powder, and mix them evenly in a fixed ratio to fill the lower half of the reaction tube, ensuring that the small holes in the tube wall are directly above. Insert conductive plugs and insulating plugs to seal the reaction tube and deposition tube respectively.

步骤二：选择合适的碳源和硅源，对两者进行前处理，制成粉末状并按固定比例均匀混合填充至反应管内下半层，保证反应管管壁小孔位于正上方，塞入导电堵头和绝缘堵头分别封闭反应管和沉积管；

[n0018]

Step 3: Fix the heating tube, reaction tube, and deposition tube to the fixing device using electrodes. Measure the resistance across the electrodes using a circuit monitoring device. Set appropriate discharge parameters in the central control system. Place the fixing device inside the vacuum chamber and below the infrared thermometer. Close the vacuum chamber door and valves. Control the discharge system to charge.

步骤三：将加热管、反应管、沉积管通过电极固定至固定装置，通过电路监测装置测量电极两端电阻，中央控制系统设定合适放电参数，将固定装置放置于真空箱内并置于红外测温仪下方，关闭真空箱箱门和阀门，控制放电系统充电；

[n0019]

Step 4: Open the vacuum pump valve and power switch to perform the vacuuming operation. Observe the reading on the vacuum pressure gauge on the vacuum chamber. After the air pressure reaches the required range, close the vacuum pump valve and power switch.

步骤四：打开真空泵的阀门和电源开关进行抽真空操作，观察真空箱上真空压力表的示数，在气压值到达要求范围后关闭真空泵阀门和电源开关；

[n0020]

Step 5: The central control system transmits a discharge command to the discharge system, first performing a short-term high-voltage discharge on the heating element, and then performing an intermittent short-term low-voltage discharge on the heating element.

步骤五：由中央控制系统向放电系统传输放电命令，先对加热管进行短时间高压放电，再对加热管进行间歇性短时间低压放电；

[n0021]

Step Six: During the discharge process, the infrared thermometer of the detection system records the temperature distribution at the heat source in real time, and transmits the measurement data to the central control system in real time. The central control system monitors the reaction temperature in real time and provides corresponding feedback: when the temperature at the heat source location is detected to be below the reaction range, it outputs a command to the discharge system to increase the discharge voltage; when the temperature is detected to be above the reaction range, it decreases the discharge voltage to bring the temperature at the heat source location back to the set range.

步骤六：放电过程中检测系统的红外测温仪实时记录热源处的温度分布，将测量情况实时记录并传输至中央控制系统，中央控制系统实时监测反应温度给出相应的反馈：检测到热源位置温度未达到反应区间时，向放电系统输出指令，增大放电电压，检测到温度超出反应区间时，减小放电电压，使热源位置温度回到设定区间内；

[n0022]

Step 7: The central control system records and organizes the reaction data of the detection system, observes the reaction through the observation port of the vacuum chamber door, and repeats step 5 as needed;

步骤七：中央控制系统记录并整理检测系统反应数据，通过真空箱箱门观察口观察反应情况，根据情况重复步骤五；

[n0023]

Step 8: Open the vacuum chamber valve to release pressure, open the vacuum chamber door, remove the heating tube and reaction tube through the slide rail separation fixing device bracket, collect the product in the deposition tube, remove the conductive plug and insulating plug, and collect the product in the reaction tube.

步骤八：打开真空箱阀门完成泄压，打开真空箱箱门，通过滑轨分离固定装置支架取出加热管和反应管，收集得到沉积管内生成物，拆卸导电堵头和绝缘堵头，收集得到反应管内产物。

[n0024]

Compared with the prior art, the present invention has at least the following advantages and effects:

本发明对于现有技术，至少具备如下优点及效果：

[n0025]

This invention utilizes the principle of Joule heating for heating, which can raise the temperature to the reaction temperature in a short time and has high thermal efficiency. It can achieve uniform heating while reducing heat loss during the conduction process.

本发明利用焦耳加热的原理进行加热，能够在短时间内将温度升高至反应温度，且具有较高的热效率，在能够实现均匀加热的同时减少了热量在传导过程中的损失；

[n0026]

This invention solves the problem that Joule heating technology is inconvenient for non-highly conductive precursors by separating the heating zone and the reaction zone, and can simultaneously prepare two different nanomaterials.

本发明通过分离加热区与反应区解决了非高导电前驱体不便采用焦耳加热技术的问题，可以同时制备两种不同的纳米材料；

[n0027]

This invention uses a layered tube design to allow the reaction products to grow in the deposition zone. Compared with the traditional vapor deposition method, it has lower equipment requirements, does not require long-term high-temperature heating, and has lower energy consumption costs.

本发明通过分层管设计使反应产物在沉积区生长，相较于传统气相沉积法对设备要求不高，不需要长期高温加热，能耗成本低；

[n0028]

This invention features a simple structure and ease of manufacturing. It can be modified at a low cost as needed to replace raw materials for the preparation of other nanomaterials. At the same time, the technical means are simple and easy to implement, and the operation is

simple and suitable for automation and large-scale production. It represents a significant and substantial improvement over existing technologies.

本发明具有结构简单、易于制造的特点，可以按需对其进行较低成本的改装，替换原料制备其他纳米材料，同时技术手段简便易行，操作简单适合自动化、规模化，相对于现有技术具有突出的实质性进步。

[0032]

Attached Figure Description

附图说明

[n0029]

Figure 1 is a schematic diagram of the overall structure of an integrated equipment for the simultaneous preparation of graphene and silicon carbide nanowires by flash Joule heating.

图1是一种闪蒸焦耳热法同时制备石墨烯及碳化硅纳米线的一体化设备整体结构示意图；

[n0030]

In Figure 1, the numbers represent: 1-vacuum system; 2-detection system; 3-central control system; 4-discharge system; 5-heating tube; 6-reaction tube; 7-deposition tube; 1-1-fixing device; 1-2-vacuum chamber; 1-3-vacuum pump; 2-1-vacuum pressure gauge; 2-2-infrared thermometer; 2-3-circuit monitoring device.

图1中编号表示：1-真空系统；2-检测系统；3-中央控制系统；4-放电系统；5-加热管；6-反应管；7-沉积管；1-1-固定装置；1-2-真空箱；1-3-真空泵；2-1-真空压力表；2-2-红外测温仪；2-3-电路监测装置。

[n0031]

Figure 2 is a process flow diagram of an integrated equipment and method for simultaneously preparing graphene and silicon carbide nanowires by flash Joule heating.

图2是一种闪蒸焦耳热法同时制备石墨烯及碳化硅纳米线的一体化设备及方法工艺流程图。

[n0032]

Figure 3 is a circuit diagram of the discharge system 4;

图3是放电系统4的电路示意图；

[n0033]

In Figure 3, the numbers indicate: 4-1-Inductor; 4-2-Charging device; 4-3-Electrode.

图3中编号表示：4-1-电感装置；4-2-充电装置；4-3-电极。

[n0034]

Figure 4 is a schematic diagram of the structure of heating tube 5, reaction tube 6, deposition tube 7, and electrode 4-3; in the figure, washer 1-1-1, spring 1-1-2, nut 1-1-3, conductive plug 1-1-4, and insulating plug 1-1-5 belong to the fixing device 1-1 part;

图4是加热管5、反应管6、沉积管7、电极4-3的结构示意图；图中垫圈1-1-1、弹簧1-1-2、螺母1-1-3、导电堵头1-1-4、绝缘堵头1-1-5属于固定装置1-1部分；

[n0035]

The numbers in Figure 4 represent: 5-heating tube; 6-reaction tube; 7-deposition tube; 1-1-1-washer; 1-1-2-spring; 1-1-3-nut; 1-1-4-conductive plug; 1-1-5-insulating plug; 4-3-electrode; 6-1-several frustum-shaped holes.

图4中编号表示：5-加热管；6-反应管；7-沉积管；1-1-1-垫圈；1-1-2-弹簧；1-1-3-螺母；1-1-4-导电堵头；1-1-5-绝缘堵头；4-3-电极；6-1-若干圆台形小孔。

[0040]

Detailed Implementation

具体实施方式

[n0036]

The present invention will now be described in further detail with reference to the accompanying drawings and embodiments.

下面结合附图和实施例对本发明作进一步的详细说明。

[n0037]

Example

实施例

[n0038]

As shown in Figure 1, this invention discloses an integrated device and method for simultaneously preparing graphene and silicon carbide nanowires by flash Joule heating, including a vacuum system 1, a detection system 2, a central control system 3, a discharge system 4, a heating tube 5, a reaction tube 6, and a deposition tube 7.

如图1所示，本发明公开了一种闪蒸焦耳热法同时制备石墨烯及碳化硅纳米线的一体化设备及方法，包括真空系统1、检测系统2、中央控制系统3、放电系统4、加热管5、反应管6、沉积管7；

[n0039]

Vacuum system 1 includes a fixing device 1-1, a vacuum chamber 1-2, and a vacuum pump 1-3; the fixing device is used to fix the heating tube 5, reaction tube 6, deposition tube 7 and electrode 4-3 in the vacuum chamber, and the vacuum chamber is connected to the vacuum pump to extract air from the vacuum chamber.

真空系统1包括固定装置1-1、真空箱1-2、真空泵1-3；固定装置用于固定加热管5、反应管6、沉积管7和电极4-3放置于真空箱内，真空箱连接真空泵，通过真空泵抽取真空箱内空气；

[n0040]

The detection system 2 includes a vacuum pressure gauge 2-1, an infrared thermometer 2-2, and a circuit monitoring device 2-3. The vacuum pressure gauge is connected to the vacuum

chamber 1-2 to measure the air pressure inside the vacuum chamber. The infrared thermometer is connected to the vacuum chamber to measure the temperature inside the heating tube 5, the reaction tube 6, and the deposition tube 7. The circuit monitoring device measures the voltage, current, and resistance across the conductive carbon source inside the heating tube. The detection system monitors the reaction data and transmits it to the central control system 3 via electrical signals.

检测系统2包括真空压力表2-1、红外测温仪2-2、电路监测装置2-3；真空压力表连接真空箱1-2，测量真空箱内气压值，红外测温仪连接真空箱测量加热管5、反应管6和沉积管7内温度，电路监测装置测量加热管内导电碳源两端的电压、电流和电阻，检测系统监测反应数据，通过电信号传输至中央控制系统3；

[n0041]

The central control system 3 plays the role of command transmission and data processing in the whole equipment, and connects the detection system 2 and the discharge system 4. The central control system selects appropriate discharge parameters based on the resistance data transmitted by the receiving circuit monitoring device 2-3, and transmits charge and discharge commands to the discharge system. When the discharge system discharges, the central control system receives the temperature data of the infrared thermometer 2-2 and the real-time voltage and current data of the circuit monitoring device, and makes corresponding

feedback to adjust the new discharge parameters and transmit charge and discharge commands in a timely manner. After the discharge is completed, the detection system reaction data is recorded and sorted out.

中央控制系统3在整个设备中起到命令传递、数据处理的作用，连接检测系统2、放电系统4；中央控制系统根据接收电路监测装置2-3传输的电阻数据选择合适的放电参数，向放电系统传输充放电命令，放电系统放电时，中央控制系统接收红外测温仪2-2的温度数据和电路监测装置的实时电压、电流数据，并及时做出相应反馈调节新的放电参数并传递充放电命令，放电结束后记录并整理检测系统反应数据；

[n0042]

The discharge system 4 includes an inductor 4-1, a charging device 4-2, and an electrode 4-3. The inductor receives instructions from the central control system 3 and controls the rapid closing of the control circuit switch to control the charging and discharging time and voltage of the charging device. The charging device is composed of capacitors and is used to store and release electrical energy. The charging device is connected to the electrode, which is made of pure copper and has threads. It can be fixed to the fixing device 1-1 by the nut 1-1-3, and at the same time clamps the conductive carbon source in the heating tube 5. The resistance is controlled by the degree of clamping.

放电系统4包括电感装置4-1、充电装置4-2、电极4-3；电感装置接收中央控制系统3指令，控制电路开关的迅速闭合实现控制充电装置的充放电时间和电压，充电装置由电容组成，用于存储和释放电能，充电装置连接电极，电极由纯铜制成，带有螺纹，可通过螺母1-1-3固定于固定装置1-1，同时夹紧加热管5内的导电碳源，通过夹紧程度控制电阻；

[n0043]

The heating tube 5 is made of quartz and contains a conductive carbon source. When the discharge system 4 discharges, the current passes through the conductive carbon source to generate Joule heat, and the heat is transferred to the reaction tube 6 and the deposition tube 7.

加热管5由石英制成，管内装有导电碳源，放电系统4放电时，电流通过导电碳源产生焦耳热，热量通过热传递至反应管6和沉积管7内；

[n0044]

The reaction tube 6 is made of quartz, and conductive plugs 1-1-4 are installed on both sides of the tube opening. The conductive plugs are made of pure copper and are connected to electrodes 4-3. They have an auxiliary Joule heating effect on the mixed carbon source and

silicon source inside the tube. There is a row of several frusto-shaped holes 6-1 on the top of the tube wall. The reactants inside the tube react with heat and sublimate into silicon carbide gas, which enters the deposition tube 7 through the holes for deposition.

反应管6由石英制成，管口两侧装有导电堵头1-1-4，导电堵头由纯铜制成，连接电极4-3，对管内混合碳源和硅源具有辅助焦耳加热作用，管壁正上方具有一排若干圆台形小孔6-1，管内反应物受热反应并升华成碳化硅气体，通过小孔进入沉积管7进行沉积；

[n0045]

The deposition tube 7 is made of quartz, and insulating plugs 1-1-5 are installed on both sides of the tube opening. The insulating plugs are made of glass fiber and are used to fix the reaction tube 6 and the deposition tube, while preventing the reaction gas from escaping. The inner wall of the tube is coated with a catalyst. After the reaction product gas enters the deposition tube, it adheres to the inner wall of the tube and deposits to form the product.

沉积管7由石英制成，管口两侧装有绝缘堵头1-1-5，绝缘堵头由玻璃纤维制成，用于固定反应管6和沉积管，同时防止反应气体逃逸，管壁内涂有催化剂，反应生成物气体进入沉积管后附着在管壁内沉积生成产物。

[n0046]

A detailed process for an integrated device and method for simultaneously preparing graphene and silicon carbide nanowires via flash Joule heating includes the following steps:

一种闪蒸焦耳热法同时制备石墨烯及碳化硅纳米线的一体化设备及方法的具体流程，包括以下步骤：

[n0047]

Step 1: Select carbon black as the conductive carbon source, heat and dry the carbon black to make it into powder, which is called material A. Take 1g of material A and fill it into the heating tube, and press it through the electrode.

步骤一：选择炭黑作为导电碳源，对炭黑进行加热干燥处理，制成粉末状，记作A料，取A料1g填充至加热管内，通过电极压紧；

[n0048]

Step 2: Select carbon nanotubes as the carbon source and silicon powder as the silicon source. Take 1g of carbon nanotubes and 4g of silicon powder, add them to 100g of distilled water and stir for 30min. Place them in an oven at 140°C and dry for 6h. Take them out and make them into powder, which is called material B. Take 1.5g of material B and fill it into the lower half of the reaction tube, ensuring that the small hole in the wall of the reaction tube is

directly above. Insert conductive plugs and insulating plugs to seal the reaction tube and the deposition tube respectively.

步骤二：选择碳纳米管作为碳源，硅粉作为硅源，取1g碳纳米管和4g硅粉加入100g蒸馏水中搅拌30min，放入烘箱140°C烘干6h后取出制成粉末状，记作B料，取B料1.5g填充至反应管内下半层，保证反应管管壁小孔位于正上方，塞入导电堵头和绝缘堵头分别封闭反应管和沉积管；

[n0049]

Step 3: Fix the heating tube, reaction tube, and deposition tube to the fixing device using electrodes. Measure the resistance across the electrodes using the circuit detection system to be 8±0.5Ω. Set the high voltage (160V) discharge time to 0.5s and the low voltage (120V) discharge time to 50ms, with a discharge cycle of 0.1s and 5 discharge cycles. Place the fixing device inside the vacuum chamber and below the infrared thermometer. Close the vacuum chamber door and valves, and control the discharge system to charge.

步骤三：将加热管、反应管、沉积管通过电极固定至固定装置，通过电路检测系统测量电极两端电阻在8±0.5Ω，中央控制系统设定高电压160V放电时长0.5s，设定低电压120V放电时长50ms、放电周期0.1s、放电次数5次，将固定装置放置于真空箱内并置于红外测温仪下方，关闭真空箱箱门和阀门，控制放电系统充电；

[n0050]

Step 4: Open the vacuum pump valve and power switch to perform the vacuuming operation. Observe the reading of the vacuum pressure gauge on the vacuum chamber. After the air pressure reaches a vacuum level of 0.1MPa, close the vacuum pump valve and power switch.

步骤四：打开真空泵的阀门和电源开关进行抽真空操作，观察真空箱上真空压力表的示数，在气压值到达真空度0.1MPa后关闭真空泵的阀门和电源开关；

[n0051]

Step 5: The central control system transmits a discharge command to the discharge system, first performing a short-term high-voltage discharge on the heating element, and then performing an intermittent short-term low-voltage discharge on the heating element.

步骤五：由中央控制系统向放电系统传输放电命令，先对加热管进行短时间高压放电，再对加热管进行间歇性短时间低压放电；

[n0052]

Step Six: During the discharge process, the infrared thermometer of the detection system records the temperature distribution at the heat source in real time, and transmits the

measurement data to the central control system in real time. The central control system monitors the reaction temperature in real time and provides corresponding feedback: when the temperature inside the heating tube is not 2700±100°C, and the temperature in the reaction tube and the deposition tube is not 1400±100°C, it outputs a command to the discharge system to increase the 5V discharge voltage; when the temperature is detected to be outside the reaction range, it decreases the 5V discharge voltage to bring the temperature at the heat source back to the set range.

步骤六：放电过程中检测系统的红外测温仪实时记录热源处的温度分布，将测量情况实时记录并传输至中央控制系统，中央控制系统实时监测反应温度给出相应的反馈：检测到加热管内温度未达到2700±100°C、反应管和沉积管未达到1400±100°C时，向放电系统输出指令，增大5V放电电压，检测到温度超出反应区间时，减小5V放电电压，使热源位置温度回到设定区间内；

[n0053]

Step 7: The central control system records and organizes the reaction data of the detection system. The reaction is observed through the observation port of the vacuum chamber door. If there is little product in the deposition tube, repeat step 5.

步骤七：中央控制系统记录并整理检测系统反应数据，通过真空箱箱门观察口观察反应情况，沉积管内生成物较少则重复步骤五；

[n0054]

Step 8: Open the vacuum chamber valve to release pressure, open the vacuum chamber door, remove the heating tube and reaction tube through the slide rail separation fixing device bracket, collect the B material product in the deposition tube, disassemble the conductive plug and insulating plug, and collect the A material product in the reaction tube.

步骤八：打开真空箱阀门完成泄压，打开真空箱箱门，通过滑轨分离固定装置支架取出加热管和反应管，收集得到沉积管内B料生成物，拆卸导电堵头和绝缘堵头，收集得到反应管内A料产物。

[n0055]

Obviously, the above embodiments of the present invention are merely examples for clearly illustrating the present invention, and are not intended to limit the implementation of the present invention.

显然，本发明的上述实施例仅仅是为了清楚说明本发明所作的举例，而并非是对本发明的实施方式的限定。

For those skilled in the art, various obvious changes, readjustments, and substitutions can be made without departing from the scope of protection of this invention.

对于所属领域的普通技术人员来说，能够进行各种明显的变化、重新调整和替代而不会脱离本发明的保护范围。

It is neither necessary nor possible to exhaustively list all possible implementation methods here.

这里无需也无法对所有的实施方式予以穷举。

Any modifications, equivalent substitutions, and improvements made within the spirit and principles of this invention shall be included within the scope of protection of the claims of this invention.

凡在本发明的精神和原则之内所作的任何修改、等同替换和改进等，均应包含在本发明权利要求的保护范围之内。