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DESCRIPTION CN120081420A

A natural mineral-based microwave absorbing material synthesized by flash sintering process and its preparation method

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一种基于闪烧工艺合成的天然矿物基吸波材料及其制备方法

[0001]

Technical Field

技术领域

[n0001]

This invention relates to the field of microwave absorbing materials technology, specifically to a natural mineral-based microwave absorbing material synthesized by flash calcination process and its preparation method.

本发明涉及吸波材料技术领域，具体涉及一种基于闪烧工艺合成的天然矿物基吸波材料及其制备方法。

[0003]

Background Technology

背景技术

[n0002]

Molybdenite is a molybdenum disulfide mineral and an important molybdenum ore resource. It also often contains rhenium and is the main mineral for refining molybdenum and rhenium.

辉钼矿是钼的二硫化矿物，是重要的钼矿资源，还常含有铼，是提炼钼、铼的主要矿物。

However, the use of molybdenite resources is mostly limited to refining after mining to obtain metals or their compounds, which is costly, and direct utilization is rare.

但辉钼矿资源的使用大多是开采后进行提炼得到金属或者其化合物，成本较高，而直接利用的少。

[n0003]

With the development of science and technology, electromagnetic waves are being used in more and more widespread applications. The use of communication equipment such as mobile phones, base stations, satellite broadcasting, and local area networks is becoming more and more frequent, leading to increasingly serious electromagnetic pollution problems.

随着科学技术的发展，电磁波的应用场景越来越广泛，移动电话、基站、卫星广播、局域网等通讯设备的使用越来越频繁，导致电磁污染问题愈发严重。

This has led to the increasingly widespread application of electromagnetic wave absorbing materials, and the demand is gradually increasing.

这也使得电磁波吸收材料的应用日趋广泛，需要量也逐步增加。

MoS_NER1 has a unique two-dimensional sheet structure and a large specific surface area, which makes it a promising candidate for application in the field of electromagnetic protection.

MoS₂具有独特的二维片层结构和较大的比表面积，使得其在电磁防护领域有很好的应用前景。

Traditional methods for preparing microwave absorbing materials from molybdenum disulfide (MoS₂) typically require multi-step synthesis, such as high-temperature sulfidation or complex physicochemical purification processes. This results in complex preparation processes, long preparation times, low efficiency, and high costs.

在二硫化钼(MoS₂)制备吸波材料的传统方法中，通常需要多步骤合成，如高温硫化或复杂的物理化学提纯过程，导致吸波材料的制备流程复杂、制备时间长、制备效率低且成本高。

[0006]

Summary of the Invention

发明内容

[n0004]

The purpose of this invention is to provide a natural mineral-based microwave absorbing material synthesized by flash calcination process and its preparation method, thereby solving the problems of complex process, high efficiency and low cost of existing preparation methods.

本发明的目的在于提供一种基于闪烧工艺合成的天然矿物基吸波材料及其制备方法，解决现有制备方法流程复杂、效率低成本高的问题。

[n0005]

To solve the above-mentioned technical problems, the present invention adopts the following technical solution: a method for preparing natural mineral-based microwave absorbing materials based on flash calcination process, characterized by the following steps:

为解决上述的技术问题，本发明采用以下技术方案：一种基于闪烧工艺合成的天然矿物基吸波材料的制备方法，其特征在于步骤如下：

[n0006]

S1.

S1.

Molybdenite powder and iron oxide powder are thoroughly mixed in a mortar to obtain a mixed raw material;

将辉钼矿粉末和氧化铁粉末在研钵中充分混合均匀，得到混合原料；

[n0007]

S2.

S2.

The mixed raw materials are loaded into a corundum ring, connected to a platinum wire, and then fixed.

将混合原料装入刚玉环中，接上铂丝后固定；

[n0008]

S3.

S3.

The corundum ring is placed in a muffle furnace, and after heating, the platinum wire is connected to the power supply for flash burning.

将刚玉环放置于马弗炉中，升温后铂丝连接电源，进行闪烧；

[n0009]

S4.

S4.

After natural cooling, the resulting product is the MoS₂/Fe₃O₄/MoO₂ composite microwave absorbing material.

自然冷却后，所得产物，即为MoS₂/Fe₃O₄/MoO₂复合吸波材料。

[n0010]

A further technical solution is that the molar ratio of molybdenite powder (calculated as molybdenum disulfide) to iron oxide powder is 1:0.5-2, the particle size of molybdenite powder is ≤0.25mm, and the particle size of iron oxide powder is ≤0.2mm.

更进一步的技术方案是所述辉钼矿粉末(以二硫化钼计)和氧化铁粉末用量摩尔比为1:0.5～2，辉钼矿粉末粒径≤0.25mm，氧化铁粉末粒径≤0.2mm。

[n0011]

A further technical solution is to seal both ends of the corundum ring with graphite sheets, and then fix it with a clamp after connecting the platinum wire to the graphite sheets.

更进一步的技术方案是所述刚玉环两端用石墨片封口，铂丝与石墨片连接后，用夹具固定。

[n0012]

A further technical solution is that in step S3, the muffle furnace is heated to 400°C at a heating rate of 10°C/min.

更进一步的技术方案是所述步骤S3中马弗炉以10°C/min的升温速率升温至400°C。

[n0013]

A further technical solution is that the voltage during flash burning in step S3 is 600V, the current is 1.5A, and the flash burning time is 3min.

更进一步的技术方案是所述步骤S3中闪烧中电压为600V，电流1.5A，闪烧时间3min。

[n0014]

The present invention also discloses a natural mineral-based microwave absorbing material synthesized by a flash calcination process, wherein the microwave absorbing material is prepared by the above-described preparation method.

本发明还公开一种基于闪烧工艺合成的天然矿物基吸波材料，所述吸波材料由上述制备方法制得。

[n0015]

Compared with the prior art, the beneficial effects of the present invention are:

与现有技术相比，本发明的有益效果是：

[n0016]

1.

1.

Using natural molybdenite as raw material, it is mixed with iron oxide powder and subjected to a flash calcination process. During this process, an electric current passes through the sample to generate Joule heat, which rapidly increases the sample temperature, promotes the transport of matter, and induces a rapid reaction between the two, resulting in rapid densification of the sample. This process enables the preparation of MoS₂ /Fe₃O₄/MoO₂ composite microwave absorbing materials. The process is simple and efficient, improving preparation efficiency while reducing production costs and minimizing environmental pollution.

利用天然辉钼矿作为原料，与氧化铁粉末混合，采用闪烧工艺，过程中电流通过样品产生焦耳热，迅速提高样品温度，促进物质的传输，促使两者快速反应，引起样品快速致密化，从而实现MoS₂/Fe₃O₄/MoO₂复合吸波材料的制备，工艺简单高效，在提高制备效率的同时降低生产成本，减少环境污染。

[n0017]

2.

2.

This invention uses molybdenite, a natural raw material, which is abundant in China and has good electromagnetic wave absorption characteristics. The prepared MoS₂

/Fe₃O₄/MoO₂ composite absorbing material can be widely used as an electromagnetic wave absorbing material in corresponding electromagnetic protection and microwave stealth fields.

本发明选用天然原料辉钼矿，国内储量丰富，且具有较好的电磁波吸收特性，制备的MoS₂/Fe₃O₄/MoO₂复合吸波材料可作为电磁波吸收材料广泛应用于相应的电磁防护以及微波隐身领域。

[0021]

Attached Figure Description

附图说明

[n0018]

Figure 1 is the XRD pattern of MoS₂/Fe₃O₄/MoO₂-3.0 prepared in Example 3 of the present invention.

图1为本发明实施例3制备得到的MoS₂/Fe₃O₄/MoO₂-3.0的XRD图。

[n0019]

Figure 2 shows the microwave absorption performance of the MoS₂/Fe₃O₄/MoO₂-1.0 sample of Example 1 of the present invention.

图2为本发明实施例1的MoS₂/Fe₃O₄/MoO₂-1.0样品的吸波性能图。

[n0020]

Figure 3 shows the microwave absorption performance of the MoS₂/Fe₃O₄/MoO₂-2.0 sample of Example 2 of the present invention.

图3为本发明实施例2的MoS₂/Fe₃O₄/MoO₂-2.0样品的吸波性能图。

[n0021]

Figure 4 shows the microwave absorption performance of the MoS₂/Fe₃O₄/MoO₂-3.0 sample of Example 3 of the present invention.

图4为本发明实施例3的MoS₂/Fe₃O₄/MoO₂-3.0样品的吸波性能图。

[n0022]

Figure 5 shows the microwave absorption performance of the MoS₂/Fe₃O₄/MoO₂-4.0 sample of Comparative Example 1 of the present invention.

图5为本发明对照例1的MoS₂/Fe₃O₄/MoO₂-4.0样品的吸波性能图。

[n0023]

Figure 6 shows the microwave absorption performance of the MoS₂/Fe₃O₄/MoO₂-5.0 sample of Comparative Example 2 of the present invention.

图6为本发明对照例2的MoS₂/Fe₃O₄/MoO₂-5.0样品的吸波性能图。

[0028]

Detailed Implementation

具体实施方式

[n0024]

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

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

It should be understood that the specific embodiments described herein are merely illustrative of the invention and are not intended to limit the invention.

应当理解，此处所描述的具体实施例仅仅用以解释本发明，并不用于限定本发明。

[n0025]

The following embodiments are only for further detailed description of the present invention, but do not constitute any limitation on the present invention; the materials used in the

following embodiments are molybdenite from the Yumu Mountain in Myanmar; the purity of the molybdenite is ⩾96.00%.

下述实施例仅对本发明作进一步详细说明，但不构成对本发明的任何限制；下述实施例中所用的材料，辉钼矿产地：缅甸蓄木山；辉钼矿纯度：⩾96.00%。

Unless otherwise specified, all other materials were purchased from conventional chemical reagent companies and raw material suppliers.

其他材料如无特殊说明，均购自常规化学试剂公司和原料供应商。

[n0026]

Example 1

实施例1

[n0027]

A MoS₂/Fe₃O₄/MoO₂ composite material based on flash-synthesized natural molybdenite and hematite, and its preparation method, comprising the following steps:

一种基于闪烧合成的天然辉钼矿和赤铁矿的MoS₂/Fe₃O₄/MoO₂复合材料及其制备方法，包括以下步骤：

[n0028]

1) Weigh 7.5 mmol (calculated as molybdenum disulfide) of molybdenite powder (particle size ⩽ 0.25 mm) and 7.5 mmol (calculated as iron oxide) of hematite (particle size ⩽ 0.2 mm) using an analytical balance, and mix them thoroughly in a mortar.

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1)使用分析天平称量7.5mmol(以二硫化钼计)辉钼矿粉末(粒径≤0.25mm)和7.5mmol(以氧化铁计)的赤铁矿(粒径≤0.2mm)，在研钵中充分混合均匀；

[n0029]

2) The mixture obtained in 1) is loaded into a prefabricated corundum ring, the upper and lower ends are sealed with graphite sheets, platinum wire is attached, and it is fixed with a clamp;

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2)将1)所得混合物装进预制的刚玉环中，上下端口使用石墨片封口，接上铂丝，用夹具固定；

[n0030]

3) Place the device described in 2) into a muffle furnace and heat the muffle furnace to 400°C at a rate of 10°C/min;

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3)将2)所述装置放入马弗炉中，按10°C/min的速率，使马弗炉升温至400°C；

[n0031]

4) Connect the platinum wire to the power supply, set the voltage to 600V, the current to 1.5A, and the flash burn time to 3 minutes;

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4)铂丝连接电源，设置电压600V，电流1.5A，闪烧时间3分钟；

[n0032]

5) Natural cooling yields the MoS₂/Fe₃O₄/MoO₂ composite material, labeled as MoS₂/Fe₃O₄/MoO₂-1.0.

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5)自然降温，所得产物即为MoS₂/Fe₃O₄/MoO₂复合材料，标示为MoS₂/Fe₃O₄/MoO₂-1.0。

[n0033]

Example 2:

实施例2：

[n0034]

The difference from Example 1 is that: in step 1), 10 mmol of molybdenite powder and 5 mmol of hematite powder were weighed, and the resulting product was labeled as MoS₂/Fe₃O₄/MoO₂-2.0; all other aspects are the same as in Example 1.

与实施例1的区别在于：步骤1)中辉钼矿粉末称取10mmol，赤铁矿粉末取5mmol，所得产物标示为MoS₂/Fe₃O₄/MoO₂-2.0；其它均同实施例1。

[n0035]

Example 3:

实施例3：

[n0036]

The difference from Example 1 is that: in step 1), 5 mmol of molybdenite powder and 10 mmol of hematite powder were weighed, and the resulting product was labeled as MoS₂/Fe₃O₄/MoO₂-3.0; all other aspects are the same as in Example 1.

与实施例1的区别在于：步骤1)中辉钼矿粉末称取5mmol，赤铁矿粉末取10mmol，所得产物标示为MoS₂/Fe₃O₄/MoO₂-3.0；其它均同实施例1。

[n0037]

Figure 1 shows the XRD pattern of the MoS₂/Fe₃O₄/MoO₂-3.0 sample in Example 3.

图1为实施例3中MoS₂/Fe₃O₄/MoO₂-3.0样品的XRD图。

As shown in Figure 1, the MoS₂/Fe₃O₄/MoO₂ composite material obtained in this invention exhibits 2H-MoS₂ as the main phase, while Fe₃O₄(PDF#89-6088) and MoO₂ (PDF#32-0671) exist as newly formed phases.

由图1可知：本发明所得产品中MoS₂/Fe₃O₄/MoO₂复合材料表现为保持着2H-MoS₂作为主相，Fe₃O₄ (PDF#89-6088)、MoO₂(PDF#32-0671)作为新生相存在。

[n0038]

Compare with Example 1:

对照例1：

[n0039]

The difference from Example 3 is that step 4) the voltage parameter is changed to 400V; everything else is the same as Example 1.

与实施例3的区别在于：步骤4)电压参数改为400V；其它均同实施例1。

[n0040]

Compare with Example 2:

对照例2：

[n0041]

The difference from Example 3 is that step 4) the voltage parameter is changed to 500V;

everything else is the same as Example 1.

与实施例3的区别在于：步骤4)电压参数改为500V；其它均同实施例1。

[n0042]

Electromagnetic absorption characteristic test:

电磁吸收特性测试：

[n0043]

The prepared MoS₂/Fe₃O₄/MoO₂

composite material with electromagnetic wave absorption properties was used as a sample,

and the dielectric and electromagnetic properties of the material were analyzed using a vector network analyzer (VNA, Agilent N5234A).

取所制得的具备电磁波吸收性能的MoS₂/Fe₃O₄/MoO₂复合材料作为样品，利用矢量网络分析仪(VNA，Agilent N5234A)对材料进行介电性能和电磁性能的分析。

The specific method is as follows: Paraffin wax and the sample are mixed at a mass ratio of 3:7 and pressed into a ring-shaped sample with an outer diameter of 7mm, an inner diameter of 3mm, and a thickness of 2.5mm for testing. The absorption performance of different samples is simulated and tested using a vector network analyzer.

具体做法是：利用石蜡与样品按照3：7的质量比进行混合，并压制成外径7mm，内径3mm，厚度2.5mm的环状样品进行测试，利用矢量网络分析仪模拟测试不同样品的吸波性能。

[n0044]

The microwave absorption performance curve of the MoS₂/Fe₃O₄/MoO₂-1.0 composite material prepared in Example 1 is shown in Figure 2. The numbers in the figure represent the sample thickness simulated by the network vector analyzer.

实施例1制得的MoS₂/Fe₃O₄/MoO₂-1.0复合材料的吸波性能曲线，如图2所示，图例中的数字代表网络矢量分析仪所模拟的样品厚度。

The RL_NER83 of the MoS₂/Fe₃O₄/MoO₂-1.0 composite material reaches -22.85dB (f is 12.56GHz, d is 2.1mm).

MoS₂/Fe₃O₄/MoO₂-1.0复合材料的RL_{min}达到-22.85dB(f为12.56GHz，d为2.1mm)。

The RL_NER88 of the MoS₂/Fe₃O₄/MoO₂-2.0 composite material reaches -28.14dB (f is 11.88GHz, d is 2.2mm).

MoS₂/Fe₃O₄/MoO₂-2.0复合材料的RL_{min}达到-28.14dB(f为11.88GHz，d为2.2mm)。

The RL_NER93 of the MoS₂/Fe₃O₄/MoO₂-3.0 composite material reaches -47.85dB (f is 11.54GHz, d is 2.0mm).

MoS₂/Fe₃O₄/MoO₂-3.0复合材料的RL_{min}达到-47.85dB(f为11.54GHz，d为2.0mm)。

The above results indicate that RL_{min} is affected by the ratio of molybdenite and hematite, while RL_{min} performs best in the MoS₂/Fe₃O₄/MoO₂-3.0 range.

以上结果表明，RL_{min}受到辉钼矿和赤铁矿比例的影响，而RL_{min}在MoS₂/Fe₃O₄/MoO₂-3.0中表现最佳。

[n0045]

Figure 5 shows the electromagnetic wave absorption performance curves of the MoS₂/Fe₃O₄/MoO₂ composite material with electromagnetic wave absorption properties prepared in Comparative Example 1.

对照例1制得的具备电磁波吸收性能的MoS₂/Fe₃O₄/MoO₂复合材料吸波性能曲线如图5所示。

Figure 6 shows the electromagnetic wave absorption performance curves of the MoS₂/Fe₃O₄/MoO₂ composite material with electromagnetic wave absorption properties prepared in Comparative Example 2.

对照例2制得的具备电磁波吸收性能的MoS₂/Fe₃O₄/MoO₂复合材料吸波性能曲线如图6所示。

As shown in Figures 5 and 6, the microwave absorption performance of the sample prepared in the control example is worse than that of the sample prepared in Example 3. This indicates that the lower voltage leads to a decrease in the microwave absorption performance of the MoS₂/Fe₃O₄/MoO₂ composite material.

由图5图6可知：与实施例3制得的样品相比，对照例制得的样品的吸波性能较差，这说明较低的电压导致MoS₂/Fe₃O₄/MoO₂复合材料的吸波性能减弱。

[n0046]

The foregoing has shown and described the basic principles, main features, and advantages of the present invention.

以上显示和描述了本发明的基本原理、主要特征及本发明的优点。

Those skilled in the art should understand that the present invention is not limited to the above embodiments. The embodiments and descriptions in the specification are merely

illustrative of the principles of the present invention. Various changes and modifications can be made to the present invention without departing from its spirit and scope, and all such changes and modifications fall within the scope of the present invention as claimed.

本行业的技术人员应该了解，本发明不受上述实施例的限制，上述实施例和说明书中描述的只是说明本发明的原理，在不脱离本发明精神和范围的前提下，本发明还会有各种变化和改进，这些变化和改进都落入要求保护的本发明范围内。

The scope of protection of this invention is defined by the appended claims and their equivalents.

本发明要求保护范围由所附的权利要求书及其等效物界定。