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(54) METHOD FOR PREPARING HEAT DISSIPATION COMPONENT WITH HIGH FLEXIBILITY MADE OF GRAPHITE OR GRAPHENE MATERIAL

(57) The present disclosure disclose a method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material, which includes that follow steps: 1) plasma cleaning a graphite or graphene raw material; 2) taking preparation materials of an activator including the following components in percentage by weight: 10-20% of sulfuric acid, 0.05-1% of an OP-10 surfactant, 0.05-1% of sodium dodecyl sulfate, and the balance of water; 3) continually cleaning the graphite or graphene raw material with the activator; 4) cleaning the graphite or graphene raw material with deionized water; 5) conducting a electroplating process on a surface of the graphite or graphene raw material to form a copper film layer; 6) continually cleaning the graphite or graphene raw material with deionized water; 7) forming a protective film on the graphite or graphene raw material by soaking; and 8) drying the graphite or graphene raw material electroplated with the copper film layer. Compared with the prior art, in the present disclosure, the surface of graphite or graphene treated with the activator has a uniform copper film layer with good binding quality during electroplating, and the flexibility of the prepared heat dissipation component is enhanced.

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Description

TECHNICAL FIELD

[0001] The present disclosure belong to the field of preparation of parts made of a graphite or graphene material, and particularly relate to a method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material.

BACKGROUND

[0002] Graphite is a two-dimensional carbon nanomaterial presented with hexagonal honeycomb lattices and composed of carbon atoms in sp2 hybrid orbitals. Graphite has a very good thermal conduction performance. The pure and defect-free single-layer graphite has thermal conductivity up to 5300 W/mK, and is the carbon material with the highest thermal conductivity so far, and its thermal conductivity is higher than those of a single-walled carbon nanotube (3500 W/mK) and a multi-walled carbon nanotube (3000 W/mK). When it is used as a carrier, the thermal conductivity can also reach 600 W/MK. Furthermore, the ballistic thermal conductivity of graphite can lower the lower limit of the ballistic thermal conductivity of a carbon nanotube of unit circumference and length.

[0003] All kinds of electronic elements in electronic products need to dissipate heat. When traditional graphite or graphene is applied for heat dissipation of the electronic elements, it mainly fixes a metal layer on the surface of graphite or graphene, and in particular the metal layer is fixed onto graphite or graphene by bonding or electroplating, wherein electroplating is better than bonding in heat conduction efficiency.

[0004] In order to ensure the electroplating quality of the metal layer on the surface of graphite or graphene, it is usually necessary to subject the surface of graphite or graphene to multiple passes of cleaning processes before electroplating. During the cleaning process, after the surface of graphite or graphene is treated with an OP-10 surfactant, the smoothness of the surface of graphite or graphene is general, which leads to poor binding effect and uneven thickness of the metal layer electroplated on the surface of graphite or graphene, and thus affects the flexibility of the prepared heat dissipation component, and the surface of the heat dissipation component is easy to generate creases.

SUMMARY

[0005] An objective of the present disclosure is to provide a method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material, which includes the following steps: firstly, plasma cleaning a surface of graphite or graphene, and then treating the surface of graphite or graphene with an activator compounded by sulfuric acid, an OP-10 surfactant and sodium dodecyl sulfate, so that the surface of the material has good smoothness, and it ensures a copper film layer electroplated on the surface of graphite or graphene has good binding quality and uniform, and enhances the flexibility of the prepared heat dissipation component, and the surface of the heat dissipation component is not easy to generate creases.

[0006] In order to achieve the aforementioned objective, the present disclosure adopts the following technical solution: a method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material, including the following steps:

1) plasma cleaning a graphite or graphene raw material;
2) taking preparation materials of an activator including the following components in percentage by weight: 10-20% of sulfuric acid, 0.05-1% of an OP-10 surfactant, 0.05-1% of sodium dodecyl sulfate, and the balance of water;
3) mixing the aforementioned components of the activator to prepare the activator, and continually cleaning the graphite or graphene raw material with the activator;
4) continually cleaning the graphite or graphene raw material with deionized water;
5) conducting a electroplating process on a surface of the graphite or graphene raw material to form a copper film layer;
6) continually cleaning the graphite or graphene raw material electroplated with the copper film layer on the surface thereof with deionized water;
7) forming a protective film on the graphite or graphene raw material by soaking; and
8) drying the graphite or graphene raw material electroplated with the copper film layer.

[0007] As a further description of the aforementioned technical solution:

[0008] In the step 1), the graphite or graphene raw material is placed in a plasma cleaning machine for cleaning.

[0009] As a further description of the aforementioned technical solution:

[0010] In the step 2), the activator includes the following components in percentage by weight: 12-16% of sulfuric acid, 0.05-0.5% of an OP-10 surfactant, 0.05-0.5% of sodium dodecyl sulfate, and the balance of water.

[0011] As a further description of the aforementioned technical solution:

[0012] in the step 2), the activator includes the following components in percentage by weight: 15% sulfuric acid, 0.1% of the OP-10 surfactant, 0.1% of sodium dodecyl sulfate, and the balance of water.

[0013] As a further description of the aforementioned technical solution:

[0014] in the step 5), the graphite or graphene raw material is subjected to the electroplating process twice with an electroplating potion, and the electroplating potion includes the following components in percentage by weight: 5% of copper ions; 14% of sulfuric acid; 0.8% of a brightener; 0.06% of an adjuvant; 0.06% of a leveling agent; and the balance of water.

[0015] As a further description of the aforementioned technical solution:

[0016] when the graphite or graphene raw material is electroplated with the electroplating potion for the first time, the temperature is 40 celsius degrees and the time is 20 minutes; and when the graphite or graphene raw material is electroplated with the electroplating potion for the second time, the temperature is 40 celsius degrees and the time is 15 minutes.

[0017] As a further description of the aforementioned technical solution:

in the step 6), the graphite or graphene raw material is firstly soaked in a 5 g/L methyl benzotriazole solution for 20-30 seconds to form a first layer of protective film, and then put into a 0.5 g/ L cetylpyridinium bromide solution for 20-30 seconds to form a second layer of protective film.

[0018] In view of the above, by employing the aforementioned technical solution, the present disclosure has the following beneficial effects.

1. in the present disclosure, the graphite or graphene raw material is firstly placed in a plasma cleaning machine for plasma cleaning, and then the surface of graphite or graphene is treated with an activator compounded by sulfuric acid, the OP-10 surfactant and sodium dodecyl sulfate. Sulphuric acid can wash away oil stains from the graphite or graphene raw material, the OP-10 surfactant improves the smoothness of the surface of the graphite or graphene raw material, and sodium dodecyl sulfate increases the dispersibility of graphite or graphene and further increases the smoothness of the surface of the graphite or graphene raw material, so that the electroplated copper film layer has good binding quality and is uniform, which enhances the flexibility of the prepared heat dissipation component, and the surface of the heat dissipation component is not easy to generate creases, and the heat dissipation and acid and alkali resistance performances of the heat dissipation component are improved.
2. In the present disclosure, after the copper film layer is formed on the graphite or graphene raw material by electroplating, the graphite or graphene raw material electroplated with the copper film layer is first soaked in a 5 g/L methyl benzotriazole solution for 20-30 seconds to form a first layer of protective film on the copper film layer, and then put into a 0.5 g/L cetylpyridinium bromide solution for 20-30 seconds to form a second layer of protective film on the copper film layer, which effectively prevents the copper film layer from color changing.

DETAILED DESCRIPTION OF THE EMBODIMENTS

[0019] Exemplary examples of the present disclosure will be described in more detail below. Although exemplary examples of the present disclosure are shown, it should be understood that the present disclosure may be implemented in various forms, and should not be limited by the examples set forth herein. On the contrary, these embodiments are provided to enable a more thorough understanding of the present disclosure and to fully convey the scope of the disclosure to those skilled in the art.

Example 1:

[0020] This example of the present disclosure provided a method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material, including the following steps:

1) placing a graphite or graphene raw material in a plasma cleaning machine for plasma cleaning, wherein the plasma cleaning machine had an air pressure of 2 MPa and a power of 550-600 W, and the time for the plasma cleaning was 30 min;
2) taking preparation materials of an activator including the following components in percentage by weight: 15% of sulfuric acid, 0.1% of an OP-10 surfactant, 0.1% of sodium dodecyl sulfate, and the balance of water;
3) mixing the aforementioned components of the activator to prepare the activator, and continually cleaning the graphite or graphene raw material with the activator;
4) continually cleaning the graphite or graphene raw material with deionized water;
5) conducting a electroplating process on a surface of the graphite or graphene raw material twice to form a copper film layer;

6) continually cleaning the graphite or graphene raw material electroplated with the copper film layer on the surface thereof with deionized water;
7) firstly, soaking the graphite or graphene raw material plated with the copper film layer in a 5 g/L methyl benzotriazole solution for 20-30 seconds to form a first layer of protective film on the copper film layer, and then putting into a 0.5 g/L cetylpyridinium bromide solution for 20-30 seconds to form a second layer of protective film on the copper film layer to effectively prevent the copper film from color changing;
8) drying the graphite or graphene raw material electroplated with the copper film layer.

[0021] In the step 5), it included two electroplating procedures: firstly, the graphite or graphene raw material was subjected to primary electroplating with an electroplating potion; and secondly, the graphite or graphene raw material was subjected to secondary electroplating with the electroplating potion. The electroplating potion included the following components in percentage by weight: 5% of copper ions; 14% of sulfuric acid; 0.8% of a brightener; 0.06% of an adjuvant; 0.06% of a leveling agent; and the balance of water. During the electroplating process, pickling could be carried out at the same time, so that the flatness of the heat dissipation component was good.

[0022] Various process parameters of Example 1 were shown in the table below:

Electroplating process	Component name	Potion concentration	Temperature	Time
Activation	sulfuric acid	15%	25°C	3 minutes
	Surfactant A (OP-10)	0.10%		
	sodium dodecyl sulfate	0.10%		
Washing with pure water	deionized water		25°C	20 seconds
Pre-plating of copper	Copper ions	50 g/l	40°C	20 minutes
	sulfuric acid	14%		
	Additive A (brightener)	0.80%		
	Additive B (adjuvant)	0.06%		
	Additive C (leveling agent)	0.06%		
Electroplating of copper	Copper ions	50 g/l	40°C	15 minutes
	sulfuric acid	14%		
	Additive A (brightener)	0.80%		
	Additive B (adjuvant)	0.06%		
	Additive C (leveling agent)	0.06%		
Cleaning with pure water	deionized water		25°C	20 seconds
Protection 1	methyl benzotriazole	5g/l	25°C	20 seconds
Protection 2	cetylpyridinium bromide	0.5g/l	25°C	20 seconds
Drying	drying oven		80°C	2 minutes

Example 2

[0023] This example of the present disclosure provided a method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material, including the following steps:

1) placing a graphite or graphene raw material in a plasma cleaning machine for plasma cleaning, wherein the plasma cleaning machine had an air pressure of 2 MPa and a power of 550-600 W, and the time for the plasma cleaning was 30 min;
2) taking preparation materials of an activator including the following components in percentage by weight: 16% of sulfuric acid, 0.2% of an OP-10 surfactant, 0.2% of sodium dodecyl sulfate, and the balance of water;
3) mixing the aforementioned components of the activator to prepare the activator, and continually cleaning the graphite or graphene raw material with the activator;
4) continually cleaning the graphite or graphene raw material with deionized water;

5) conducting a electroplating process on a surface of the graphite or graphene raw material twice to form a copper film layer;
6) continually cleaning the graphite or graphene raw material electroplated with the copper film layer on the surface thereof with deionized water;
7) firstly, soaking the graphite or graphene raw material plated with the copper film layer in a 5 g/L methyl benzotriazole solution for 20-30 seconds to form a first layer of protective film on the copper film layer, and then putting into a 0.5 g/L cetylpyridinium bromide solution for 20-30 seconds to form a second layer of protective film on the copper film layer to effectively prevent the copper film from color changing;
8) drying the graphite or graphene raw material electroplated with the copper film layer.

[0024] In view of the above, compared with the prior art, the method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material as provided by the aforementioned examples has the following advantages. The graphite or graphene raw material is firstly placed in a plasma cleaning machine for plasma cleaning, and then the surface of graphite or graphene is treated with an activator compounded by sulfuric acid, the OP-10 surfactant and sodium dodecyl sulfate. Sulphuric acid can wash away oil stains from the graphite or graphene raw material, the OP-10 surfactant improves the smoothness of the surface of the graphite or graphene raw material, and sodium dodecyl sulfate increases the dispersibility of graphite or graphene and further increases the smoothness of the surface of the graphite or graphene raw material, so that the electroplated copper film layer has good binding quality and is uniform, which enhances the flexibility of the prepared heat dissipation component, and the surface of the heat dissipation component is not easy to generate creases, and the heat dissipation and acid and alkali resistance performances of the heat dissipation component are improved. After the copper film layer is formed on the graphite or graphene raw material by electroplating, the graphite or graphene raw material electroplated with the copper film layer is first soaked in a 5 g/L methyl benzotriazole solution for 20-30 seconds to form a first layer of protective film on the copper film layer, and then put into a 0.5 g/L cetylpyridinium bromide solution for 20-30 seconds to form a second layer of protective film on the copper film layer, which effectively prevents the copper film layer from color changing.

[0025] The aforementioned description is only preferred specific embodiments of the present disclosure, and the claimed scope of the present disclosure is not limited thereto. Equivalent substitutions or modifications can be made by those of skills in the art according to the technical solution and inventive concept of the present disclosure, without departing from the technical scope disclosed by the present disclosure. These substitutions or modifications all fall within the claimed scope of the present disclosure.

Claims

1. A method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material, comprising the following steps:

   1) plasma cleaning a graphite or graphene raw material;
   2) taking preparation materials of an activator comprising the following components in percentage by weight: 10-20% of sulfuric acid, 0.05-1% of an OP-10 surfactant, 0.05-1% of sodium dodecyl sulfate, and the balance of water;
   3) mixing the aforementioned components of the activator to prepare the activator, and continually cleaning the graphite or graphene raw material with the activator;
   4) continually cleaning the graphite or graphene raw material with deionized water;
   5) conducting a electroplating process on a surface of the graphite or graphene raw material to form a copper film layer;
   6) continually cleaning the graphite or graphene raw material electroplated with the copper film layer on the surface thereof with deionized water;
   7) forming a protective film on the graphite or graphene raw material by soaking; and
   8) drying the graphite or graphene raw material electroplated with the copper film layer.

2. The method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material according to claim 1, wherein in the step 1), the graphite or graphene raw material is placed in a plasma cleaning machine for cleaning.

3. The method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material according to claim 1, wherein in the step 2), the activator comprises the following components in percentage by

weight: 12-16% of sulfuric acid, 0.05-0.5% of the OP-10 surfactant, 0.05-0.5% of sodium dodecyl sulfate, and the balance of water.

4. The method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material according to claim 3, wherein in the step 2), the activator comprises the following components in percentage by weight: 15% sulfuric acid, 0.1% of the OP-10 surfactant, 0. 1% of sodium dodecyl sulfate, and the balance of water.

5. The method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material according to claim 1, wherein in the step 5), the graphite or graphene raw material is subjected to the electroplating process twice with an electroplating potion, and the electroplating potion comprises the following components in percentage by weight: 5% of copper ions; 14% of sulfuric acid; 0.8% of a brightener; 0.06% of an adjuvant; 0.06% of a leveling agent; and the balance of water.

6. The method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material according to claim 5, wherein in the step 5), when the graphite or graphene raw material is electroplated with the electroplating potion for the first time, the temperature is 40 celsius degrees and the time is 20 minutes; and when the graphite or graphene raw material is electroplated with the electroplating potion for the second time, the temperature is 40 celsius degrees and the time is 15 minutes.

7. The method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material according to claim 1, wherein in the step 6), the graphite or graphene raw material is firstly soaked in a 5 g/L methyl benzotriazole solution for 20-30 seconds to form a first layer of protective film, and then put into a 0.5 g/L cetylpyridinium bromide solution for 20-30 seconds to form a second layer of protective film.

Amended claims in accordance with Rule 137(2) EPC.

1. A method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material, comprising the following steps:

    1) plasma cleaning a graphite or graphene raw material;
    2) taking preparation materials of an activator comprising the following components in percentage by weight: 10-20% of sulfuric acid, 0.05-1% of an OP-10 surfactant, 0.05-1% of sodium dodecyl sulfate, and the balance being water;
    3) mixing the aforementioned components of the activator to prepare the activator, and continually cleaning the graphite or graphene raw material with the activator;
    4) continually cleaning the graphite or graphene raw material with deionized water;
    5) conducting a electroplating process on a surface of the graphite or graphene raw material to form a copper film layer;
    6) continually cleaning the graphite or graphene raw material electroplated with the copper film layer on the surface thereof with deionized water;
    7) forming a protective film on the graphite or graphene raw material by soaking, comprising: the graphite or graphene raw material is firstly soaked in a 5 g/L methyl benzotriazole solution for 20-30 seconds to form a first layer of protective film, and then put into a 0.5 g/L cetylpyridinium bromide solution for 20-30 seconds to form a second layer of protective film; and
    8) drying the graphite or graphene raw material electroplated with the copper film layer.

2. The method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material according to claim 1, wherein in the step 1), the graphite or graphene raw material is placed in a plasma cleaning machine for cleaning.

3. The method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material according to claim 1, wherein in the step 2), the activator comprises the following components in percentage by weight: 12-16% of sulfuric acid, 0.05-0.5% of the OP-10 surfactant, 0.05-0.5% of sodium dodecyl sulfate, and the balance being water.

4. The method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material according to claim 3, wherein in the step 2), the activator comprises the following components in percentage by

weight: 15% sulfuric acid, 0.1% of the OP-10 surfactant, 0.1% of sodium dodecyl sulfate, and the balance being water.

5. The method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material according to claim 1, wherein in the step 5), the graphite or graphene raw material is subjected to the electroplating process twice with an electroplating solution, and the electroplating solution comprises the following components in percentage by weight: 5% of copper ions; 14% of sulfuric acid; 0.8% of a brightener; 0.06% of an adjuvant; 0.06% of a leveling agent; and the balance being water.

6. The method for preparing a heat dissipation component with high flexibility made of a graphite or graphene material according to claim 5, wherein in the step 5), when the graphite or graphene raw material is electroplated with the electroplating solution for the first time, the temperature is 40 celsius degrees and the time is 20 minutes; and when the graphite or graphene raw material is electroplated with the electroplating solution for the second time, the temperature is 40 celsius degrees and the time is 15 minutes.

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EUROPEAN SEARCH REPORT

Application Number
EP 21 19 9008

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	CN 109 748 267 B (ONTAP PLATING PREC ELECT CO LTD) 14 May 2021 (2021-05-14)	1-6	INV. C25D3/38
A	* abstract * * examples 1-5 * * paragaph "summary of the invention" *	7	C25D5/10 C25D5/54
A	CN 108 823 615 A (JIAXING ZHONGYI CARBON TECH CO LTD) 16 November 2018 (2018-11-16) * abstract * * examples 1-4 *	1-7	
A	CN 103 943 281 B (UNIV ZHEJIANG) 4 May 2016 (2016-05-04) * abstract * * example 1 *	1-7	

TECHNICAL FIELDS SEARCHED (IPC)

C25D

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	29 March 2022	Lange, Ronny

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

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ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 21 19 9008

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
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29-03-2022

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
CN 109748267 B	14-05-2021	NONE	
CN 108823615 A	16-11-2018	NONE	
CN 103943281 B	04-05-2016	NONE	

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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82