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Zhang et al.

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(54) **METHOD FOR MAKING SEMIMETAL COMPOUND OF PT**

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This patent is subject to a terminal disclaimer.

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Sep. 28, 2016 (CN) 201610862880.2

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C30B 9/06 (2006.01)
C30B 23/00 (2006.01)

(Continued)

(52) **U.S. Cl.**

CPC **C30B 9/06** (2013.01); **C01B 19/007** (2013.01); **C30B 23/00** (2013.01); **C30B 25/00** (2013.01);

(Continued)

(58) **Field of Classification Search**

CPC C01B 19/007; C01P 2002/72; C01P 2002/82; C01P 2004/01; C01P 2004/60;
(Continued)

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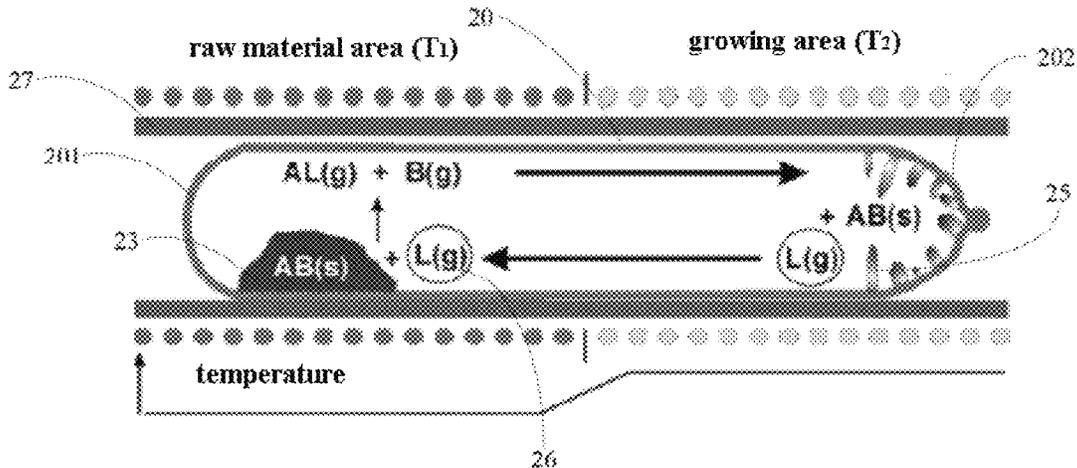
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(57) **ABSTRACT**

The disclosure relates to a method for making semimetal compound of Pt. The semimetal compound is a single crystal material of PtSe₂. The method comprises: placing pure Pt and pure Se in a reacting chamber as reacting materials; evacuating the reacting chamber to be vacuum less than 10 Pa; heating the reacting chamber to a first temperature of 600 degrees Celsius to 800 degrees Celsius and keeping for 24 hours to 100 hours; cooling the reacting chamber to a second temperature of 400 degrees Celsius to 500 degrees Celsius at a cooling rate of 1 degrees Celsius per hour to 10 degrees Celsius per hour and keeping for 24 hours to 100 hours to obtain a crystal material of PtSe₂; and separating the excessive reacting materials from the crystal material of PtSe₂.

11 Claims, 19 Drawing Sheets



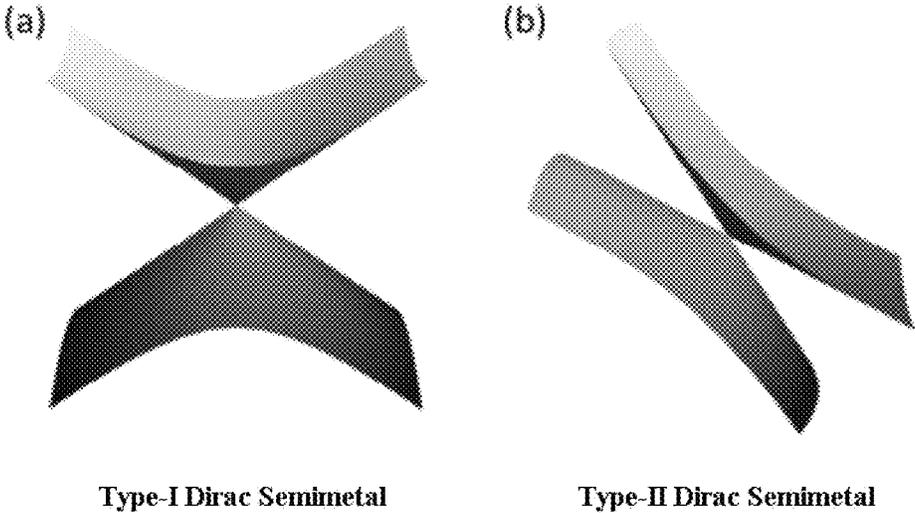
- (51) **Int. Cl.**
C01B 19/00 (2006.01)
C30B 25/00 (2006.01)
C30B 29/46 (2006.01)
- (52) **U.S. Cl.**
CPC *C30B 29/46* (2013.01); *C01P 2002/72*
(2013.01); *C01P 2002/82* (2013.01); *C01P*
2004/01 (2013.01); *C01P 2004/60* (2013.01)
- (58) **Field of Classification Search**
CPC C30B 23/00; C30B 25/00; C30B 29/46;
C30B 9/06
See application file for complete search history.

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(PRIOR ART)

FIG. 1

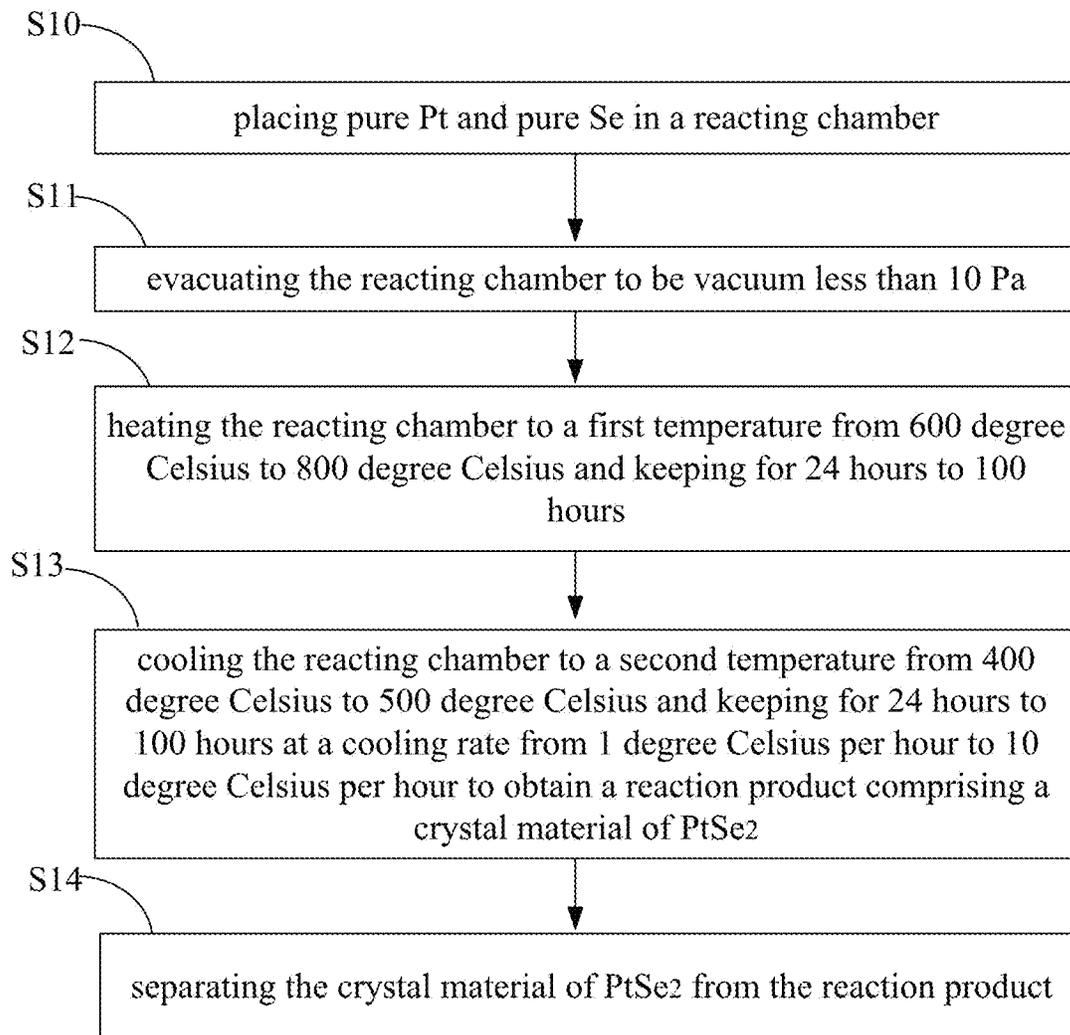


FIG. 2

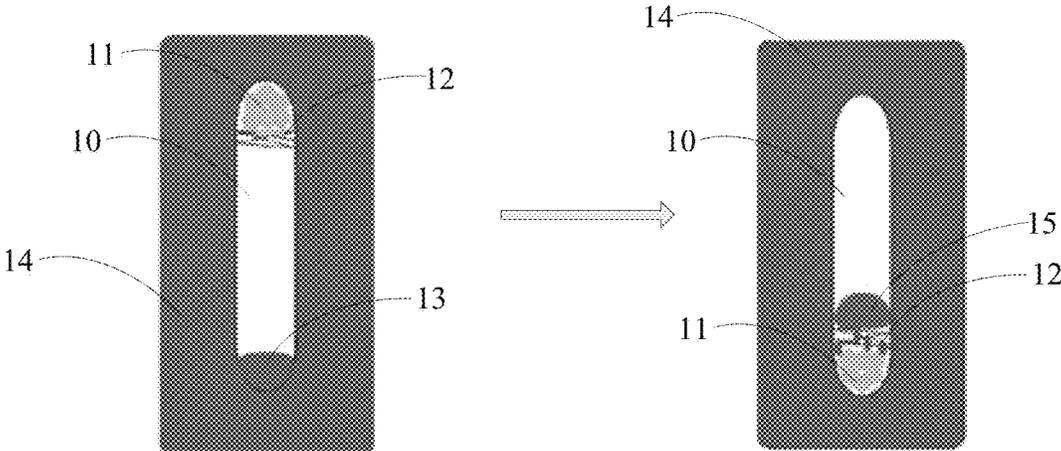


FIG. 3

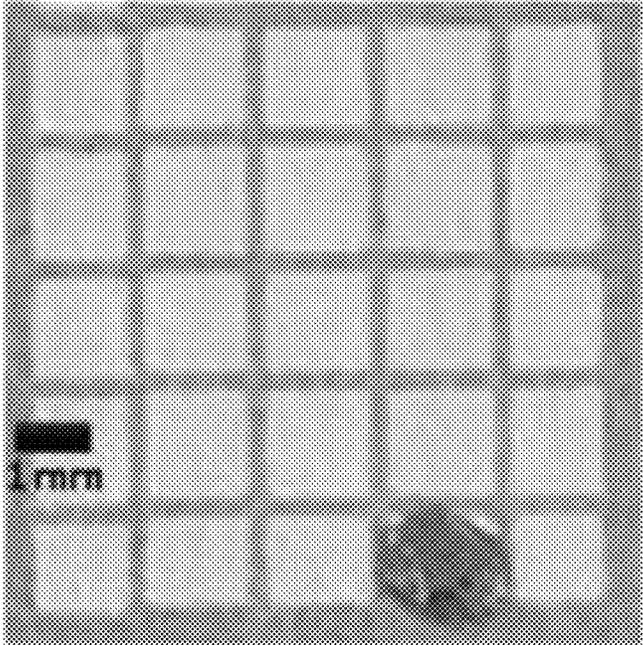


FIG. 4

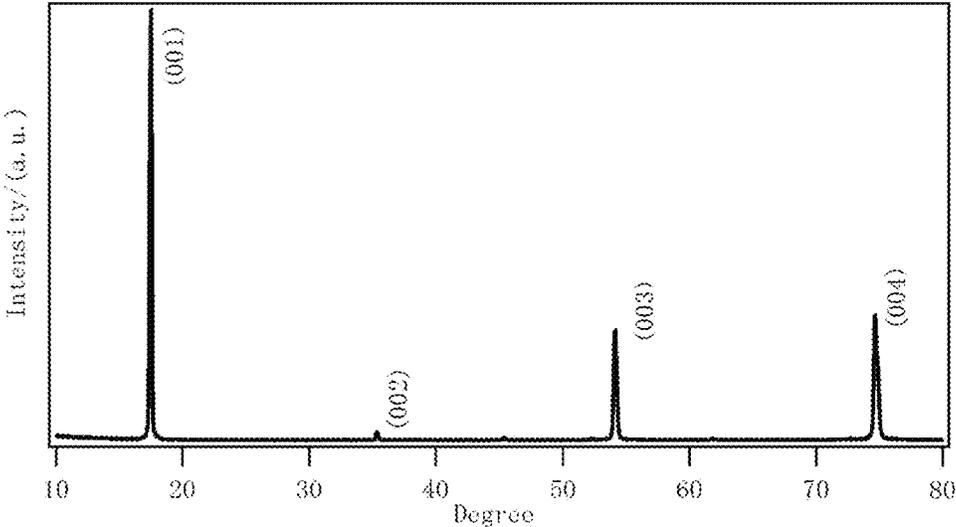


FIG. 5

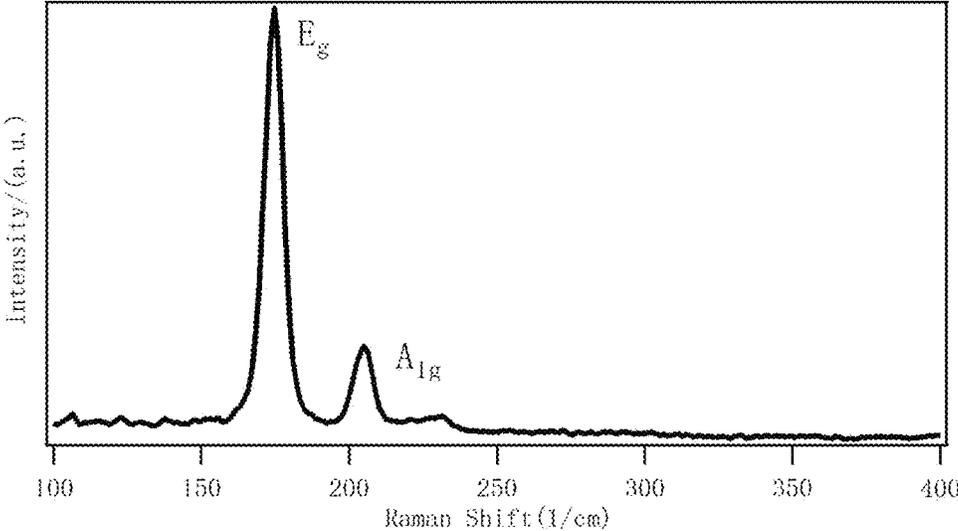


FIG. 6

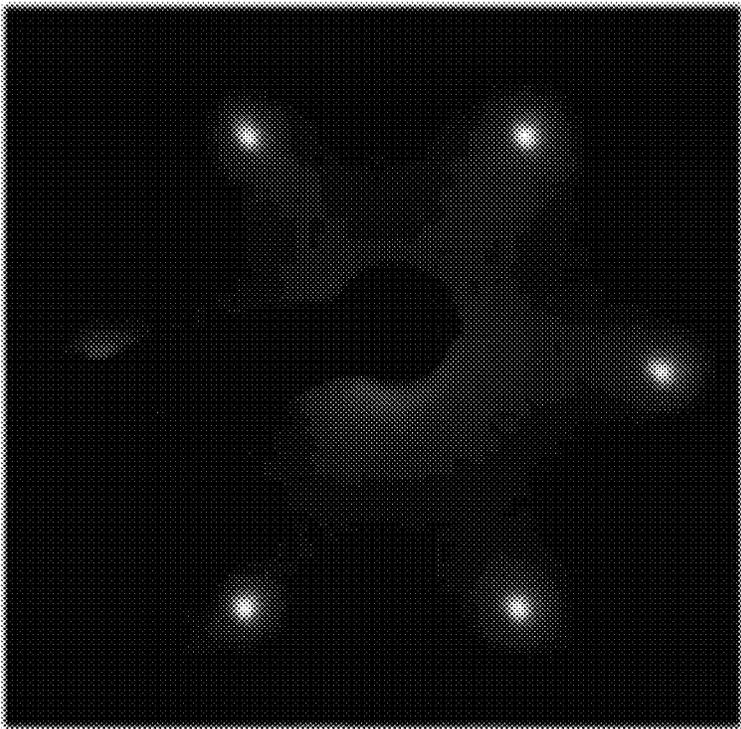


FIG. 7

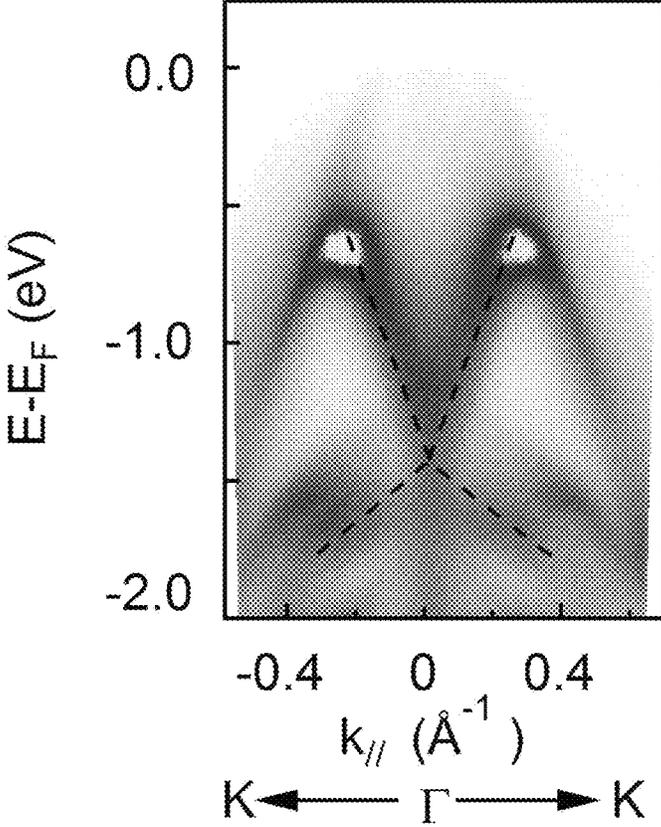


FIG. 8

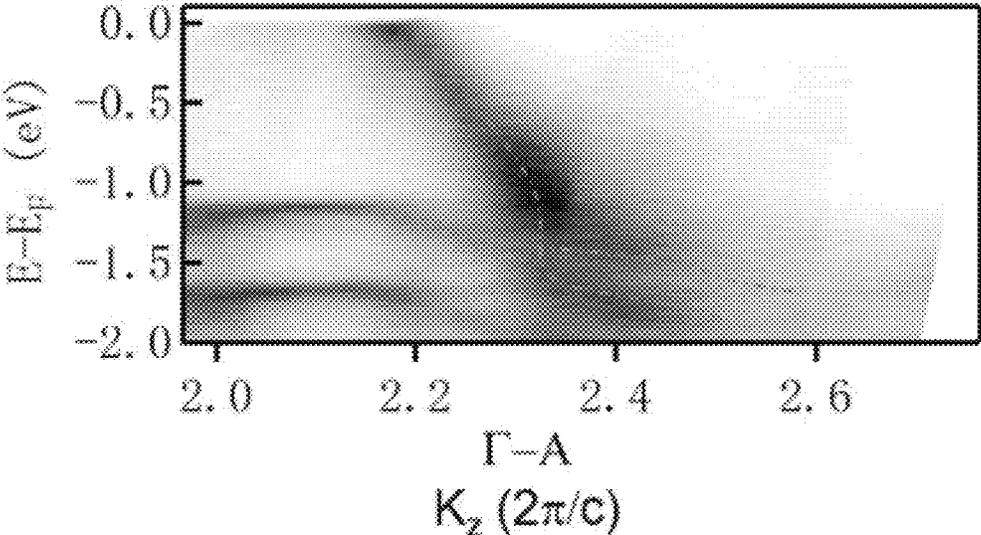


FIG. 9

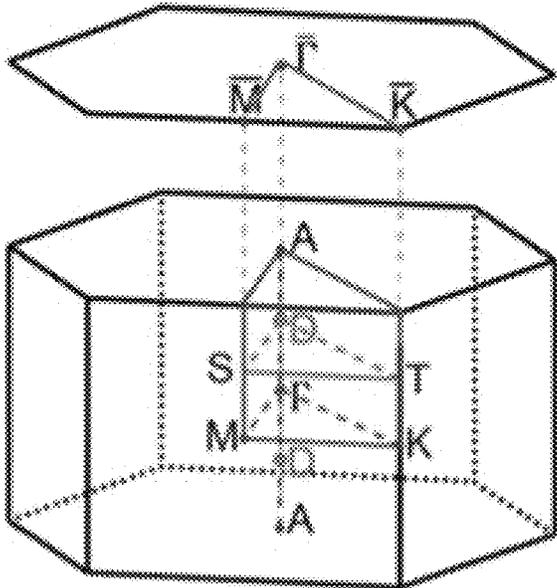


FIG. 11

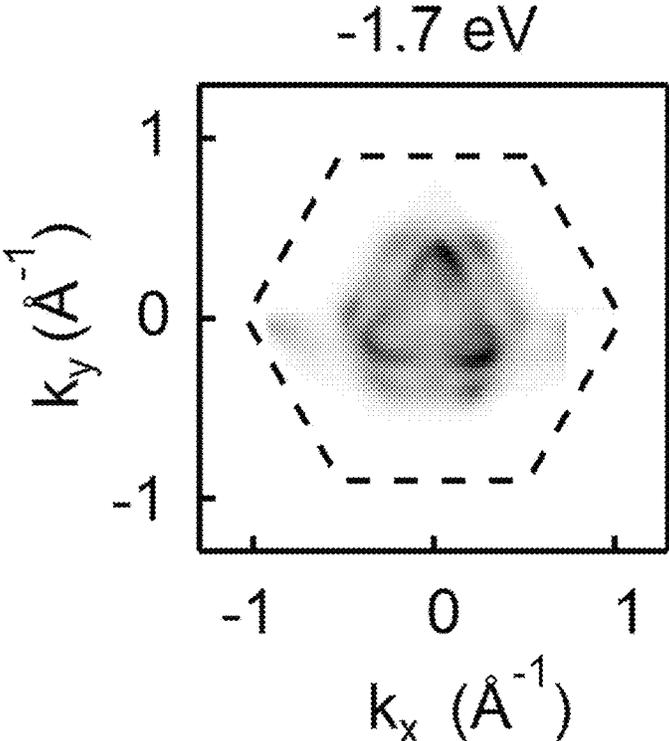


FIG. 12

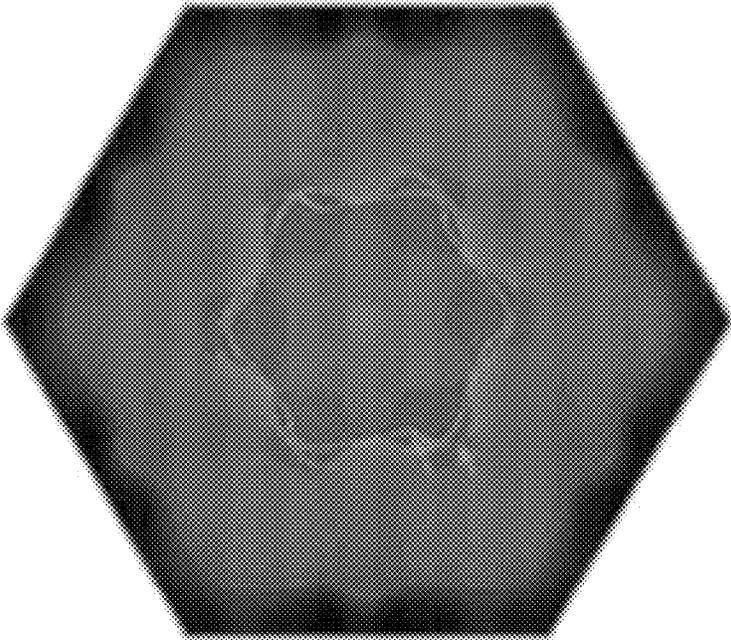


FIG. 13

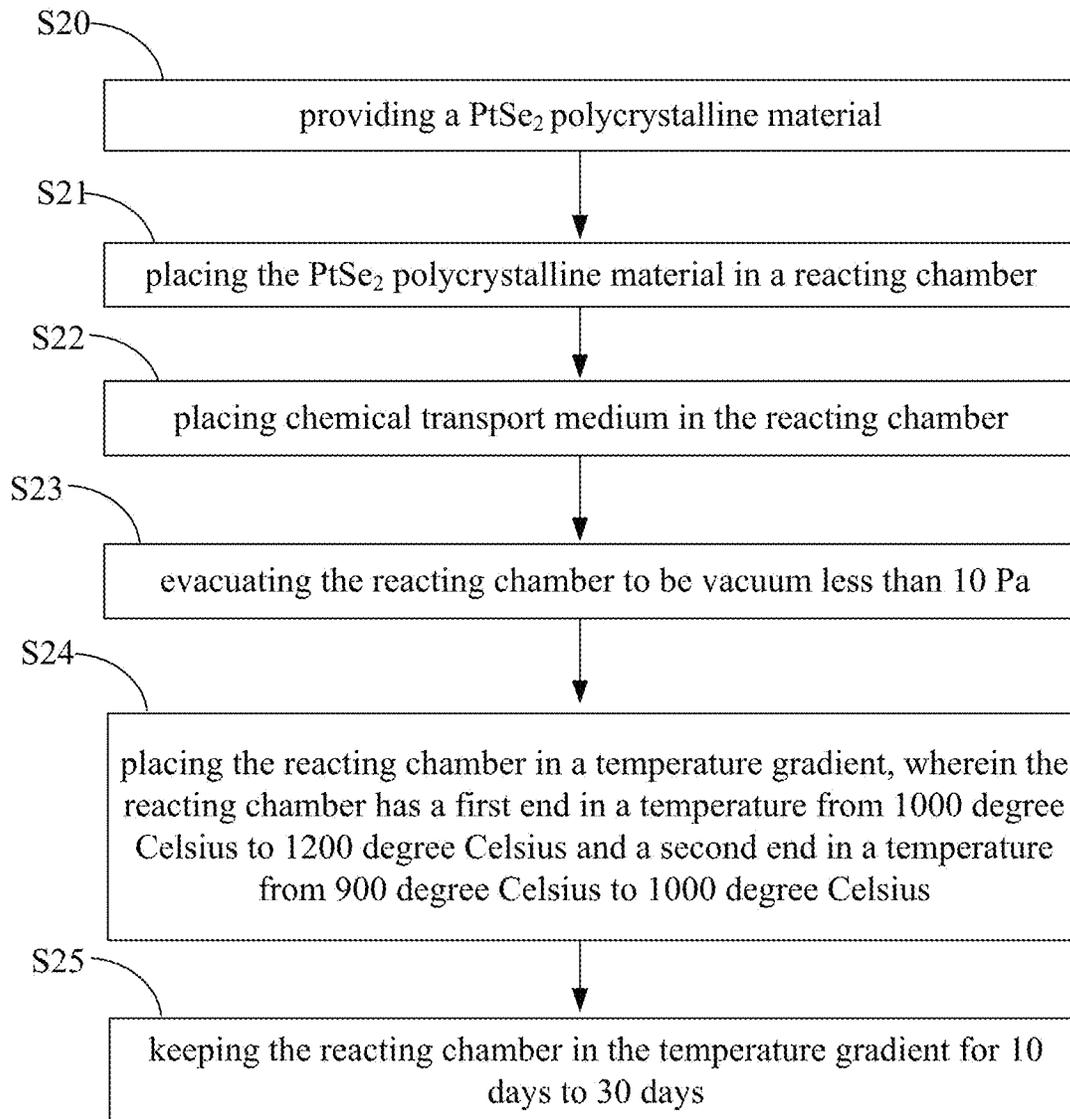


FIG. 14

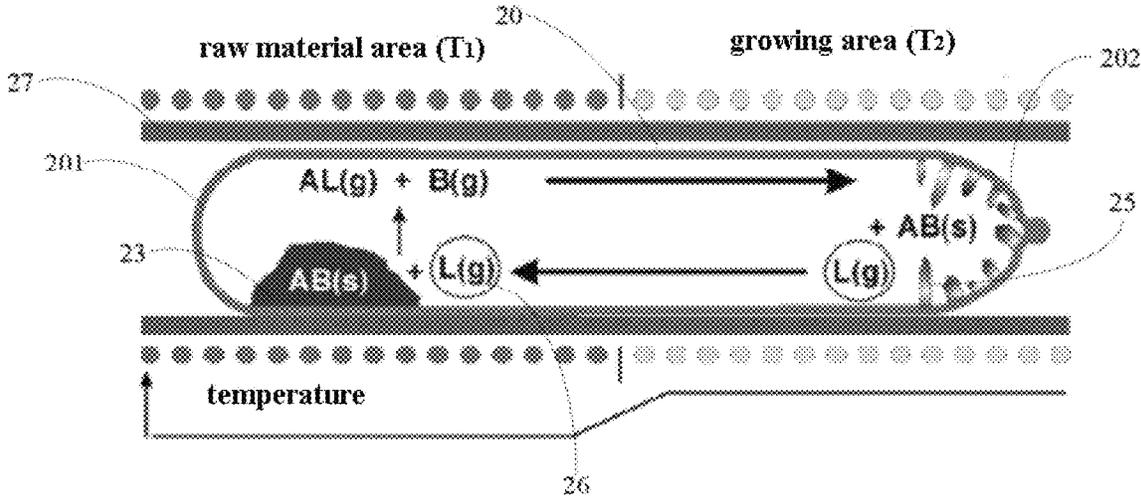


FIG. 15

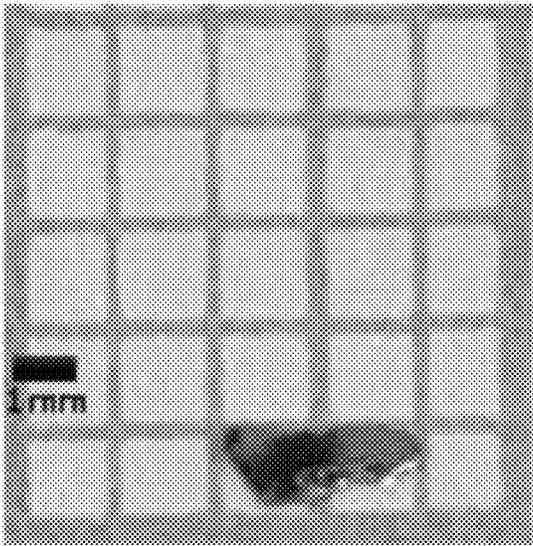


FIG. 16

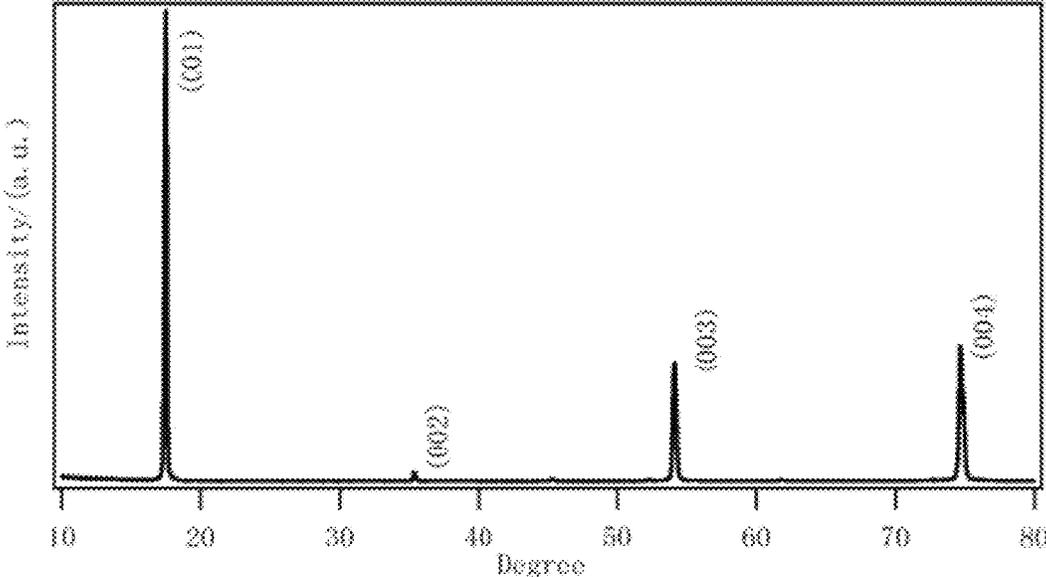


FIG. 17

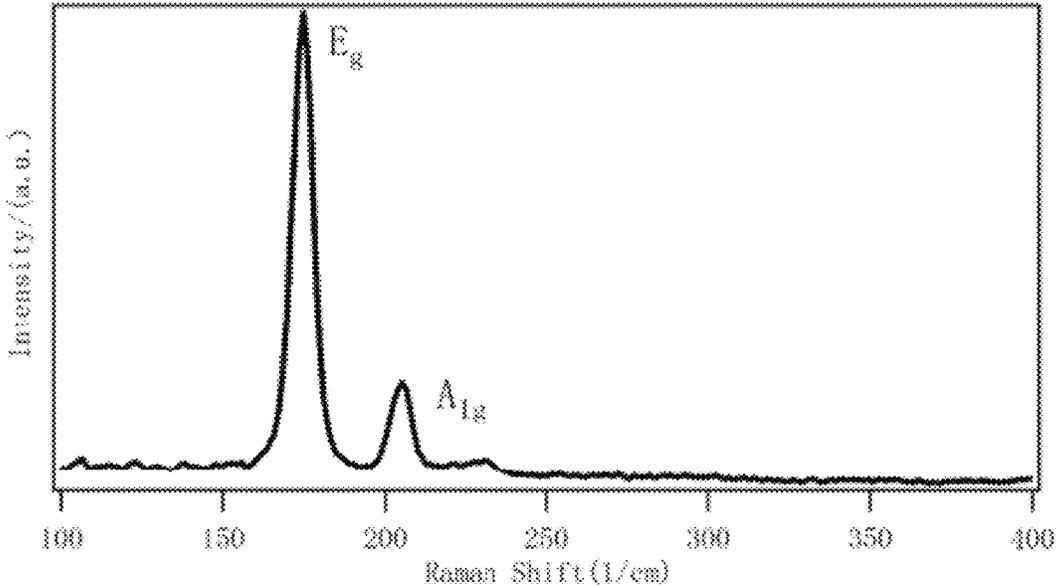


FIG. 18

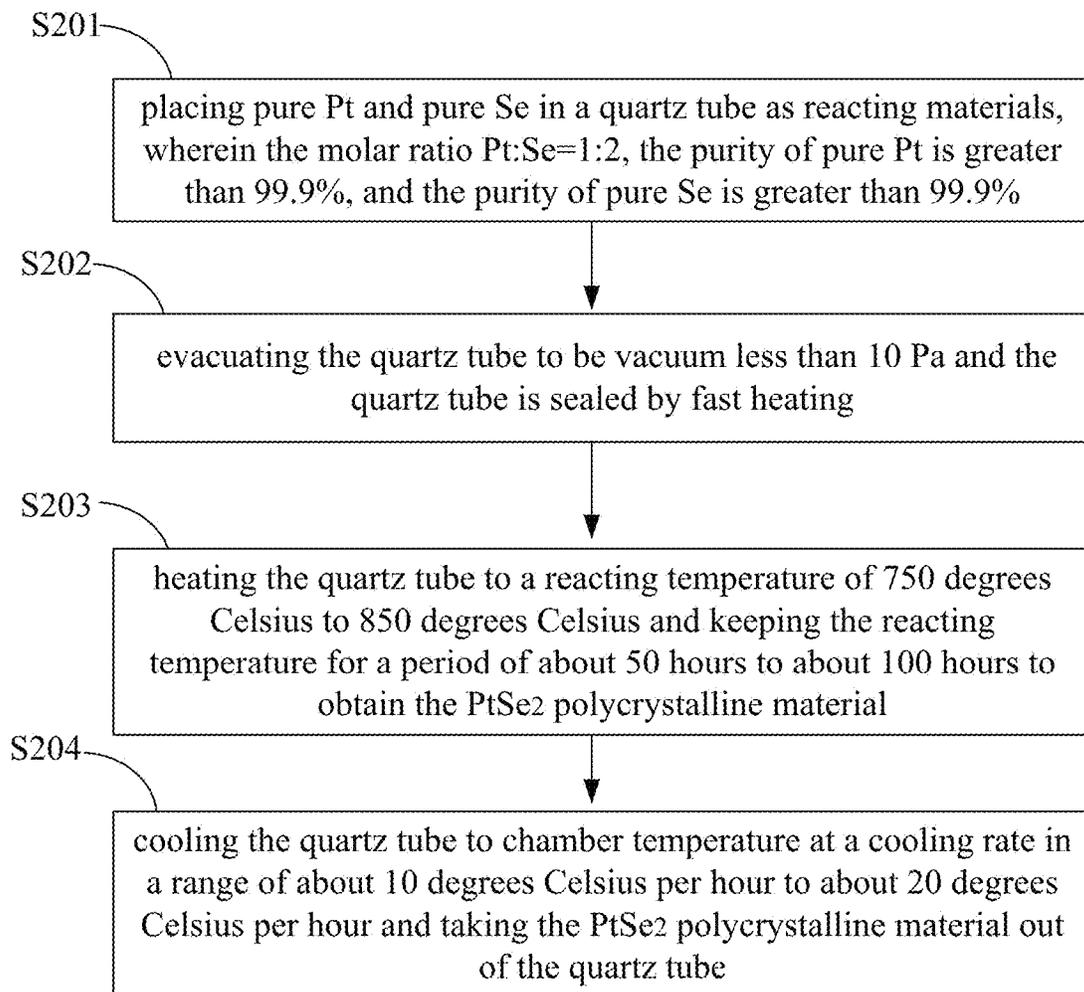


FIG. 19

METHOD FOR MAKING SEMIMETAL COMPOUND OF PT

CROSS-REFERENCE TO RELATED APPLICATIONS

This application is a continuation application of U.S. patent application Ser. No. 15/642,412, filed on Jul. 6, 2017, entitled "METHOD FOR MAKING SEMIMETAL COMPOUND OF Pt," which claims all benefits accruing under 35 U.S.C. § 119 from China Patent Application No. 201610862880.2, filed on Sep. 28, 2016, in the China Intellectual Property Office, the disclosure of which is incorporated herein by reference.

BACKGROUND

1. Technical Field

The present disclosure relates to a semimetal compound of Pt and method for making the same.

2. Description of Related Art

Three-dimensional semimetals are important hosts to exotic physical phenomenon such as giant diamagnetism, linear quantum magnetoresistance, and quantum spin Hall effect. Three dimensional Dirac fermions can be viewed as three dimensional version of graphene and have been realized in Dirac semimetals.

Cd_3As_2 , Na_3Bi , K_3Bi , and Rb_3Bi are found as three-dimensional Dirac semimetals. However, all the Cd_3As_2 , Na_3Bi , K_3Bi , and Rb_3Bi are type-I Dirac semimetals having a vertical cone of electron energy band as shown in FIG. 1. The type-I Dirac semimetals shows spin degenerate conical dispersions that cross at isolated momentum points (Dirac points) in three dimensional momentum space. In a topological Dirac semimetal, the massless Dirac fermions are stabilized by crystal symmetry and could be driven into various topological phases. When breaking the inversion or time-reversal symmetry, the doubly de-generate Dirac points can be split into a pair of Weyl points with opposite chiralities, and a Dirac fermion splits into two Weyl fermions. Weyl fermions were originally proposed in high energy physics, and their condensed matter physics counterparts have been recently realized. Weyl semimetals exhibit intriguing properties, with open Fermi arcs connecting the Weyl points of opposite chiralities. Both Dirac and Weyl semimetals obey Lorentz invariance and they exhibit anomalous negative magnetoresistance.

Recently, a new type of Weyl semimetal (type-II Dirac semimetals) have been predicted. In type-II Dirac semimetals, the Weyl points arise from the topologically protected touching points between electron and hole pockets, and there are finite density of states at the Fermi level. Type-II Dirac semimetals have strongly tilted cone and thus violate the Lorentz invariance. However, so far, the spin-degenerate counterpart of type-II Dirac semimetals have not been realized.

What is needed, therefore, is a type-II Dirac semimetals and method for making the same that overcomes the problems as discussed above.

BRIEF DESCRIPTION OF THE DRAWINGS

Many aspects of the embodiments can be better understood with reference to the following drawings. The com-

ponents in the drawings are not necessarily drawn to scale, the emphasis instead being placed upon clearly illustrating the principles of the embodiments. Moreover, in the drawings, like reference numerals designate corresponding parts throughout the several views.

FIG. 1 is a schematic section views of electron energy band of type-I Dirac semimetal and type-II Dirac semimetal.

FIG. 2 is a flowchart of example I of a method for making a semimetal $PtSe_2$.

FIG. 3 is a schematic section view of example I of a device for making a semimetal $PtSe_2$.

FIG. 4 is a photo image of the semimetal $PtSe_2$ of example I.

FIG. 5 is an X-ray diffraction (XRD) result of the semimetal $PtSe_2$ of example I measured at room temperature.

FIG. 6 is a Raman spectrum result of the semimetal $PtSe_2$ of example I measured at room temperature.

FIG. 7 is a low energy electron diffraction (LEED) pattern of the semimetal $PtSe_2$ of example I taken at beam energy of 70 eV.

FIG. 8 shows a measured in-plane two-dimensional Dirac cone of the semimetal $PtSe_2$ of example I.

FIG. 9 shows a measured out-plane two-dimensional Dirac cone of the semimetal $PtSe_2$ of example I.

FIG. 10 shows a calculated simulation result of two-dimensional Dirac cone of the single crystal semimetal $PtSe_2$.

FIG. 11 shows a Brillouin zone of the semimetal $PtSe_2$ of example I.

FIG. 12 shows a measured three-dimensional Dirac cone of the semimetal $PtSe_2$ of example I.

FIG. 13 shows a calculated simulation result of three-dimensional Dirac cone of the single crystal semimetal $PtSe_2$.

FIG. 14 is a flowchart of example II of a method for making a semimetal $PtSe_2$.

FIG. 15 is a schematic section view of example II of a device for making a semimetal $PtSe_2$.

FIG. 16 is a photo image of the semimetal $PtSe_2$ of example II.

FIG. 17 is an X-ray diffraction (XRD) result of the semimetal $PtSe_2$ of example II measured at room temperature.

FIG. 18 is a Raman spectrum result of the semimetal $PtSe_2$ of example II measured at room temperature.

FIG. 19 is a flowchart of example II of a method for making a $PtSe_2$ polycrystalline material.

DETAILED DESCRIPTION

It will be appreciated that for simplicity and clarity of illustration, where appropriate, reference numerals have been repeated among the different figures to indicate corresponding or analogous elements. In addition, numerous specific details are set forth in order to provide a thorough understanding of the embodiments described herein. However, it will be understood by those of ordinary skill in the art that the embodiments described herein can be practiced without these specific details. In other instances, methods, procedures, and components have not been described in detail so as not to obscure the related relevant feature being described. The drawings are not necessarily to scale, and the proportions of certain parts may be exaggerated better illustrate details and features. The description is not to be considered as limiting the scope of the embodiments described herein.

The term “outside” refers to a region that is beyond the outermost confines of a physical object. The term “inside” indicates that at least a portion of a region is partially contained within a boundary formed by the object. The term “substantially” is defined to essentially conforming to the particular dimension, shape or other word that substantially modifies, such that the component need not be exact. For example, substantially cylindrical means that the object resembles a cylinder, but can have one or more deviations from a true cylinder. The term “comprising” means “including, but not necessarily limited to”; it specifically indicates open-ended inclusion or membership in a so-described combination, group, series and the like. It should be noted that references to “an” or “one” embodiment in this disclosure are not necessarily to the same embodiment, and such references mean at least one.

References will now be made to the drawings to describe, in detail, various embodiments of the present type-II Dirac semimetals and method for making the same.

Example I

Referring to FIGS. 2-3, a self-flux method for making the semimetal PtSe₂ comprises following steps:

step (S10), placing pure Pt and pure Se in a reacting chamber 10 as reacting materials 13;

step (S11), evacuating the reacting chamber 10 to be vacuum less than 10 Pa;

step (S12), heating the reacting chamber 10 to a first temperature of 600 degrees Celsius to 800 degrees Celsius and keeping the first temperature for a period of about 24 hours to about 100 hours;

step (S13), cooling the reacting chamber 10 to a second temperature of 400 degrees Celsius to 500 degrees Celsius and keeping the second temperature for a period of about 24 hours to about 100 hours at a cooling rate from about 1 degree Celsius per hour to about 10 degrees Celsius per hour to obtain a reaction product 15 comprising a crystal material of PtSe₂; and

step (S14), separating the crystal material of PtSe₂ from the reaction product 15.

In step (S10), the reacting chamber 10 is a quartz tube having an open end and a sealed end opposite to the open end. The quartz tube is filled with quartz slag 11 so that the quartz slag 11 form a support at the sealed end. The support has a thickness in a range of about 0.5 centimeter to about 3 centimeters. In one embodiment, the thickness of the support is about 2 centimeters. The particle diameter of the quartz slag 11 is less than 1 millimeter. The inner diameter of the quartz tube is about 8 millimeters, and the outer diameter of the quartz tube is about 10 millimeters. The quartz tube is further filled with quartz wool 12 so that the quartz wool 12 forms a filter on the quartz slag 11. The filter has a thickness in a range of about 0.5 centimeter to about 2 centimeters. In one embodiment, the thickness of the filter is about 1 centimeter. The diameter of the quartz wool 12 can be in a range of about 1 micrometer to about 10 micrometers. In one embodiment, the diameter of the quartz wool 12 is about 4 micrometers. The quartz slag 11 and the quartz wool 12 separate the crystal material of PtSe₂ from the reaction product 15. The quartz slag 11 and the quartz wool 12 are optional. If the quartz slag 11 and the quartz wool 12 are omitted, the reaction product 15 should be separated by special method, such as using a metal filter or ceramic filter. The pure Pt and pure Se are filled in the quartz tube after the quartz tube is filled with the quartz slag 11 and the quartz wool 12. The molar ratio of Pt element and Se element is

about 2:80~2:120. In one embodiment, the molar ratio is Pt:Se=2:98. The pure Pt and pure Se form the reacting materials 13. The purity of pure Pt is greater than 99.9%, and the purity of pure Se is greater than 99.99%.

In step (S11), the quartz tube is evacuated by a mechanical pump and then the open end of the quartz tube is sealed by fast heating the open end by a flame of natural gas and oxygen gas. In one embodiment, the pressure of the quartz tube can be less than 1 Pa. The natural gas can be replaced by propane gas or hydrogen gas.

In step (S12), the quartz tube is reversed and vertically located so that the reacting materials 13 is located at the bottom of the quartz tube, and the quartz slag 11 and the quartz wool 12 are located at the top of the quartz tube. The quartz tube is further accommodated in a steel sleeve 14. The fire-resistant cotton is filled between the quartz tube and the steel sleeve 14 to fix and keep the temperature of the reaction product 15 in the quartz tube.

The steel sleeve 14, having the quartz tube therein, is placed in a muffle furnace. The quartz tube is heated by the muffle furnace to a point where the inside thereof reaches 700 degrees Celsius and kept at 48 hours at 700 degrees Celsius in the muffle furnace. During heating, the reacting materials 13 is kept at the bottom of the quartz tube and the quartz slag 11 and the quartz wool 12 are kept at the top of the quartz tube. The quartz tube can also be heated by other heating device rather than muffle furnace.

In step (S13), in one embodiment, the muffle furnace is cooled down to 480 degrees Celsius at a cooling rate of 5 degrees Celsius per hour and kept at 48 hours at 480 degrees Celsius to obtain the reaction product 15. The reaction product 15 includes the crystal material of PtSe₂ and the excess reacting materials. The cooling rate can be in a range of about 1 degree Celsius per hour to about 10 degrees Celsius per hour.

In step (S14), the steel sleeve 14, having the quartz tube therein, is taken out of the muffle furnace. Then the steel sleeve 14 is reversed and vertically located so that the quartz slag 11 and the quartz wool 12 are located at the bottom of the quartz tube, and the reaction product 15 is filed by the quartz wool 12. Thus, the excess reacting materials are separated from the crystal material of PtSe₂.

Furthermore, the steel sleeve 14, having the quartz tube therein, can be centrifuged for a period in a range of about 1 minute to about 5 minutes at a speed in a range of about 2000 revolutions per minute (rpm) to about 3000 rpm. In one embodiment, the centrifuged period is about 2 minutes, and the centrifuged speed is about 2500 rpm. The excessive reacting materials can also be separated from the crystal material of PtSe₂ by other methods after the reaction product 15 is taken out of the quartz tube.

The quartz tube is taken out of the steel sleeve 14 after natural cooling. Then the crystal material of PtSe₂ is taken out of the quartz tube, washed by chemical reagent to remove residual Se element, and then rinsed by water to obtain pure crystal material of PtSe₂. The chemical reagent can be hydrogen peroxide, dilute hydrochloric acid, sodium hydroxide.

The pure crystal material of PtSe₂ of example I was tested. FIG. 4 is a photo image of the semimetal PtSe₂ of example I. As shown in FIG. 4, the semimetal PtSe₂ of example I is macroscopic visible and has a length about 2 millimeters and a thickness about 10 micrometers to about 100 micrometers. FIG. 5 is a XRD result of the semimetal PtSe₂ of example I measured at room temperature of about 15 degrees Celsius to about 25 degrees Celsius. FIG. 6 is a Raman spectrum of the semimetal PtSe₂ of example I measured at room tem-

perature. FIG. 7 is a LEED pattern of the semimetal PtSe₂ of example I taken at beam energy of 70 eV. As shown in FIGS. 5 and 7, the semimetal PtSe₂ of example I confirm the high quality of the single crystals. As shown in FIG. 7, the semimetal PtSe₂ of example I has a symmetrical structure. The Raman spectrum in FIG. 6 shows Eg and Ag vibrational modes of the semimetal PtSe₂ of example I at about 170 cm⁻¹ and 210 cm⁻¹ respectively, which are typical for 1T structure.

FIG. 8 shows a measured in-plane two-dimensional Dirac cone of the semimetal PtSe₂ of example I measured at 22 eV. FIG. 9 shows a measured out-plane two-dimensional Dirac cone of the semimetal PtSe₂ of example I measured at 22 eV. FIG. 10 shows a calculated simulation result of two-dimensional Dirac cone of the single crystal semimetal PtSe₂. FIG. 11 shows a Brillouin zone of the semimetal PtSe₂ of example I. FIG. 12 shows a measured three-dimensional Dirac cone of the semimetal PtSe₂ of example I. FIG. 13 shows a calculated simulation result of three-dimensional Dirac cone of the single crystal semimetal PtSe₂. As shown in FIGS. 8-13, it is experimentally and theoretically confirmed that the semimetal PtSe₂ of example I belong to Type-II Dirac semimetals.

The semimetal PtSe₂ of example I has physical properties such as negative magnetoresistance, quantum spin Hall effect, and linear quantum magnetoresistance. As Type-II Dirac semimetals, the semimetal PtSe₂ of example I exhibits anomalous negative magnetoresistance.

Example II

Referring to FIGS. 14-15, a chemical vapor transport method for making the semimetal PtSe₂ comprises following steps:

- step (S20), providing a PtSe₂ polycrystalline material;
- step (S21), placing the PtSe₂ polycrystalline material in a reacting chamber 20 as reacting materials 23;
- step (S22), placing chemical transport medium 26 in the reacting chamber 20;
- step (S23), evacuating the reacting chamber 20 to be vacuum less than 10 Pa;
- step (S24), placing the reacting chamber 20 at a temperature gradient, wherein the reacting chamber 20 has a first end 201 at a temperature of about 1200 degrees Celsius to about 1000 degrees Celsius and a second end 202 opposite to the first end 201 and at a temperature of about 1000 degrees Celsius to about 900 degrees Celsius; and
- step (S25), keeping the reacting chamber 20 in the temperature gradient for 10 days to 30 days to obtain the reaction product 25 at the second end 202.

In FIG. 15, AB represents the element of PtSe₂, and L represents the element of the chemical transport medium 26.

In step (S20), the PtSe₂ polycrystalline material can be provided by any method. In one embodiment, as shown in FIG. 19, the PtSe₂ polycrystalline material is made by following steps:

- step (S201), placing pure Pt and pure Se in a quartz tube as reacting materials, wherein the molar ratio Pt:Se=1:2, the purity of pure Pt is greater than 99.9%, and the purity of pure Se is greater than 99.9%;
- step (S202), evacuating the quartz tube to be vacuum less than 10 Pa and the quartz tube is sealed by fast heating;
- step (S203), heating the quartz tube to a reacting temperature of 750 degrees Celsius to 850 degrees Celsius and keeping the reacting temperature for a period of about 50 hours to about 100 hours to obtain the PtSe₂ polycrystalline material; and

step (S204), cooling the quartz tube to chamber temperature at a cooling rate in a range of about 10 degrees Celsius per hour to about 20 degrees Celsius per hour and taking the PtSe₂ polycrystalline material out of the quartz tube.

In step (S204), the PtSe₂ remains polycrystalline because the cooling rate is higher than 10 degrees Celsius per hour.

In step (S21), the reacting chamber 20 is a quartz tube having an open end and a sealed end opposite to the open end. The inner diameter of the quartz tube is about 8 millimeters, and the outer diameter of the quartz tube is about 10 millimeters. The reacting materials 23 are located at the sealed end of the quartz tube.

In step (S22), the chemical transport medium 26 can be located adjacent to the reacting materials 23. The chemical transport medium 26 can be SeBr₄, I₂, Br₂, Cl₂, SeCl₄. The concentration of the chemical transport medium 26 can be in a range of about 5 mg/mL to about 20 mg/mL. In one embodiment, the chemical transport medium 26 is SeBr₄ with a concentration 10 mg/mL.

In step (S23), the quartz tube is evacuated and sealed by fast heating as described above. The pressure of the quartz tube can be lower than 1 Pa.

In step (S24), the quartz tube is horizontally located in the tubular furnace 27. The reacting materials 23 are located at the first end 201 of the quartz tube. The temperature of the first end 201 is higher than the temperature of the second end 202 so that the temperature gradient is formed between the first end 201 and the second end 202. In one embodiment, the first end 201 of the quartz tube is heated by the tubular furnace 27 to a point where the inside thereof reaches 1050 degrees Celsius, and the second end 202 of the quartz tube is heated by the tubular furnace 27 to a point where the inside thereof reaches 970 degrees Celsius.

In step (S25), the reaction product 25 can be obtained at the second end 202 of the quartz tube after keeping the temperature gradient for 10 days to 30 days. The reaction product 25 is taken out of the quartz tube and washed using alcohol to remove the chemical transport medium to obtain pure crystal material of PtSe₂. In one embodiment, the reacting chamber 20 is kept in the temperature gradient for 20 days.

The pure crystal material of PtSe₂ of example II is tested. FIG. 16 is a photo image of the semimetal PtSe₂ of example II. As shown in FIG. 16, the semimetal PtSe₂ of example II is macroscopic visible and has a size about 2 millimeters. FIG. 17 is a XRD result of the semimetal PtSe₂ of example II measured at room temperature. FIG. 18 is a Raman spectrum result of the semimetal PtSe₂ of example II measured at room temperature. As shown in FIG. 17, the semimetal PtSe₂ of example II confirm the high quality of the single crystals. The Raman spectrum in FIG. 18 shows Eg and Ag vibrational modes of the semimetal PtSe₂ of example I at about 170 cm⁻¹ and 210 cm⁻¹ respectively, which are typical for 1T structure. It is experimentally and theoretically confirmed that the semimetal PtSe₂ of example II belong to Type-II Dirac semimetals.

It is to be understood that the above-described embodiments are intended to illustrate rather than limit the disclosure. Any elements described in accordance with any embodiments is understood that they can be used in addition or substituted in other embodiments. Embodiments can also be used together. Variations may be made to the embodiments without departing from the spirit of the disclosure. The above-described embodiments illustrate the scope of the disclosure but do not restrict the scope of the disclosure.

Depending on the embodiment, certain of the steps of methods described may be removed, others may be added,

and the sequence of steps may be altered. It is also to be understood that the description and the claims drawn to a method may include some indication in reference to certain steps. However, the indication used is only to be viewed for identification purposes and not as a suggestion as to an order for the steps.

What is claimed is:

1. A method for making semimetal compound of Pt, the method comprising:

placing Pt and Se in a reacting chamber as reacting materials, wherein a first purity of the Pt is greater than 99.9%, and a second purity of the Se is greater than 99.99%;

evacuating the reacting chamber to a vacuum level of a pressure lower than 10 Pa;

heating the reacting chamber to a first temperature of 600 degrees Celsius to 800 degrees Celsius and maintaining the reacting chamber at the first temperature for a first time period of 24 hours to 100 hours;

cooling the reacting chamber to a second temperature of 400 degrees Celsius to about 500 degrees Celsius at a cooling rate of 1 degrees Celsius per hour to 10 degrees Celsius per hour;

maintaining the reacting chamber at the second temperature for a second time period of 24 hours to 100 hours to obtain a reaction product comprising a crystal material of PtSe₂; and

separating the crystal material of PtSe₂ from the reaction product.

2. The method of claim 1, wherein a molar ratio of Pt element and Se element is from 2:80 to 2:120.

3. The method of claim 2, wherein the molar ratio of Pt element and Se element is 2:98.

4. The method of claim 1, wherein the reacting chamber is evacuated to lower than 1 Pa.

5. The method of claim 1, wherein the separating the crystal material of PtSe₂ from the reaction product further comprises centrifuging the reaction product.

6. The method of claim 5, wherein a third time period of the centrifuging the reaction product is in a range from 1 minute to 5 minutes.

7. The method of claim 5, wherein a speed of the centrifuging the reaction product is in a range of 2000 revolutions per minute (rpm) to 3000 rpm.

8. A method for making semimetal compound of Pt, the method comprising:

placing Pt and Se in a reacting chamber as reacting materials, wherein a first purity of the Pt is greater than 99.9%, and a second purity of the Se is greater than 99.99%;

evacuating the reacting chamber to a vacuum level of a pressure lower than 10 Pa;

heating the reacting chamber to a first temperature of 600 degrees Celsius to 800 degrees Celsius and maintaining the reacting chamber at the first temperature for a first time period of 24 hours to 100 hours;

cooling the reacting chamber to a second temperature of 400 degrees Celsius to 500 degrees Celsius at a cooling rate of 1 degrees Celsius per hour to 10 degrees Celsius per hour; and

maintaining the reacting chamber at the second temperature for a second time period of 24 hours to 100 hours to obtain a reaction product comprising a crystal material of PtSe₂.

9. The method of claim 8, wherein a molar ratio of Pt element and Se element is from 2:80 to 2:120.

10. The method of claim 9, wherein the molar ratio of Pt element and Se element is 2:98.

11. The method of claim 8, wherein the reacting chamber is evacuated to lower than 1 Pa.

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