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SOLID THIN FILM SPECIMEN FORMING APPARATUS

Filed Oct. 17, 1957

2 Sheets-Sheet 1

Fig. 1

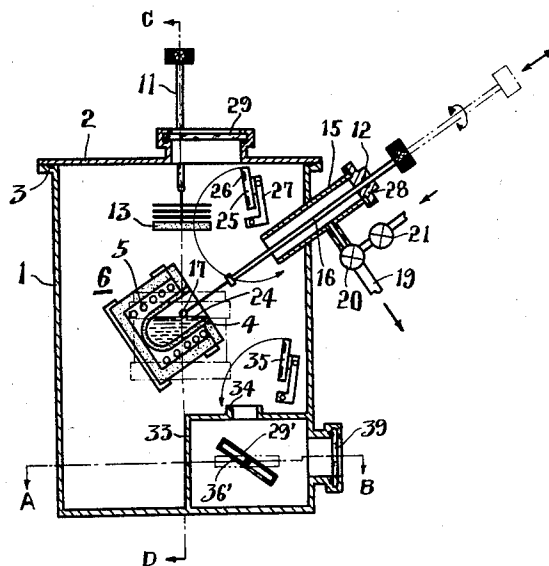
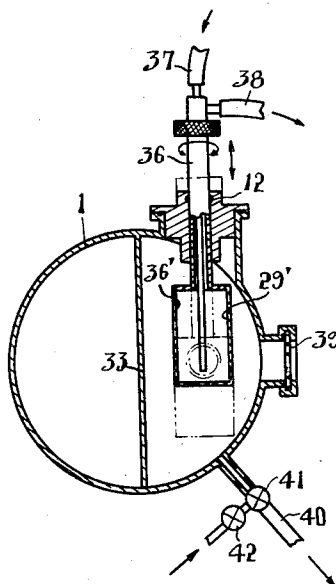


Fig. 2



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Fig. 3

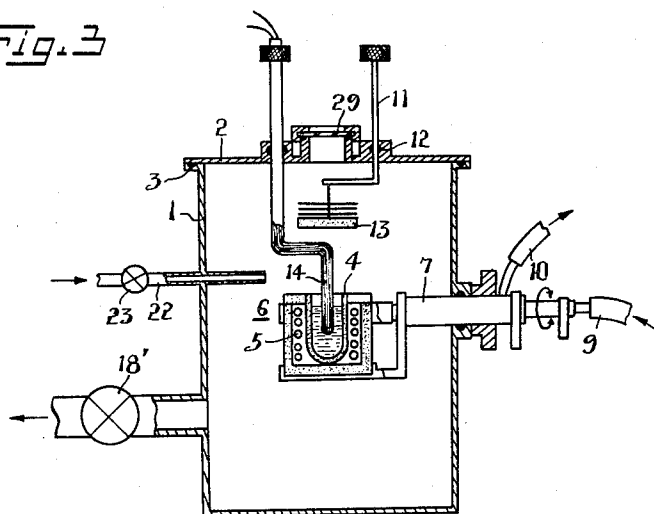


Fig. 4

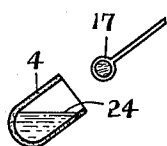


Fig. 5

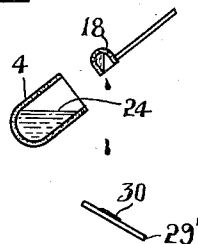
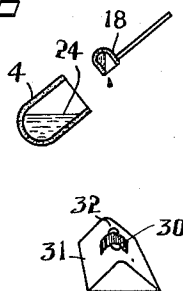


Fig. 6



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SOLID THIN FILM SPECIMEN FORMING APPARATUS**Noboru Takahashi, Kofu-shi, and Kenji Kazato, Kanichi Ashinuma, and Masaru Watanabe, Tokyo, Japan****Application October 17, 1957, Serial No. 690,806****Claims priority, application Japan October 22, 1956****5 Claims. (Cl. 22—73)**

This invention relates to a solid thin film specimen forming apparatus which enables to form the thin film in a vacuum or any desired gas atmosphere for an electron microscope and more particularly to such an apparatus which facilitates the formation of a thin film used as a specimen for an electron microscope, an electron diffraction apparatus, X-ray diffraction apparatus, an optical instrument or the like.

It is an object of this invention to provide a solid thin film specimen forming apparatus which enables to make a specimen which is suitable to observe its surface construction of a metal, solid alloy or the like, for an electron microscope and the like in a very simple and easy manner.

It is another object of this invention to provide a solid thin film specimen forming apparatus in which a thin solid film can simply be made without any particular skillfulness.

It is a further object of this invention to provide such an apparatus which enables to make a non-oxidized solid thin film in a vacuum or a solid thin film subjected to any desired gas.

Other object, features and advantages of this invention will be more apparent from the following detailed description taken in connection with the accompanying drawings, in which—

Fig. 1 is a longitudinally sectional view of an apparatus embodying this invention.

Fig. 2 is a cross sectional view taken along the line A—B of the apparatus shown in Fig. 1.

Fig. 3 is a sectional view taken along the line C—D of the apparatus shown in Fig. 1.

Fig. 4 illustrates a partial view showing a manner in which a solid thin film is made on a loop.

Fig. 5 is a partial view showing another manner in which a solid thin film is formed on a plate, and

Fig. 6 is a similar view showing a further manner in which a solid thin film is formed on a prism.

Referring to the drawings, 1 is a sealed vessel and 2 a cover, between which a rubber packing 3 is inserted. In the sealed vessel arranged an electric furnace 6 having a crucible 4 for melting a desired specimen material such as a metal or an alloy and an electric heating coil 5 wound around the crucible. For supporting the furnace to the vessel 1 a supporting axis 7 is inserted horizontally through the wall of the vessel 1 and lead wires of the heating coil are passing through the supporting axis. This supporting axis is rotatably mounted on the wall of the vessel so that the furnace can be inclined at any desired time. 9 shows an inlet pipe of cold water, 10 an outlet pipe of the same which serves to prevent the supporting axis from overheating.

A handle 11 is inserted into the vessel through the cover 2 by means of a Wilson seal 12 and the lower end of the handle has a cover 13 for the furnace 6. The Wilson seal is a pressure seal which will permit axial and rotational movement of an operating rod 16 without the escape of gas past the seal. Similarly a pipe

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14 is inserted into the vessel through the cover 2 by means of a Wilson seal 12 and to the lower end of the pipe 14 is attached a thermocouple for measuring the temperature in the furnace, if desired.

5 A cylinder 15 is attached to a desired position of the wall of the vessel, the axis thereof being inclined at a suitable angle to the vertical. An operating apparatus or an operating rod 16 for handling a solid thin film forming apparatus as referred hereinafter is inserted into the cylinder 15 coaxially, and sealed by means of a Wilson seal 12 at the outer end of the cylinder 15 as shown in the drawing. A loop 17 or a spoon 18 which forms a solid thin film forming member is detachably inserted into the tubular end of the operating rod 16 so that the dipping and taking up operation of the loop or spoon with respect to the molten material in the crucible 4 can be made from the outside of the vessel 1.

15 In order to evacuate the vessel 1 a vacuum pump 18' is provided and in order to evacuate locally the cylinder 15, the latter is connected with a tube 19 which is, in turn, connected with another vacuum pump not shown. 20 is a valve, 21 a leak cock.

20 In order to introduce a desired gas including inert gas into the vessel 1, if desired, another pipe 22 is connected with the vessel 1 through a valve 23.

In the above arrangement according to this invention, in order to form a solid thin film by means of the loop 17, the supporting axis 7 is rotated so that the furnace 6 is inclined as shown in Fig. 1, after a specimen in the crucible 4 is molten. Then the operating rod 16 is pushed to dip the loop 17 which is attached to the end of the operating rod into the molten solution 24, and again the loop is lifted from the molten solution by pulling the operating rod 16. Then the molten specimen is hanged and spread over the loop 17 owing to its surface tension, in the form of a thin film, which soon coagulates as it is. This condition can be best seen from the illustration in Fig. 4.

40 To remove the solid thin film thus obtained, the operating rod 16 is more pulled, as shown by the chain line, until the loop 17 enters into the cylinder 15. Then inner end of the cylinder is covered by a cover 25 which has a rubber packing 26 to be put on the inner end of the cylinder 15 and which is supported by a supporter 27, which is also constructed to be operated from the outside of the vessel 1. This operating construction taken in connection with the supporter 27 may be easily made by any desired means well known by those skilled in the art.

50 After the cylinder end is covered by the cover 25 only the vacuum of the cylinder 15 is broken by opening the leak cock 21 and then an outer cover 28 of the cylinder 15 is removed to pick up the loop 17 from the operating rod 16. Now a new loop is put into the tubular end of the operating rod 16 and the cover 28 is again put on the outer end of the cylinder 15. Thus the cylinder 15 is again evacuated to ready to the next thin film forming operation. By using the cylinder 15 the vacuum operation is performed speedily and exactly because of the smaller volume in the cylinder. This condition can be observed through a window 28.

55 Although the film thus formed has not equal thickness over the whole area thereof, it can be recognized that at least one part of this film is thin enough to be examined by an electron microscope and the like.

60 According to this invention other means for making a solid thin film by applying the surface tension of the molten material are provided. That is, a spoon 18 is substituted for the loop 17 and inserted into the tubular end of the operating rod 16. The molten specimen in

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the crucible is picked up by the spoon 18 and thrown onto an inclined plate 29' which is arranged below in the vessel, as shown in Fig. 5. The drop of the molten specimen is spread over the plate and at the same time subjected to the surface tension to form a thin film 30.

Fig. 6 shows another method of forming a solid thin film. In this case the drop is thrown down onto the top line 32 of the prism member 31 and then the drop flows downwardly along the left and right side walls from the top line. Thus a solid thin film 30 is formed across the both inclined side walls of the prism member 31 under the surface tension of the molten specimen and the decrease of fluidity thereof. The inclined plate 29' is preferably enclosed in a small chamber 33 formed in the vessel 1 as shown in Fig. 1. The molten specimen is dropped from an opening 34 of the chamber 33 onto the plate 29' and then a cover 35 is put onto the opening 34. This cover 35 may similarly be constructed to be operated as above mentioned cover 25 from the outside of the vessel 1.

In order to rotate the plate 29' and adjust the inclination thereof from the outside of the vessel 1, a sleeve 36 is inserted horizontally into the vessel 1 through a Wilson seal 12 attached to the wall of the vessel as shown in Fig. 2. The sleeve 36 may be moved along mainly its axis as shown by the arrow. This construction enables to drop the molten specimen at every point of the plate 29. It is preferable that a case 36' is provided in the chamber 33 and the upper surface of the case 36' is used as the plate 29'. In this case a cold liquid is circulated into the case 36' in order to cool the same through an inlet pipe 37 and an outlet pipe 38, the pipes being formed coaxially and integrally with the sleeve 36 as shown in the drawing. The plate 29' can also be heated by an electric heating coil instead of cooling, if desired. A solid thin film which is formed on the plate 29' can be removed from the small chamber 33 by only breaking the vacuum of the chamber 33 after its opening 34 is covered with a cover 35. 39 is an observing window, 40 is a vacuum pipe for the chamber 33 and 41 a cock.

According to this invention, as above described, specimen solid thin films can be formed easily and speedily.

Although we have explained particular embodiments of our invention, it will be understood, of course, that we do not wish to be limited thereto since many modifications may be made and we, therefore, contemplate by the appended claims to cover any such modifications as within the spirit and scope of our invention.

What is claimed is:

1. A solid thin film specimen forming apparatus for an electron microscope and the like comprising a furnace composed of a crucible and an electric heater wound around said crucible for heating the solid material put into said crucible, a sealed vessel for enclosing said furnace to maintain the molten material in the vacuum, a specimen holder having a loop shaped member at the end thereof which is inserted into said sealed vessel and having an end outside of said vessel, and a pressure seal for enabling said specimen holder to be operated from the outside of said sealed vessel to dip said loop shaped member into the crucible and thereafter to lift up the same therefrom.

2. A solid thin film specimen forming apparatus for an electron microscope and the like comprising a furnace composed of a crucible and an electric heater wound around said crucible for heating the solid material put

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into said crucible, a sealed vessel for enclosing said furnace to maintain the molten material in an inert gas atmosphere, an exhaust pipe connected with said sealed vessel, a gas inlet valve for introducing a gas into said sealed vessel, a specimen holder extending outside of said vessel and having a loop shaped member at the end thereof which is inserted into said sealed vessel, and a seal between said vessel and said specimen holder for enabling said specimen holder to be operated from the outside of said sealed vessel to dip said loop shaped member into said crucible and thereafter to lift up the same therefrom.

3. A solid thin film specimen forming apparatus for an electron microscope and the like comprising a furnace composed of a crucible and an electric heater wound around said crucible for heating the solid material put into said crucible, a sealed vessel for enclosing said furnace to maintain the material in the vacuum, a specimen holder extending outside of said sealed vessel and having a loop shaped member at the end thereof which is within said sealed vessel, a seal for enabling said specimen holder can be operated from the outside of said sealed vessel to dip said loop shaped member into the crucible and thereafter to lift up the same therefrom, and means defining an opening into said vessel for adding additional material into said crucible during melting operation.

4. A solid thin film specimen forming apparatus for an electron microscope and the like comprising a furnace composed of a crucible and an electric heater wound around said crucible for heating the solid material put into said crucible, a sealed vessel for enclosing said furnace to maintain the molten material in the vacuum, a specimen holder extending outside of said vessel and having a spoon at the end thereof which is within said sealed vessel, a seal for enabling said specimen holder to be operated from the outside of said sealed vessel to dip said spoon into the crucible and thereafter to lift up the same therefrom, a film forming plate which is disposed beneath said spoon in said sealed vessel, and a small chamber for enclosing said film forming plate having an opening through which said molten material is dropped from said spoon onto said film forming plate.

5. A solid thin film specimen forming apparatus for an electron microscope or the like comprising means for containing a molten material, a sealed vessel for enclosing said containing means for molten material, a specimen holder having a loop shaped member at one end thereof which is within said sealed vessel and having an operating member at the other end which is outside of said vessel, means defining an opening in said vessel of a size to accommodate the passage of said specimen holder and loop shaped member for insertion into the vessel, closure means for said opening, and sealing and supporting means for said specimen holder mounted on said closure means and sealingly supporting said specimen holder for manipulation of the loop shaped member from outside of said sealed vessel.

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