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N. J. MASKALICK
METHOD OF MAKING SINGLE CRYSTAL FILMS AND THE
PRODUCT RESULTING THEREFROM
Filed Oct. 26, 1964

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FIG. 1

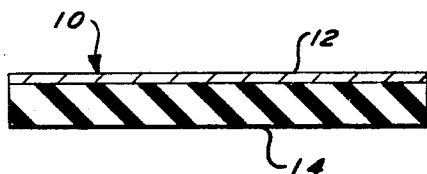
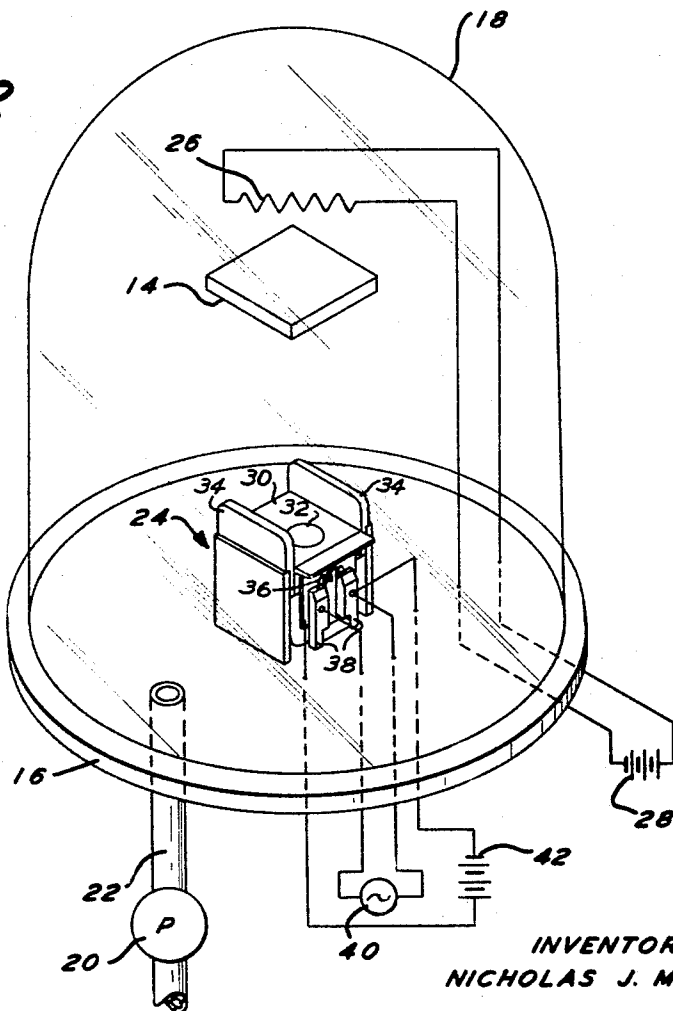


FIG. 2



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3,392,056

METHOD OF MAKING SINGLE CRYSTAL FILMS

AND THE PRODUCT RESULTING THEREFROM

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5 Claims. (Cl. 117--227)

The present invention relates to a method of making single crystal films and the product resulting therefrom. More particularly, the present invention relates to a method of coating an electrical insulating substrate with a thin film of single crystalline silicon or tungsten and the resulting product.

In recent years many types of electrical components have been developed using single crystalline materials. With the recent trend toward miniaturized electrical circuits, such as integrated circuits and the like, it has been found desirable to have such single crystalline materials in the form of thin films which are coated on a base substrate of an electrical insulating material.

It is an object of the present invention to provide a novel method of making thin films of single crystal material.

It is another object of the present invention to provide a method of coating a base substrate of an electrical insulating material with a thin film of a single crystal material.

It is a further object of the present invention to provide a method of coating a base substrate of an electrical insulating material with a thin film of single crystalline silicon or tungsten.

It is still another object of the present invention to provide a base substrate of an electrical insulating material coated with a thin film of single crystalline silicon or tungsten.

Other objects will appear hereinafter.

The invention accordingly comprises the several steps and the relation of one or more of such steps with respect to each of the others, and the article possessing the features, properties, and the relation of elements, which are exemplified in the following detailed disclosure, and the scope of the invention will be indicated in the claims.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following detailed description taken in connection with the accompanying drawing in which:

FIGURE 1 is a sectional view of the article of the present invention.

FIGURE 2 is a schematic view of an apparatus for carrying out the method of the present invention.

Referring to FIGURE 1 of the drawing, the article of the present invention, generally designated as 10, comprises a thin film 12 of single crystalline silicon or tungsten coated on the surface of a substrate 14 of sapphire, such as manufactured by Linde Company, Division of Union Carbide Corporation. According to the present invention, the thin film 12 of single crystalline silicon or tungsten is formed on the sapphire substrate 14 by evaporating silicon or tungsten in a vacuum and condensing the silicon or tungsten vapors on the heated surface of the sapphire substrate. To form a film of single crystalline silicon, the surface of the sapphire substrate should be heated to a temperature of between 1200° C. and 1350° C. To form a film of single crystalline tungsten, the surface of the sapphire substrate should be heated to a temperature of between 1300° C. and 1500° C. It has been found that at substrate temperatures above the maximum temperature of each of these ranges there is a tendency for the metal to chemically react with the material of the substrate, and at temperatures below the minimum temperature the crystalline deposit is not completely per-

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fectured. The deposition is carried out in an ultra-high vacuum, no greater than 10^{-7} millimeters of mercury. For best results, the sapphire substrate should be cut so that the individual crystals of which the substrate is comprised are oriented with their "C" axes being in the plane of the substrate surface on which the single crystalline metal is to be deposited. Also, the rate of condensation of the metal vapors on the substrate should generally be maintained at less than one-half micron per minute. However, when forming a film of single crystalline silicon on a substrate heated to a temperature above 1250° C. a faster deposition rate, approximately 20 microns per minute, should be used to minimize any chemical reactions between the silicon and the sapphire.

Referring to FIGURE 2 there is shown a schematic view of an apparatus which can be used to carry out the method of the present invention. The apparatus comprises a base plate 16 and a cover 18, such as a bell jar, seated on and sealed to the base plate so as to provide a chamber therebetween. An exhaust pump 20 is connected to the chamber within the cover 18 by means of a pipe 22 extending through the base plate 16. Exhaust pump 20 can be of any well known type which can draw and maintain the desired vacuum in the chamber. An evaporator 24 is mounted on the base plate 16 within the chamber. The sapphire substrate 14 is mounted by any suitable means over the evaporator 24. A resistance heater 26 is mounted over and in close proximity to the substrate 14 so as to heat the substrate. The heater 26 is connected to a suitable source of electrical current 28 which is outside the chamber.

Although the evaporator 24 can be any of the types well known in the art, the evaporator shown is an electron beam heating gun manufactured by Varian Associates of Palo Alto, Calif. The electron beam gun comprises a water cooled crucible 30 having a depression 32 for receiving the material to be evaporated. The legs 34 of a focusing magnet extend upwardly along opposite sides of the crucible 30. A filament 36 is connected between terminals 38 mounted on a side of the crucible between the legs 34 of the magnet. The terminals 38 are connected to a suitable source of A.C. electrical current 40 outside the chamber. Also, the filament 36 and crucible 30 are connected to a suitable source of very high voltage D.C. current 42 outside the chamber, and the crucible is grounded.

In the use of the apparatus to form the film 12 of single crystalline silicon or tungsten on the surface of the sapphire substrate 14, a charge of silicon or tungsten is placed in the depression 32 of the crucible 30. The exhaust pump 20 is turned on to evacuate the chamber within the cover 18 to a pressure slightly lower than the desired pressure. When the desired pressure is obtained, the heater 26 is turned on to heat the substrate 14 to the proper temperature, 1200° C. to 1350° C. for silicon and 1300° C. to 1500° C. for tungsten. The temperature of the substrate 14 can be determined by any suitable means, such as an optical pyrometer or a thermocouple. A high negative voltage is applied to the filament 36 from the source 42 and the A.C. current to the filament 36 of the evaporator 24 is then turned on. The filament 36 gives off a beam of electrons which is deflected by the arms 34 of the focusing magnet to impinge on the charge in the crucible. The bombardment of the charge by the electron beam heats the charge so as to melt and evaporate the silicon or tungsten. For this purpose the charge is heated to a temperature of at least the melting temperature of the metal, 1410° C. for silicon and 3370° C. for tungsten. The vapors of the charge diffuse upwardly toward the substrate 14 and the vapors which contact the substrate condense on the surface thereof to form the film 12 of single crystalline silicon or tungsten. The rate of

condensation of the vapors on the substrate can be determined by any well known means, such as a quartz crystal oscillator deposition monitor, mounted within the chamber. The rate of condensation can be controlled by controlling the rate of evaporation of the charge by varying the temperature of the charge. In the apparatus shown, this is accomplished by suitable adjustment of both the current through the filament 36 and the electron beam current bombarding the charge in the crucible. The rate of condensation can also be controlled by suitably locating the substrate 14 with respect to the charge. The closer the substrate 14 is to the charge, the higher the deposition rate and vice versa.

Thus, there is provided by the method of the present invention a thin film of single crystalline silicon or tungsten adhered to a substrate of an electrical insulating material, sapphire. The thin single crystalline film can be used to form microcircuits in any manner well known in the art. For example, in a film of single crystalline silicon transistors and/or diodes can be formed and connected together to provide a desired electrical circuit. The substrate provides a support for the thin film for ease of further processing the film and for use in mounting the completed device in a housing with the device being insulated from the housing.

The present invention may be embodied in other specific forms without departing from the spirit or essential attributes thereof, and accordingly, reference should be made to the appended claims, rather than to the foregoing specification as indicating the scope of the invention.

I claim:

1. A method of forming on the surface of sapphire a thin film of a single crystalline material selected from the group consisting of silicon and tungsten comprising the steps of placing the substrate in an enclosed chamber, evacuating and maintaining said chamber at a pressure of no greater than 10^{-7} millimeters of mercury, heating said substrate to a temperature of between 1200° C. and 1350° C. for forming a film of single crystalline silicon and between 1300° C. and 1500° C. for forming a film of single crystalline tungsten, evaporating a charge of said material in said chamber, and condensing the vapors of said material on a surface of said substrate.

2. A method in accordance with claim 1 including forming the substrate with the crystals of the sapphire being preferentially oriented with their "C" axes being in the

plane of the surface on which the material film is to be formed.

3. A method of forming on the surface of a substrate of sapphire a thin film of a single crystalline silicon comprising the steps of placing the substrate in an enclosed chamber, evacuating and maintaining said chamber at a pressure no greater than 10^{-7} millimeters of mercury, heating said substrate to a temperature of between 1200° C. and 1250° C., evaporating a charge of said silicon in said chamber, condensing the silicon vapors on a surface of said substrate, and maintaining the rate of condensation of the silicon vapors on the substrate at less than one-half micron per minute.

4. A method of forming on the surface of a substrate of sapphire a thin film of single crystalline silicon comprising the steps of placing the substrate in an enclosed chamber, evacuating and maintaining said chamber at a pressure no greater than 10^{-7} millimeters of mercury, heating said substrate to a temperature of between 1250° C. and 1350° C., evaporating a charge of said silicon in said chamber, condensing the silicon vapors on a surface of said substrate, and maintaining the rate of condensation of the silicon vapors on the substrate at approximately 20 microns per minute.

5. A method of forming on the surface of a substrate of sapphire a thin film of single crystalline tungsten comprising the steps of placing the substrate in an enclosed chamber, evacuating and maintaining said chamber at a pressure no greater than 10^{-7} millimeters of mercury, heating said substrate to a temperature of between 1300° C. and 1500° C., evaporating a charge of tungsten in said chamber, condensing the tungsten vapors on a surface of said substrate, and maintaining the rate of condensation of the tungsten vapors on the substrate at less than one-half micron per minute.

References Cited

UNITED STATES PATENTS

3,312,572	4/1967	Morton et al.	117—106
3,335,038	8/1967	Doo	117—201

OTHER REFERENCES

Manasevit et al.: J. Applied Physics, vol. 35, No. 4, April 1964.

WILLIAM L. JARVIS, *Primary Examiner*.

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,392,056

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Nicholas J. Maskalick

It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

In the heading to the printed specification, lines 4 and 5, "IRC, Inc., a corporation of Delaware" should read -- TRW Inc., a corporation of Ohio --.

Signed and sealed this 3rd day of March 1970.

(SEAL)

Attest:

Edward M. Fletcher, Jr.

Attesting Officer

WILLIAM E. SCHUYLER, JR
Commissioner of Patents