United States Patent [19]

Phinney et al.

[54] CRUCIBLE FOR EVAPORATION OF METALLIC FILM

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[56] References Cited U.S. PATENT DOCUMENTS

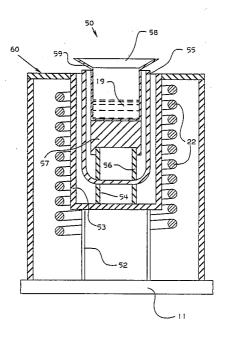
2,643,201	6/1953	Chadsay, Jr. et al 219/10.49 X
3,036,888	5/1962	Lowe 219/10.49 R
3,227,431	1/1966	Steeves 118/726 X
3,470,304	9/1969	Geringer 219/10.49 R
3,551,115	12/1970	Jameison et al 219/10.49 R
3,593,775	7/1971	Privott, Jr. et al 219/10.49 R X
4,242,553	12/1980	Berkman et al 219/10.49 R

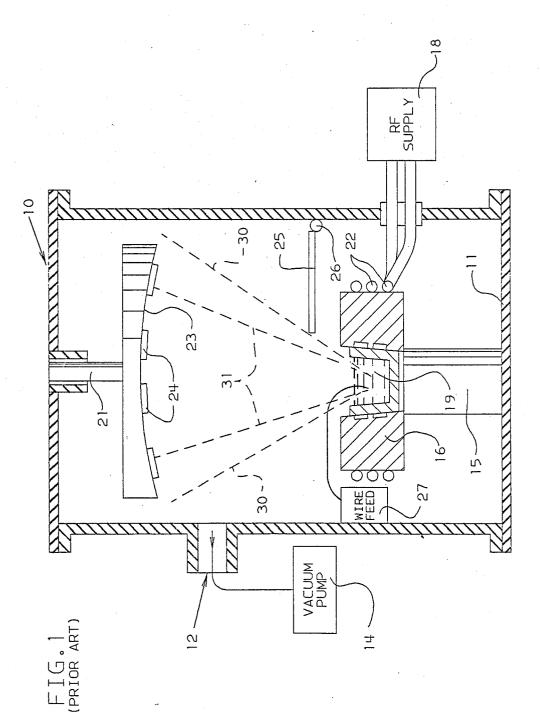
Primary Examiner—Philip H. Leung Attorney, Agent, or Firm—Francis J. Thornton

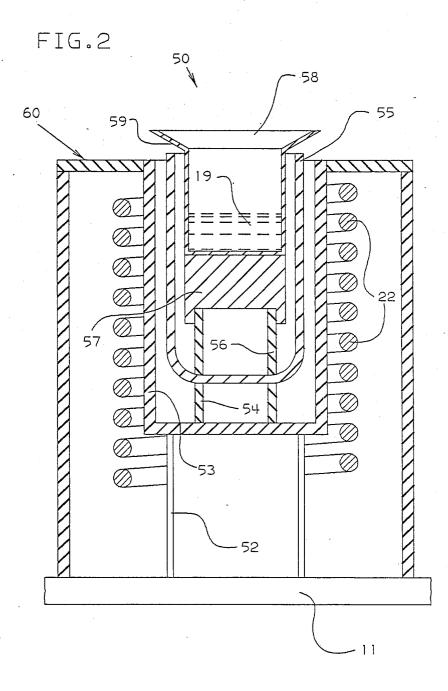
[57] ABSTRACT

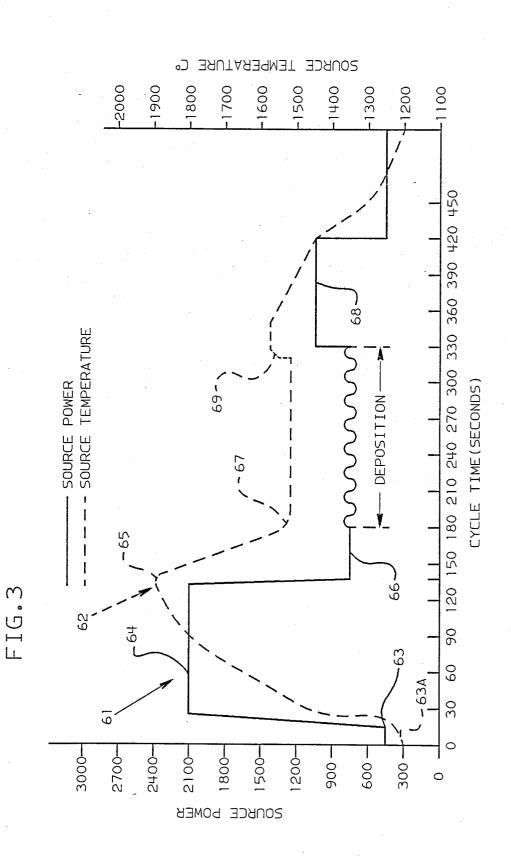
An evaporation source for RF heated evaporators in which the crucible has a susceptor made of a solid block of graphite carbon having a volume commensurate with the volume of molten material prior to evaporation. Moreover, the crucible has a depth that is less than twice its diameter. These features enhance the self-fractionation of the molten material prior to evaporation to thus enhance the properties of the film as-deposited.

13 Claims, 3 Drawing Sheets









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CRUCIBLE FOR EVAPORATION OF METALLIC FILM

BACKGROUND OF THE INVENTION

This invention relates generally to material deposition apparatus, and more particularly to an evaporation source used in a RF evaporation apparatus.

Evaporation apparatus is well known in the art. U.S. 10 Pat. No. 3,401,055 to J. L. Langdon et al, issued Sept. 10, 1968, and assigned to the IBM Corporation, describes how such evaporation apparatus is used in semiconductor integrated circuit processing technology. Specifically, this patent teaches a method of depositing 15 a metal such as a solder onto a plurality of small areas simultaneously on a semiconductor device.

As semiconductor integrated circuit chips have become denser, i.e., a million or more semiconductor devices per chip, evaporated films must meet more 20 are a plurality of RF heating coils 22. One suitable exacting criteria. Specifically, the films must have a thickness that does not appreciably vary either across one wafer or wafer to wafer, and the amount of conductivity-detracting impurities such as oxides must be kept at a minimum.

Using the evaporation apparatus and techniques of the prior art, for example as in the above identified U.S. Pat. No. 3,401,055, fabrication of a uniform formation of (e.g. copper) is extremely difficult. Thus, there is a need in the art for an evaporation apparatus that can efficiently deposit low vapor pressure metallic films.

SUMMARY OF THE INVENTION

Accordingly, it is a general object of this invention to eliminate the disadvantages associated with prior art evaporation apparatus so as to provide uniform high quality evaporated thin films as required by dense semiconductor integrated circuit chips. 40

A more particular object of the invention is to provide an evaporation source for use in an RF evaporation apparatus which results in the deposition of high quality, uniform thin films.

tion source for evaporating high quality, uniform metallic films onto a semiconductor workpiece (e.g. a wafer). The evaporation source of the present invention comprises an RF coupling susceptor having a volume commensurate with or greater than the amount of charge 50disposed within an overlaying crucible. More particularly, the source of the invention includes a pair of coaxial, cupped heat shields surrounding a flared lip crucible that is supported away from the base of the 55 material in the crucible being evaporated, into the crucimetallic susceptor. The flared lip of the crucible overlaps and covers the exposed edges of the heat shields. By utilizing the source of the invention, the charge being evaporated from within the crucible can be rap- 60 is described above in only the most rudimentary form. idly heated to promote self-fractionation, thus eliminating conductivity-detracting impurities from the deposited film while providing a film of uniform thickness.

The foregoing and other objects, features and advantages of this invention will be apparent from the follow- 65 ing more particular description of a preferred embodiment of the invention as illustrated in the accompanying drawings, wherein:

DESCRIPTION OF THE DRAWINGS

FIG. 1 (Prior Art) shows a cross-sectional schematic of a conventional evaporation apparatus.

FIG. 2 shows in detailed cross-section the crucible of the present invention.

FIG. 3 shows a plot of the time/temperature cycle used in the present invention.

DETAILED DESCRIPTION OF THE INVENTION

FIG. 1 (Prior Art) shows, in some detail, the general structures associated with conventional evaporation apparatus. A vacuum chamber 10 has a base 11 and a porthole 12 which is connected to a source of vacuum 14. Seated on a pedestal 15 affixed to base 11 is a crucible 16 containing a charge 19 of material to be evaporated. Wrapped around crucible 16 and connected to a suitable RF source 18 (such as 3 kilowatt power supply) power supply is a EN1 PS 300 as sold by the EN1 Corporation of Rochester, N.Y. A holder 23, which can be rotated, if desired, is supported by a rod 21 descending from the top of the chamber 10, and a plurality of work-25 pieces 24 are disposed on the holder 23. Each workpiece 24 is, for example, a semiconductor wafer, such as silicon, having a plurality of previously created chip sites that are to be coated with metallic layers. Each high quality thin films of low vapor pressure materials 30 or the like, that clearly delineates the metallic pattern to wafer may bear a protective coating, such as photoresist be deposited on the semiconductor surface. Alternatively, the metallic material may be deposited on the wafers in the absence of such protective coating, in which case the pattern will be defined by subsequent 35 subtractive etching.

> An interposing shutter 25 comprising a solid plate of stainless steel can be selectively positioned between the substrates 24 and the crucible 16 by motor 26. The shutter 25 can be moved to the left to block the partially or totally emission of evaporant from the crucible 16. During the evaporation phase the path of the evaporant is generally cone shaped as if the evaporant came form a point source, as indicated by dashed lines 30.

If desired, the evaporation source can be disposed in The invention is directed toward a unique evapora- 45 an entirely separate chamber below the evaporation chamber 10. In this manner the sources may be heated independently of the workpieces. An example of this system is the Balzers SCS 800 isolated source load lock evaporation system. Such portioned apparatus is well known to the prior art. It is preferred to provide the evaporation source of the invention to this type of evaporation system.

> A mechanism 27 is provided within the evaporation chamber 10 for automatically feeding a wire, of the ble to automatically replenish the charge 19 as it becomes diminished by evaporation.

> It should be noted that such an evaporation apparatus is presently well known and commercially available and

> In the prior art, when depositing a low vapor pressure metal such as copper, it was found that during the initial heating and melting phase any impurities contained within the charge would, during the evaporation thereof, emit particles of impurity material (e.g. oxide) from the molten evaporant source. This phenomenon (known as "spitting") results in large particles of the evaporant and oxide in a solid, liquid or semisolid state

being transmitted to the workpiece. These impurity particles of materials cause irregularities, such as large spheres, lumps, or voids, to appear in the evaporated film. These irregularities reduce the conductivity of the film, causing electrical defects and the like that result in 5 a lower production yield of finished work.

In addition, prior art evaporant crucibles were generally designed to be relatively deep with respect to their diameter, i.e., their depth was generally at least twice their diameter. This causes, as the charge becomes di- 10 minished, a narrowing of the stream of evaporant material, as shown by dashed lines 31. Because of this narrowing of evaporant the center workpieces 24 accumulate a significantly larger amount of material than does any of the workpieces on the perimeter of the holder 23, 15 also leading to a lack of uniformity of deposit.

The present inventors succeeded in overcoming these disadvantages of the prior art by redesigning the evaporation source. Specifically, the inventors found that when the charge is quickly heated to a temperature at 20 which it self-fractionates (i.e., pure metal is separated from the oxide impurities), and thereafter is deposited onto the workpieces, the oxide impurities are eliminated from the film as-deposited. An evaporation source that is compatible with such a self-fractionation heating 25 process will be described in detail below in conjunction with FIG. 2.

The evaporation source 50, shown in FIG. 2, is designed to be substituted for the post 15 and crucible 16 in FIG. 1, and basically comprises a thermally isolating 30 column or spacer 52 on base 11, which in turn supports an outer heat shield 53. Contained within the outer heat shield 53 is a second thermally isolating spacer 54 which supports and thermally isolates an inner heat shield 55 from the outer heat shield 53. Within the inner heat 35 shield 55 there is disposed a third thermally isolating spacer 56 upon which there is disposed a susceptor 57 which in turn supports a crucible 58. This crucible is provided with a flared lip 59 which extends from the inner edge of the crucible above the lips or upper edges 40 of the two heat shields 53 and 55. A multiplicity of RF coils 22 matched to the power supply, as is well known in the RF heating industry, surround the above described structure.

As shown in FIG. 2, there is also provided an outer 45 casing 60 coupled to the outer heat shield 53 to surround and enclose the coils 22. The casing 60 prevents any of the evaporated material from depositing on the coils 22, the outer heat shield 53, or the thermal isolating column 52. This casing need not be used and can be 50 heat shield 55 from the outer heat shield 53. This inner eliminated, but because it aids in extending the life of the coils 22 and eliminates the need of cleaning the column and outer heat shield after every evaporation run, it is very desirable.

In operation, when the RF power supply 18 is acti-55 vated, the coils 22 emit RF energy which is concentrated within the confines of the coil. This energy is absorbed by the material adjacent the coil, and the material begins to heat due to eddy currents developed therein by the emitted frequencies.

Because it is necessary to heat the charge 19 to controlled temperatures at which it will self-fractionate and evaporate in a relatively short time, and since it is desirable that this be accomplished with the lowest possible energy expenditure, it is necessary to select both the 65 configurations and compositions of the susceptor 57 and crucible 58 so as to absorb the maximum amount of energy and convert it into heat, and to transfer the heat `

by radiation and conduction to the material 19 within the crucible 58. The other components of the source 50 are selected to minimize this RF absorption so that the level to which they become heated is minimized.

The susceptor 57 is made of a solid, columnar block of graphite carbon $1\frac{1}{8}$ inches in diameter and $\frac{1}{2}$ inches high. The crucible 58 is 13/16 inches in diameter and 11/16 inches deep, and is made of a material (e.g. molybdenum) that has a lower vapor pressure than the material to be evaporated (e.g. copper). The crucible is filled with approximately 30 grams of material, such that it is partially filled (i.e., the material within the crucible forms a pool approximately 5/16 inches high). The inventors found that when the initial volume of charge to be deposited is commensurate with (e.g. $\frac{5}{8}$) the volume of the suceptor 57, the charge can be completely self-fractionated. The self-fractionation efficiency increases as the amount of charge decreases (i.e. as the charge/susceptor volume ratio decreases). On the other hand, if the charge/susceptor volume ratio is increased by increasing the charge size or charge volume, or decreasing the susceptor, or a combination of both, the self-fractionation process will not be as efficient. At very high charge/susceptor ratios, self-fractionation does not occur to an extent sufficient to improve the properties of the film as-deposited.

The susceptor 57, being a relatively large mass and being very receptive to heating by RF frequencies, assures that the charge 19 contained within the crucible 58 is heated to the desired levels necessary to melt and evaporate the charge 19 in the crucible 58. Such susceptors are especially required when the charge is a low vapor pressure material (e.g. copper or gold) that is not very absorbant of RF energies.

The base 11, upon which the unit under discussion is mounted, is usually metallic and relatively large. It is therefore very radiative (i.e., it acts as a heat sink). Since it is necessary to heat the contents of the crucible rapidly, a quartz thermal insulator 52, in the form of a hollow column having an outside diameter of $1\frac{1}{2}$ inches, a height of $1\frac{3}{8}$ inches, and an inner diameter of $1\frac{1}{4}$ inches, is disposed to support the outer heat shield 53 from the base 11. This outer heat shield 53 is a cup formed of high density silica having a wall thickness of 1 inch on outer diameter of 1 11/16 inches and a height of $1\frac{3}{4}$ inches. Similarly, the quartz spacer 54 also in the form of a 1/16 inch ring 1 5/16 inches in diameter and 3/16 inches in height is disposed in the bottom of the cup shaped outer shield 53 to maintain and thermally isolate the inner heat shield is formed of pyrolytic boron nitride and has an outer diameter of 13 inches in diameter, a height of 1 9/16 inches and a wall thickness of 1/16 inches. Quartz and pyrolytic boron nitride are used for most of the thermal isolators and the inner heat shield because such materials are transparent to radio frequencies emitted by the coil 22 and thus becomes heated very slowly. Other material having similar properties could be used. Also, such materials can be made in very pure form 60 such that they do not provide any contaminants in the system.

The outer heat shield 53 is formed of silica which has improved thermal characteristics over those of quartz. The inner shield 55 is formed of pyrolytic boron nitride.

Pyrolytic boron nitride is used as the inner heat shield because it prevents infrared radiation from being emitted from either the susceptor or the crucible. In this way the inner shield acts to retain heat within itself, i.e. in the crucible area and susceptor area, while shielding the outer shield 53 from infrared radiation.

A boron nitride support ring 56, 15/16 inches in diamtter which is $\frac{1}{2}$ inch in height and 1/16 inches thick is disposed within the inner heat shield 55 to support a 5 carbon susceptor 57 which in turn supports the crucible 58. Pyrolytic boron nitride is used for this support ring 56 not only because it is a good thermal isolator but also because it is formed with the highest purity and does not temperatures at which source is operated.

It should be noted that the crucible 58 has an outwardly directed flared lip 59. This lip 59 protects the top exposed edges of the shields 53 and 55 and prevent any of the material being evaporated from within the 15 crucible from depositing on the inner edges of the heat shield 55. Moreover, note that the depth of the crucible is less than its diameter. If the depth/diameter ratio is increased to the point where the depth is greater than problems result.

With reference now to FIG. 3, there is shown the RF power curve (solid line 61) and the temperature curve (dotted line 62) associated with the self-fractionation and deposition processes that can be carried out using 25 the evaporation source of the invention. A 3 kilowatt RF power supply was used, and a charge of 30 grams of copper was disposed within the crucible. At the beginning of the cycle, the workpieces are located in the vacuum chamber and the chamber is evacuated. Four 30 hundred fifty watts of RF power, as shown by that portion of the power curve marked 63, is then applied to the coils. The susceptor 57 begins to heat and to transfer this heat by a radiation and conduction process into the charge in the crucible, and the charge begins to melt 35 with little or no evaporation occurring. This power level is maintained until the temperature of the charge stabilizes at 1200° C., as shown by that portion of the temperature curve marked 63A. During this time, to protect the workpieces from any undesired evaporation 40 or contamination, the shutter 26 is moved to the left from the position. When the workpieces are to be coated, the RF power is ramped to between 2 kilowatts and 2.1 kilowatts, and is maintained there for approximately 135 seconds. This is shown by that portion of the 45 power curve marked 64. Subsequently the power level is reduced to 780 watts (portion 66). During this time the temperature of the charge 19 is increased and selffractionation, i.e. fractional distillation, of the charge 19 occurs. As this fractional distillation begins, any con- 50 and scope of the invention. taminants in the charge (such as oxides or the like) adhere to the surface of the melt to form a slag-like surface layer. As the RF power is increased, the temperature of the charge also increases and the fractional distillation continues until the contaminants collected 55 on the liquid surface of the melted charge become either sublimed or vapored due to limits set by respective vapor pressures of the material used in the charge and its oxides and other contaminants. The impurities migrate to the edge of the crucible where they are drawn 60 up the side of the crucible. In copper, this occurs because a high vapor pressure of copper evaporant is created, which pushes the surface contaminants and the like to the sidewalls of the crucible (which are at a temperature slightly higher than that of the melted 65 depth of said crucible is less than twice said diameter charge). Once this fractionation process is completed the temperature of the crucible has peaked at 1900° C. (reference numeral 65 on curve 62). The RF power is

then decreased to a power level of 780 watts as indicated by numeral 66 on curve 61. This power level corresponds to the desired rate of deposition of 4 angstroms per second of the charged material on the surface of the workpieces 24 when the workpiece is 28 inches away from the crucible 58. Forty-five seconds after this decrease in RF power, when the temperature of the charge 19 stabilizes at about 1510° C., as indicated by numeral 67 on curve 62, the evaporant shield or interact with the carbon of the susceptor 57 at the high 10 shutter 24 is withdrawn from in front of the workpiece to permit the evaporant to coat the workpiece. This power level is maintained for an additional 150 seconds at which time the shutter 24 is then positioned in front of the workpiece to prevent further deposition on the workpieces. Simultaneously with the shutter 24 closing, the power level is raised to 1000 watts as indicated by reference numeral 68, and is maintained at this level for 90 seconds. This causes a slight rise in temperature of the charge 19 as indicated by numeral 69 on curve 62. twice the diameter, the above-described uniformity 20 Twenty-four seconds after the shutter closes and the power level rises, wire is fed from the wire feed 27 into the crucible to replenish the charge therein. As the wire melts and replenishes the charge the temperature of the charge decreases as indicated by numeral 70 on curve

At the end of 90 seconds the power is again decreased to the standby power level of 450 watts and the temperature of the charge continues to decline to 1200° C. at which temperature it again stabilizes.

By supporting all of the units of the invention within the coils 22, as described above, the crucible 58 is heated in a thermally controlled manner to provide self-fractionation. This self-fractionation phenomenon eliminates any spitting of copper or copper oxides and eliminates the need for utilizing ultra pure material as a source. Self-fractionation of the charge is particularly important when the material of the charge has a large amount of impurities that are introduced during its replenishment.

By utilizing the present invention as described above, copper films free of voids or inclusions and with less than 0.001% by weight of non-metallic contaminants contained therein have been successfully deposited on semiconductor wafers.

While the invention has been particularly shown and described with reference to a preferred embodiment thereof, it will be understood by those skilled in the art that the forgoing and other changes in form and details may be made therein without departing from the spirit

What is claimed is:

- 1. An evaporation source, comprising:
- a crucible having a given depth and a given diameter, said crucible containing a volume of molten material to be evaporated;
- a susceptor abutting said crucible, said susceptor being a solid columnar block, the volume of said molten material within said crucible prior to evaporation of said molten material being five eights or less of the solid volume of said susceptor; and
- a conductive coil disposed about said susceptor and said crucible for coupling RF energy to said susceptor and to said crucible.

2. The evaporation source of claim 1, wherein said thereof.

3. The evaporation source of claim 1, wherein said susceptor comprises a block of graphite carbon.

4. In a metal evaporation apparatus, a RF heated source comprising:

- a crucible disposed within said RF coil, said crucible having a depth that is less than twice its diameter, said crucible containing a volume of molten material to be evaporated; and
- a susceptor abutting and supporting said crucible, said susceptor comprising a solid columnar block 10 of material, the volume of said molten material prior to evaporation being five eights of the volume of the susceptor.

5. The apparatus of claim 4, further comprising a plurality of coaxial heat shields separating said crucible and said susceptor from said RF coil, each of said plurality of coaxial heat shields having an upper edge.

6. The apparatus of claim 5 wherein crucible has a flared mouth that extends above said upper edges of 20 each of said plurality of heat shields.

7. The apparatus of claim 4 wherein said susceptor comprises a block of graphite carbon.

8. In an evaporation apparatus, an evaporation source 25 or promoting self-fractionation of molten material to be evaporated,

a radio frequency heating coil;

- a plurality of coaxial heat shields each having an upper edge, said shields being supported within said coil;
- a solid columnar susceptor block of a given volume disposed within an innermost one of said plurality of coaxial shields;
- a crucible disposed on and abutting said susceptor and containing a volume of molten material to be evaporated, said crucible having a depth that is less than twice its diameter,
- the volume of said molten material, being less than five eights of the volume of the susceptor.

 The apparatus of claim 8 further comprising a plurality of coaxial heat shields separating said crucible
and said susceptor from said RF coil, each of said plurality of coaxial heat shields having an upper edge.

10. The apparatus of claim 9 wherein crucible has a flared mouth that extends above said upper edges of each of said plurality of heat shields.

11. The apparatus of claim 9, wherein said susceptor comprises a block of graphite carbon.

12. The apparatus of claim 11 wherein an outermost one of said plurality of heat shields comprises a cupshaped member made of silica.

13. The apparatus of claim 12, wherein an innermost one of said plurality of heat shields comprises a cupshaped member made of pyrolytic boron nitride.

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a RF coil;