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METHOD OF PRODUCING HYPERPURE SILICON

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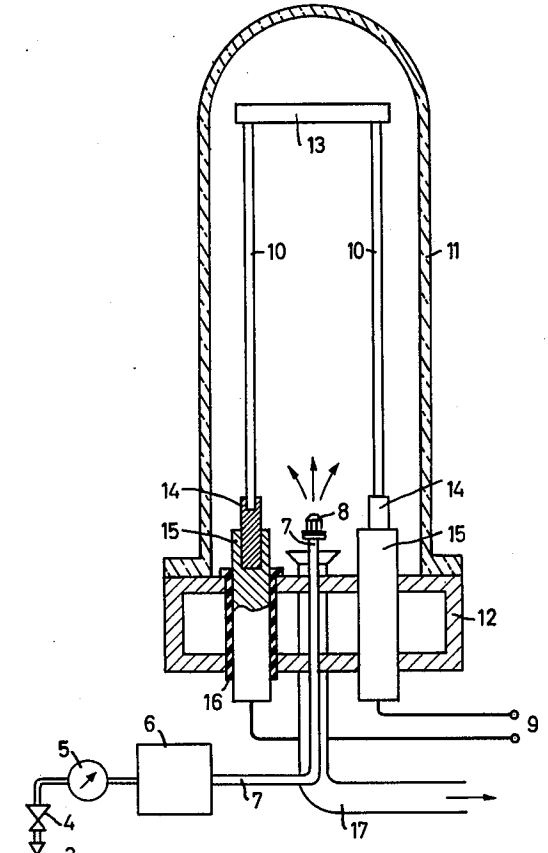

FIG. 1

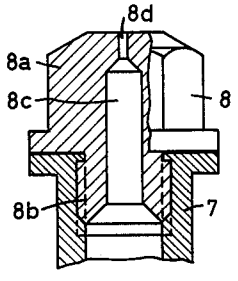

FIG. 2

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METHOD OF PRODUCING HYPERPURE SILICON

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2 Claims. (Cl. 117—201)

Our invention relates to improvements in the production of hyperpure semiconductor material, particularly silicon, for electronic purposes. Application, Serial No. 90,291, filed February 20, 1961, and now Patent No. 3,099,534, a division of application, Serial No. 665,086, filed June 11, 1957, now Patent No. 3,011,877, and the parent application respectively claim methods and devices for preparing semiconductor material. According to these methods and devices, solid core rods of the same material are mounted on a common base and are electrically heated by passing current through the rods, while the rods are mounted in a sealed vessel traversed by reaction mixture comprising a gaseous compound of the semiconductor material to be precipitated and a gaseous reaction agent such as hydrogen. By means of electric current the rods are kept at a high temperature, for example between 900 and 1200° C. for silicon, at which the gaseous semiconductor compound becomes pyrolytically reduced and the resulting semiconductor material is precipitated upon the core rods, which gradually increase in thickness. The inlet for the gaseous reaction mixture in such devices is located on or in the base structure carrying the holders for the core rods. In a particular embodiment of the devices, the gas inlet has a nozzle-shaped design so that the fresh gas mixture is caused to flow from the location of the core-rod holders along the core rods in the longitudinal direction of the rods.

It is an object of our invention to improve the performance of such methods and devices toward an optimum rate of crystalline growth of the material being precipitated.

We have discovered, according to our invention, that this advantage is achieved if the speed of the reaction gas mixture entering into the pyrolytic reaction chamber of a sealed apparatus is maintained above 100 m./sec. (meters per second) but below 200 m./sec. at the mouth of the inlet nozzle. The significance of this rate of gas supply will be understood from the following.

For attaining large quantities of precipitated semiconductor material, it is generally desirable to keep the speed of the gaseous semiconductor compound entering into the reaction vessel as large as feasible, because the quantity of semiconductor material precipitating onto the cored rods is dependent, among other things, upon the throughput of gas.

We have found that by keeping the gas-entering speed below about 200 m./sec., the semiconductor material has a uniform thickness over the entire length of the core rods during the processing period. This is of advantage particularly for a monocrystalline growth of the semiconductor material being precipitated, because such a monocrystalline rod can be sawed or cut, without further fabrication, into a multiplicity of discs or wafers all having the same size and thus being all suitable as monocrystalline base bodies for electronic semiconductor devices of a great variety of types. However, we have found that the economy of operation becomes appreciably impaired if the gas-entering speed becomes too small, and that the speed range within which the desired improvement is achieved is substantially between 100 and 200 m./sec. Maintaining the gas-entering speed below about 200 m./sec. has

the additional advantage of preventing breakage of equipment, for example of a quartz bell forming the upper portion of the hermetically sealed vessel structure, or lifting of the quartz bell from the mounting base constituting the bottom wall of the reaction chamber, in the event of a high-pressure gas build-up in the reaction space, caused by clouding or narrowing of the gas outlet.

An embodiment of equipment by means of which the above-described can be performed in a simple manner is illustrated by way of example on the accompanying drawing in which:

FIG. 1 shows schematically and partly in section an apparatus for the production of hyperpure silicon; and

FIG. 2 shows separately an inlet nozzle for the gaseous reaction mixture which forms part of the apparatus according to FIG. 1.

In the illustrated apparatus, hydrogen, to be employed as carrier and reaction gas, is supplied from a gas bottle 2 through a shut-off valve 3 and a plural-stage pressure-reduction valve 4 from which the gas passes through a gas-flow meter 5 to an evaporator 6. In the evaporator 6 a liquid semiconductor compound, preferably silico-chloroform $SiHCl_3$ or silicon tetrachloride $SiCl_4$, is evaporated. In the evaporator 6, therefore, the hydrogen becomes mixed with the evaporated silicon compound, such as silico-chloroform, and passes through a gas pipe 7 to a nozzle-type inlet device 8 and thence into the reaction space. The inlet nozzle 8 may have an inner diameter of about 2 mm. for example and is designed to produce a turbulent flow of gas along core rods 10 of hyperpure silicon. The rods, for example, have a thickness of 3 mm. and a length of about 400 mm. The rods 10 are mounted in a reaction vessel comprising an hermetically sealed quartz cylinder or bell 11 whose inner diameter, for example, is approximately 150 mm., and whose height, for example, is approximately 550 mm. It will be understood that the numerical values here given are properly correlated to one another, but may be modified in accordance with the requirements or desiderata of a particular method or apparatus. The bottom of the reaction chamber is closed by a metallic base plate 12, which is hollow and preferably cooled in its interior by a flowing or circulating coolant. The lower ends of the two rods 10 are inserted in respective bores of holders 14 consisting, for example of spectral carbon which in turn are fastened in cylindrical supports 15, inserted into the base plate 12, for example, by having a screw thread in engagement with threaded bores of the base plate 12. At least one of the cylindrical bodies 15 is insulated from the base plate 12 by an insulating sleeve 16. The cylindrical bodies 15 are connected to terminals 9 to be attached to a supply of electrical current for heating the core rods 10. The upper ends of the rods 10 are electrically interconnected by a bridge piece 13 which preferably consists of the same semiconductor material, namely silicon, as the core rods 10.

The turbulent flow of gases issuing from the orifice of the nozzle 8 passes along the rods 10 toward the top of the reaction space where the gas flow becomes reversed. The spent residual gases leave the reaction vessel through an outlet pipe 17, which concentrically surrounds the nozzle 8. The nozzle 8 is so designed that the entire reaction gas mixture entering into the reaction vessel, for example in a quantity of about 2.5 $m.^3/h.$, is whirled about in the reaction space so that a largest possible proportion of the gases enter into contact with the surface of the core rods 10. This promotes a uniform growth of semiconductor material on the carrier rods. Then the rods are heated electrically to the above-mentioned pyrolytic temperature, for example, in the neighborhood of 1000 to 1100° C. As mentioned, the speed at which the gaseous mixture enters through the nozzle into the

reaction space is below 200 m./sec. and is, for example, in the neighborhood of 150 m./sec.

As shown in FIG. 2, the nozzle head 8*a* may have a threaded neck portion 8*b* screwed onto the top end of the inlet pipe 7. A bore 8*c* of the nozzle has a diameter of 5 to 10 mm. but is constricted at the nozzle mouth 8*d* to a diameter of about 0.5 to 4 mm. over a length of about 2 to 5 mm.

While reference is made in the foregoing description to the precipitation of silicon, the method of the invention is applicable in the same manner for the produtcion of other semiconductor materials, for example germanium, silicon carbide SiC, indium antimonide InSb, or gallium arsenide GaAs, in which cases the same advantages, uniform product, and trouble-free operation are simultaneously secured.

We claim:

1. A process for producing a silicon body by reaction of a gaseous mixture of hydrogen and a gaseous compound of silicon in a reaction chamber, comprising heating a silicon core body of approximately the same purity as the silicon to be precipitated to glowing temperature, the heated body effecting the reaction, introducing said gaseous mixture, at a velocity of at least 100 m./sec. and below 200 m./sec. into the reaction chamber to produce a high degree of turbulence whereby the gas mixture flows longitudinally along said core body to effect efficient reaction into silicon and uniformly thicken said core body.

2. A process for producing a silicon body by reaction of a gaseous mixture of hydrogen and a gaseous silicon compound selected from the group consisting of silicochloroform and silicon tetrachloride in a reaction chamber, comprising heating a silicon core body of approximately the same purity as the silicon to be precipitated to glowing temperature, the heated body effecting the reaction, introducing said gaseous mixture, at a velocity of at least 100 m./sec. and below 200 m./sec., into the reaction chamber to produce a high degree of turbulence whereby the gas mixture flows longitudinally along said core body to effect efficient reaction into silicon and uniformly thicken said core body.

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