TO: KSI/Scientific & Technical Information Division
Attn: Miss Winnie M. Morgan

FROM: GP/Office of Assistant General Counsel for Patent Matters

SUBJECT: Announcement of NASA-Owned U.S. Patents in STAR

In accordance with the procedures agreed upon by Code GP and Code KSI, the attached NASA-owned U.S. Patent is being forwarded for abstracting and announcement in NASA STAR.

The following information is provided:

U.S. Patent No.: 3,796,592

Government or Corporate Employee: U.S. Government

Supplementary Corporate Source (if applicable): 

NASA Patent Case No.: ERC-10073-1

NOTE - If this patent covers an invention made by a corporate employee of a NASA Contractor, the following is applicable:

YES / NO

Pursuant to Section 305(a) of the National Aeronautics and Space Act, the name of the Administrator of NASA appears on the first page of the patent; however, the name of the actual inventor (author) appears at the heading of each page of the Specification, following the words "...with respect to an invention of..."

Bonnie L. Woerner

Enclosure
METHOD AND APPARATUS FOR STABLE SILICON DIOXIDE LAYERS ON SILICON GROWN IN SILICON NITRIDE AMBIENT
Filed Sept. 9, 1969

Fig. 1.

Fig. 2.

Fig. 3.

INVENTORS
RONALD A. COHEN &
ROY K. WHEELER

ATTORNEYS
A method and apparatus for thermally growing stable silicon dioxide layers on silicon is disclosed. A previously etched and baked silicon nitride tube placed in a furnace is used to grow the silicon dioxide. Pure oxygen is allowed to flow through the tube to initially coat the inside surface of the tube with a thin layer of silicon dioxide. After the tube is coated with the thin layer of silicon dioxide, the silicon is oxidized thermally in a normal fashion. If the tube becomes contaminated, the silicon dioxide is etched off thereby exposing clean silicon nitride and then the inside of the tube is recoated with silicon dioxide.

The silicon nitride tube can also be used as an ambient for the pyrolytic decomposition of silane and ammonia to form thin layers of clean silicon nitride. This method of forming thin layers of clean silicon nitride avoids the problem of mobile ions that is encountered in the prior art method of epitaxial deposition of silicon nitride in quartz tubes, because tubes can contain and also transmit mobile ions.

A still further object of this invention is to provide a method for forming thin layers of silicon nitride. And a still further object of this invention is to provide the apparatus for forming thin layers of silicon nitride.
the silicon nitride tube 1. Silicon dioxide is then thermally grown on the silicon wafer 3 in a normal fashion. The silicon wafer 3 having been placed inside the tube 1 prior to the initial step of forming a coating of silicon dioxide on the inside of the silicon nitride tube 1. The statement that silicon dioxide is grown on the cleaned silicon wafer 3 in a normal manner means that the silicon dioxide is grown on the silicon wafer 3 by any suitable well known prior art method such as described in the review article entitled “The Si—SiO₂ Solid-Solid Interface System,” A. G. Reveze and K. H. Zawinger, RCA Review pp. 22–76, March 1968.

FIG. 2 shows the apparatus of the invention for forming thin layers of silicon nitride. The apparatus of FIG. 2 is identical to the apparatus of FIG. 1 except that the liquid oxygen tank 7 of FIG. 1 has been replaced by a first tank 9 containing ammonia and a second tank 9 containing silane has also been connected to the valve 6 by means of a section of tubing 11. Also, a nitrogen tank 12 has been added for flushing purpose and a hydrogen chloride and/or hydrogen tank can be added for optional in situ cleaning of the silicon wafer. In addition, the wafer or slice 3 can be made of any suitable material including silicon, germanium, gallium arsenide, etc. Wafer 3 serves as a substrate upon which the silicon nitride is formed.

In the apparatus of FIG. 2, the inside of the silicon nitride tube 1 is initially coated with a thin layer of silicon dioxide. In order to form the silicon nitride layer, the valve 6 is turned on permitting the silane and ammonia to flow through the tube 1 with the furnace 2 at a temperature of 800° C. to 1200° C. A layer of silicon nitride is formed on the substrate 3 by the pyrolytic decomposition of the ammonia and silane.

The relative positions of the elements inside the silicon nitride tube 1 are clearly shown in FIG. 3. As shown, the inside of the silicon nitride tube 1 is coated with a layer of silicon dioxide 13. The platform or boat 4 is generally centrally located inside the tube 1 and the substrate 3 which is shown as being a silicon slice in this figure is seated on the boat 4.

While the invention has been described with reference to specific embodiments, it will be obvious to those skilled in the art that various changes and modifications can be made. For example, the silicon nitride container can be used as a noncontaminating environment for other chemical reactions, such as deposited alumina, Al₂O₃ and as a tube for sintering aluminum contacts to silicon integrated circuits.

What is claimed is:

1. A method for growing stable silicon dioxide layers on a silicon substrate comprising the steps of: placing said substrate in a silicon nitride tube; heating said tube to a temperature in the range of 1000° C. to 1200° C.;
flowing pure oxygen through said tube when heated to said temperature range to thereby provide a coating of silicon dioxide on the inside surface of said silicon nitride tube; and
thermally growing a layer of silicon dioxide on the silicon wafer inside said tube by thermal oxidation.

2. A method for growing thin films of silicon nitride on a substrate comprising the steps of: placing the substrate in a silicon nitride tube; heating said tube to a temperature in the range of 1000° C. to 1200° C.;
flowing pure oxygen through said tube when in said temperature range to thereby provide a coating of silicon dioxide on the inside surface of said tube;
flowing a mixture of silane and ammonia through said tube when maintained in a temperature range of 800° C. to 1200° C. to thereby form a layer of silicon nitride on said substrate by the pyrolytic decomposition of the ammonia and silane.

References Cited

UNITED STATES PATENTS


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ALFRED L. LEAVITT, Primary Examiner
M. F. ESPOSITO, Assistant Examiner

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117–106 R, 106 A, 201, Digest 12