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[54] CONTINUOUS VAPOR PROCESSING APPARATUS AND METHOD

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[51] Int. Cl. .... C23c 11/08

[58] Field of Search 117/106 A, 106 R, 107.1, 107.2, 117/54; 118/48, 49, 50

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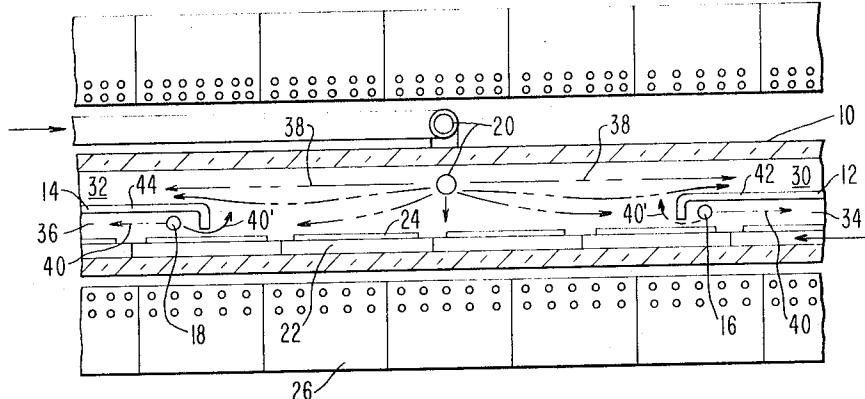
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[57] ABSTRACT

Apparatus for effecting uniform and continuous mass transport reactions, such as oxidation, diffusion, etching, etc., between a gaseous phase reactant and semiconductor substrates. The apparatus comprises a longitudinal process tube, which includes a reaction zone flanked on either side by a combination entrance-exhaust section and an exit-exhaust section. Reactant gas is provided to the reaction zone at a fixed flow rate and allowed to escape from the reaction zone axially through the two exhaust zones. Accurate isolation of the reaction zone is accomplished by passing additional gases into the entrance and exit zones at a rate sufficient to cause some gas to flow into the reaction zone where it is carried away through the exhaust zones by the axially flowing reacting gas.

12 Claims, 4 Drawing Figures



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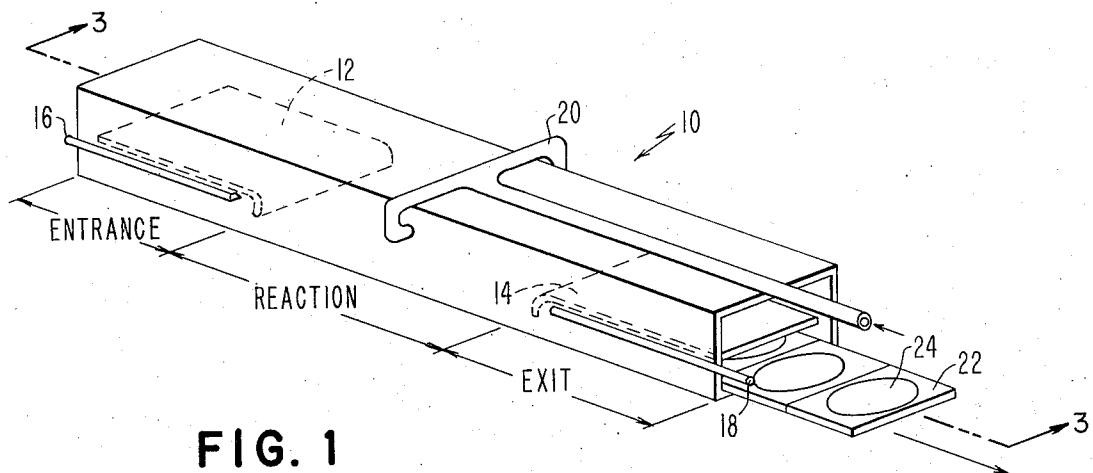


FIG. 1

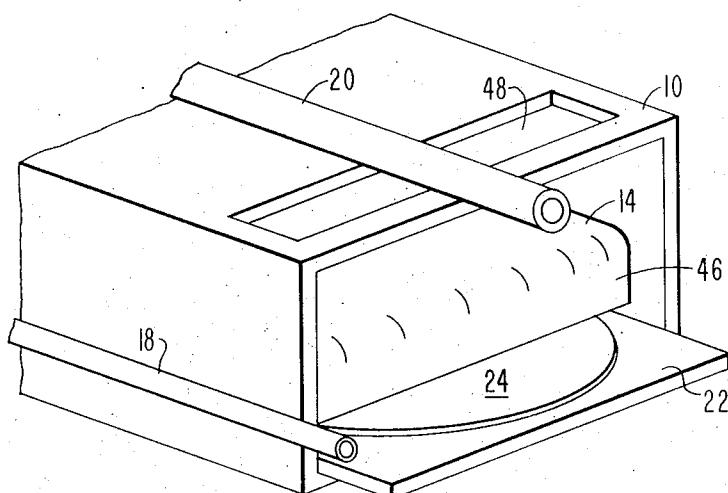


FIG. 3

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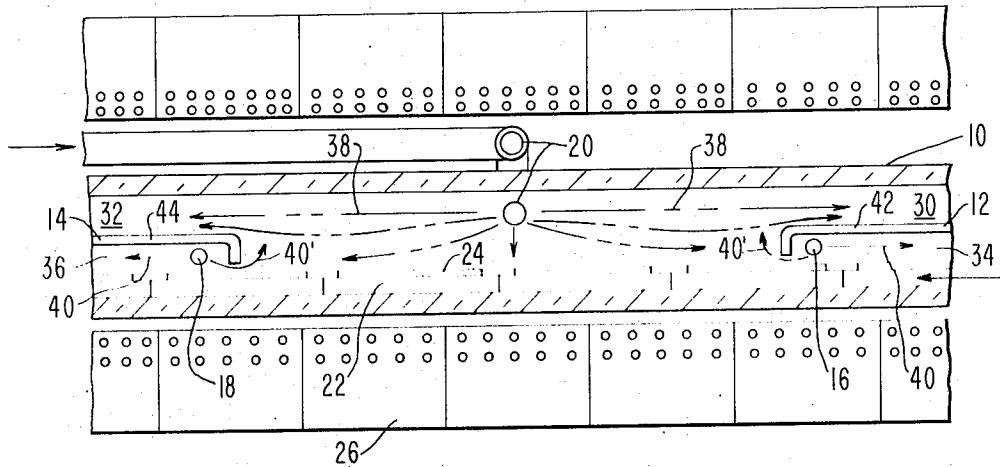


FIG. 2

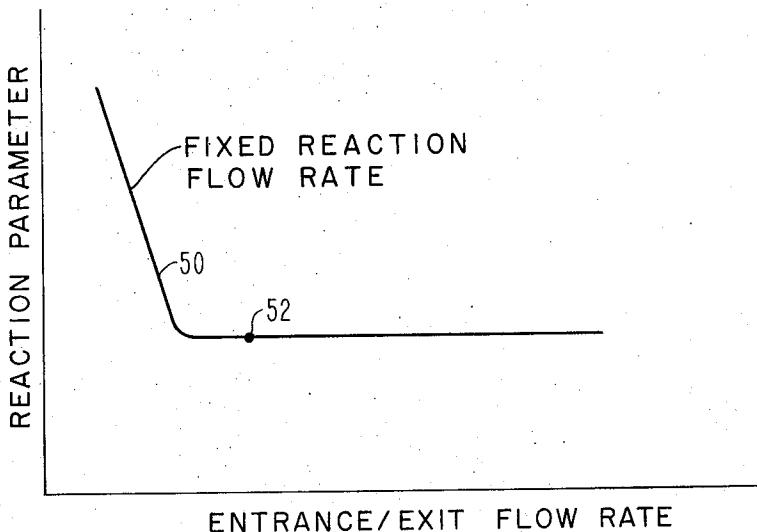


FIG. 4

## CONTINUOUS VAPOR PROCESSING APPARATUS AND METHOD

### BACKGROUND OF THE INVENTION

This invention relates to the processing of semiconductor devices and more particularly to apparatus for continuously carrying out mass transfer operations between gaseous phase materials and solid substrate surfaces.

In the manufacture of semiconductor devices there are numerous occasions for utilizing mass transport reactions between gaseous phase reactants and semiconductor substrates to produce additive processes, such as the production of oxide films, doped oxide layers for diffusions, etc., as well as, subtractive processes such as etching. In recent years much emphasis has been placed on the importance of providing extremely uniform integrated circuit products on a mass production basis. In the early years it was commonplace to utilize batch processing for almost all semiconductor manufacturing steps. Such processing included frequent use of the well known closed and open tube processes.

Recent emphasis has been placed in the development of various methods and apparatus capable of operating on a continuous, or in line, basis. Prior art apparatus for achieving continuous processing such as oxidation, deposition and etching, etc., have proved to be extremely expensive and require elaborate hardware configurations, special handling techniques and special support equipment.

Because of the extreme criticality of processing parameters necessary for the production of semiconductor devices, substantial development effort has been directed to devising methods and apparatus for accurately controlling such parameters. For example, in processes requiring critical thicknesses of oxides, it is necessary to insure that the length of the reaction zone of a continuous oxidation tube be exactly maintained. Prior art techniques used to isolate processing steps include the use of gas curtains, usually directed perpendicular to the movement of the substrates, positive or negative pressure devices and various types of vapor locks including such mechanical items as sliding door locks, etc.

In summary, no continuous processing equipment simple in design and adaptable to existing support equipment has been devised.

### SUMMARY OF THE INVENTION

It is therefore an object of the instant invention to improve the uniformity of processing semiconductor devices and other substrates in continuous mass production.

It is another object of the invention to reduce the cost of producing semiconductor devices and other products by providing a basic processing tube adaptable to many applications.

It is yet another object of this invention to provide a processing tube and method of operation which are adaptable to existing support equipment without substantial modification.

The basic aspects of the instant invention provide for substantial improvement in the quality of mass produced articles through the use of a process tube of simplified construction and operation. The process tube essentially comprises a centralized reaction zone

flanked on either side by a multi-functional region formed by a simple longitudinal partition dividing that portion of the tube into two isolated chambers. On one side of the reaction zone there is provided an exhaust and an entrance zone and on the other side an exhaust and an exit zone. Reactant gas is provided to the reaction zone at a constant flow rate. The structure of the process tube causes the reacting gas to flow axially through the reaction zone and out both ends. A flow of 10 purge gas, or other compatible gas, is provided to both the entrance and exit zones at a rate sufficient to cause some gas to flow into the reaction zone where it immediately is swept out through its associated exhaust zone by the axially flowing reactant gas. Substrates are 15 passed sequentially through the entrance, reaction and exit zones and are exposed to the reactant for a precisely predetermined time which provides extremely uniform processing.

The foregoing and other objects, features and advantages 20 of the invention will be apparent from the following more particular description of the preferred embodiments of the invention, as illustrated in the accompanying drawings.

### BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is an isometric view of the preferred embodiment of the invention showing the general layout and structure of the process tube.

FIG. 2 is a partial sectional view of a preferred embodiment of the invention showing the required gas flow patterns in the vicinity of the reaction zone.

FIG. 3 is a partial isometric view of one end of a second embodiment of the invention showing the structure of a modified partition used to isolate the exit zone 35 from one of the exhaust zones.

FIG. 4 is a plot of the effect of a reaction parameter 40 versus the flow rate of gas in the entrance/exit zones showing the independence of the reaction parameter to flow rate above a critical flow rate.

### DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring to FIG. 1 there is shown an isometric view 45 of the preferred embodiment of process tube 10. Although the preferred embodiment is specifically designed for the processing of semiconductor wafers, other items, or substrates, may be equally suitable to treatment where exacting uniformity of the produced product is a necessity. The basic supporting structure

50 for process tube 10 is a commercially procured rectangular quartz tube nominally measuring  $\frac{3}{4}$  inch high, 2  $\frac{1}{2}$  inches wide, and 72 inches long. A flat tube is preferred as it facilitates stacking of multiple tubes in a standard furnace thereby increasing throughput.

55 Mounted inside the ends of tube 10 and designated by phantom lines, there are provided longitudinally extending quartz partitions 12 and 14. As can be seen, the partitions divide process tube 10 into three separate portions. The central portion, between partitions 12

60 and 14, defines the reaction zone, as indicated in FIG. 1. A second portion of tube 10 contains partition 12 defining an substrate entrance region, a third portion defines a gas exit region. These last mentioned portions are described more fully in connection with FIG. 2. 65 Mounted on the side of tube 10 there is provided two gas inlet tubes 16 and 18 through which gases may be passed into the entrance and exit zones. In order to sup-

ply a gaseous reactant to the reaction zone there is provided a gaseous reactant inlet tube 20. Representative substrate carriers 22 and semiconductor wafers 24 are shown at the exit end of tube 10 and normally would be moved either continuously or incrementally through tube 10 during processing.

Referring now to FIG. 2 there is shown a partial sectional view of process tube 10 mounted inside a commercial furnace consisting of heating blocks 26 and elements 28. The gaseous reactant introduced through inlet 20 is supplied at a fixed volumetric rate to the reaction zone, defined by the ends of partitions 12 and 14, and allowed to pass freely out both exhaust zones. In order to isolate the reactant gas sufficiently to fix the effective length of the reaction zone to which semiconductor wafers 44 are exposed, a non-reactant, or purge gas, is applied to inlets 16 and 18 of entrance zone 34 and exit zone 36. This purge gas may conveniently be an inert gas or may be any other reactant compatible with the primary reactant supplied to the reactant zone.

FIG. 2 also shows the gas flow pattern necessary to isolate the reactant gas without a need for complicated pressure regulating or metering devices. Reactant gas flow is indicated by long-short dashed lines 38 and 25 purge gas flow by dashed lines 40 and 40'.

In order to limit the flow rate of the purge gas into the reaction zone it is preferred to restrict the size of the openings between the reaction zone and entrance zone 34 and between the reaction zone and exit zone 36. This is accomplished conveniently by providing extensions 42 and 44 to partitions 12 and 14. It is necessary to provide a sufficient flow of purge gas to the entrance and exit zones to cause at least some gas to pass into the reaction zone as indicated by dashed lines 40'. This flow pattern physically limits the effective length of the reaction zone. In order to insure that the purge gas does not penetrate further into the reaction zone by diffusion it is preferred that the axial flow rate of reactant gas down the process tube toward the exhaust zones exceed the static diffusion rate of the purge gas into the reactant gas, preferably by at least one order of magnitude. Since the total amount of purge gas passing under extension 42 and 44 is immediately swept away from the reaction zone through the exhaust zones, semiconductor wafers 24 passing through the reaction zone are exposed to a constantly refreshed supply of reactant.

Referring briefly to FIG. 3, there is shown a partial isometric view of one end of a modified process tube. In some applications it is desirable to further limit the flow rate of the purge gas from the entrance and exit zones by providing a second extension 46 to partitions 12 and 14. Also shown in FIG. 3 is an exhaust port 48 which allows for more convenient removal of exhaust and purge gases through an overhead hood. It is not normally necessary to provide for further isolation of exhausted gases as the ambient temperature at the ends of the process tube is usually not sufficiently high to support a reaction. In situations where low temperature reactions are utilized further isolation is necessary.

In order to determine the necessary gas flow rates to minimize control requirements while maintaining optimum operating conditions, the following procedure, described in conjunction with FIG. 4 is preferred. Initially, process tube 10 is placed in a furnace in which a desired temperature profile may be maintained throughout the length of the tube. A reactant gas sup-

ply 15 connected through a control valve to gaseous reactant inlet tube 20. A supply of purge, or other desired gas, is connected to gas inlet tubes 16 and 18. Semiconductor wafers 24 are placed on carriers 22 and passed 5 at a constant rate through process tube 10. Reactant gas is passed into the reaction zone at a fixed rate allowing it to pass into both exhaust zones as well as the entrance and exit zones. Purge gas is then passed into, for example the entrance zone at a relatively high but 10 carefully regulated flow rate, not exceeding that of the reactant gas. The flow rate of purge gas into the exit zone is then incrementally increased from zero, allowing sufficient time between changes to allow sample wafers to pass completely through the reaction zone. 15 Processed semiconductor wafers are examined to determine the conditions at which the purge gas flow rate no longer influences the reaction product. For example, such parameters as oxide thickness, diffusion depth or flatband voltage may be measured. Referring now to 20 FIG. 4 there is shown a plot of a typical reaction parameter versus entrance/exit flow rate for a fixed reactant flow in a symmetrical process tube. Curve 50 initially shows a linear dependence on purge gas flow rate. As sufficient back pressure is developed by the purge gas to cause some of the gas to flow into the reaction zone, the reaction parameter is no longer dependent upon 25 purge gas flow rate. An operating point, such as point 52 should be chosen such that expected variations in 30 purge gas flow rate will not effect the reaction. If the process tube is symmetrical, in that both entrance and exit zones are of identical construction, the above determined operating point may be used to determine the proper flow rate in both the entrance and exit zones as 35 they will be equal. If, however, structural variations exist between entrance and exit zones, it is preferable to repeat the above procedure by varying the entrance flow rate and monitoring reactant parameters. Alternatively, both entrance and exit purge gas flow rates may be varied simultaneously to establish an operating 40 point.

In order to insure a controlled distribution of reactant gas in the reactant zone, the flow of reactant and purge gases in each exhaust zone must be kept substantially equal. If this condition is not met by providing a 45 symmetrical tube or by throttling exhaust flow rates, an undesirable net reactant flow in only one direction through the tube will be established destroying much of the effectiveness of the isolation capable of being produced. Attempts to utilize extremely high flow rates or 50 a great differential between entrance and exit flow rates may also produce unstable conditions.

Those skilled in the art will recognize that many mass transport processes well known in the art are suitable 55 for use in the instant process tube. Reference is made to those processes disclosed in U.S. Pat. No. 3,650,042 of Boerger et al., issued Mar. 21, 1972, and assigned to the same assignee as the instant invention, for typical examples of such processes.

In order to demonstrate the principal of operation of 60 the subject invention the following experiment was performed.

A process tube constructed in accordance with the 65 above description and having a reaction zone measuring 26 1/2 inches between partitions was mounted in a commercial diffusion furnace. A flat temperature profile of 1100°C was maintained over about 28 inches, including all of the reaction zone. Partially processed

semiconductor wafers were placed on quartz carriers and passed incrementally at 120 second intervals, 1  $\frac{1}{4}$  inch at a time, through the process tube. Dry oxygen was supplied to both the entrance zone inlet and reaction zone inlet at a flow rate of 2000 cubic centimeters per minute. The nitrogen supply was connected to the exit zone inlet, but no nitrogen flow was initially supplied. This condition allowed oxygen to pass into the exit zone creating an extended oxidation zone. An initial oxide thickness of 870 angstrom units was obtained. Nitrogen was then passed into the exit zone at increasing rates and the oxide thickness at various flow rates was determined. At 500 cubic centimeters per minute nitrogen a thickness of 775 angstrom units was produced. At higher nitrogen flow rates, for example, at 2000 and 4000 cubic centimeters per minute the oxide thickness remained constant at 770 plus or minus 2 angstrom units indicating that the flat portion of curve 50 (FIG. 4) had been reached and that oxygen was no longer penetrating into the exit zone. A nitrogen flow rate of about 700 cubic centimeters per minute was found to provide effective isolation between the exit zone and reaction zone. The experiment clearly demonstrates the ability of the process tube to accurately achieve uniform reaction products and isolation without precise control of gas flow rates.

Although the instant invention has been described only in connection with a thermal oxidation process, many other processes such as diffusions, drive-in, and etching processes may be successfully achieved. In addition, it will also be recognized that the sequential processing may also be achieved by providing different reactive gases through the entrance, reaction and/or exit zones.

While the invention has been particularly shown and described with reference to preferred embodiments thereof, it will be understood by those skilled in the art that various changes in form and details may be made therein without departing from the spirit and scope of the invention.

What is claimed is:

1. Apparatus for continuously effecting mass transport between a gaseous phase reactant and a solid substrate surface comprising:

an elongated process tube divided into at least a first, second and third longitudinal region, said first region including a longitudinal partition dividing said first region into a gas exhaust zone and a substrate inlet zone, said third region also having a longitudinal partition dividing said third region into a gas exhaust zone and a substrate exit zone, said second region being a reaction zone;

means for passing substrates sequentially through said inlet, reaction and exit zones;

means for continuously introducing a gaseous phase reactant into said reaction zone, said gaseous phase reactant being axially exhausted substantially equally through each of said exhaust zones after passing through said reaction zone; and

means for introducing a second gaseous phase material into each of said substrate inlet and substrate exit zones at a pressure in excess of that of said gaseous phase reactant in said reaction zone to provide control over the effective length of said reaction zone.

2. Apparatus for continuously effecting mass transport between a gaseous reactant and solid substrate surfaces comprising:

a process tube having a longitudinal axis a longitudinal first portion of said tube defining a reaction zone;

a longitudinally extending first partition means mounted adjacent to one end of said process zone, said first partition means dividing a longitudinal second portion of said process tube into an upper first gas exhaust zone and a lower substrate entrance zone,

a longitudinally extending second partition means mounted adjacent to the other end of said process zone, said second partition dividing a longitudinal third portion of said process tube into an upper second gas exhaust zone and a lower substrate exit zone;

a gas inlet means in said reaction zone for providing a continuous flow of a first gaseous reactant through said reaction zone and said first and second exhaust zones;

a gas means in said entrance zone for continuously providing a first gas, other than said gaseous reactant, to said entrance zone at a rate sufficient to cause at least some of said first gas to flow into said reaction zone and said first exhaust zone; and

a gas inlet means in said exit zone for continuously providing a second gas, other than said gaseous reactant, to said exit zone at a rate sufficient to cause at least some of said second gas to flow into said reaction zone and said second exhaust zone.

3. The apparatus of claim 2 wherein the linear flow rate of said first gaseous reactant through said reaction and exhaust zones is greater than the diffusion velocity of said first and second gases in said gaseous reactant.

4. The apparatus for claim 2 wherein said first and second gases are inert to said first gaseous reactant.

5. The apparatus of claim 3 wherein the flow rate of said gaseous reactant exceeds the diffusion velocity of said first and second gases by a factor of at least 10 to 1.

6. The apparatus of claim 2 wherein said first gas is a second gaseous reactant.

7. The apparatus of claim 2 wherein said second gas is a second gaseous reactant.

8. The apparatus of claim 2 wherein both said first and second gases comprise a second and third gaseous reactant.

9. The apparatus of claim 2 including heating means to maintain said reaction zone at a predetermined temperature.

10. The apparatus of claim 2 wherein the ends of said partitions adjacent to said reaction zone include means to restrict the flow of said first and second gases between said entrance and exit zones and said reaction zone.

11. The apparatus of claim 9 wherein means are provided at the ends of said partition not adjacent to said reaction zone to restrict the flow of said first and second gases out of said entrance and exit zones.

12. The method of continually effecting mass transport between a gaseous phase reactant and a solid substrate in a process tube including a substrate entrance, reaction, and exit zones, comprising the steps of:

providing a constant flow rate of a first gaseous reactant to said reaction zone;  
providing a constant flow rate of said first gas to said entrance zone;  
passing substrates sequentially through said entrance, reaction, and exit zones;  
providing a flow of a second gas in increasing increments, starting from zero flow rate, to said exit

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zone until the effect of said first gaseous reactant on said substrates remains constant and independent of the flow rate of said second gas; and varying the flow rate of said first gas until the effect of said first gaseous reactant on said substrates remains constant and independent of the flow rate of said first gas.

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