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EP 0731497 A2 **JP 610051409 A** **JP 610040371 A**
JP 400038826 A **US 5492597 A** **US 5413670 A**
US 5228950 A

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(54) Abstract Title

Cleaning semiconductor fabrication equipment using heated NF_3/oxygen mixture

(57) Quartzware used in semiconductor fabrication equipment is cleaned with thermally activated nitrogen trifluoride (NF_3) and a source of oxygen at 300-1000°C. The NF_3 and oxygen are present in a concentration of 1-50% and 1-30% of the etchant mixture respectively. The oxygen source may be oxygen (O_2), ozone (O_3), water (H_2O), nitric oxide (NO), nitrous oxide (N_2O), nitrogen dioxide (NO_2) and/or silicon dioxide (SiO_2). The etchant mixture may include an inert gas eg nitrogen (N_2), argon (Ar) and/or helium (He).

This print incorporates corrections made under Section 117(1) of the Patents Act 1977.

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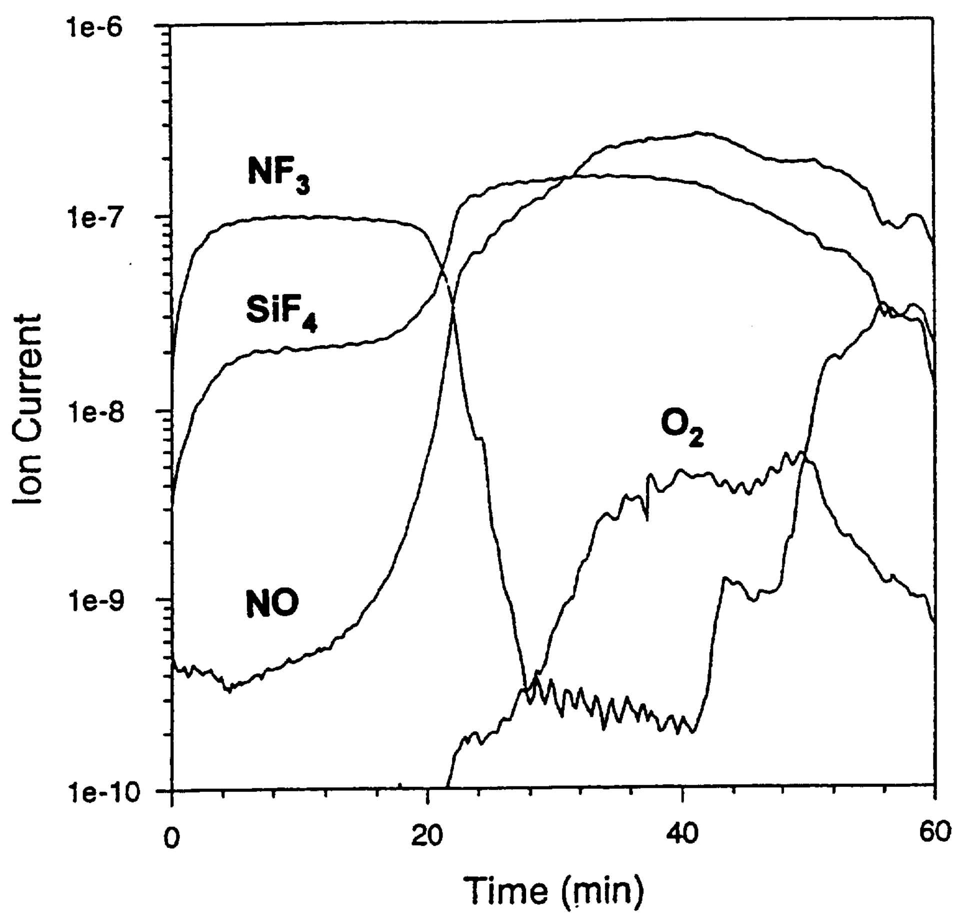

FIG.1

INTERRUPTED ETCH

(600°C, 500 torr (65 kPa), 30%, 3.0 SLM)

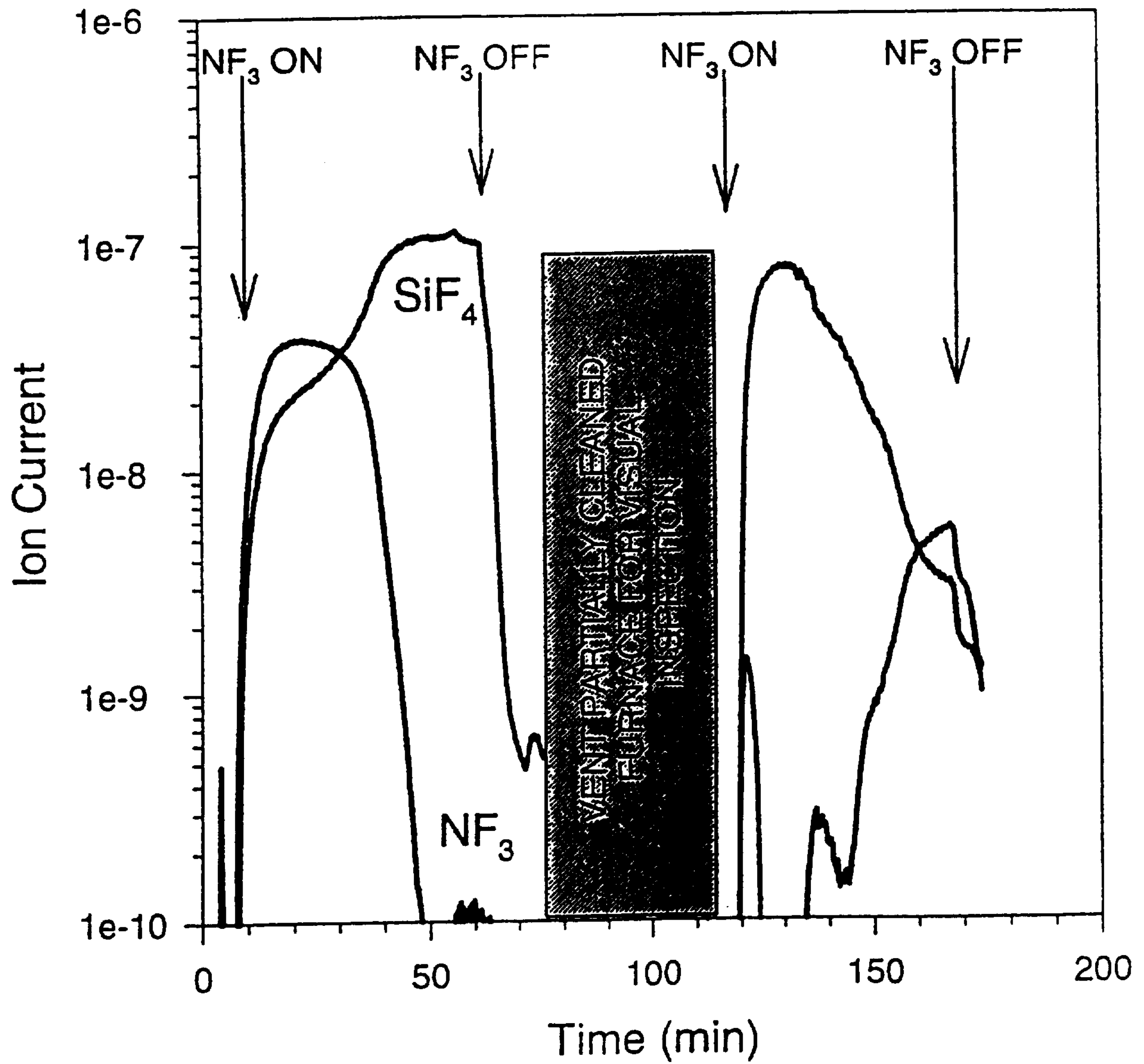

FIG. 2

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NITROGEN TRIFLUORIDE-OXYGEN THERMAL CLEANING PROCESS

The present invention is directed to the field of thermal cleaning with nitrogen trifluoride and an oxygen source etchant mixture. More specifically, the present invention is directed to cleaning various silicon-containing semiconductor substances from wafers and chemical vapor deposition equipment, including reaction vessels and hardware, using a dynamic flow of nitrogen trifluoride and an oxygen source at elevated temperatures with a unique process.

The semiconductor industry has experienced a long-felt need to solve the problem of cleaning chemical vapor deposition furnaces and quartz tubes in the furnaces as well as quartz hardware of various undesired substances such as silicon nitride, polycrystalline silicon, titanium silicide, tungsten silicide and various other silicides, as well as silicon dioxide, which are typically present as undesired films on furnaces and quartz hardware during their deposition on silicon wafers and chips being processed as electronic materials and integrated circuits.

The normal procedure for cleaning is to remove the parts, such as those made of quartz, metals or other materials from a furnace, such as quartz furnace tubes, and perform a wet chemical cleaning. O-ring seals would typically have to be replaced in such an operation, as well as cleaning of lines, doors and other vacuum components. Another alternative is to use another piece of equipment and perform a plasma cleaning. The wet chemical cleaning is very costly and time consuming. When the equipment is shut down to pull the quartz parts, it can require up to 18 hours of time. Also, the system necessitates reverification for its operating integrity before it can be used again. Direct cleaning application requires special chemicals, equipment and sinks to perform this cleaning.

Another disadvantage is that the quartzware is attacked at accelerated rates which affects its reproducibility and reliability of operation. This is especially true when cleaning quartz racks or wafer holders. These components have special slots cut in the quarts to hold the parts being coated, and when the wet chemical clean attacks the quartz, it affects the dimensions of these slots. When the slots are affected, the parts being held are not coated uniformly, which requires the quartz be replaced at accelerated scheduling.

The plasma cleaning method requires the use of another piece of equipment especially designed to perform a cleaning of quartz tubes in place within the chemical vapor deposition furnace equipment. First, the plasma cleaning method does not clean the other quartz parts which are used in the chemical vapor deposition furnace system. This means these parts will need to be chemically wet cleaned, separate and apart from plasma cleaning methods. Also, the plasma cleaning equipment takes up space in the manufacturing area, and may prevent other tubes from being used in the system while it is being utilized. If the quartz parts are replaced with another material like silicon carbide, it will make the plasma system application unreliable.

Methods of using nitrogen trifluoride have been attempted but never have been brought to the marketplace. One such method was performed in a static mode that allowed by-products to condense on the cooler walls of the system. This caused a dangerous condition when these by-products were not evacuated before returning the system to the atmosphere. This called for extended purging times and reduced the benefits of this method. It also allowed for possible dangerous gas mixtures to develop in the system's vacuum components that may mix with gases which are normally utilized to deposit films on the wafers. One

nitrogen trifluoride cleaning method is discussed in UK Patent Appln. GB2183204 where nitrogen trifluoride is used in a static mode and suggestion for use in a continuous flowing mode is also set forth. Said patent application does not address the means or methods for removing cleaning by-products, the treatment of by-products once removed or the use of nitrogen trifluoride with any other gases.

US Patent 5,421,957 discloses a nitrogen trifluoride cleaning process for semiconductor process reactors and apparatus by controlling the moisture content during the clean operation to be less than 10 parts per million by volume. Inert carrier gases for the nitrogen trifluoride are disclosed such as nitrogen, helium or argon. The concentration of the inert was from 95.5% to 80% of the etchant mixture.

U.S. Reissue Patent No. 30,505 discloses a process for plasma etching of a solid material with a binary mixture comprising essentially oxygen and a halocarbon wherein at least one carbon atom in said molecule is linked to a predominance of fluorine atoms. At the temperatures recited (25-300°C) there is no reaction between the binary gas mixture and the solid material to be etched. Temperatures in excess of 1000°C are necessary to thermally dissociate halocarbons, making this gas impractical for thermal cleaning of semiconductor process equipment.

U.S. Patent No. 4,374,698 describes an etch process for differential etching of silicon nitride from silicon dioxide using the combination of carbon tetrafluoride and a halofluorocarbon. The gas etchant may include oxygen or nitrous oxide. A plasma is necessary to dissociate the halocarbon into species that will react with the solid material. The role of the oxygen source in this patent is to volatilize the carbon products into CO and CO_2. Without oxygen, this process would coat the process equipment with

teflon-like material, defeating the usefulness of the process for cleaning.

U.S. Patent No. 4,522,681 discloses a method for
5 etching holes in silicon dioxide wherein a dry plasma etch gas of argon, nitrogen trifluoride and oxygen may be used. Polymeric photoresist materials such as polymethyl methacrylate, ethyl methacrylate, methyl isopropyl ketone as well as copolymers thereof with methacrylic acid may be
10 used. This class of photoresist materials was required, versus standard novel AC photoresists, to successfully practice the invention. Plasma is necessary to dissociate the fluorine compound into specie that will etch the substrate. The role of the oxygen is to etch the
15 photoresist and not the substrate.

U.S. Patent No. 4,568,410 discloses a dry plasma etch process for etching silicon nitride using nitrogen trifluoride and oxygen. Good results for etching silicon
20 nitride were found with relative percentages of the nitrogen trifluoride to oxygen recited at column 5, line 65 to be 10-20 SCCM of NF_3 in comparison to 20-35 SCCM for oxygen. The gases are also disclosed as capable of etching common resists.

25

U.S. Patent No. 4,787,957 is directed to a method for plasma desmear and etchback of epoxy and polyimide materials from a multilayered or double sided printed circuit board using a plasma gas composition in the range
30 of 20-45% NF_3, the remainder being O_2.

The prior art has failed to address a commercially successful process for cleaning semiconductor materials or equipment using a gaseous source to produce volatile
35 cleaning by-products which are readily removed from the materials or equipment after cleaning is accomplished. In addition, the prior art has not addressed a viable method

for removal of cleaning by-products and collection of such by-products (which typically may be reactive and toxic) for safe, easy disposal when servicing chemical vapor deposition equipment and materials treated in such equipment. The prior art has not addressed a method for using nitrogen trifluoride in combination with other gases so as to permit elevated furnace temperatures with controlled etch rates and with minimum down time for etch cleaning. The prior art has not utilized oxygen to enhance the thermal etch of semiconductor materials with nitrogen trifluoride. The present invention as set forth below overcomes these drawbacks of the prior art.

A process has been developed for in-situ thermal cleaning of equipment such as process chambers, boats and/or tool pieces in a semiconductor electronic fabrication facility using nitrogen trifluoride and a source of oxygen at elevated temperature. The present invention achieves rapid initiation of the clean or etch process because of the synergistic action of oxygen with the nitrogen trifluoride. The presence of oxygen, particularly at the initiation of the clean process, provides for rapid initiation of the clean without the delays of the prior art clean processes.

The present invention reduces the cost to clean commonly used quartzware parts and metal roughing lines used in semiconductor and electronic manufacturing equipment by allowing their surfaces to be cleaned in-situ without removal of such surfaces from the process system and without significant reduction in the temperature that the equipment operates at under normal manufacturing operation and by reducing the time required to effect cleaning. This is uniquely possible with the process of the present invention because the nitrogen trifluoride with the added source of oxygen does not require or experience any substantial initiation time before the thermal cleaning

reaction begins at commercially desirable rates. This is a significant benefit because process equipment downtime is critical to economic operation and having to include an extended initiation time is undesirable.

5

The present invention does not require a retrofit that utilizes a radio frequency power supply and means of applying radio frequency to surfaces for activation energy to decompose nitrogen trifluoride to produce cleaning
10 reagent such as required in plasma processes. The present invention also eliminates the use of other equipment used for current methods of cleaning, such as to remove quartzware or roughing lines for cleaning in aqueous acids and deionized water in large, semi-automated ventilated
15 tanks. The present invention eliminates what chemical wastes and possible operator exposure to hazardous acids and solvents used in current cleaning systems.

The present invention is a thermal process for
20 cleaning equipment surfaces in semiconductor manufacturing using diluted nitrogen trifluoride, comprising:

(a) initially evacuating a zone contacting the equipment surfaces;

(b) maintaining the zone at an elevated temperature
25 sufficient to thermally disassociate nitrogen trifluoride; and

(c) flowing an etchant mixture of nitrogen trifluoride and a source of oxygen being through the zone, the source of oxygen present in the range of 1% to 30%, of
30 the etchant mixture by volume, whereby the equipment surfaces are cleaned by chemical reaction of the nitrogen trifluoride and/or fluorine cleaning reagents disassociated from nitrogen trifluoride with undesired substances on said surfaces to form volatile reaction products.

35

Preferably, the elevated temperature of step (b) is in the range of 300 to 1000°C.

More preferably, the elevated temperature of step (b) is in the range of 400 to 600°C.

Most preferably, the flow of the etchant mixture is in
5 the range of 1 to 10 standard liters per minute.

Preferably, the source of oxygen is selected from oxygen, ozone, water, nitric oxide, nitrous oxide, nitrogen dioxide, silicon dioxide and mixtures thereof.
10

Preferably, the etchant mixture includes an inert gas selected from nitrogen, argon, helium and mixtures thereof.

Preferably, the initial evacuation is at a pressure of
15 no greater than 600 torr (80 kPa), the flow of diluted nitrogen trifluoride is at a pressure of no greater than 750 torr (100 kPa) and the heating is at a temperature of no greater than 400-600°C.

20 Preferably, the nitrogen trifluoride is present in the range of 1 to 50% by volume of the etchant mixture.

Preferably, the undesired substances are selected from silicon nitride, siliconoxynitride, silicon dioxide,
25 polysilicon and mixtures thereof.

The present invention involves thermally cleaning a typical process chamber including the parts arrayed within a chamber of a semiconductor or electronic fabrication
30 facility by evacuating the chamber. Nitrogen trifluoride with a source of oxygen, preferably diluted with an inert gas such as nitrogen, argon, helium or mixtures thereof to provide an etchant mixture is then introduced via a gas supply means, such as a gas control manifold, into the
35 process chamber and associated roughing manifold and the cleaning process usually is conducted at a pressure in the range of 400-750 torr (50-100 kPa) using the thermal energy

of 300° to 1000°C, preferably 400° to 600°C, available from the existing equipment heating system to decompose or disassociate the nitrogen trifluoride and produce a fluorine cleaning reagent, such as ionic or free fluorine.
5 The fluorine cleaning reagent produced from the decomposition or disassociation of nitrogen trifluoride under thermal degradation are effective against deposits of silicon nitride, siliconoxynitride, silicon dioxide, polycrystalline silicon, titanium silicide, tungsten
10 silicide, refractory metals and their various silicides. The fluorine cleaning reagent produced from the decomposition or disassociation of nitrogen trifluoride has minimal effect on quartz tube furnaces or quartzware used within such furnaces. The source of oxygen can be oxygen
15 or a source of oxygen such as ozone, water, nitric oxide, nitrous oxide, nitrogen dioxide, silicon dioxide or mixtures thereof. The oxygen source is preferably present in a range of 1% to 30% by volume of the etchant mixture.

20 Upon completion of the cleaning process, the nitrogen trifluoride source is turned off and usually the system pumped under vacuum, preferably with heated and temperature controlled pressurized gas, for 15 minutes followed by a purge with nitrogen (or other inert gas) for at least one-
25 half hour while under vacuum.

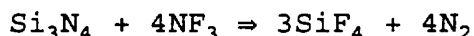
The following is a description by way of example only and with reference to the accompanying drawings of a presently preferred embodiment of the invention. In the
30 drawings:-

FIGURE 1 is a graph of nitrogen trifluoride, silicon tetrafluoride, nitric oxide and oxygen during a nitrogen trifluoride clean not using the process of the present
35 invention. The graph is clean time vs. ion content from the clean zone. The etch was at a pressure of 520 torr (70

kPa), 600°C, 30% nitrogen trifluoride and a flow of 3.0 standard liters per minute.

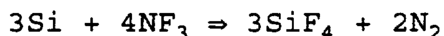
FIGURE 2 is a graph of a nitrogen trifluoride clean under similar conditions of FIGURE 1 but in which the clean was stopped and then restarted. On the restart, no delay in clean was noticed indicating that oxygen accelerated the clean initiation.

This invention enhances the thermal NF_3 process used to clean low pressure chemical vapor deposition (LPCVD) Si_3N_4 and polysilicon tubes. The current (prior art) process uses a NF_3(30%)/N_2 gas mixture at a temperature of 500°C to 600°C and a pressure of 400 to 600 torr (50-80 kPa). The stoichiometry of the overall reaction that removes the Si_3N_4 film is:



$$Si_3N_4 + 4NF_3 \Rightarrow 3SiF_4 + 4N_2$$

and for the polysilicon film is:



$$3Si + 4NF_3 \Rightarrow 3SiF_4 + 2N_2$$

Because NF_3 and SiF_4 are, respectively, etch reactants and products, the Si_3N_4 etch rate is directly proportional to the NF_3 consumption and SiF_4 partial pressure. Figure. 1 shows the gas composition while cleaning 5 μm of Si_3N_4 from an LPCVD tool. The process temperature and pressure are 600°C and 520 torr (70 kPa), respectively, and NF_3(30%)/N_2 is introduced at time zero. The NF_3 partial pressure remains high for about 20 min. after which time it decreases by a factor of 500. Accompanying the dramatic NF_3 drop is a tenfold increase in the SiF_4 concentration. The partial-pressure profile shows that the Si_3N_4 etch rate increases dramatically after 20 minutes, resulting in consumption of all of the NF_3 (i.e., the Si_3N_4 etch rate is now limited by the NF_3 flow). The cause of this etch

increase is a clearing of the Si_3N_4 film at the tube inlet to expose the underlying quartz (SiO_2). Support for this hypothesis is the appearance of NO and O_2 in the profile (Figure. 1) which is evidence of quartz etching.

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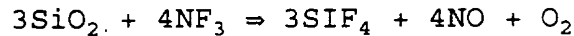


$$3SiO_2 + 4NF_3 \Rightarrow 3SIF_4 + 4NO + O_2$$

In addition to this evidence from the QMS profile, a visual inspection after the initiation step showed the NF_3 clean to be incomplete.

10

Figure. 2 shows the time evolution of the NF_3 and SiF_4 partial pressure while cleaning 5 μm of Si_3N_4. The process temperature and pressure are 600°C and 500 torr (65 kPa), respectively, and NF_3(30%)/N_2 is introduced after 10 minutes. An initiation period lasting about 30 minutes was again observed before all of the NF_3 is consumed and the accompanying tenfold increase in the SiF_4 partial pressure and Si_3N_4 etch rate. At the start of the main etch (40 minutes), the clean is interrupted by turning off the NF_3 gas flow and purging etch products from the system with N_2. The furnace tube was then vented to atmosphere and the wafer boat removed. While the Si_3N_4 film was starting to clear from the top of the boat, most surfaces were still coated with Si_3N_4. The wafer boat was loaded back into the furnace and the clean restarted. The NF_3(30%)/N_2 process gas was reintroduced after 120 minutes. When the clean was restarted, no initiation time was observed. The profile of this partial clean (Figure. 2) shows that when the underlying quartz is exposed and O_2 is added to the process gas, the initiation time is eliminated.

15 20 25 30

These observations suggest that the underlying quartz supplies oxygen to the process gas, increasing the Si_3N_4 etch rate by more than a factor of 10. By deliberately doping the process gas with oxygen, e.g., NF_3(30%)/O_2/N_2, the initiation period can be eliminated and the time to

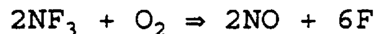
35

clean the LPCVD tube shortened by a factor of two. It is believed that the added oxygen provides additional pathways for the volatilization of Si_3N_4:

5

$$Si_3N_4 + O_2 + NF_3 \Rightarrow SiF_4 + NO_x + N_2$$

or decomposition of NF_3:



$$2NF_3 + O_2 \Rightarrow 2NO + 6F$$

10

The enhanced thermal NF_3/O_2 clean process involves:

1. Cooling the furnace to the cleaning temperature (400°C to 600°C).
2. Isolate LPCVD tube from the process pump with a bypass valve. (15)
3. Fill the LPCVD tube with N_2 (or other inert gas) while pumping with a vacuum source.
4. Introduce the process gas:

20

NF_3(30%)/Oxygen source (1-30%)/N_2
Pressure 400 to 600 torr (50-80 kPa)
Gas flow 1 to 10 SLM

25

5. Flow process gas until all the Si_3N_4 or polysilicon has been removed.
6. Stop flowing process gas.
7. Purge etch products from system with N_2 (or other inert gas).

30

The most significant feature of the present invention is that the process is executed in a dynamic flow mode using nitrogen fluoride and a source of oxygen at approximate process temperatures with only thermal activation. (35)

Typically the nitrogen trifluoride can be thermally activated to degrade and produce the fluorine cleaning reagent at elevated temperatures between 300°C and 1000°C, preferably 400° to 600°C.

5

During the course of chemical vapor deposition of materials in the quartz tubes, deposition occurs on various appliances and carriers within the quartz tubes, as well as on the quartz tube surfaces themselves. At some point in
10 time one or more of the appliances and/or tubes must be taken out of service for cleaning. Although in the present illustration, the cleaning process will be exemplified by all of the quartz tubes being cleaned simultaneously, it is understood by appropriate manifolding, a quartz tube or
15 appliances may be independently or separately cleaned, while other tubes remain in service.

Having described the present invention with regard to a preferred embodiment, it is apparent that the nitrogen
20 trifluoride and oxygen source etchant mixture cleaning process of the present invention provides a number of benefits over the prior art, including: using thermal activation with heat sources typically already in place in quartz tube furnaces; avoidance of requirements for energy
25 sources of plasma systems, the ability to clean system components in place; reduction in maintenance down time for cleaning; lower cost of cleaning and capital investment to effect appropriate cleaning procedures; the elimination of wet chemical cleaning materials and procedures; providing a
30 cleaning method which does not require recalibration of the process chamber or furnace of the semiconductor fabrication facility prior to reinitiation of production runs; provision of a safe method for cleaning by using an isolated evacuation system from the manufacturing process
35 apparatus vacuum system; providing a beneficial method of cleaning system quartzware and boats with minimal damage and handling; providing a method to ensure fast by-product

removal and economic cleaning process cycles, and providing a method which allows for rapid initiation of the clean or etch process at commercially attractive rates. In addition, the cleaning method of the present invention can be used to clean other types of process equipment, including stand alone cleaning equipment, stainless steel door flanges, vacuum conduits, exhaust piping, equipment of stainless steel, aluminum and ceramic.

In addition, the total time required to be off-line from process operation is shorter using the nitrogen trifluoride and source of oxygen etchant mixture of the present invention at elevated or process temperatures, because of the elimination or significant reduction in the lengthy time to cool a furnace and reheat the furnace for safe temperature operation if concentrated or pure nitrogen trifluoride were used and rapid initiation of clean or etch rates without the typical delayed rates of the prior art. Maintaining a furnace at or near steady-state temperatures is also more advantageous for the useful life of an expensive quartz furnace tube, wherein the stress of significant temperature changes is avoided.

The present invention has been described with reference to a preferred embodiment, however, it should be appreciated that the present invention is not limited to said embodiment and that numerous modifications and variations can be made without departing from the scope of the invention as defined in the following claims.

CLAIMS

1. A thermal process for cleaning equipment surfaces in semiconductor manufacturing using nitrogen trifluoride,
5 comprising:

(a) initially evacuating a zone contacting said equipment surfaces;

(b) maintaining said zone at an elevated temperature sufficient to thermally disassociate
10 nitrogen trifluoride; and

(c) flowing an etchant mixture of nitrogen trifluoride and a source of oxygen through said zone, said source of oxygen being in the range of 1% to 30% of the etchant mixture, whereby said equipment
15 surfaces are cleaned by chemical reaction of said nitrogen trifluoride and/or fluorine cleaning reagents disassociated from nitrogen trifluoride with undesired substances on said surfaces to form volatile reaction products.

20

2. A process as claimed in Claim 1, wherein said elevated temperature of step (b) is in the range of 300°C to 1000°C.

25 3. A process as claimed in Claim 2, wherein said elevated temperature of step (b) is in the range of 400°C to 600°C.

4. A process as claimed in any one of the preceding
30 claims, wherein flow of said etchant mixture is in the range of 1 to 10 standard liters per minute.

5. A process as claimed in any one of the preceding claims, wherein said source of oxygen is selected from
35 oxygen, ozone, water, nitric oxide, nitrous oxide, nitrogen dioxide, silicon dioxide and mixtures thereof.

6. A process as claimed in any one of the preceding claims, wherein said etchant mixture includes an inert gas selected from nitrogen, argon, helium and mixtures thereof.

5 7. A process as claimed in any one of the preceding claims, wherein said initial evacuation is at a pressure of no greater than 80 kPa (600 torr), said flow of etchant mixture is at a pressure of no greater than 100 kPa (750 torr) and said heating is at a temperature of 400-600°C.

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8. A process as claimed in any one of the preceding claims, wherein the flow of etchant material is at a pressure of 50-100 kPa (400-750 torr).

15 9. A process as claimed in any one of the preceding claims, wherein said nitrogen trifluoride is present in the range of 1 to 50% by volume of the etchant mixture.

10. A process as claimed in any one of the preceding
20 claims, wherein said undesired substances are selected from silicon nitride, siliconoxynitride, silicon dioxide, polysilicon and mixtures thereof.

11. A process as claimed in any one of the preceding
25 claims, wherein step (a) is conducted by filling the zone with nitrogen while pumping with a vacuum source.

12. A process as claimed in any one of the preceding claims, wherein, after step (c), etch products are purged
30 from the zone with nitrogen.

13. A process as claimed in any one of the preceding claims, wherein said equipment is quartzware.

35 14. A process as claimed in Claim 1 and substantially as hereinbefore described with reference to Figure 2.

15. The use of a gaseous oxygen source to reduce etch initiation of a nitrogen trifluoride etchant mixture used to clean equipment surfaces in semiconductor manufacture.

5 16. The use of a gaseous oxygen source to reduce etch initiation of a nitrogen trifluoride etchant mixture used to clean silicon nitride, siliconoxynitride, silicon dioxide, polysilicon or a mixture of any two or more thereof from the surface of a quartz substrate.

10

17. A use as claimed in Claim 15 or Claim 16, wherein the cleaning is conducted by a process as defined in any one of Claims 1 to 14.

15 18. A use as claimed in Claim 15 or Claim 16 and substantially as hereinbefore described with reference to Figure 2.

The Patent Office

Application No: GB 9800970.7
Claims searched: 1-18

Examiner: Graham Russell
Date of search: 28 April 1998

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.P): B6J (JP, JQD, JQY)

Int Cl (Ed.6): H01L 21/306, 21/311

Other: Online: WPI

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
A	EP 0731497 A2	(AIR PRODUCTS) see column 5 lines 21-26	1,15,16
X	JP 610051409 A	(HITACHI DENSHI) see WPI abstract Acc No: 94-212920/199426	15,16
X	JP 610040371 A	(FUJITSU) see WPI abstract Acc No: 94-203803/199425	15,16
X	JP 400038826 A	(TOSHIBA) see WPI abstract Acc No: 92-153856/199219	15,16
X	US 5492597	(MICRON SEMICONDUCTOR) see column 2 lines 19-21	15,16
X	US 5413670	(AIR PRODUCTS) see column 5 Table 1	15,16
X	US 5228950	(APPLIED MATERIALS) see column 3 lines 30-50	15,16

X Document indicating lack of novelty or inventive step
Y Document indicating lack of inventive step if combined with one or more other documents of same category.
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