

Description

FIELD OF THE INVENTION

This invention relates to a method and an apparatus for evaluating the gas purifying ability of a gas purifier of the Getter type that is suitable for making semiconductor devices.

BACKGROUND OF THE INVENTION

Process gases used for making semiconductor devices require higher purity than gases used for general industry, in order to enhance the reliability of the semiconductor devices. In particular, the purity of the Ar gas generally used in sputtering technology, has a great influence on the reliability of the aluminum wire. Accordingly gas purifiers are generally used for purifying Ar gas. Furthermore, in order to monitor the gas purifying ability of the gas purifiers, a sampling method that extracts Ar gas into a gas container and detects small amounts of impurities by an analysis apparatus such as a gas chromatograph, is traditionally used.

However, as stated above, the sampling method comprising extracting the process gas such as Ar gas into the gas container and detecting impurities such as N_2 , O_2 , and H_2O to evaluate the gas purifying ability of the gas purifier require much time. For example, one day is required for extraction and one week for detection, and great cost is entailed. It is therefore difficult to evaluate the gas purifying ability at periodic intervals.

Accordingly, users cannot decide when the gas purifier has to be used, and have the times for use indicated by the manufacturers of the gas purifiers.

However, gas purifiers which have leaks in the gas lines, will cause the densities of impurities such as N_2 , O_2 , and H_2O to increase. If such purifiers are used continually, then the fatal flaws of face leaks and detachment will be caused because of the entry of impure gas into the semiconductor devices. This has been a problem.

SUMMARY OF THE INVENTION

Accordingly, it is an object of the present invention to provide a simple method and apparatus for evaluating the gas purifying ability of a gas purifier of a process gas such as Ar gas, within a short time.

The foregoing object is accomplished by providing a method of evaluating the gas purifying ability of gas purifiers, comprising the steps of:

sealing a gas purifier while reducing the inner gas pressure thereof;

providing a tank containing gas or gases to be extracted by said gas purifier, said gas or gases

within said tank being at a higher pressure than the inner gas pressure of said gas purifier;

releasing into the gas purifier the gas or gases in said tank; and

measuring the change of pressure in said tank over a given period.

The invention also provides an apparatus for evaluating the gas purifying ability of a gas purifier, comprising:

a sealable gas purifier means;

means for reducing the inner gas pressure in said gas purifier means;

a tank storing gas or gases to be extracted by said gas purifier, said gas or gases in the tank being at a higher gas pressure than the inner gas pressure of the gas purifier;

a valve means located between the gas purifier means and said tank for connecting the one to the other; and

a measuring means for measuring the change of pressure in said tank over a given period.

In the invention, when the gas purifying ability of the gas purifier decreases, the gas releasing ability of the impure gas from the tank having a higher inner gas pressure than the gas purifier, to the gas purifier, also decreases in accordance with decreasing gas purifying ability. Accordingly, by measuring the gas releasing ability, namely by measuring changes in the inner gas pressure of the tank, the gas purifying ability of the gas purifier is evaluated.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention is illustrated diagrammatically in the following drawings wherein:

Figure 1 is an entire flow diagram of a vacuum apparatus using a carrier gas;

Figure 2 is a graph showing the relationship between the degree of vacuum of an auxiliary tank and the release time of the auxiliary tank;

Figure 3 is a graph showing the relationship between the release time of the auxiliary tank and the working time of a gas purifier;

Figure 4 is a graph showing the relationship between the impurity density and the release time of the auxiliary tank.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring to the embodiment of the invention in the drawings. Fig. 1 shows a schematic view of an entire gas use apparatus. In Fig. 1, a process gas, such as Ar gas which is fed from a gas line 1 such as a gas container flows along a main pipe 2, and is purified through a gas purifier 3, and is then introduced into a process chamber 4 such as a

sputtering apparatus. A valve 5 is mounted between the gas line 1 of said main pipe 2 and the gas purifier 3, and a valve 6 and a gas flow regulator 7 are mounted between the gas purifier 3 and the process chamber 4 respectively. A release pipe 8 of the process chamber 4 is connected via a valve 9 to a vacuum pump 10. The vacuum pump 10 is operated when the process gas such as Ar gas is introduced into the process chamber, the valves 5, 6 and MFC7 of the main pipe 2 and the valve 9 of the release pipe 8 are open. Therefore, the gas which is purified through the gas purifier 3 from the gas line 1, is introduced to the process chamber 4, wherein the gas is used for forming a semiconductor member. The used gas is discharged from the process chamber 4.

An auxiliary pipe 11 branches from the main pipe 2 on the outlet side of said gas purifier 3, and an auxiliary tank 12 is mounted on the auxiliary pipe 11. An auxiliary pump 13 is connected to the outlet side of the auxiliary tank 12, and a vacuum gauge 14 such as an ionization vacuum gauge, is mounted at the leading edge of the auxiliary pipe 11, and a valve 15 and a vacuum gauge 16 such as a Pirani gauge are mounted respectively, in series between the leading edge and the auxiliary tank 12. In addition, a valve 17 is mounted between said auxiliary pump 13 and the auxiliary pipe 11, and a valve 18 is mounted at the point farthest from the connection of the auxiliary pipe 11 and the auxiliary pump 13.

The operation for monitoring the purifying ability of said gas purifier 3 is as follows.

First, the valves 5 and 6 are closed (in this case, valve 9 may be either open or closed), and the valves 15 and 17 of the auxiliary pipe 11 are opened, and by operating the auxiliary pump 13 the gas is discharged from the gas purifier 3, the auxiliary tank 12 and the pipe system therebetween. In this case, the degree of vacuum inside them is about $1 \times 10^{-1} \sim 1 \times 10^{-3}$ Pa and is measured by the vacuum gauge 14.

Secondly, the valves 15 and 17 are closed, and the valve 18 is opened from where N_2 gas is introduced into the auxiliary tank 12. Therefore, the gas pressure within the auxiliary tank 12 becomes about 1 ~ 100 Pa which is higher than that within the gas purifier 3 and is measured by the vacuum gauge 16.

Thirdly, the valve 18 is closed and the N_2 gas is shut within the auxiliary tank 12. Then by opening the valve 15 and releasing N_2 gas into the gas purifier 3, the released N_2 gas is removed (the purifying process) by the gas purifier 3.

In this operation, the N_2 gas flows from the auxiliary tank 12 to the gas purifier 3, as a result the gas pressure of the auxiliary tank 12 decreases, as shown in line (a) in Fig. 2. The N_2 gas

volume (gas releasing ability) which flows from the auxiliary tank 12 to the gas purifier 3 is in accordance with the gas purifying ability of the gas purifier 3. That is to say, when the gas purifying ability of the gas purifier lowers, the gas purifier 3 is not able to remove the N_2 gas introduced from the auxiliary tank 12 to the gas purifier 3, and accordingly, the ability to release gas from the tank 12 to the gas purifier 3 decreases as shown by line (2) in Fig. 2. In Fig. 2, the time between the point where line (a) and 1×10^{-1} Pa intersect and the point where line (b) and 1×10^{-1} Pa intersect, is about one hour.

Also, the results in Fig. 3 are acquired by monitoring the gas purifying ability of the gas purifier 3 for a long time with said monitoring apparatus. That is to say, in Fig. 3 it is understood the gas purifying ability is normal in the initial stage, but then deteriorates with extended use.

Fig. 4 shows the relationship between the time represented logarithmically for releasing until the gas pressure is 1×10^{-1} Pa and the gas analysis value obtained by sampling the impurities within the Ar gas which initially includes 10 ppm impurities and is then purified by the gas purifier 3. In Fig. 4, the gas purifying ability has a good relationship with the releasing ability.

In the gas purifier 3, in general by heating an alloy consisting of zirconium (Zr), iron (Fe), and vanadium (V) at about 400°C , impurity gases such as N_2 , O_2 , H_2O , NO, CO, and CH_4 react to be incorporated into the alloy, thus accomplishing the gas purifying process. Accordingly for measuring the gas releasing ability, gases except N_2 gas may be introduced into the gas purifier 3.

In above mentioned embodiment, the auxiliary tank 12 and the auxiliary pump 13 is used but alternatively, the process chamber 4 of the sputtering apparatus may serve as a storage tank for the impurity gas, and connected to a pipe for introducing the impurity gas such as N_2 gas, so that the impurity gas such as N_2 gas is introduced into the process chamber 4 (tank) by the suction operation of the vacuum pump 10. Additionally, when the auxiliary tank 12 is much smaller than the gas purifier 3, for example about

$$\frac{1}{1000}$$

to

$$\frac{1}{10000},$$

it is possible to shut the air into the auxiliary tank, at atmospheric pressure. In this case, when the valve 15 is opened, the pressure within the gas

purifier 3 is about 10 to 1000 Pa.

Claims

1. A method of evaluating the gas purifying ability of gas purifiers, characterized by comprising the steps of:
 - sealing a gas purifier (3) while reducing the inner gas pressure thereof;
 - providing a tank (12) containing gas or gases to be extracted by said gas purifier, said gas or gases within said tank being at a higher pressure than the inner gas pressure of said gas purifier;
 - releasing into the gas purifier (3) the gas or gases in said tank (12); and
 - measuring the change of pressure in said tank (12) over a given period.
2. The method of Claim 1 wherein the gas to be extracted by said purifier (3) is N₂ gas.
3. An apparatus for evaluating the gas purifying ability of a gas purifier, characterized by comprising:
 - a sealable gas purifier means (3);
 - means for reducing the inner gas pressure in said gas purifier means (3);
 - a tank (12) storing gas or gases to be extracted by said gas purifier, said gas or gases in the tank (12) being at a higher gas pressure than the inner gas pressure of the gas purifier (3);
 - a valve means (15) located between the gas purifier means (3) and said tank (12) for connecting the one to the other; and
 - a measuring means (16) for measuring the change of pressure in said tank (12) over a given period.
4. The apparatus of Claim 3, wherein the gas purifier means (3) has an alloy to incorporate the gas or gases to be extracted by said gas purifier means.

Patentansprüche

1. Verfahrens zum Bewerten der Gasreinigungsfähigkeit von Gasreinigern, dadurch gekennzeichnet, daß es die Schritte umfaßt:
 - Abdichten eines Gasreinigers (3), während der innere Gasdruck desselben verringert wird;
 - Bereitstellen eines Tanks (12), der ein Gas oder Gase enthält, die mittels des besagten Gasreinigers abgezogen werden sollen, wobei das besagte Gas oder die besagten Gase innerhalb des besagten Tanks einen höheren Druck aufweisen, als der innere Gasdruck des

besagten Gasreinigers;

Freigeben des Gases oder der Gase in dem besagten Tank (12) in dem Gasreiniger (3); und

Messen der Druckänderung in dem besagten Tank (12) über einen vorgegebenen Zeitraum.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das von dem besagten Reiniger (3) abzuziehende Gas N₂-Gas ist.
3. Vorrichtung zum Bewerten der Gasreinigungsfähigkeit eines Gasreinigers, dadurch gekennzeichnet, daß sie umfaßt:
 - eine abdichtbare Gasreinigervorrichtung (3);
 - Mittel zum Verringern des inneren Gasdrucks in der besagten Gasreinigervorrichtung (3);
 - einen Tank (12), in welchem ein Gas oder Gase gelagert sind, die mittels des besagten Gasreinigers abgezogen werden sollen, wobei das besagte Gas oder die besagten Gase in dem Tank (12) einen höheren Gasdruck aufweisen, als der innere Gasdruck des Gasreinigers (3);
 - eine zwischen der Gasreinigervorrichtung (3) und dem besagten Tank (12) angeordnete Ventilvorrichtung (15), um die eine mit dem anderen zu verbinden; und
 - eine Meßvorrichtung (16) zum Messen der Druckänderung in dem besagten Tank (12) über einen vorgegebenen Zeitraum.
4. Vorrichtung nach Anspruch 3, dadurch gekennzeichnet, daß die Gasreinigervorrichtung (3) eine Legierung besitzt, um das Gas oder die Gase aufzunehmen, die mittels der besagten Gasreinigervorrichtung abgezogen werden sollen.

Revendications

1. Procédé d'évaluation de la capacité de purification de gaz de purificateurs de gaz, caractérisé en ce qu'il comprend les étapes consistant à:
 - sceller un purificateur de gaz (3) tout en réduisant sa pression de gaz interne;
 - fournir une cuve (12) contenant un ou plusieurs gaz à extraire par ledit purificateur de gaz, le(s)dit(s) gaz dans ladite cuve étant à une pression supérieure à la pression de gaz interne dudit purificateur de gaz;
 - libérer dans le purificateur de gaz (3) le(s) gaz dans ladite cuve (12); et
 - mesurer la modification de pression dans



purifier 3 is about 10 to 1000 Pa.

Claims

1. A method of evaluating the gas purifying ability of gas purifiers, characterized by comprising the steps of:
 - sealing a gas purifier (3) while reducing the inner gas pressure thereof;
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besagten Gasreinigers;

Freigeben des Gases oder der Gase in dem besagten Tank (12) in dem Gasreiniger (3); und

Messen der Druckänderung in dem besagten Tank (12) über einen vorgegebenen Zeitraum.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das von dem besagten Reiniger (3) abzuziehende Gas N₂-Gas ist.
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 - eine abdichtbare Gasreinigervorrichtung (3);
 - Mittel zum Verringern des inneren Gasdrucks in der besagten Gasreinigervorrichtung (3);
 - einen Tank (12), in welchem ein Gas oder Gase gelagert sind, die mittels des besagten Gasreinigers abgezogen werden sollen, wobei das besagte Gas oder die besagten Gase in dem Tank (12) einen höheren Gasdruck aufweisen, als der innere Gasdruck des Gasreinigers (3);
 - eine zwischen der Gasreinigervorrichtung (3) und dem besagten Tank (12) angeordnete Ventilvorrichtung (15), um die eine mit dem anderen zu verbinden; und
 - eine Meßvorrichtung (16) zum Messen der Druckänderung in dem besagten Tank (12) über einen vorgegebenen Zeitraum.
4. Vorrichtung nach Anspruch 3, dadurch gekennzeichnet, daß die Gasreinigervorrichtung (3) eine Legierung besitzt, um das Gas oder die Gase aufzunehmen, die mittels der besagten Gasreinigervorrichtung abgezogen werden sollen.

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ladite cuve (12) sur une période donnée.

2. Procédé de la revendication 1 dans lequel le gaz à extraire par ledit purificateur (3) est le gaz N₂. 5
3. Appareil pour évaluer la capacité de purification de gaz d'un purificateur de gaz, caractérisé en ce qu'il comprend:
 - un organe purificateur de gaz scellable (3); 10
 - un moyen pour réduire la pression interne de gaz dans ledit organe purificateur de gaz (3);
 - une cuve (12) emmagasinant le(s) gaz à extraire par ledit purificateur de gaz, le(s)dit(s) gaz dans la cuve (12) étant à une pression de gaz supérieure à la pression de gaz interne du purificateur de gaz (3); 15
 - un organe de type soupape (15) situé entre l'organe purificateur de gaz (3) et ladite cuve (12) pour relier l'un à l'autre; et 20
 - un organe de mesure (16) pour mesurer la modification de pression dans ladite cuve (12) sur une période donnée. 25
4. Appareil de la revendication 3, dans lequel l'organe purificateur de gaz (3) comporte un alliage pour incorporer le(s) gaz à extraire par ledit organe purificateur de gaz. 30

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