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ION ASSISTED ETCHING: INFLUENCE OF THE ION CHARGE SIGN

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The relative influence of the ions of different signs on the ion-assisted etching of silicon is under investigation. The fluorine radicals are produced by the direct current glow discharge with pressure gradient. The beam of positive or negative ions is produced by PIG source. The flows of radicals and ion beam are met on the surface of the silicon placed in high vacuum. The positive ions can be converted into the fast neutral atoms by method of resonance exchanging in an own gas. It is shown that the fast neutral atoms have the highest stimulating ability. The catalytic influence of the positive ions is in two times less. The negative ions have the intermediate parameters. It is found that some kinds of ions (for example, molecular oxygen) decelerate the etching that is they behave itself like an inhibitor.

Introduction

The ion assisted plasma etching was discovered more than 20 years ago [1, 2], and widely employed in microelectronics [3]. The essence of the phenomenon is the following. The physical sputtering occurs when enough energetic ions bombard the surface of the silicon. A sputtering coefficient is on the level of ~ 1 for the energy ranges 500 - 1000 eV. This process is called in microelectronic "sputtering" or "ion etching" and characterized by the etch rate $V_{ph} = dh/dt$, where h is a silicon plate thickness. Another extreme situation is a chemical etching. This comes about when spontaneous reaction between neutral species and substrate material forms volatile gaseous reaction products. A well-known example is silicon etching by fluorine atoms $Si + 4F \rightarrow SiF_4$. The product of the reaction is a gas and leaves the surface spontaneously. In this case the plasma is used just for the radical production from the initial stable and safe species (CF_4, SF_6 and so on). The etching goes on with some rate V_{ch}. It depends on a halogen radical concentration and a surface temperature. Now, if we subject a surface of a silicon by a simultaneous influence of the ion and radical fluxes, the etch rate V_i can exceed the sum ($V_{ph} + V_{ch}$) by a few times. The process has clear synergetic character and, consequently, general scientific worth [4, 5]. In most gas-solid combination which react to form volatile products, the etch rate is increased when energetic ion bombardment is superimposed on the reactive neutral gas flux. Nevertheless we concentrate our attention on the Si-F system, since it is most widely used and so most carefully studied.

Plasma etching can be viewed as a several sequence of steps:

1.Etchant formation: $e + CF_4 \rightarrow e + CF_3 + F$ or $e + SiF_6 \rightarrow e + SiF_5 + F$.

2. Etchant transport to the surface.
3. Adsorption/chemisorption on the surface.
4. Chemical reaction.
5. Desorption product molecule.

Items 1, 2 influence the reaction dynamics indirectly via the etchant composition. So the most of authors consider items 3, 4, 5 only. Now we consider the probable consequences of the ion bombardment for these processes. For item 3 it can cause: a) an increase of nondissociative chemisorption by means of distraction Si-Si bonds in a silicon lattice; b) dissociative adsorption of the complex radicals on a surface (such as CF_3 or SF_5). In item 4 the ion beam can increase an etch rate due to two effects: a) formation the dangling Si bonds; b) direct mechanical penetration of the reactive species in a silicon bulk. In item 5 the ions can cause compulsory desorption of: a) an intermediate product of the reaction (such as volatile SiF_2); b) involatile products.

Arguments have been presented supporting each of these mechanisms. The problem is that the results of each certain series of experiment supporting one of a mechanism rejected another. For example, authors of articles [4, 6] attributed the etch rate acceleration to the dissociative

chemisorption. Moreover they presented the experimental results calling in question the positive influence of Si-Si bond destruction. This is in a contradiction with the results of [7-9] where a surface ripping is considered as a main effect. In 1978 Mauer showed that the ion bombardment could cause a removing from a surface the intermediate products of the reaction, such as volatile SiF_2 [10]. This mechanism is very popular now. Moreover, the ability of the ion-solid collision cascade to rearrange bonded fluorine has been demonstrated in the chemically assisted ion beam etching of Si at low temperatures using condensed SF_6 as the fluorine source [11].

Thus, we haven't now successfully understood of ion assisted etching mechanism, although the investigation has been carried out more than 20 years and had an overall character. So the situation requires new conceptions and methods of the research. The attempt to realize one of them and first results are presented below.

Problem statement and experimental system

We put attention on a following fact. As it is known, the silicon etching can be accelerated by energetic particle bombardment – by ions and electrons. The difference is that the ions accelerate the process at any cases, but the electrons in some only. For example, an electron beam with a current density of 2 - 4 mA/cm^2 and energy of a few keV accelerates an etching process in 4 - 6 times for plasma-forming gases such as CF_4 or CF_3Cl. This isn't observed for XeF_2 or SF_6 [6, 12]. It gives a reason to investigate an influence of an ion charge sign on an etching assistance. Compression of the positive and negative ion ability is also interesting due the proposals to use the negative ions for advanced plasma processing of semiconductors [13].

Finally, if we compare the negative and positive ions, it seems to be reasonable to involve fast neutral atoms in investigation, which can be considered as an ion with a charge equal to zero.

So, we need the fluxes of a positive and negative ions and also fast neutral atoms of one and the same chemical element with a comparable intensity. The fluorine isn't suitable for these purposes. The cold cathode PIG provides mainly negative ions F^-. The quantity of positive ions is negligible. The oxygen is free from this disadvantage. One the other hand, the chemical properties of oxygen weakly influence on the etching process, if it is used in combination with SF_6 and its partial pressure doesn't exceed 50 %.

Positive ions and fast neutral atoms of argon were used as examinant elements.

The system described in [4], was taken as a prototype. The idea was to take two independent sources, one of which produces an ion beam and second a flux of chemical active species. Two fluxes are met on a surface of silicon located in a high vacuum. In such situation practically all parameters of a system are independent. We have carried out experiments in a high vacuum chamber pumped up to background pressure 2 · 10^{-3} Pa. The experimental arrangement allocated inside is shown schematically in Fig. 1.

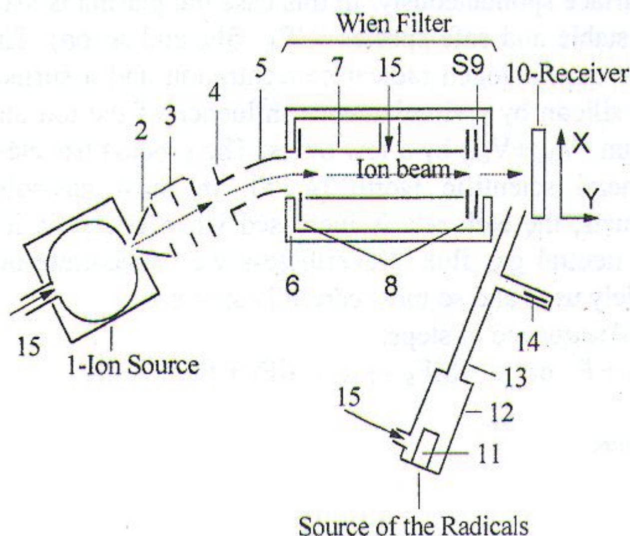

Fig. 1. Scheme of the experimental arrangement: *1* - ion source; *2* – extractor; *3* - bipotential lens; *4* - decelerating electrode; *5* - electrostatic bending magnet; *6, 7* - magnetic yoke and electrostatic capacitor of a Wien filter; *8* - entrance and output slots of the filter; *S9* - auxiliary electrode; *10* – receiver; *11* – cathode; *12* - quartz vessel; *13* - glass tube; *14* - auxiliary anode; *15* - gas inlet; *i* - trajectory of the ion beam.

For the ion beam production we use the cold cathode PIG similar to [14]. The body of a source is hermetically sealed and is coupled with a high vacuum chamber through a hole (for ion extraction) of 1.5 mm diameter only. Such design allows keeping a pressure in a source 2 - 10 Pa when a pressure in a vacuum chamber is less than 10^{-2} Pa. A magnetic field in a source of ~ 0.07 T was provided by permanent magnets. The source has such parameters: diameter of a cathode is 2 cm; length of an anode is 2.2 cm; discharge voltage is 600 - 750 V; discharge current is 60 - 120 mA. Argon, oxygen and sometime CF_4 are used as a plasma forming gas. The ions are extracted across a magnetic field. A voltage on an extractor is 2.5 kV. Beam is focused by bipotential lens and then decelerated up to 1 keV. The ion beam is deflected on an angle ~ 12° by cylindrical electrostatic capacitor and then get in the Wien filter.

Flying about 2 cm after Wien filter, the band ion beam get on a grounded receiver (Fig. 2). Upper part of a beam is used for a silicon treatment. Lower part is used for express control of beam parameters. The latter is carried out through a hole of 2.5 mm diameter. The system is equipped by auxiliary electrode ES that can either suppress or extract all secondary electrons from a collector in dependence of applied potential.

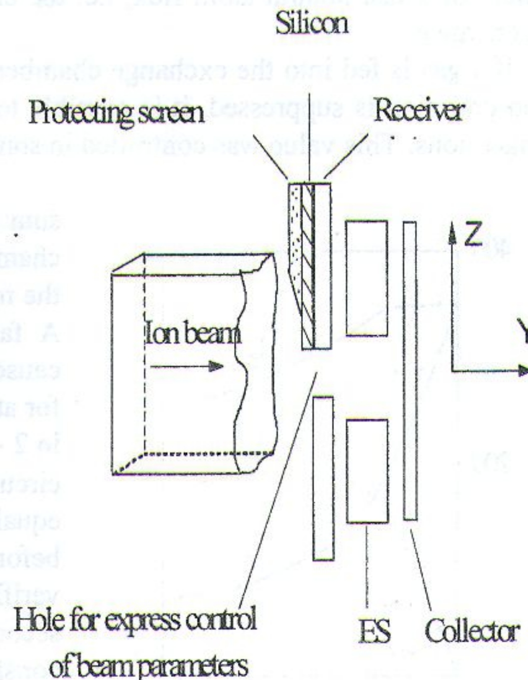

Fig. 2. Scheme of the receiver unit. ES - electrode for extracting or suppression of secondary electron emission. (Axis X-Y in Fig. 1 and Z-Y in Fig. 2 are the components of one and the same left-handed coordinate system).

The Wien filter separates a beam either by mass (O_2^+ and O^+; O_2^- and O^-) or by charge (Ar^+ and Ar^{2+}). Experiments show that the PIG produces comparable amount of positive and negative ions both atomic and molecular oxygen. The argon ion beam almost whole consists of Ar^+. Experiments with CF_4 shows that out ion source produces the positive ions F^+ in a negligible amount. That doesn't allow to make a full investigation of fluorine. Nevertheless some results concerning F^- are presented below.

Fast neutral atoms Ar^o, O_2^o, O^o (energy is 1 keV) are produced from the positive ions by method of a resonant gas exchanging (Ar^+ in Ar; O_2^+ in O_2; O^+ in O_2). For this purposes the side walls of the electrostatic capacitor plates in a Wien filter are hermetically sealed and formed a rectangular tube. The middle part of a tube has the holes for gas inlet and pressure measuring. So we get an exchange chamber with a differential pumping. A gas pressure in a middle part of a tube is ~ 0.4 Pa when a pressure in a high vacuum cap is less than 10^{-2} Pa.

The charge exchange coefficient is measured in accordance with method described in [15, 16]. It is based on an assumption that the ions and fast neutral atoms knock out secondary electron from a metal with an equal efficiency, i.e. for the secondary electron emission coefficients we have $\kappa_{i-e} = \kappa_{n-e}$. Most probably this is true for an energy region, where the kinetic knocking out exceeds the potential one.

The procedure of measuring consists in the following. Initially the gas inlet in exchange chamber is turned off, auxiliary electrode S9 has ground potential, and the beam losses are negligible. The Wien filter is tuned on some certain kind of ion, for example Ar^+. Changing the polarity of a potential on the electrode ES one can either suppress secondary electron emission or extract all secondary electrons from a collector. In a first case we measure the ion beam current I_i. In

second case a current on a collector is $I_k = I_i + I_e$ (I_e – current of a secondary electrons). $\kappa_{i\text{-}e} = I_e / I_i$ = $= (I_k - I_i)/I_i$. It was checked regularly in a certain time interval during each experiment.

Then the correspondent gas is fed into an exchange chamber. Part of ions in a beam will be exchanged but a part will leave a chamber in the invariable form. Now, if one applies an enough large positive potential on an electrode S9, all non-exchanged ions will be reflected and can't leave a Wien filter. It is observed when a voltage U_{S9} exceeds 1100 V (it was verified by collector current measurement when a secondary electrons was suppressed and a gas in exchange chamber wasn't fed yet). Now we change the polarity of the potential on electrode ES and extract all secondary electrons. Since ions don't reach a collector the measured current I_e can be caused by fast neutral atoms only. I accordance with [15, 16] we assume $I_n = I_e / \kappa_{i\text{-}e}$. Here and then I_n is an electric equivalent of a fast neutral atom flux, i.e. the current which will flow if each neutral atom would have a charge e.

If a gas is fed into the exchange chamber, electrode S9 has a grounded potential, secondary electron emission is suppressed, it is possible to measure one more parameter I_{ir} - current of non-exchanged ions. This value was controlled in some auxiliary experiments only.

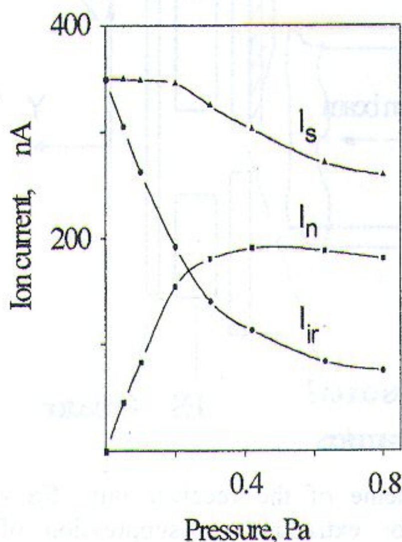

Fig. 3. Dependencies of I_n (an electric equivalent of a fast neutral atom flux), I_{ir} (current of non-exchanged ions) and their sum I_s on a pressure in a middle part of an exchange chamber.

Fig. 3 shows the dependencies of I_n, I_{ir} and their sum I_s on a pressure in a middle part of an exchange chamber. Working gas is argon. We see, that I_n achieves the maximum for P ~ 0.5 Pa and then decreases slowly. A fast atom scattering on the exchanging gas atoms causes the latter. The same dependencies are observed for atomic and molecular oxygen but for pressure higher in 2 - 3 times. Now we draw attention on two important circumstances. In a region $P \leq 0.2$ Pa I_s is practically equal to the initial ion beam current (i.e. a current was before a gas is fed into an exchange chamber). This verifies the validity of measurement procedure. The second- if (nevertheless) we assume an existence of considerable error, the real fast atom flux can't exceed a value we declared, since a sum of I_n and I_{ir} can't be large than initial current of a beam.

A dc glow discharge in gas flow with a strong pressure gradient (see Fig. 1) is employed as a source of chemically active species. Aluminum cathode (11) of 2 cm^2 area is positioned inside of a quartz vessel (12) of ~ 10 cm^3 volume. The vessel is coupled with a high vacuum chamber through a glass tube (13) of diameter 8 mm and length 22 cm. The side offset of a tube is in 6 cm from its end. It contains an auxiliary anode. It makes easier to initiate a discharge with a strong pressure gradient. For a standard gas flow 900 cm^3·Pa/s a pressure near a cathode (11) is 25 - 30 Pa and a pressure in a high vacuum chamber is ~ $7 \cdot 10^{-3}$ Pa. Plasma forming gas is SF_6. It is fed into a vessel (12). As a rule we use the following regime of a glow: a voltage is 2.5 - 3 kV, a current I_{gl} is 4 mA. The latter is distributed in approximately equal parts between an auxiliary anode and a grounded surface of a high vacuum chamber.

The atoms of fluorine are produced by dissociation of SF_6 in the discharge positive column. The measurements show that an etch rate of a silicon and a distance from a glass tube end R are connected by a relationship $V_{et} \sim 1/R^2$. An angle between the axis of a radical source and plate of receiver (as well as a silicon surface) is ~ 20°. A distance between the end of a glass tube and a middle of an ion beam (see Fig. 1) is 2 cm (see section 4). The permanent magnet is put on a receiver. The field has an induction on a magnet surface ~ $7 \cdot 10^{-3}$ T and is directed alone the axis Y

in Fig. 1. It prevents a penetration of electrons from a glow discharge in a zone where the beam and fluorine radicals interact with a silicon surface.

Method and result of measurements

An etch rate is determined by means of direct measurements of an etch depth. The silicon sample is put on a receiver plate (Fig. 2). Its upper part is protected from an influence of ion beam and fluorine radicals by screen. The height of a step (along the axis Y on Fig. 1, 2) between protected and processed parts of a surface is measured by interference microscope. As it is mentioned before, the space distribution of a radical etch rate is given by formula $V_{et} \sim 1/R^2$. So the profile of etching alone the axis X (Fig. 1) (ion beam off) has a shape shown in Fig. 4, *a*. The start point of an axis X is set on the end of a glass tube. When the ion beam is on, an additional step appears on an etching profile (Fig. 4, *b*) in a region of joint influence of the radicals and ions on a silicon. Sections (d) and (e) can be readily extrapolated and we get a value Δh. Distance R was chosen equal to 2 cm because of the following reason. It is known [17] that the atom yield of silicon per one incident ion depends on the energetic species to neutral flux ratio. When this ratio is enough low, the yield is in a neutral-saturated regime, and the yield attains a maximum. Varying the distance R, the current of dc glow and a flux of SF_6 we found that this regime is realized for any investigated kind of ion when $R \leq 2$ cm, $I_{gl} \geq 4$ mA, $Q(SF_6) \geq 900$ cm^{3}Pa/s.

The atoms yield of silicon per one incident ion (main measurable quantity) is determined in a following way. Let we have etched a silicon sample on a depth Δh during time Δt. A number of atoms removed from a unit of area is $N_{si} = \Delta h \rho N_a/\mu$, where ρ and μ are the density and the atomic weight of silicon, N_a is Avogadro number. A number of ions incident on a unit of area is $N_i = j\,\Delta t/e = I\Delta t\,/eS$, where j is an ion beam density, I and S are the current on the collector and its area, e is an electron charge. The previously described I_n plays a part of I in above-mentioned formula, when we deal with a fast neutral atoms. So we have $Y = \Delta h \rho N_a eS/\mu\, I\Delta t$. Run of measurements was done for each kind of stimulating particle. Averaged values are presented below. It is known [8] that an atom yield for silicon depends on the energy and mass of incident ion as well as of experimental conditions. Usually this value changes from 4 to 20 atom/ion [4, 17] for Ar^+ with energy of 1 keV. We have obtained $Y(Ar^+) = 5.7 \pm 1.3$ atom/ion and afterwards use it as a reference value.

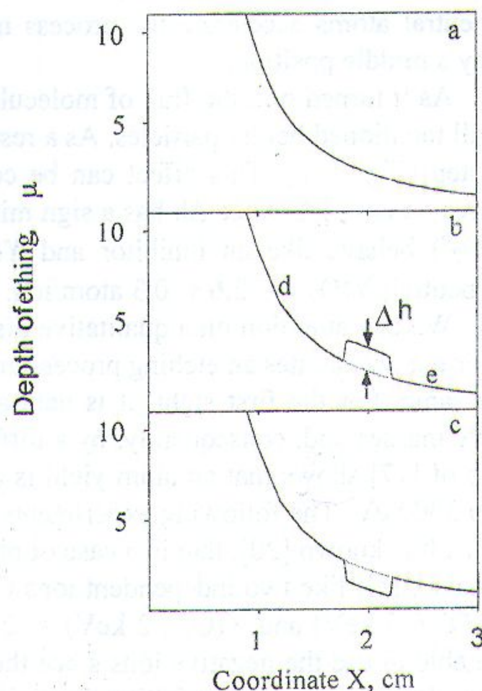

Fig. 4. Qualitative dependencies of an etching depth on the X coordinate (see Fig. 1, point X = 0 is set on the end of a glass tube). *a* – the radical source is on, the ion beam is off. *b* – both sources are on. *c* – the etch profile for the case when the process is locally decelerated somehow.

For the fast neutral atoms of argon we have obtained $Y(Ar^0) = 10.6 \pm 1.5$ atom/neutral, i.e. $Y(Ar^0)/\,Y(Ar^+) \cong 1.85$. The result is unexpected because of two reasons. As it was mentioned in the introduction, the stimulating influence of an ion bombardment is attributed now mainly to the surface ripping, distraction of Si-Si bonds and forced desorption of a intermediate product of the reaction SiF_x (x = 1, 2, 3). Existence of these effects and its contribution to the process doesn't call

a doubt. However this is an exactly mechanical influence and shouldn't depend on a state of outermost electron shell (energy of ion is 1 keV but energy of all transitions in outermost electron shell is less than twenty eV). It makes us to assume an existence of a reaction branch, which isn't taken into account before. Secondly, when an ion approaches a surface, it becomes neutralized usually through an Auger or resonant tunneling process [18, 19]. This generally happens before the ion interacts strongly with the hard potential wall of the atoms in a solid. So at practically all cases the surface of silicon is subjected by influence of fast neutral atoms. The difference is the place, the time and the mechanism of neutralization. It makes us to doubt in a method of I_n measurement. However, as it was emphasized in a previous section, the fast atom flux can't exceed a value we declared, since it should contradict to the current balance in a system. But $Y \sim 1/j$ and, consequently, the presented value of $Y(Ar^0)$ is actually its lower limit. Thus, we can claim that the stimulating ability of an ion depends on a population of its outermost electron shell as well as of its mass and energy.

Experiments show that all kinds of atomic oxygen accelerate the etching process: $Y(O^+) = 1.9 \pm 0.2$ atom/ion; $Y(O^o) = 3.4 \pm 0.5$ atom/neutral; $Y(O^-) = 2.1 \pm 0.5$ atom/ion. Again, the fast neutral atoms accelerate the process most strongly and $Y(O^o)/Y(O^+) \cong 1.8$. Negative ions occupy a middle position.

As it turned out, the flux of molecular oxygen ions decelerate an etching process in contrast with all mentioned before particles. As a result the fall-through appears on an etching profile instead of a step (Fig. 4, *c*). This effect can be considered as a negative catalysis and characterized by negative values of Y since Δh has a sign minus. It was found that all kinds of the oxygen ions (O_2^+, O_2^-, O_2^o) behave like an inhibitor and $Y(O_2^+) = -2.9 \pm 0.3$ atom/ion; $Y(O_2^o) = -2.3 \pm 0.2$ atom/neutral; $Y(O_2^-) = -2.6 \pm 0.3$ atom/ion.

We pay attention on a qualitative distinction in a behavior of atomic and molecular oxygen. The former accelerates an etching process and the latter decelerates it although the chemical element is the same. For the first sight, it is natural to suppose that this is caused by a difference of the particle masses and, consequently, by a difference of momentum at the same energy. However, the results of [17] shows that an atom yield is a weak function of ion energy at list for the range from 500 to 3000 eV. The following experiments were carried out in order to conform or reject this point of view. It is known [20], that in a case of physical sputtering the particle O_2^+ with energy 1 keV has to behave itself like two independent ions O^+ with energy 500 eV. So we can expect $Y(O_2^-, 1\ keV) = 2Y(O^-, 0.5\ keV)$ and $Y(O_2^+, 2\ keV) = 2Y(O^+, 1\ keV)$. In an energy region less than 1keV it is reasonable to use the negative ions since their intensity is higher (when we decrease ion energy the current density on a target falling down almost linearly). The experiments show $Y(O^-, 0.5\ keV) = 1.6 \pm 0.9$ atom/ion at expected (- 1.3). However, $Y(O_2^+, 2\ keV) = -0.5 \pm 0.3$ atom/ion instead of (+ 3.8). It means that the chemical binding energy (seems to be negligible value) causes a qualitatively new effects. Again, the stimulating ability of an ion depends on a status of its outermost electron shell, but this time - on presence or absence of chemical bond.

At the first sight the inhibition can be attributed to formation of SiO_2 on a silicon surface. However the etch rate doesn't decrease if we feed an oxygen in an exchange chamber when the ion beam is off. The second - deceleration isn't observed when we mixed O_2 with SF_6 in proportion less than 1 : 2 and feed them in a radical source. The latter is well known effect and we just verify it for our system. So, the passivation seems to be negligible for our experiments. The assumption that passivation (due to the forming SiO_2) can be caused by the energetic particles only seems to be artificial and doesn't explain a difference in a behavior of the atomic and molecular oxygen.

As it was mentioned above our PIG source produces ions F^+ from CF_4 in a minute quantity and it was a reason why we choose an oxygen. However the flux of F^- was enough large. It seems to be natural to measure the stimulating ability of these particles. We found that $Y(F^-, 1\ keV) = 3.7 \pm 0.6$ atom/ion. The atom yield keeps a positive sign when the ion energy is reduced at least up to

250 eV, where we registered of Y(F^-, 0.25 keV) = 2.1 ± 0.4 atom/ion. Further diminishing of energy is unreasonable because of the low intensity of a beam.

Conclusion

Thus, we investigated an influence of an ion charge sign on its ability to stimulate a gas-surface reaction of Si-F type. The first results are:

1. The fast neutral atoms have the highest stimulating ability among the fast particles assisted plasma etching of silicon. The stimulating ability of the positive ions is in two times less approximately. Negative ions occupy a middle position.

2. It has been found that some kinds of ions don't accelerate an etch process, but decelerate it. So they behave it like the inhibitors. For the energy range around 1 keV all varieties of molecular oxygen (O_2^+, O_2^-, O_2^o) reveal such property.

3. The presented results make us able to claim that the surface ripping, distraction of Si-Si bonds and forced desorption of a intermediate product of the reaction SiF_x (x = 1, 2, 3) don't explain comprehensively the influence of ion bombardment on an etching process. The branch of the reaction exists which efficiency depends on the status of outermost electron shell (sign of an ion charge and presence of a chemical bond).

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ІОННО-СТИМУЛЬОВАНЕ ТРАВЛЕННЯ КРЕМНІЮ: ВПЛИВ ЗНАКА ЗАРЯДУ ІОНА

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Досліджується відносний вплив іонів різних знаків заряду на стимуляцію травлення кремнію у плазмових умовах. Радикали фтору створюються в жевріючому розряді з градієнтом тиску. Пучок позитивних або негативних іонів формується за допомогою джерела іонів пенінгівського типу. Потік радикалів фтору і пучок іонів взаємодіють на поверхні кремнію у високому вакуумі. Позитивні іони конвертуються у швидкі нейтральні атоми методом резонансної перезарядки. Показано, що найбільшу каталітичну здатність у процесі травлення мають швидкі нейтральні атоми. Ефективність реакції при опромінюванні поверхні позитивними іонами в два рази нижча. Негативний знак заряду іонів суттєво на процес не впливає.

ИОННО-СТИМУЛИРОВАННОЕ ТРАВЛЕНИЕ КРЕМНИЯ: ВЛИЯНИЕ ЗНАКА ЗАРЯДА ИОНА

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Исследуется относительное влияние ионов разных знаков заряда на стимуляцию травления кремния в плазменных условиях. Радикалы фтора производятся тлеющим разрядом с градиентом давления. Пучок положительных или отрицательных ионов создается с помощью источника ионов пеннинговского типа. Поток радикалов фтора и пучок ионов совмещаются на поверхности кремния, расположенного в высоковакуумном объеме. Положительные ионы могут быть конвертированы в быстрые нейтральные атомы методом резонансной перезарядки и собственном газе. Показано, что наибольшей каталитической способностью обладают быстрые нейтральные атомы. Каталитическое влияние положительных ионов примерно в два раза ниже. Отрицательные ионы занимают промежуточное положение. Впервые обнаружено, что некоторые виды ионов (например, молекулярный кислород) не ускоряют, а замедляют процесс травления, т.е. ведут себя как ингибиторы.