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Study of Conditions for Anisotropic Plasma Etching of Tungsten and Tungsten Nitride Using SF_6/Ar Gas Mixtures

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Results of the study of reactive ion etching of tungsten, tungsten nitride, and silicon in SF_6/Ar gas mixtures are presented. For plasma diagnostics, optical emission spectroscopy (actinometry) was used. Using the actinometry technique, it was possible to show that etching mechanisms were different for Si-F and W-F chemistries. Anisotropic etching of tungsten/tungsten nitride using conventional reactive ion etcher has been obtained, and conditions of achieving anisotropic etching have been analyzed. A correlation is found between anisotropy of tungsten etching and the ratio of Si/W etch rates. Mechanisms of fluorine redistribution between the bottom/sidewall surfaces due to surface diffusion and/or reflection are proposed as possible reasons for the observed correlation.
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The use of tungsten and tungsten nitride thin films in microfabrication attracts much attention due to their high thermal stability, good adherence (for tungsten nitride), and low sheet resistance. Due to the continuous shrinking of microdevice dimensions, demands on the dimensional control and anisotropy of etch profiles are constantly increasing. For etching of W and WN_x films, fluorine-containing gases (in particular SF_6 or CF_4) are commonly used which provide high etch rates.[1-4] For conventional reactive ion etching (RIE) reactors, considerable undercutting or formation of concave sidewall profiles due to spontaneous etching of tungsten by fluorine in fluorine-containing plasmas was usually reported.[1-3] To avoid this, various techniques have been employed including passivation of sidewalls by carbon-containing species using polymer-forming gases[1,4] or substrate cooling.[5] Anisotropic etching was obtained in SF_6/Ar using low-pressure high-density etchers characterized by relatively high ion-to-radical flux ratios.[2] Usually low anisotropy is achieved with conventional low-density (*i.e.*, a low ion-to-radical flux ratio) RIE etchers. In some cases, anisotropic etching in RIE experiments was obtained.[3] However, conditions for anisotropic tungsten etching still are not well understood. The objective of the present study was to find and to model conditions of anisotropic etching of W and WN_x in a SF_6/Ar plasma at normal temperatures, without applying sidewall passivation techniques. A conventional RIE medium pressure reactor was used in this study.

The characteristic of a RIE process is a specific combination of chemical and physical (ion-induced) etching mechanisms. The relative contribution of chemical/physical components of the process determines the profile and anisotropy of the etched features. For a better understanding of etching mechanisms it is important to characterize quantitatively particle fluxes at the surface. Here, in order to characterize the density of fluorine radicals in the plasma, an optical emission spectroscopy (the actinometry technique)[6] was employed.

In order to provide considerable variation of radical/ion fluxes to the processed surfaces, the SF_6/Ar flow ratio was varied widely in the experiments. The specific problem of capacitively coupled RF plasma sources is that the main plasma parameters are strongly interrelated. In particular, if the rf power is kept unchanged, the changes in the gas composition (*i.e.*, SF_6/Ar ratio) result in a considerable variation of a dc self-bias as well. The self-bias, built up between the powered electrode and the plasma, determines the energy of ions arriving at the electrode surface. To consider possible effects of an ion bombardment energy on the etching process, we performed two sets of experiments keeping either the rf power or the dc bias constant while changing the SF_6/Ar ratio. To further widen the range of fluorine radical flux variation, some additional experiments were performed at a higher pressure (190 mTorr) or without the Si wafer used as a sample holder (actinometry data showed that removal of the Si wafer resulted in a three-fold rise of the fluorine atomic density in the plasma).

Experimental

In the experiment, a capacitively coupled rf-driven (13.56 MHz) asymmetric plasma reactor made of stainless steel was used. RF power was applied to a smaller water-cooled Al electrode (12 cm in diameter). The larger electrode was electrically connected to the chamber walls. The lower powered electrode was usually covered by a 10 cm diam Si wafer used to avoid redeposition of only slightly volatile products of Al etching (or sputtering) on the samples. A dc potential between electrodes (which is nearly equal to the self-bias, U_{sb}, existing between the plasma and the smaller electrode in an asymmetric reactor) was measured.

Thin films of W and WN_x (with ~10% nitrogen) were deposited on GaAs substrates by magnetron sputtering of W in an Ar or Ar/N_2 atmosphere, respectively. The thickness of films varied from 800 to 900 nm. Samples were patterned by an AZ 5214 photoresist mask, and the etch depth was measured after removal of the mask by a stylus profilometer Dektak. Small samples (area of ~0.2 cm^2) of tungsten, tungsten nitride, and p-type silicon (100) were placed in the center of the Si wafer used as a sample holder.

For etching, mixtures of SF_6 and Ar were used with the gas pressure ranging from 50 to 190 mTorr. In order to characterize the density of fluorine radicals (atoms) in the plasma, an optical emission spectroscopy (actinometry technique) was employed. For actinometry purposes, a 703 nm F line and a 750 nm Ar line were used. Comparing the intensities of these two lines, the fluorine atomic density (N_F) in the plasma can be characterized relatively. It should be noted that for this method to be accurate, the characteristics of excitation for the pair of lines chosen should be similar.[6] Unlike fluorine, an argon atom has two metastable states (11.55 and 11.73 eV) which can be highly populated in plasmas. Thus depending on plasma conditions, a two-step excitation through metastable states may be significant. However, the contribution of the two-step excitation was shown to be practically negligible for the 750 nm Ar line.[7]

Results and Discussion

The results showing the etch rate dependence on the atomic fluorine number density (N_F), as determined from the actinometry data, are presented in Fig. 1, 2, and 3 for Si, W, and WN_x, respectively. The results confirm that the silicon etch rate is as nearly proportional

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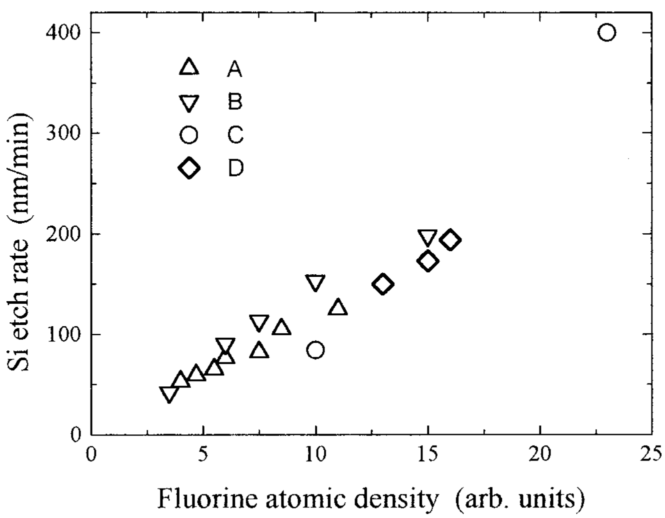

Figure 1. Si etch rate *vs.* fluorine atomic density. Processes A, B, and C: SF_6 percentage varied. A, 50 mTorr, 20 W; B, 50 mTorr, P varied (15 → 54 W); U_{sb} = 106 V; C, 190 mTorr, P varied (30 → 80 W); U_{sb} = 106 V. Process D, SF_6 percentage fixed, 190 mTorr, P varied (30 → 71 W), U_{sb} varied (48 → 158 V).

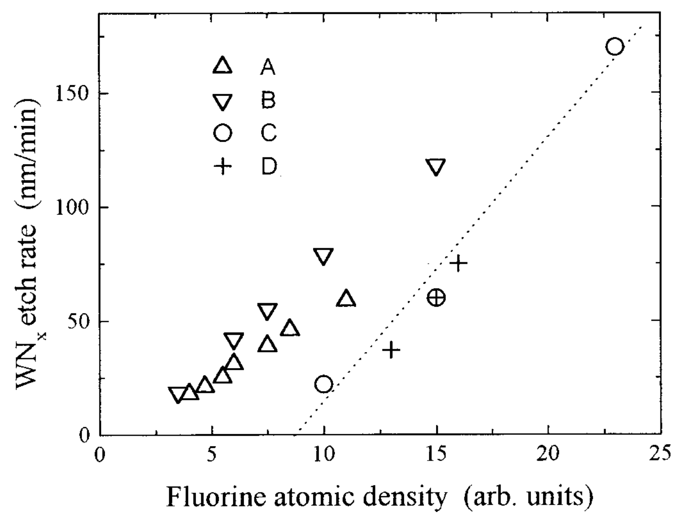

Figure 3. WN_x etch rate *vs.* fluorine atomic density (SF_6 percentage varied). Conditions of the processes A, B, C, and D are the same as for Fig. 1.

to N_F as can be expected[5] within the whole range of plasma parameters investigated, see Fig. 1. Only minor effect of the dc bias (*i.e.*, of the ion bombarding energy) was observed. This confirms that under the present conditions, a chemical (spontaneous) etching is the dominant mechanism in a Si-F system. In contrast, for the tungsten etching the role of ion-induced effects was quite evident (tungsten nitride shows practically the same behavior, compare Fig. 2 and 3). Indeed, at higher dc biases, tungsten etching was considerably faster. The difference between etch rate values obtained at the same fluorine radical densities N_F but differing dc bias was up to 50% at a pressure of 50 mTorr. At 190 mTorr, the tungsten etch rate obtained at the same fluorine density dropped up to 2.5 times as compared with the 50 mTorr case, at about the same dc bias value (again, in striking contrast to the behavior of the Si etch rate). Possible reasons for this may include (*i*) reduction of the ion flux coming to the surface and (*ii*) changes in transport of ions through the sheath. At higher pressures, the sheath becomes collisional, *i.e.*, ions undergo a number of collisions on the way from the plasma boundary to the electrode, partially losing their kinetic energy. Thus the mean energy of ions arriving at the surface at higher pressures is a fraction of the dc bias. Due to different sensitivity to ion bombardment, the pressure effect is notably different for silicon and tungsten etching.

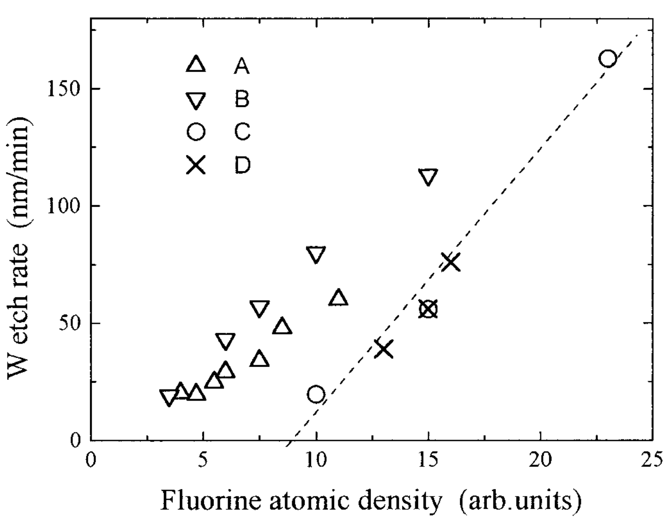

Figure 2. W etch rate *vs.* fluorine atomic density (SF_6 percentage varied). Conditions of the processes A, B, C, and D are the same as for Fig. 1.

The effect of electrode material (Si *vs.* Al) on etching is shown in Fig. 4, where the results of experiments performed at a constant dc bias value (106 V) are presented. As follows from the actinometry data, the removal of the Si wafer (normally covering most of the Al electrode surface area) results in a sharp (~three-fold) rise of fluorine radical densities in the plasma. Thus under the present conditions, losses of fluorine radicals in the plasma are mostly due to their consumption on the Si wafer surface followed by desorption as SiF_x etch products. The effect of the N_F increase in the plasma due to the wafer removal is different for silicon and tungsten etching. As Si etching by fluorine is basically spontaneous, the Si etch rate continues to rise linearly with the fluorine density. In contrast, W and especially WN_x etch rate dependencies on N_F show considerable deviation from a simple linear behavior. The observed drop of the WN_x etch rate was much stronger than that for W, so that the difference between the W and WN_x etch rates was as high as ~50%. In all other experiments (see Fig. 2 and 3), this difference did not exceed 10%. Note that practically no changes were observed in the dc bias after the wafer removal, if other plasma conditions were kept the same.

Another important feature of W (and WN_x) etch rate dependence on N_F is its threshold behavior. This effect is not so evident for the gas pressure of 50 mTorr. In contrast, it is well pronounced for the pressure of 190 mTorr (see Fig. 2 and 3), where the tungsten etch rate starts to grow fast after the fluorine atomic density exceeds some critical value. The threshold appears to depend on ion bombardment energies which tend to decrease with gas pressure (as the self-bias decreases with the pressure). This indicates that ion bombardment provides considerable enhancement of surface processes including surface diffusion leading to efficient formation and desorption of volatile WF_6 etch products, and the effect is stronger for higher ion energies. The likely mechanism is an associative desorption accelerated by ion bombardment[2,5]

$$WF_{x(ads.)} + WF_{y(ads.)} \xrightarrow[\text{(ion)}]{} WF_6\uparrow + F_{x+y-6(ads.)}$$

Note that the threshold must be higher for a spontaneous associative desorption which occurs without ion bombardment and is responsible for lateral etching. All experiments presented in Fig. 4

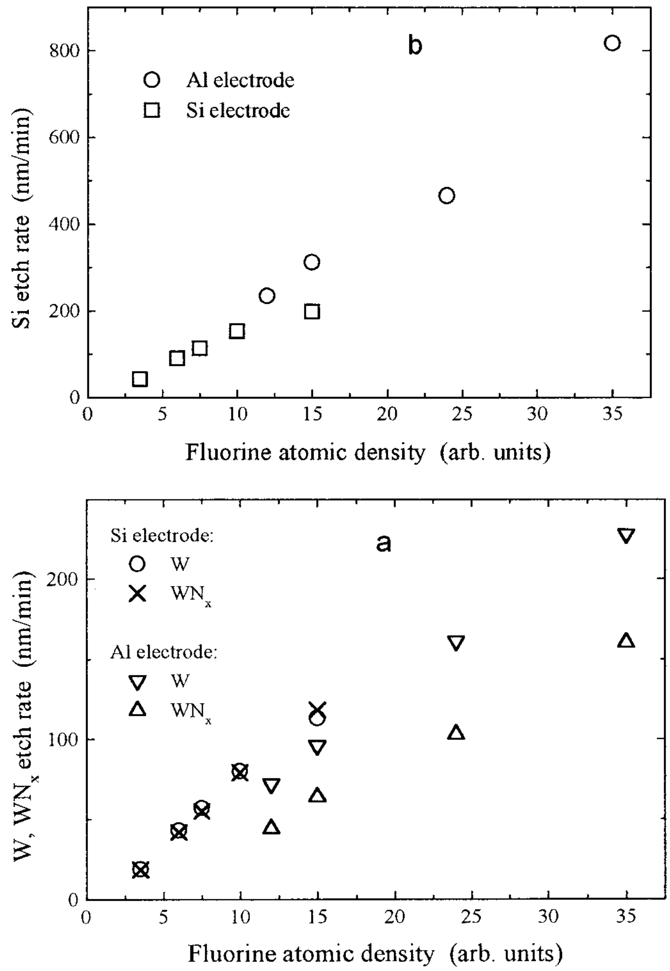

Figure 4. Effect of the electrode material (Al/Si) on etching characteristics of W and WN_x (a) and Si (b). Plasma conditions: P varied (15 → 54 W), U_{sb} = 106 V, 50 mTorr.

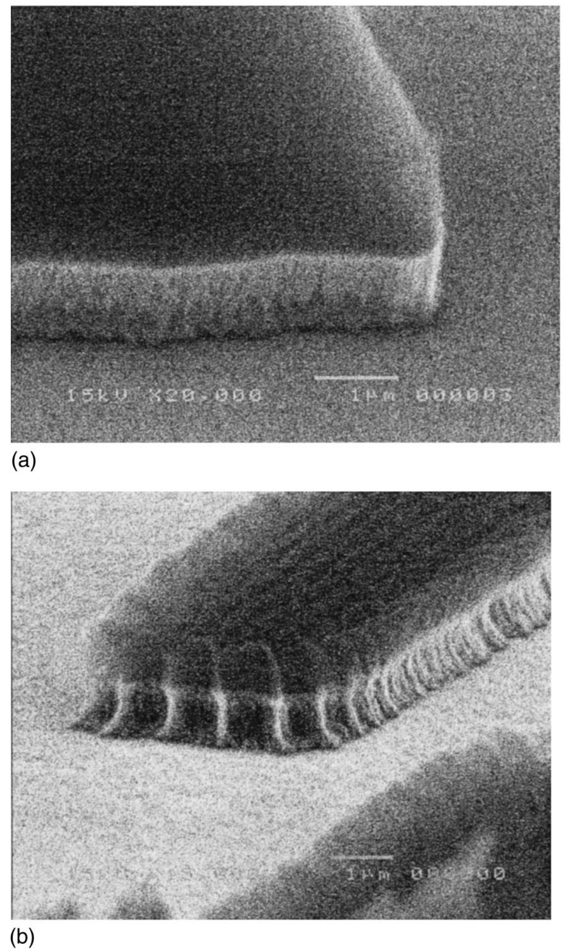

Figure 5. SEM pictures of an etched structure in WN_x (photoresist film is not removed). Plasma conditions: 190 mTorr; (a) SF_6/Ar = 10/35 sccm, 65 W, 122 V; (b) SF_6/Ar = 20/35 sccm, 80 W, 100 V. Note vertical (a) and concave (b) etch profiles.

were performed at the same dc bias, *i.e.*, the same ion bombarding energy. Therefore the sharp drop of the WN_x (W) etch rate observed with the Si wafer removal can be attributed to a considerable slowing down of surface diffusion in the presence of hardly volatile Al products redeposited on the tungsten surface from the plasma. The effect appears to be more pronounced for WN_x, probably due to higher porosity of a tungsten nitride surface than that of tungsten. No notable threshold was observed for the Si etch rate dependence on fluorine density in the plasma, in accordance with the conclusion that ion-induced effects are of minor importance for this case.

In most experiments performed at 50 mTorr, highly anisotropic etching in tungsten and tungsten nitride have been achieved. Moreover, for Ar-rich mixtures anisotropic etching was possible also at higher pressures. One example, shown in Fig. 5a, was obtained at 190 mTorr with the rf power of 65 W, the self-bias of 122 V, and gas flows of SF_6/Ar = 10/35 sccm. In this case, the vertical etch rate was relatively high (75 nm/min). For comparison, an example of a concave etch profile obtained for the same pressure (other parameters are: rf power of 80 W, self-bias of 100 V, gas flows SF_6/Ar = 20/35 sccm) is shown in Fig. 5b. For this case, the lateral etch rate can be estimated as ~30 nm/min.

These results show that anisotropic etching of tungsten is possible even at a relatively high fluorine flux which is characteristic of a high-pressure plasma. Thus it is likely that the anisotropy of etching depends not solely on the fluorine radical flux but on the ratio of radical and ion fluxes. Further, in order to analyze conditions of anisotropic etching of tungsten in more detail, we now consider a simple model of surface processes during etching. This consideration shows that it is instructive to compare etch rates for tungsten and silicon which depend on fluorine and ion fluxes in different ways. Results of the present work and other studies[2,3] on W and Si etching by SF_6 are compared.

For the case of reactive ion etching, a simple model, which takes into account synergistic effects of ion-neutral interaction on the surface,[8,9] gives the following expression for etch rate

$$V = J_i Y(E_i, \Theta)\Theta/\rho \quad [1]$$

where J_i is the ion flux to the surface, $Y(E_i, \Theta)$ is the effective sputtering yield (the number of substrate atoms removed per incident ion), E_i is the incident ion energy, Θ is the surface coverage by reactive radicals, and ρ is the substrate density. For tungsten, the atomic fluorine adsorption was shown to be monolayer-like,[5] with the adsorption rate being proportional to $s(1 - \Theta)$, where s is the sticking coefficient for fluorine atoms on a bare surface. Due to a strong repulsive interaction between fluorine atoms, adsorption of fluorine is strongly reduced when the surface coverage is close to saturation ($\Theta \approx 1$). In this case, fluorine atoms coming from the plasma are mostly reflected. Surface coverage of tungsten by fluorine in a steady state is determined by[9]

$$\Theta = (1 + \eta J_i / s J_F)^{-1} \quad [2]$$

where η is the number of fluorine atoms removed from the surface per incident ion, and J_F is the atomic fluorine flux to the surface.

Depending on the J_i/J_F ratio, surface conditions can vary widely. At a relatively high radical flux ($\eta J_i \ll sJ_F$), saturation of the surface coverage can be obtained, with $\Theta \approx 1$. For the other limiting case ($\eta J_i \gg sJ_F$), the surface coverage is low, and the expression for it reduces to $\Theta \approx sJ_F/\eta J_i$, then the etch rate appears to depend solely on the fluorine flux

$$V_W = s_W J_F/\chi_W \rho_W \qquad [3]$$

where χ_W is the stoichiometry of tungsten etch products ($\chi_W = \eta_W/Y_W$). Note that the strength of a F-W bond is much higher than that for a F-F bond (5.7 and 1.65 eV, respectively[10]). Thus it is likely that F atoms adsorbed on a tungsten surface do not recombine and are eventually desorbed from the surface mostly in the form of etch products. For the W-F chemistry, the main etch product is known to be WF_6,[2,5] so that $\chi_W \approx 6$. As the silicon etching by fluorine is basically chemical, the silicon etch rate is determined predominantly by a fluorine flux and can be presented in a form, similar to Eq. 3

$$V_{Si} = s_{Si} J_F/\chi_{Si} \rho_{Si} \qquad [4]$$

A variety of Si etch products (from SiF_2 to SiF_4) is possible in the case of Si-F chemistry, however the main etch product is known to be SiF_4,[5] so that χ_{Si} is close to 4. Under strong ion bombardment, not fully saturated silicon etch products can be produced, but one can expect that the average χ_{Si} value to be no less than 3.

Finally, from Eq. 3 and 4, the ratio of the Si and W etch rates can be found for the case $\eta J_i \gg sJ_F$ (low surface coverage)

$$V_{Si}/V_W = (s_{Si}/s_W)(\chi_W/\chi_{Si})(\rho_W/\rho_{Si}) \qquad [5]$$

Therefore, under the conditions of a radical-flux-limited regime, the Si/W etch rates ratio turns out to be independent of the plasma parameters. Sticking coefficients for Si and W were shown to be about the same.[5] Then, taking $\rho_W/\rho_{Si} = 1.26$,[10] the estimates give the ratio $V_{Si}/V_W \approx 1.7$. For average values of χ_{Si} in the range from 4 to 3, one can obtain slightly higher ratios, *i.e.*, $1.7 \leq V_{Si}/V_W \leq 2.3$.

Note that the Eq. 3 was obtained for the case of low Θ. As Θ approaches saturation, the fraction of the fluorine flux J_F adsorbed on the tungsten surface, begins to reduce. As a result, the V_{Si}/V_W ratio starts to grow and, closer to saturation, may considerably exceed the value corresponding to the low Θ limit.

Now, two important points should be underlined: (*i*) for $\Theta \ll 1$, the V_{Si}/V_W ratio takes the lowest value, close to 2.0, (*ii*) for $\Theta \approx 1$, the ratio must be considerably higher than 2.0. In other words, if experimentally obtained V_W is considerably smaller than $V_{Si}/2$, this indicates that the W surface coverage is close to saturation. Below it is shown that our results and the data available in the literature for W and Si etching in SF_6 with different plasma sources[2,3] are consistent with the present analysis. The minimum values of the V_{Si}/V_W ratio obtained under different plasma conditions, vary in the range of 1.7-2.0. Furthermore, a certain correlation can be found between the V_{Si}/V_W ratios and the etching anisotropy.

In Ref. 2, the experiments were carried out with a microwave high-density low-pressure (<0.2 mTorr) plasma source, with W etch rates in the range 10-40 nm/min. The power and the dc bias applied to the sample holder were kept constant (600 W and −70 V, respectively). Production of atomic fluorine in the plasma was varied by changing gas pressure. At lower fluorine densities (apparently, at a low surface coverage), the ratio of W/Si etch rates in SF_6 was close to 1.75 (see Fig. 1 in Ref. 2). At higher fluorine densities (and growing surface coverage), a tendency of saturation for W etch rate was observed, resulting in a considerable growth of the V_{Si}/V_W ratio. At low fluorine fluxes (low Θ), highly anisotropic W etching was reported, while a notable lateral etching (undercutting) started at the V_{Si}/V_W ratio exceeding 2.3.

In Ref. 3, in the experiments performed with an rf driven (13.56 MHz) magnetically enhanced middle-pressure plasma source, etch rates in SF_6 were measured for W and polycrystalline Si (our experiments have shown that in a SF_6 plasmas, the etch rate for poly-Si is close to that of mono-Si[11]). Two modes of etching were used, an RIE mode (samples placed on the powered electrode) and a plasma etching or PE mode (samples placed on the grounded electrode). Relatively high W etch rates, about 500 nm/min, were obtained in both cases. The $V_{Si\text{-}poly}/V_W$ ratio was close to 1.8 and to 2.5 in RIE and PE modes, respectively (see Fig. 7 of Ref. 3). No undercutting was observed in the RIE mode, while an isotropic etching was reported for the PE mode. As the plasma conditions in the experiment were kept unchanged (the rf power of 400 W and the gas pressure of 10 mTorr), the difference between the two modes was basically in a ratio of fluxes J_i/J_F coming to the etched surface, with the fluorine flux J_F presumably being about the same. The fact that the etch anisotropy differed dramatically for the two modes, gives strong evidence that lateral etching depends not only on the fluorine flux coming directly from the plasma, but on the conditions on the bottom surface as well.

Finally, in the present study, the V_{Si}/V_W ratio was close to 2.0 in most experiments performed at a lower pressure, *i.e.*, at 50 mTorr (see Fig. 1-3), and the minimum value obtained was 1.7. Higher ratios (up to 4.1) were obtained at a pressure of 190 mTorr. High anisotropy of etching was achieved not only for experiments at 50 mTorr, but also in some experiments at a higher pressure (190 mTorr), see Fig. 5a. An important point is that for all cases of anisotropic etching, V_{Si}/V_W values did not exceed 2.5. This shows the same tendency as observed in Ref. 2 and 3 under widely differing plasma conditions; higher anisotropy correlates with lower V_{Si}/V_W ratios (and thus, with lower surface coverage by fluorine). Let us discuss possible mechanisms which may explain this effect.

Due to geometrical reasons, sidewalls of etched features receive smaller flux of fluorine than the bottom/horizontal surface (we consider small etch depths). Lateral etching of sidewalls occurs mainly due to a spontaneous mechanism as only a small fraction of the ions reflected from the bottom surface can reach the walls of etched features. However, lateral etching occurs not only due to fluorine coming directly from the plasma, it may also depend on the fluorine supply from the bottom surface adjacent to the wall. This effect should be much stronger when the bottom surface is saturated with adsorbed fluorine.

There are some mechanisms through which the bottom surface conditions may affect a lateral etching. First, the fluorine radical flux coming from the plasma to the saturated bottom surface is mostly reflected and eventually may be adsorbed at a wall surface. Second, surface diffusion from the bottom (upward diffusion) may contribute to a rise of fluorine surface density at the walls, especially if the bottom surface is saturated with fluorine. The characteristic length of fluorine diffusion at a tungsten surface at a normal temperature was reported to be ~0.5 μm, being comparable with the size of etched features.[5] Third, under certain conditions the fluorine surface density may be higher at the vertical surfaces (walls) than at the bottom. This may happen if the J_i/J_F ratio is relatively high so that the steady-state coverage of the bottom surface is low because the ion flux efficiently removes fluorine from the bottom surface with etch products. Then the direction of diffusion may change, coming downward from the walls to the bottom. In this case, the vertical etch rate will be enhanced as compared with that given by Eq. 3, determined solely by the fluorine flux coming directly from the plasma. Furthermore, the lateral etching will be suppressed (and the anisotropy improved) due to downward diffusion that depletes the wall surface coverage by fluorine.

So, using a simple model of etching, important information on a tungsten surface coverage by fluorine can be obtained from a comparison of W and Si etch rates. Based on this analysis, mechanisms involving fluorine redistribution between the bottom/sidewall surfaces due to diffusion or reflection have been discussed. These mechanisms may be responsible for the experimentally observed correlation between the etching anisotropy and the ratio of etch rates for W and Si obtained under the same plasma conditions. The discussed mechanisms should be taken into account in modeling of a sidewall profile evolution in etching experiments (see, for example, Ref. 12).

Conclusions

In the present work, results of a comparative study of W, WN_x, and Si RIE in SF_6/Ar are presented. For plasma diagnostics, an optical actinometry technique was employed which allowed us to evaluate the dependence of the fluorine atomic density on the plasma parameters. By using actinometry, etching mechanisms are shown to be different for Si-F and W-F chemistries. Using a simple model for etching, etch rates have been compared for Si and W under the same plasma conditions. It is shown that the ratio of Si/W etch rates depends essentially on a tungsten surface coverage by fluorine. In the case of low coverage, the ratio takes minimum values close to 1.7-2.0. The ratio grows well above 2.0 as the coverage gets closer to saturation.

Conditions of achieving anisotropic etching to obtain vertical etch profiles in W/WN_x are analyzed. A correlation is found between anisotropy of tungsten etching and the ratio of Si/W etch rates (which in turn, depend on a tungsten surface coverage by fluorine). Mechanisms of fluorine redistribution between the bottom/sidewall surfaces due to diffusion or reflection are proposed as a possible reason for the observed correlation. Based on the present consideration of surface processes, more accurate modeling of sidewall profile evolution during etching is possible.

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