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MECHANISMS OF SELECTIVITY LOSS DURING TUNGSTEN CVD

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ABSTRACT

The tungsten subfluoride mechanism as well as other proposed mechanisms of selectivity loss are reviewed. To further demonstrate the viability of the tungsten subfluoride mechanism, we have extended the measurement of the tungsten subfluoride production rate down to 450°C. We also report results from some preliminary experiments designed to identify the selectivity loss mechanism when elemental silicon is available for reaction. Comments regarding the origins of the insulator effect and selectivity loss for silane reduction are offered.

INTRODUCTION

Although selective chemical vapor deposition (CVD) of tungsten [1-6] holds much promise for metallization during microelectronics fabrication, its use in manufacturing has been hindered by the inability to maintain selectivity for a sufficient growth duration. While there are a number of extrinsic (or extraneous) causes of selectivity loss [7], there is evidently at least one intrinsic selectivity loss mechanism. The intrinsic selectivity loss mechanism(s) exhibits a "proximity" effect [8-10] whereby the rate of tungsten nucleation on an oxide surface is effected by its proximity to an area of tungsten deposition. This phenomenon also leads to an autocatalytic nucleation process in which the tungsten particle density appears to grow exponentially in time [8,11,12]. The effected oxide surface need not be contiguous with the deposition surface, i.e. they may be on separate wafers. This observation clearly indicates that a volatile product or byproduct of the CVD process is responsible for initiating selectivity loss. A number of candidate species have been proposed including HF, SiF_4, SiF_x ($x < 4$), and WF_x ($x < 6$). We will review the supporting evidence for each of these species.

In general, there are two types of experiments that have been (and can be) performed to study selectivity loss. In one type of experiment the "degree" of selectivity loss is measured (usually by monitoring tungsten particle density) as some experimental condition (e.g. temperature, pressure, wafer composition, etc.) is varied. Although this type of study can be extremely valuable, it is indirect by its very nature. Therefore, results from this type of study can rarely, if ever, be used to unambiguously prove any particular mechanism of selectivity loss, although such observations may serve to rule out proposed selectivity loss mechanisms and may set important criteria that a proposed mechanism must meet. In a second type of experiment, diagnostics (e.g. mass spectroscopy, Auger spectroscopy, optical spectrscopies, etc) are used with the goal of directly examining the intermediates responsible for selectivity loss. Ideally, the diagnostics are used in situ, or in a environment which closely mimics CVD conditions. We have primarily focused our efforts on this second type of experiment and made direct measurements of key reaction intermediates.

EXPERIMENTAL

Experiments were performed in dual chambered UHV systems as described previously [13,14]. The systems consisted of a reaction chamber, an analysis chamber, and a manipulator used to move a sample between the chambers. The analysis chamber contained Auger electron spectroscopy (AES), and a quadrupole mass spectrometer for temperature programmed desorption (TPD) and gas-phase sampling of the reaction chamber via differential pumping. A novel aspect of the experiments is that two independently heated samples were mounted in the reaction chamber. One sample was typically an oxidized silicon substrate while the other sample was a tungsten foil. For some experiments the tungsten foil was replaced with a silicon sample. The tungsten and silicon samples served as sources of chemical intermediates which could then interact with the nearby oxide sample. The oxide surface could then be examined with the diagnostics in the analysis chamber. This experimental apparatus and procedure allowed for a stepwise determination of the selectivity loss mechanism. Further experimental details are described in refs. 13,14.

RESULTS

The Role of Hydrogen Fluoride

Hydrogen fluoride is the primary reaction product formed by the hydrogen reduction of WF_6, i.e. $3H_2(g) + WF_6(g) \rightarrow W(s) + 6HF(g)$, and is an obvious candidate for initiating selectivity loss. Since HF is evolved from the depositing tungsten surfaces, any selectivity loss mechanism involving it will automatically exhibit the proximity effect. In fact, Kwakman et al. [8] quantitatively measured tungsten nuclei densities on oxide surfaces and showed that they indeed correlated with the predicted HF gas-phase concentration above the wafer surface. Although this observation is suggestive, it does not prove a cause and effect relationship for HF, as any species evolved from the tungsten surface will also show a similar correlation (giving rise to the proximity effect).

To the contrary, attempts to accelerate selectivity loss by adding additional HF to the gas stream have been somewhat contradictory or inconclusive [7,8,13,15]. These latter results lead us to believe that HF does not play a major role in the selectivity loss mechanism. By working through possible chemical mechanisms we can show why HF is not a likely candidate for the selectivity loss precursor. HF may attack SiO_2 under some conditions, yielding H_2O and SiF_4 (see reaction 1). Surface hydroxyl and SiF groups are likely surface intermediates of the reaction.

$$4\ HF(g) + SiO_2(s) \rightarrow 2\ H_2O(g) + SiF_4(g), \qquad \Delta H = -41\ \text{kcal/mol}, \qquad (1)$$

$$WF_6(g) + H_2O(g) \rightarrow WOF_4(g) + 2\ HF(g), \qquad \Delta H = 4\ \text{kcal/mol}, \qquad (2)$$

$$WF_6(g) + 3\ H_2O(g) \rightarrow WO_3(s) + 6\ HF(g), \qquad \Delta H = -2\ \text{kcal/mol}. \qquad (3)$$

Reasonable reaction scenarios can be devised where WF_6 reacts with adsorbed water or hydroxyl groups on the SiO_2 surface to yield tungsten oxides or oxyfluorides (e.g. see reaction 2 and 3). It is important to note that the oxidation state of the tungsten (and silicon) remains unchanged in these reaction schemes and it is unlikely that the tungsten oxides and oxyfluorides lead to selectivity loss. We believe that the oxidation state of tungsten must be lowered to initiate selectivity loss. As an example, we have found [16] that tungsten oxides (WO_3 and H_2WO_4) do not lead to selectivity loss and are actually etched by WF_6.

It has been suggested that the reaction of HF with silicon oxides may yield silicon subfluorides (SiF_x, $x < 4$) [8,12,17]. Silicon subfluorides are certainly capable of reducing WF_6 (see below), but all reaction pathways we have considered which produce silicon subfluorides by the HF + SiO_2 reaction are highly endothermic (in excess of 90 kcal/mol). To illustrate the significance of this, we note that at 400°C a first-order reaction rate constant with a 90 kcal/mol activation energy has a value of $\sim 10^{-31}$ s. Simply stated, silicon in SiO_2 is in a high oxidation state (+4), and since HF is an extremely poor reducing agent it is unable to produce silicon in a reduced oxidation state, i.e. SiF_3 or SiF_2. Therefore we do not believe that silicon subfluoride formation via HF/SiO_2 reactions is a viable pathway for selectivity loss.

The Role of Tungsten Subfluorides

Our previous studies [13,14,18] found evidence for a selectivity loss mechanism that is initiated by desorption of a tungsten subfluoride formed by the reaction of WF_6 with metallic tungsten surfaces. Our first experiments isolated tungsten tetrafluoride (WF_4) [13]. When a hot (~600°C) tungsten surface is exposed to WF_6 , a non-volatile subfluoride, apparently WF_4, will accumulate on a nearby SiO_2 surface held at ~30°C. Disproportionation of this tungsten subfluoride upon heating above 350°C produces a reactive, essentially metallic state of tungsten that induces rapid selectivity loss when exposed to CVD conditions. Several factors suggest that tungsten pentafluoride, WF_5, not WF_4, is the actual gas-phase intermediate involved in the tungsten transport mechanism for typical tungsten CVD conditions. These factors include its volatility, the temperature range for which it can be formed, and its propensity to disproportionate into lower subfluorides. We were able to isolate WF_5 [14] by cooling the SiO_2 surface to -45°C under experimental conditions otherwise similar to those used to isolate WF_4. Auger spectroscopic results yield a stoichiometry of $F/W = 4.9 \pm 0.5$ for this subfluoride, and TPD results are consistent with the behavior of tungsten pentafluoride. Upon heating of WF_5 multilayers, most of the compound desorbs, but some reaction and disproportionation occurs which ultimately leaves a reduced state of tungsten on the SiO_2 surface. An intermediate in the WF_5 disproportionation sequence is WF_4, which is the compound we first isolated as described above.

These results strongly support a selectivity loss mechanism based on an <u>intrinsic</u> tungsten transport mechanism that is initiated by formation of tungsten pentafluoride from the reaction of WF_6 with metallic tungsten (reaction 4). This reaction is considered an etching reaction that removes elemental tungsten (s_1 = tungsten surface) and forms a volatile subfluoride. In this reaction the tungsten in WF_6 is reduced by the metallic tungsten (the oxidation state is reduced from +6 to +5).

$$5/6\ WF_6(g) + 1/6\ W(s_1) \rightarrow WF_5(g), \qquad \Delta H = \sim 10 \text{ kcal/mol}, \tag{4}$$
(Subfluoride formation and desorption from tungsten)

$$WF_5(g) \rightarrow WF_5(s_2), \qquad \Delta H = \sim -12 \text{ kcal/mol}, \tag{5}$$
(Adsorption on SiO_2)

$$WF_5(s_2) \rightarrow 1/2\ WF_4(s_2) + 1/2\ WF_6(g), \qquad \Delta H = \sim -5 \text{ kcal/mol}, \tag{6}$$
(Disproportionation to WF_4)

$$1/2\ WF_4(s_2) \rightarrow 1/3\ WF_6(g) + 1/6\ W(s_2), \qquad \Delta H = \sim 7 \text{ kcal/mol}. \tag{7}$$
(Disproportionation to W)

Tungsten pentafluoride is volatile even at room temperature and may easily diffuse through the gas phase. Upon contact with another warm surface (s_2 = SiO_2), WF_5 may disproportionate into WF_4 and WF_6, (reaction 5-6). This step involves a reaction between at least two pentafluorides and is the source of WF_4 that was isolated in our first experiments. At temperatures greater than 200°C, tungsten tetrafluoride may disproportionate further into tungsten hexafluoride and metallic tungsten (reaction 7). Note that the sum of reactions 4-7 gives no net production of new species, the only change is that tungsten has been transported from one surface (s_1) to another (s_2). The enthalpy values given for reactions 4-7 should be considered approximate because of experimental uncertainties in the heats of formation of the subfluorides.

In these previous studies it was necessary to accentuate the selectivity loss mechanism in order to raise the concentration of the key intermediates to levels detectable with conventional surface science techniques (e.g. AES and XPS). This was generally accomplished by creating the subfluorides (reaction 4) at 600-700°C, although experiments at 500°C did produce detectable amounts of subfluoride. Since this temperature range is somewhat higher than that used for conventional tungsten CVD, the viability of the tungsten subfluoride mechanism at "conventional" temperatures is of concern. In order to address this issue we measured the rate of reaction 4 using a total and partial pressure drop technique [13] for an initial WF_6 partial pressure of ~5 mTorr. The results of these measurements are displayed in the upper three curves of Fig. 1 (ΔP data). There is a factor of ~5 variation in absolute rates measured over a period of time that is most likely due to variations in tungsten surface area caused by the etching reaction. The activation energy for reaction 4 was found to be 23 ± 2 kcal/mol considering all data sets. Extrapolation of these results down to "conventional" CVD temperatures shows that tungsten subfluoride formation reaction is still significant, being on the order of 10^{11}-10^{12} molecules $cm^{-2}\ s^{-1}$ at 300°C and 10^{12}-10^{13} molecules $cm^{-2}\ s^{-1}$ at 400°C. These rates are "significant" when you recall that selectivity loss is considered severe with tungsten particle densities of 10^8-$10^{11}\ cm^{-2}$.

By employing a different technique, we have now verified that the extrapolation of the ΔP data is correct down to at least 450°C. This technique uses a quartz crystal microbalance (QCMB) placed within a few centimeters of the heated tungsten foil. The QCMB was cooled to -65°C in order to condense the tungsten pentafluoride formed by reaction 4. Results of this experiment are given in the lower curve of Fig. 1. The activation energy calculated from this technique is slightly lower (~21 kcal/mol) than the value obtained from the pressure drop method, but in general the quantitative agreement is quite good. The absolute value of the reaction rates obtained from the QCMB method are certainly underestimated because only a fraction of the WF_5 formed is condensed on the QCMB active area. The detection limit of this technique was limited by a low volatility species present in the WF_6 source (probably WOF_4) which also condensed on the QCMB head, otherwise we believe the measurement range could be extended well below 400°C. This background condensation rate was subtracted from the data that is presented in Fig. 1. In any case, we see no reason to suspect that extrapolations of the rate of reaction 4 to 300-400°C are in error.

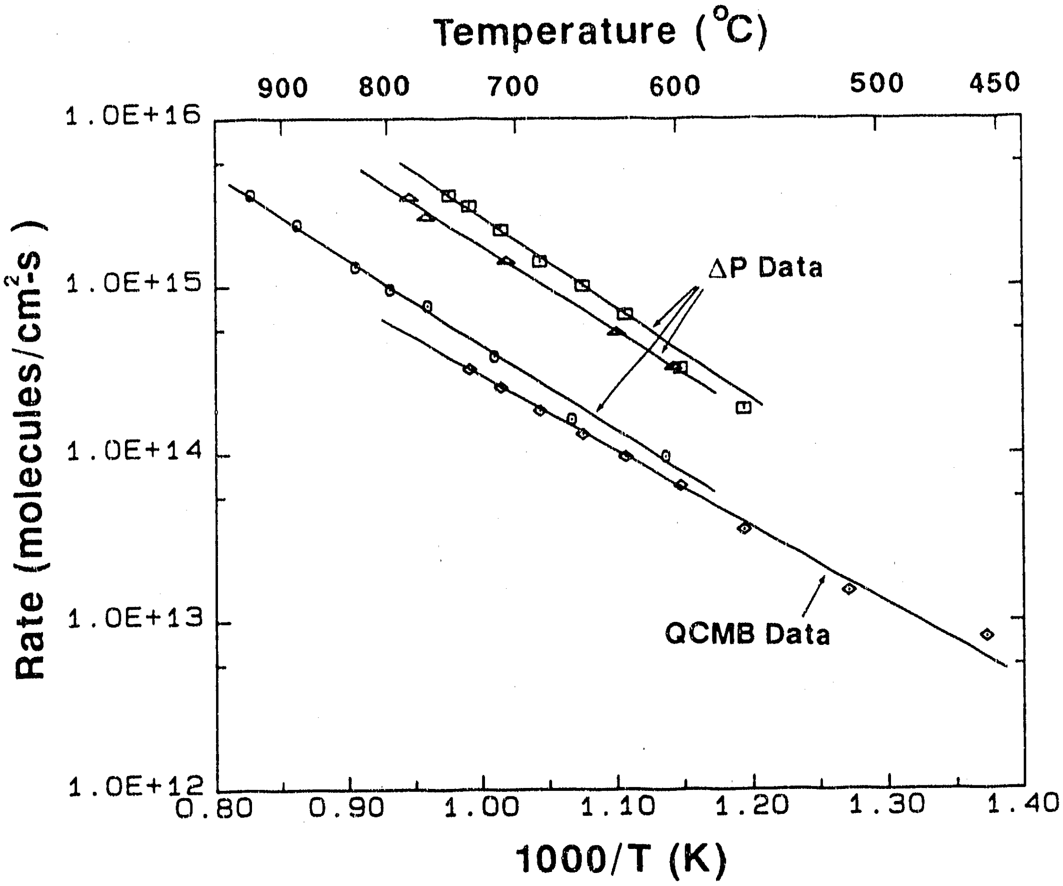

Fig. 1. Measurements of WF_5 generation using two methods, the pressure drop (ΔP) method and the condensation (QCMB) method. The WF_6 pressure was ~5 mTorr.

While selectivity loss in initiated by WF_5 formation, the overall kinetics of the process involves adsorption, desorption, and disproportionation steps, followed by growth of the tungsten particles by tungsten CVD. We have not explicitly measured the kinetics of these reactions. The kinetics of final stages of tungsten nucleation has been examined in more detail by McConica and Cooper [11] and Pattee et al. [9]. Detailed modeling that included formation of tungsten subfluorides has been performed by Ulacia et al. [19] and Arora and Pollard [20].

The Role of Silicon Tetrafluoride and Subfluorides

In some applications it is desirable to deposit tungsten directly on silicon. In this case WF_6 is directly reduced by elemental silicon to form predominately SiF_4 (see reaction 8) before other reduction processes proceed, e.g. H_2 reduction. Several researchers have noted that selectivity loss on some oxides is faster when silicon is initially present when compared to the same conditions where an equal area of tungsten or other metal is initially present [8,10,21].

$$2/3\ WF_6(g) + Si(s) \rightarrow 2/3\ W(s) + SiF_4(g), \qquad \Delta H = -111\ \text{kcal/mol.} \qquad (8)$$

This effect was quantitatively described by Hitchman et al., and the initial nucleation rate on oxide wafers facing silicon was found to be ~3.2 times faster than the nucleation rate on oxide wafers facing tungsten [21]. This observation suggests that either some product of silicon reduction enhances selectivity loss to a greater extent than tungsten subfluorides, or tungsten subfluorides are produced in greater concentration when elemental silicon is present. Silicon tetrafluoride has been suggested as a precursor for inducing selectivity loss [5,21], but we do not believe it is a viable candidate as silicon is in its highest oxidation state and cannot act as a reducing agent towards WF_6. Hydrogen reduction of SiF_4 is also extremely unlikely since it is endothermic by 130 kcal/mol.

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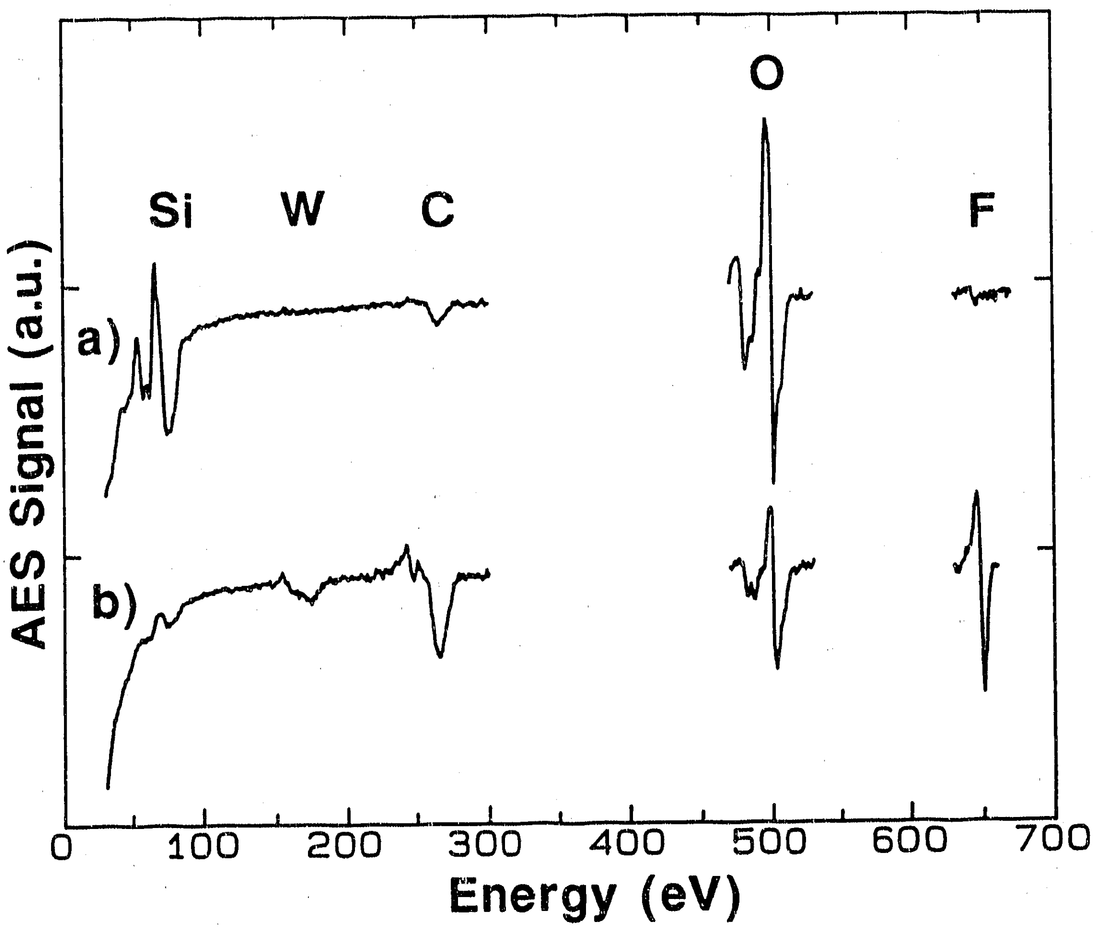

Fig. 2. AES measurements of oxide surface to test effect of silicon reduction of WF_6. Curve (a) is the initial SiO_2 surface. Curve (b) is the surface following WF_6 reaction with a nearby silicon sample.

Silicon subfluorides such as SiF_2 and SiF_3 should be capable of reducing WF_6, as illustrated in reaction 9.

$$SiF_2(g) + WF_6(g) \rightarrow WF_4(s) + SiF_4(g), \qquad \Delta H = -115\ \text{kcal/mol.} \qquad (9)$$

In fact, Hirase et al.[7] demonstrated that blanket tungsten deposition occurs (i.e. total selectivity loss) when silicon subfluorides are intentionally added during tungsten CVD. Ming Yu, et al. [22] have shown that SiF_2 is formed when low doses of WF_6 impinge upon clean silicon above 450°C, so a mechanism that forms volatile silicon subfluorides via a WF_6/Si interaction has been demonstrated. It is not clear if this mechanism of silicon subfluoride production is occuring at the higher WF_6 pressures typically used during CVD, as higher pressures would tend to drive silicon tetrafluoride formation to completion.

We have performed preliminary experiments aimed at elucidating the selectivity loss mechanism when elemental silicon is initially present. The experimental methodology was the same as that used to isolate tungsten tetrafluoride except a silicon sample (~2 cm^2 area) was substituted for the tungsten foil. The Auger spectrum of the initial SiO_2 (thermally grown) surface was measured and is displayed in Fig. 2.a. Then, with the oxide sample kept at ~30°C, WF_6 was introduced into the reaction chamber at a pressure of 5 mTorr. The silicon sample, which was ~2 cm from the oxide sample, was then heated from 30°C to 430°C over a 3 min interval. To insure that silicon reduction did occur, the evolution of SiF_4 was monitored with a differentially-pumped mass spectrometer. The evolution of SiF_4 was virtually complete by 350°C (Due to the vacuum system design, the mass spectrometer was not capable of detecting subfluorides of silicon or tungsten). After completing the silicon reduction cycle, the reaction chamber was evacuated and the SiO_2 surface was examined with AES. If a silicon subfluoride was present, it should exhibit a silicon AES signal between 76 eV (SiO_2) and 92 eV (elemental Si). As illustrated in Fig. 2.b, the silicon signal at 76 eV arising from the SiO_2 surface has been strongly attenuated by presence of an overlayer of tungsten, carbon, fluorine, and perhaps some additional oxygen. The degree of attenuation indicates an overlayer thickness of ~1.2 nm. A silicon AES signal expected for a silicon subfluoride was not detected. Instead, the tungsten and fluorine signals suggest the presence of a tungsten subfluoride with a stoichiometry approximating WF_4. The source of the additional carbon as well as its chemical binding state cannot be positively ascertained. An experiment where the silicon sample was heated to 1000°C to remove the native oxide before WF_6 introduction yielded results qualitatively similar to those displayed in Fig. 2.

The absence of silicon subfluorides and the presence of a tungsten subfluoride on the SiO_2 surface was somewhat surprising at first, but can be explained in at least two ways. The first explanation is that volatile silicon subfluorides are indeed formed by the WF_6/Si reaction. Some of the silicon subfluorides adsorb on the nearby SiO_2 surface and then react with WF_6 to form SiF_4 (which desorbs) and a non-volatile tungsten subfluoride such as WF_4. This last step must occur at temperatures as low as ~30°C to be consistent with the observations described above. The second explanation is that a volatile tungsten subfluoride is formed directly at the silicon surface, as illustrated in reaction 10.

$$WF_6(g) + 1/4\ Si(s) \rightarrow WF_5(g) + 1/4\ SiF_4(g), \qquad \Delta H = \sim -24\ \text{kcal/mol}. \qquad (10)$$

The WF_5 adsorbs and disproportionates on the SiO_2 as in reaction 6 described in the preceding section. The disproportionation step is known to proceed around ~30°C [13,14]. This mechanism is identical to the one we described in the preceding section except that WF_5 formation occurs as a result of silicon rather than tungsten reduction of WF_6. For this second explanation to be consistent with the observation that exposed silicon is ~3 times "worse" than tungsten [12] for selectivity loss, the amount of WF_5 formed via silicon reduction must be appreciably greater than that formed by tungsten reduction. This would be entirely consistent with the thermochemistry since reaction 4 is slightly endothermic while reaction 10 is exothermic. This basically says that silicon is a stronger reducing agent towards WF_6 than is tungsten. While we tend to favor this latter explanation, neither explanation can be ruled out by the existing data. We believe a conclusive resolution of this issue requires an in situ spectroscopic technique that can detect gas-phase silicon and/or tungsten subfluorides, and that's no small order. Indirect measurements that examine tungsten particle densities as experimental conditions are varied (e.g. swaping wafer types) can not be used to conclusively identify the reaction intermediates.

Origins of the Insulator Dependence

Numerous studies have shown that some insulator surfaces are much better at maintaining selectivity than others [10,23], with the phosphorus-doped oxides normally giving the best behavior. There is currently no direct experimental evidence which explains the origins of the insulator dependence. We can suggest qualitative explanations for the insulator dependence which are based upon the tungsten subfluoride selectivity loss mechanism. One explanation is simply that the binding energy of WF_5 is different on each type of insulator surface. As the binding energy of WF_5 is lowered, the coverage of WF_5 will decrease and this in turn will lower the overall nucleation rate. A second possibility is that some insulator surfaces may chemically deactivate the tungsten subfluorides. For instance, phosphate groups in the phosphorus-doped oxides might react with and oxidize the tungsten subfluorides, effectively removing them from the selectivity loss mechanism. Studies of the tungsten subfluoride + insulator interactions are obviously necessary before the nature of the insulator dependence can be conclusively determined.

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Comments on the SiH_4 Reduction of WF_6

We are not aware of any direct experimental evidence concerning the selectivity loss mechanism during the silane reduction of WF_6. Evidence of a proximity effect is also lacking and it is conceivable that homogeneous chemistry may play an important role. Some obvious candidate precursors would be silicon and tungsten subfluorides and silicon subhydrides. The mechanistic information regarding tungsten subfluorides may not be directly applicable to the silane reduction system because the state of the growing surface may be appreciably different. While it is generally believed that the tungsten surface is predominately covered with fluorine during the hydrogen reduction of WF_6, the surface may instead be covered with silicon and hydrogen species during silane reduction. This would dramatically effect the evolution of tungsten subfluorides.

SUMMARY

Several proposed mechanisms of selectivity loss have been reviewed. Direct evidence for a mechanism involving HF is lacking. Also, chemical mechanisms that have been invoked that involve HF do not appear viable, since they do not lower the oxidation state of tungsten or silicon unless a highly endothermic (>90 kcal/mol) reaction step is taken.

The tungsten subfluoride mechanism is supported by direct and indirect experimental evidence. The mechanism involves the reaction of WF_6 with elemental W to form a volatile tungsten subfluoride, WF_5. Disproportionation of WF_5 on oxide surfaces eventually produces elemental tungsten which represents the initial stage of selectivity loss. The first step in the process (WF_5 formation) is only slightly endothermic and has much experimental support in the literature. We had previously measured the rate of this reaction down to 600°C and used the measured activation energy (~23 kcal/mol) to estimate rates down to 300-400°C. The extrapolated rates indicate that subfluoride production could easily explain the magnitude of selectivity loss at "typical" CVD temperatures. Using a new technique we have verified the previous rate measurements and extended the measurement range down to 450°C.

It has been reported that when elemental silicon is present selectivity loss occurs more rapidly. Preliminary examination of selectivity loss when silicon is present did not yield direct evidence of silicon subfluorides. Instead, we isolated a small amount of a tungsten subfluoride with a nominal WF_4 stoichiometry. We presently favor the explanation that elemental silicon enhances the formation of tungsten subfluorides although we cannot conclusively rule out an alternative mechanism involving volatile silicon subfluorides.

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