

SEMI F79-0703

GUIDELINE FOR GAS COMPATIBILITY WITH SILICON USED IN GAS DISTRIBUTION COMPONENTS

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1 Purpose

1.1 The purpose of this guideline is to identify resource information on compatibility of gases in contact with silicon in the wetted path of a gas delivery system operating at typical gas stick conditions.

2 Scope

2.1 The information and conclusions provided are taken from published literature. References are cited. No opinion is made as to the validity of the published conclusions. The suggestions are specific to high purity silicon, single or poly crystal, covered by native oxide.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Limitations (Prescribed Conditions)

3.1 The guideline applies to the gas distribution system for a typical semiconductor process tool. It is assumed that silicon is in the wetted path as a coating or a silicon-based component (eg. a valve, orifice, sensor, mass flow controller) in the gas distribution system. The ambient temperature and operating temperature limits are typical for the industry. No additional energy source, thermal, radiative or ionizing, is present. The moisture content of the gas is not specified for the purpose of this guideline; the recommendations apply whether or not the conditions are anhydrous.

4 Referenced Standards

4.1 SEMI Standard

SEMI E52 — Practice for Referencing Gases Used in Digital Mass Flow Controllers

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Classification of Gases

5.1 The gases included are taken from SEMI E52; the number assigned to a gas is the same as in that document.

5.1.1 Inerts

5.1.2 Hydrogen

5.1.3 Hydrocarbons

5.1.4 Halogenated Hydrocarbons

5.1.5 Hydrides

5.1.6 Halogens, other than Fluorine

5.1.7 Halides, other than Fluorides

5.1.8 Fluorine and Fluorides

5.1.9 Organo-metallic and Siloxanes

5.1.10 Oxygen and Oxides and Sulfides

5.1.11 Nitrogen and Nitrogen Compounds

5.1.12 Acids

5.1.13 Other

6 Review of the literature

6.1 Inerts

6.1.1 References (1, 2, 3, 4, 5) are cited which indicate these gases do not react with silicon under the prescribed conditions.

6.2 Hydrogen

6.2.1 References (1, 2, 3, 4, 5) are cited which indicate these gases do not react with silicon under the prescribed conditions. Hydrogen has been reported to react with silicon (75) and silicon carbide (76) at temperatures above 1100°C.

6.3 Hydrocarbons

6.3.1 References (1, 2, 3, 4, 5, 55, 56) are cited which indicate these gases do not react with silicon under the prescribed conditions.

6.4 Halogenated Hydrocarbons

6.4.1 References (1, 2, 3, 4, 5, 35, 51, 54, 55, 56, 57, 58, 59) are cited which indicate these gases do not react with silicon under the prescribed conditions. Extensive review papers by Flamm (55) and Coburn (56) cite numerous references on the etching of silicon and other materials using halogenated hydrocarbons. The critical requirement for etching of silicon, or any material using

a halogenated hydrocarbon, is a radical specie of fluorine, chlorine or other halogen. A low pressure plasma is the typical method of creating this radical. In addition, Winters (78) points out that for an “unassisted” etching reaction to take place adsorption onto the silicon surface must be followed by four well defined steps. Should any of the steps not occur, or be interrupted, etching will not proceed.

6.4.2 Two of the most widely used halogenated hydrocarbons are CF_4 and C_4F_8 . The Matheson Gas Book (79) says this about the chemical properties of CF_4 , “...Carbon tetrafluoride is extremely stable, reacting only slightly even at the temperature of a carbon arc...”. About C_4F_8 , “...Octafluorocyclobutane is extremely stable. It is unreactive with other materials under ordinary conditions. At high temperatures (600°C), it dissociates to form carbon and carbon tetrafluoride and some toxic compounds...”

6.5 Hydrides

6.5.1 References (1, 2, 3, 4, 5, 64, 65, 66) are cited which indicate these gases do not react with silicon under the prescribed conditions.

6.6 Halogens, other than fluorine

6.6.1 The halogens represent a special class of materials. Molecular or atomic chlorine, iodine or bromine have been reported not to etch silicon at temperatures below 200°C or without the assistance of a plasma (19, 26, 50, 55, 56, 63). Schwartz (74) makes the comment. “Although Cl_2 chemisorbs spontaneously on silicon, (in a plasma) at low temperatures the reaction between Si and Cl does not proceed to the formation of SiCl_4 . That is, silicon does not etch in Cl_2 except at elevated temperature.” He goes on to note: “...except in the case of highly doped silicon, etching occurs at low temperatures in the reactive ion etching system not because of the ease of Cl attachment but because partially reacted silicon can be sputtered readily.” Schwartz then reports the observation that there was no perceptible etching after thirty minutes in a Cl_2 plasma when there was no cathode bias on the wafer; in contrast to 4.5 microns of removal when the bias was applied.

6.6.2 Flamm (55) published in 1981 and titled “The Design of Plasma Etchants”, states, “...Chlorine containing halocarbon feed gases are frequently used instead of Cl_2 because they are not hazardous (before reaction), are noncorrosive, and they decompose in the plasma to generate radicals (in addition to chlorine atoms) which induce some desired effects.” This statement applies to a plasma based system; Flamm is pointing out the difficulty of etching silicon with just Cl_2 or Br_2 in a plasma. He points out that Wang and Maydan (from a private communication in 1979) have

etched silicon in a pure Cl_2 plasma at pressures below 0.05 torr.

6.6.3 Flamm goes on to say that, “...It seems that chlorine and bromine atoms do not spontaneously etch SiO_2 or undoped single crystal silicon but do etch some forms of polycrystalline silicon and heavily n-doped single crystal and polycrystalline silicon...”

6.6.4 A conclusion here is that even with a plasma Cl_2 and Br_2 require very specialized conditions to etch silicon.

6.7 Halides, Other Than Fluorides

6.7.1 Gaseous mixtures of HCl , HBr or HI will not etch silicon at the prescribed conditions (1, 2, 3, 4, 5, 62). However, mild pitting of the silicon may be observed in HBr or HI under aqueous conditions (5). In laboratory testing (10) no reaction of the silicon was noted with 50% aqueous HBr after 45 days immersion.

6.7.2 One reason for the lack of reactivity of silicon with chlorine, bromine or iodine halides is the low vapor pressure of the reaction product. SiCl_4 has the lowest boiling point at 57°C ; SiBr_4 boils at 154°C and SiI_4 at 286°C . On the other hand, the boiling point of SiF_4 is -86°C . Practical advantages of this low vapor pressure and non-reactivity were demonstrated by Texas Instruments in their patents (24, 25) on RIE etching; controlled additions of HBr , HI , BCl_3 , among other gases, led to passivating films of SiBr_4 , etc. on the silicon side walls and allowed vertical wall etching to be achieved.

6.7.3 The moisture content of the gas stream can be a critical factor in the corrosion resistance of most materials. Typical stainless steel passivity is strongly dependent upon the amount of H_2O present, declining sharply as water content increases above 1 ppm in the presence of halogens or halides (6, 9, 10, 72). Paciej (72) reported that 0.1 ppm moisture in HCl does not attack 316L stainless, while 200 ppm moisture in HCl can significantly corrode the surface of this material. Fine (9) points out similar behavior for 316L in HBr at 0.5 ppm and 100 ppm moisture over the course of 10 days.

6.7.4 However, silicon corrosion resistance is virtually unaffected by moisture content up to and including aqueous solutions. The “RCA clean” (67) and conventional wet etches (60, 68, 69) are obvious examples of this.

6.7.5 Silicon based halides, such as SiCl_4 , SiH_2Cl_2 , etc. have not been reported to react with silicon under the prescribed conditions. (55)

6.8 Fluorine and Fluorides

6.8.1 It is documented that fluorine (F_2) and atomic fluorine, F, will attack silicon at room temperature (19, 22, 26, 42, 49).

6.8.2 Chen (49) used fluorine gas (F_2) at room temperature on freshly cleaned silicon; he measured an etch rate of about $100 \text{ \AA}/\text{min}$ at 2.3 torr and 5 sccm of F_2 flowing. He also noted a two to three hour incubation period before the etching started; the authors speculate about the causes for this incubation period.

6.8.3 Flamm (42) and Vasile (22) authored several papers detailing the mechanisms involved in atomic fluorine reacting with silicon. Flamm's data produced the following relation for the etch rate of silicon by atomic fluorine.

6.8.4 $R(\text{Si}) = 2.91 \cdot 10^{-12} \cdot n_F T^{1/2} e^{-E_{\text{etch}}/kT}$ where E_{etch} is given as 0.108 eV. At $n_F = 2.9 \cdot 10^{15} \text{ atoms/cc}$, the etch rate is reported to be about $3,000 \text{ \AA}/\text{min}$ at 25°C .

6.8.5 Flamm used an RF discharge of at least 3.8 watts to produce F from F_2 . Vasile used thermal dissociation of F_2 to produce F; at 800°C he measured 47% dissociation; below 650°C , negligible.

6.8.6 Flamm (55) also reports a similar relation for the etch rate of SiO_2 by atomic fluorine: $R(\text{SiO}_2) = 6.14 \cdot 10^{-13} \cdot n_F T^{1/2} e^{-E_{\text{etch}}/kT}$ where E_{etch} is given as 0.163 eV.

6.8.7 Flamm (55) states "...Fluorine atoms react spontaneously with all forms of silicon, SiO_2 and silicon nitride to form volatile products."

6.8.8 Ibbotson (19) examined potential etching processes which required no external energy sources. "Silicon is rapidly etched by the gas-phase halogen fluorides ClF_3 , BrF_3 , BrF_5 , and IF_5 , in analogy to XeF_2 etching silicon..... By contrast, ClF and Groups III and V fluorides such as NF_3 , BF_3 , PF_3 and PF_5 do not spontaneously etch either Si or SiO_2 under the same experimental conditions."

6.8.9 Winters (21) and Ibbotson (20) report that XeF_2 does not etch SiO_2 . Regardless, silicon, with or without additional oxide, is not recommended for use with F_2 , XeF_2 , ClF_3 and the other fluorine containing interhalogens which may spontaneously decompose to atomic fluorine at temperatures below 200°C .

6.8.10 As quoted from Ibbotson's article above, excluded from this list of reactive gases are compounds such as NF_3 , BF_3 , PF_3 and PF_5 which have been reported not to etch Si or SiO_2 without the assistance of a plasma or RF source (19) or require temperatures above 200°C (1, 35, 37, 38).

6.8.11 The ionized fluorine atom, F^+ , is reported to not react with silicon under the prescribed conditions (1, 2, 3, 4, 5, 19, 64, 65, 66). Further evidence of this is the fact that adding H_2 to fluorine containing plasmas reduces the etch rate (55). The assumed mechanism is the removal of F atoms as active species.

6.8.12 Other fluoride compounds, such as SF_6 , which do not spontaneously decompose to atomic fluorine below 200°C are not a source of reaction (33, 34, 52, 53, 55).

6.8.13 WF_6 is a special case. Numerous articles (41, 43, 44, 45, 46, 47) have been published on the CVD of tungsten films from WF_6 . Two primary mechanisms are cited; the reduction of WF_6 by silicon and the thermal decomposition in the presence of H_2 . As stated in Yarmoff's paper (43):

"The dissociative chemisorption of WF_6 on Si (111) was found to be complete, even at room temperature. The reaction is self-poisoning at room temperature, however as the fluorine liberated from WF_6 ties up the active Si sites responsible for the dissociation."

6.8.14 Tsao (41) reports that at 410°C only $200 \text{ \AA}$ of W will deposit from WF_6 onto single crystal silicon which has an oxide thickness between 5 and $15 \text{ \AA}$. C.A. van der Jeugel (46) describes the effect of doping levels on the self-limiting growth of tungsten films. It is documented (44, 45, 47) that WF_6 will not deposit on SiO_2 without the addition of hydrogen or some other initiation mechanism.

6.8.15 Using the selective deposition characteristic of WF_6 in a device, Fleming, et.al. have patented (48) a technique for producing wear resistant coatings of tungsten. Their invention requires a "clean" surface and a temperature higher than 200°C , preferably around 450°C ; exposure to WF_6 then results in a self-terminating film of 5–50 nm.

6.8.15.1 Zdunek (80) reported on electrochemical based work which indicated that unprotected silicon surfaces exhibited "no degradation" after exposure to WF_6 for 5 days at 80°C . A similar result was observed for Cl_2 gas.

6.8.16 As a point of interest, metals such as stainless steel, aluminum and nickel develop corrosion resistant coatings of the respective fluoride when exposed to atomic fluorine during the proper passivation procedure (14, 23, 27, 28, 29, 30, 31, 32, 35, 39, 40).

6.8.17 Titanium, molybdenum, tungsten, brass and columbium have been reported to be unacceptable when exposed to gases such as ClF_3 , which decompose to atomic fluorine (27, 32).

In summary, it is recommended that neither silicon, silicon dioxide nor silicon nitride be in the wetted path when fluorine, F_2 , or the halogen fluorides, ClF_3 , BrF_3 , BrF_5 , and IF_5 , plus XeF_2 are in use.

6.9 *Organo-metallic and Siloxanes*

6.9.1 References (1, 2, 3, 4, 5, 64, 65, 66) are cited which indicate these compounds do not react with silicon under the prescribed conditions.

6.10 *Oxygen and Oxides and Sulfides*

6.10.1 References (1, 2, 3, 4, 5, 64, 65, 66) are cited which indicate these gases do not react with silicon under the prescribed conditions.

6.11 *Nitrogen and Nitrogen Compounds*

6.11.1 References (1, 2, 3, 4, 5, 64, 65, 66) are cited which indicate these gases do not react with silicon under the prescribed conditions.

6.12 *Acids*

6.12.1 Aqueous HF is the only single acid reported to etch silicon (70). Combinations of fluoride ions in solution with an oxidizing acid such as HNO_3 in an aqueous solution will etch silicon at practical rates (5, 54, 60, 64, 65, 66, 67, 68, 69).

6.12.2 Madou (70) has an excellent review of the mechanisms required for aqueous based etching; he cites Hu and Kerr (72) to report an etch rate of $0.3^\circ A/min$. for n-type, 2 ohm-cm (111) silicon in a 48% HF solution at $25^\circ C$. Madou points out the strong dependence of etch rate in aqueous solutions on doping level, light conditions and relative potential.

NOTE 1: Most bases will etch silicon and silicon dioxide; the oxide is etched very slowly.

6.13 *Other*

6.13.1 As currently designated these are materials in SEMI E52-0302 not encountered in mass flow controllers under typical conditions. The manufacturer should be consulted.

7 **Cautions and Warnings**

7.1 The reader is specifically requested to consult the MSDS and the manufacturer's recommended practices of any gas or liquid prior to use and to follow the suggestions prescribed.

7.1.1 All other appropriate safety procedures should be followed as well.

8 **Related Documents**

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