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WET ETCHING OF SILICON DIOXIDE

SiO2 films have two main roles in microtechnologies: as a dielectric layer or as a doping/etching mask. In both cases, patterning is usually necessary. SiO2 is named "thermal" when obtained in a high temperature oven by oxidation of silicon substrate. Otherwise, it is obtained by Chemical Vapor Deposition (CVD) as an added layer , with no need of a silicon substrate.

The oxide thickness usually ranges between 100 nm and 1000 nm (that is respectively 1000 Å and 10000 Å, being the use of angstrom units still very popular in thin film technology).

It can be easily etched with chemicals having negligible effect on silicon. Moreover, many silicon etchants do not effect the oxide. Such possibilities are extensively used in all silicon-based microtechnologies.

If the oxide is used as a high temperature mask for silicon processing, it has to be previously patterned at low temperature, with a resist-based microlithographic process.

When a room temperature process must be made on silicon, the use of the oxide mask could be avoided, that is the resist can act direct as a mask for silicon. However, there are chemicals which can easily etch the resist as well as the silicon. A typical example is given by the anisothopic etch of silicon, which is usually made in KOH (potassium hydroxide). In this case, diluited KOH is also the typical development liquid for positive resist. Hence the etching process should be carried out in yellow ligh, to which the resist is insensitive. This is not practical. But even so, since an acceptable etching rate for silicon require concentrated KOH, again the resist cannot be used, because undiluited KOH removes the resist even if it has not been exposed.

A very "selective" chemical for SiO2 - i.e. does not etch silicon at all - is hydrofluoric acid (HF). If used directly such etchant has a too fast and aggresive action on the oxide, making very difficult the undercut and the linewidth control. For such reason, HF is universally used as a "buffered" solution, which can keep the etch rate low and constant, by moderating the PH level of the bath. This allows the etching time to be reliably correlated to the etching depth.

The industry standard buffered hydrofluoric acid solution (BHF) has the following formulation:

- 6 volumes of ammonium floride (NH4F, 40% solution) - 1 volume of HF.

This can be prepared, for example, by mixing 113 g of NH4F in 170 ml of H2O, and adding 28 ml of HF. The etch rate at room temperature can range from 1000 to 2500 Å/min. This depends on the actual density of the oxide which, as an amorphous layer, can have a more compact structure (if thermally grown in is oxygen) or less compact (if grown by CVD). The following etching reaction holds:

$SiO_2 + 6HF \rightarrow H_2SiF_6 + H_2O$

where H_2SiF_6 is water soluble.

Sometimes the BHF is prepared and stored according to the formulation given above, but it is 7:1 diluited in water just before being used. This allows an even better control of the etching rate. It is good practice to use the diluited BHF only once and then discard it, in order to assure process

repeatability. If a diluited BHF bath at 35°C is used, the etching rate for thermal oxide is around 800Å/min.

Another popular etching formulation is the P-etch:

60 volumes of H_2O + 3 vol. of HF + 2 vol. of HNO_3, that is:

300 ml of H_2O + 15 ml of HF + 10 ml of HNO_3.

The P-etch action is strongly dependent on oxide density, as it results from the growth technique. An example is reported in the literature (W.A. Pliskin, J.Vac.Sci Technol., vol. 14, p.1064, 1977), indicating 120 Å/min for thermal oxide and 250-700 Å/min for sputtered oxide.

A slow etching bath is preferred for opening mask windows for a silicon substrate. However, the etching process could be used just for removing the oxide film from the whole surface. In this case the etching speed is not critical, and a fast solution can be used, such as HF diluited 1:10 in water. The etching time can be easily evaluated by visually inspecting the surface. Once the oxide film is removed, the metal-grey color of the silicon surface appears.

Sometimes a very light etch is required, for removing just a few atomic layers. This is the case of surface cleaning and decontamination. HF diluited 1 : 50 in water can be used. The etching speed will be around 70 Å / min. For example, a typical 50 Å "native" oxide on silicon can be removed with a 45 - 50 sec light etch.

Note that both BHF and diluited HF must be handled and used only in polyethylene or Teflon containers. Any glass material would be etched as well. This, besides the potential danger for the operator, would be a pollution source for the etching.
