

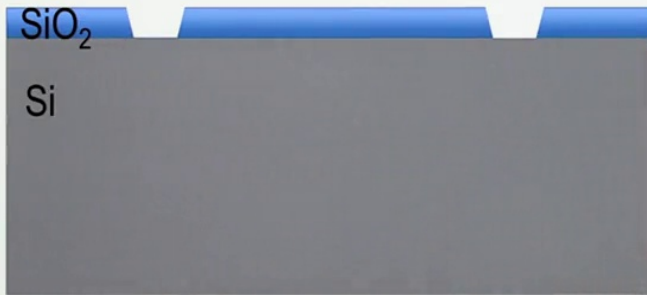


- Supercritical drying
- Microstructures for quantifying stress in thin layers

Micro and Nanofabrication (MEMS)

In our introductory lesson on Wet Etching, we have already introduced the concept of *surface micromachining* which consisted in removing a silicon dioxide thin film from underneath a polysilicon layer so that the latter becomes a suspended functional structure. In this lesson, we will present an issue with this technique that is related to the surface tension of the drying etching liquid which exerts a force on the suspended silicon structure so that the latter can be permanently deformed. *Supercritical drying* is a technique in which such collapse of the functional silicon structure can be avoided. Another major issue with surface micromachining is the existence of stress in the functional layer which becomes apparent after removal of the sacrificial layer and results in undesirable deformed microstructures. Here, we will introduce some test microstructures that are dedicated for quantifying stress in thin layers.

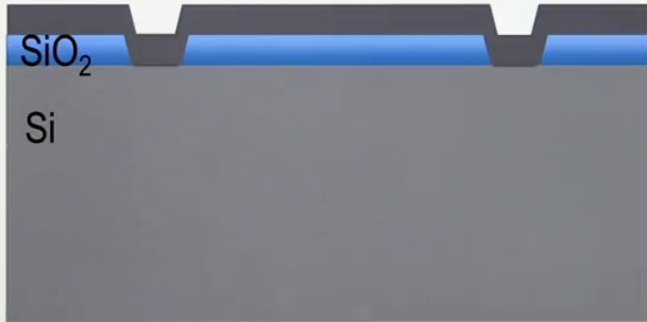
Etching of sacrificial layers



- Surface machining requires removing a sacrificial layer beneath a functional layer
- Step 1: patterning of SiO₂ sacrificial layer

We sketch here again a typical surface micromachining sequence. In surface micromachining, one needs to remove the sacrificial layer from beneath the functional layer. Step one of such a process is the patterning of the silicon dioxide sacrificial layer. So we have patterned it, and there are two parts where the silicon dioxide has been removed.

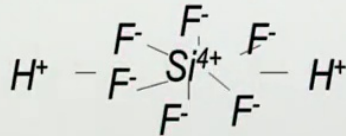
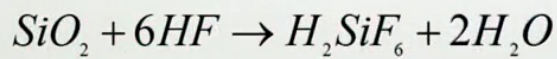
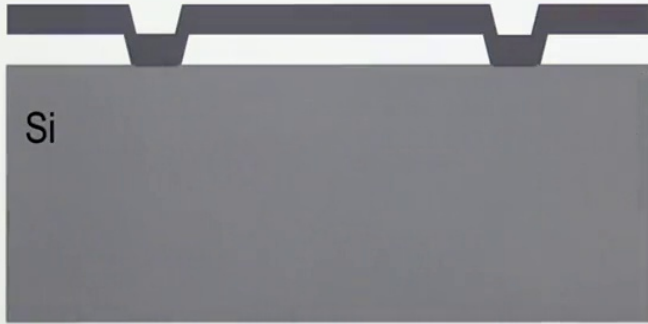
Etching of sacrificial layers



- Surface machining requires removing a sacrificial layer beneath a functional layer
- Step 1: patterning of SiO₂ sacrificial layer
- Step 2: deposition of polySi layer (for example by LPCVD)

Step two is the deposition of a polysilicon layer, which is a deposition that is conformal to the texture which is already present on the substrate, and it can be performed, for example, by a low-pressure chemical vapor deposition step.

Etching of sacrificial layers



- Surface machining requires removing a sacrificial layer beneath a functional layer
- Step 1: patterning of SiO₂ sacrificial layer
- Step 2: deposition of polySi layer (for example by LPCVD)
- Step 3: wet etching of SiO₂ in a HF bath forming fluorosilicic acid (H₂SiF₆) and water

Etching in the HF liquid is based on the formation of the fluorosilicic acid, schematically represented here, that goes into the solution. The drawing here is shown after removal of the wafer out of the etching bath and after rinsing in deionized water. This is the ideal case where everything worked out fine.

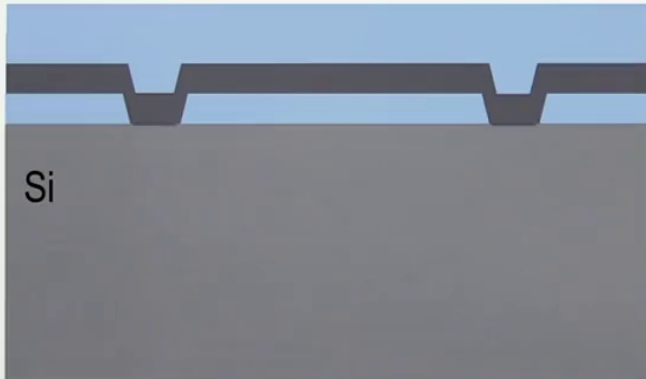
Etching of sacrificial layers



- Reality may be different from the ideal case: during drying of the etching or rinsing solution, the microstructure is pulled down by capillary forces, often leading to unwanted permanent deformation
- Solution to this problem: **supercritical point drying**

However, in reality, the situation may be different. During drying of the etching or rinsing solution, a limited amount of liquid underneath the functional structure can pull down the latter towards the substrate by capillary forces. Often, after such collapse, the functional structure cannot be released again and can, hence, not be used in the application. A solution to avoid such problem is the technique of *supercritical point drying*.

Supercritical point drying

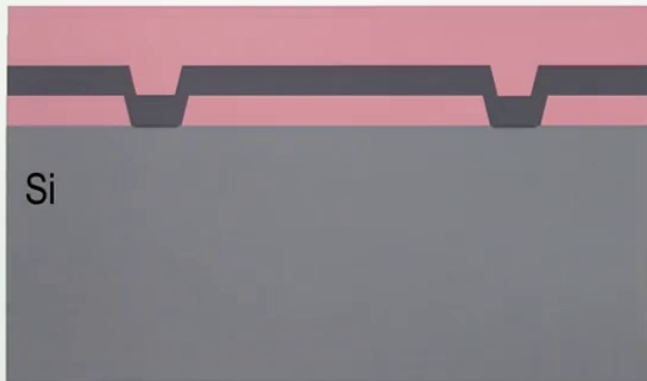


- Sacrificial layer is etched in **HF bath**
- HF is replaced by **deionized water**

Micro and Nanofabrication (MEMS)

In this technique we etch the sacrificial layer in HF bath as schematically illustrated here. Then we replace the HF by deionized water, whereby, the liquid is replaced by a liquid, and no drying is involved.

Supercritical point drying



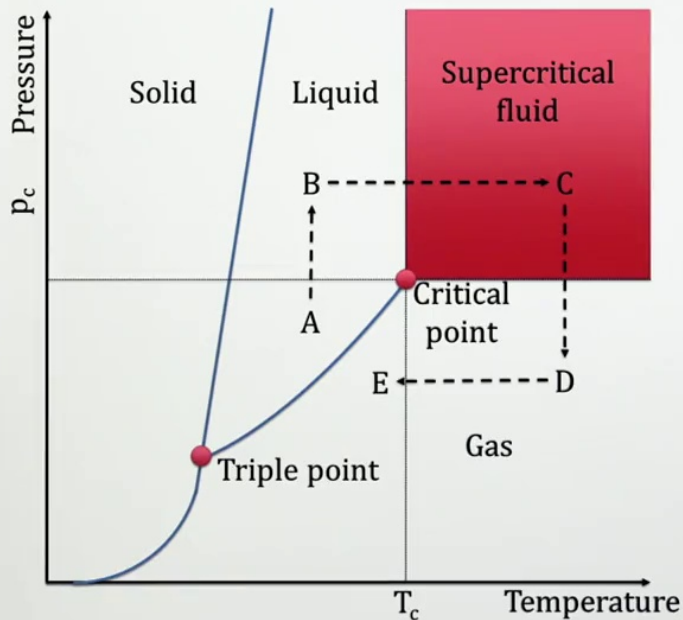
- Sacrificial layer is etched in **HF bath**
- HF is replaced by **deionized water**
- Water is replaced by **ethyl alcohol**
- The wafer is placed in a closed chamber that is filled with **CO₂ gas**, which will be condensed into a liquid



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In the next step, in a similar way, we replace the water by *ethyl alcohol*, which is a liquid with lower surface tension and higher vapor pressure than water. This wafer is then placed in a closed chamber that can be filled with CO₂ gas, to which one can apply high pressure so that the gas is condensed in liquid CO₂ that will occupy, then, also, the space between the functional layer and the substrate. So this will be replaced, this liquid, by liquid CO₂. The picture shows the stainless steel pressurizable vessel in which the wafer is positioned.

Supercritical point drying

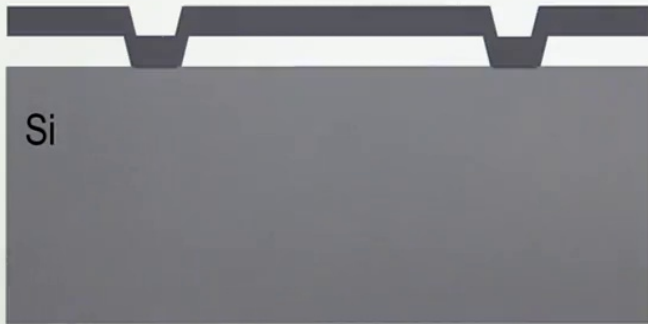


- The chamber is brought at $T=10\text{ }^{\circ}\text{C}$
- It is pressurized to 75 bar (A→B)
- The temperature is raised to $T=31\text{ }^{\circ}\text{C}$ (critical point temperature of CO_2) (B→C); the pressure rises to 93 bar
- A supercritical fluid is formed, there is no distinction between liquid and gas phase, hence no surface tension
- The pressure is dropped during a 4 min process (C→D), the CO_2 evaporates
- The chamber is brought to room temperature (D→E)

Micro and Nanofabrication (MEMS)

Here we show a phase diagram for CO_2 . On the *x-axis*, there is the temperature, and on the *y-axis*, the pressure. At high temperature and low pressure, the CO_2 is in the gas state, while at low temperature, the CO_2 is in the solid state, and in the intermediate temperature range, the CO_2 is in the liquid state. At low pressure there can be transfer from the solid to the gas phase by sublimation. Let us now discuss the *supercritical point drying process*. The chamber is brought to 10 degrees Celsius and then the chamber is pressurized to 75 bar. This means we go from A to B in the phase diagram. Then we raise the temperature to 31 degrees Celsius, which is the critical point temperature of CO_2 , this temperature. This means that we go from B to C in the diagram, and, in fact, our point C is just above this critical point temperature. Due to this heating, the pressure rises further to 93 bar in the chamber. As when it's just above the critical point of CO_2 , a supercritical fluid is formed in which there is no distinction between the liquid and gas phase anymore and, hence, there is no surface tension. Then one releases the pressure and one brings the CO_2 in the gas phase going from C to D, during which, the CO_2 evaporates. Finally, after removal of all CO_2 , the chamber is brought to room temperature going from D to E, this region. We have now avoided the evaporation of CO_2 from the liquid phase during the drying process, and we have removed all CO_2 .

Stress in surface-machined microstructures



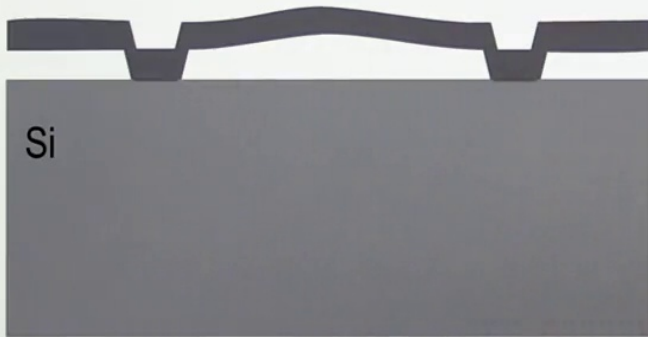
- Deposition of the polySi layer is done at elevated temperature (for example by LPCVD)
- When cooling down to room temperature and after release of the sacrificial layer, due to different thermal dilatation between film and substrate, stress may develop

• Stress may be compressive

Micro and Nanofabrication (MEMS)

Another issue in surface micromachining is the presence of stress in the surface machined microstructures. This results from the fact that the polysilicon layer is deposited at a high temperature, and that, when cooling down to room temperature, the thermal expansion coefficient of the thick substrate and the thin deposit layer is not the same. The stress becomes most apparent after removal of the sacrificial layer and it is visible by deformed membranes and beam structures.

Stress in surface-machined microstructures

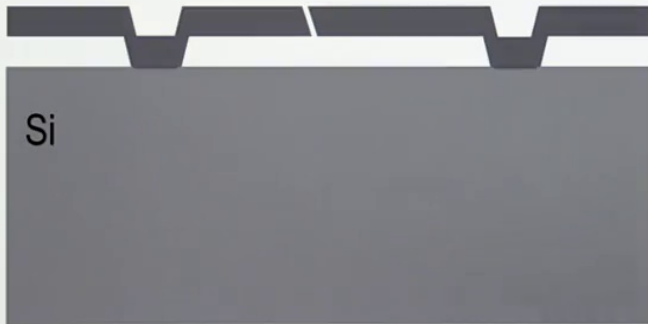


- Deposition of the polySi layer is done at elevated temperature (for example by LPCVD)
- When cooling down to room temperature and after release of the sacrificial layer, due to different thermal dilatation between film and substrate, stress may develop
- Stress may be **compressive**

Micro and Nanofabrication (MEMS)

The stress may be compressive, and this happens when the substrate, during cooling, shrinks down more than the thin film, resulting in the type of structure shown here in the schematic diagram.

Stress in surface-machined microstructures



- Deposition of the polySi layer is done at elevated temperature (for example by LPCVD)
- When cooling down to room temperature and after release of the sacrificial layer, due to different thermal dilatation between film and substrate, stress may develop
- or **tensile**, which is less visible, but eventually the microstructure breaks
- Stress is to be avoided → adaptation of thin film process needed

Micro and Nanofabrication (MEMS)

The stress can also be tensile, and this happens when the thin film would like to shrink down more than the substrate, but it's kept under tension by the contact with the substrate at these two points. In such case, it may be that one cannot see the tensile stress because it will result in a straight beam or straight membrane, just like a microstructure without stress. Only in case the stress is so high that catastrophic failure of the thin film occurs, it becomes visible. Hence, generally, it is hard to detect tensile stress as it is less easy to observe. Suppose one can detect stress, then this information will be used to fine-tune the deposition process, vary parameters, and eventually do annealing treatments of the structures to reduce the stress.

Ring crossbar test structures



- Series of suspended microstructures made in polySi (the functional material in general) and of increasing size, each fixed to the substrate by two anchor points
- If there is no stress in the polySi, the microstructures stay completely flat after removal of the sacrificial layer underneath

Micro and Nanofabrication (MEMS)

So-called *ring crossbar test structures* are very useful for detecting and quantifying the stress in the thin film material. The size of these structures is in the range of between tens of microns to several hundreds of microns and one designs them typically in a row of structures of increasing size. The microstructures consist of a ring with connecting crossbar that are suspended because one has removed the sacrificial layer underneath. Fixation on the substrate is achieved by two anchor points for each ring. If there is no stress in the suspended polysilicon ring crossbar structure, and if one removes the sacrificial layer underneath, the structures stay nicely flat.

Ring crossbar test structures

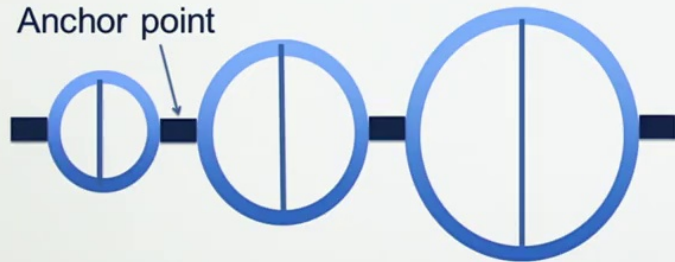


- **Tensile stress:** the ring shrinks down, but is fixed at the two anchor points, hence shrinks down more than the crossbar in the y-direction
- Both the ring and crossbar are deformed
- The bigger the microstructure, the more easy deformation is. The size at which deformation occurs allows quantifying the tensile stress

Micro and Nanofabrication (MEMS)

If these structures are under tensile stress, all suspended materials want to shrink down. The ring is fixed at two anchor points, hence, will shrink down more than the crossbar in the *y-direction*. So the shrinkage of the *x-direction* is, in a way, transferred to the *y-direction*. A result is that the crossbar in the middle will be deformed. It will shrink down too, but not as much as the ring structure in this direction. Planar microstructures are easily recognizable as such when looking through a microscope. Also, the bigger the microstructure, the more easy deformation is. That is why one makes a series of microstructures of increasing size, to look at the size of the ring crossbar structure where deformation will first develop. In this case, this ring is okay, and here, this is the first ring which shows deformation. This allows quantifying the tensile stress.

Ring crossbar test structures



- **Compressive stress:** the ring expands, but is fixed at the two anchor points, hence expands more than the crossbar in the y-direction
- The ring is deformed but the crossbar remains straight
- The bigger the microstructure, the more easy deformation is. The size at which ring deformation occurs allows quantifying the compressive stress

Micro and Nanofabrication (MEMS)

We draw here again the test structures without stress. If these structures are under compressive stress, all suspended materials want to expand. The ring is again fixed at the two anchor points, hence, it will expand more than the crossbar in the *y-direction*. A result is that the crossbar will stay straight while the ring will be non-planar. The bigger the microstructure, the more easy deformation is. That is why one makes, again, this series of microstructures: to look at the size of the ring crossbar structure where deformation will first develop, and this allows quantifying the compressive stress. The ring crossbar test structure, hence, is a universal detector of stress that can either be of tensile or compressive nature.

Summary



- Mechanism of supercritical drying using CO_2 for avoiding collapse of surface-micromachined structures
- Ring crossbar structures for diagnosis of tensile or compressive stress in surface-micromachined functional layers

Micro and Nanofabrication (MEMS)

In this lesson we have explained the technique of supercritical drying using CO_2 by which evaporation from the liquid phase underneath a functional microstructure can be avoided, leading to surface micromachine structures that do not collapse towards a substrate because of the absence of surface tension-driven bending forces. Also, we introduced the phenomenon of intrinsic stress in the functional thin film materials and explained how ring crossbar microstructures can be used for diagnosis of both tensile and compressive stress.
