Thin Films

Thin films are an important basic element of microsystem design. They are used for masking in etching processes, as a diffusion stop layer, and as functional elements such as electrical conductors, membranes, and beams. Thin films may consist of nearly every rigid material. Typical examples are metals, polymers, oxides, and nitrides. The thickness of thin films typically is in the range of 50 nm – 10 μm. The lower limit is due to the problem that layers with an average thickness of less than 50 nm hardly are made homogeneously, because they tend to form separated clusters. The upper limit is a kind of convention. Films that are thicker than 10 μm are no longer considered to be thin in microtechnology, and they cannot be generated easily by processes such as sputtering and evaporation but need to be produced by, e.g., electroplating.

When dealing with thin films, stress is an important parameter which may make the difference between success and a flop of a newly developed microsystem. That is why stress control is important (or even a design which works at any stress level). This chapter describes how thin film stress affects their behavior, what are the consequences for parts fixed to the film, and how the stress can be altered.

It is almost impossible to deposit a thin film onto a substrate without any residual stress. The only exception is epitaxial growth. The reason for this is that the molecules of the deposited layer walk around on the substrate surface until an energetically low position is found. The mismatch in the crystalline structure of substrate and thin film material results in a strain of the crystal lattice of the thin film, and, therefore, generates some stress.

If a polymer layer is deposited by a process such as spin-coating or just painting, a different mechanism is working. A solvent is evaporating from the thin film and its dimension is reduced. As a consequence, some tensile stress is generated in the thin film.

Residual stress bends the substrate a little bit. Typically, the deflection by bending of a silicon wafer with a diameter of 100 mm and a thickness of 500 μm is on the order of 100 μm when the thickness of the thin film is 100 nm. On the macroscopic scale, this is of no importance. When a bridge is painted, nobody cares about the change in shape entailed with this. On the microscopic scale, however, this is an effect which needs to be considered.

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Substrate bowing is used to measure residual stress σ_0 of thin films. When the thickness d_f of the film is much less than the thickness d_{Su} of the substrate, the residual stress can be calculated approximately with Stoney's equation [9]:

$$\sigma_0 = \frac{E_{Su}}{6\,(1 - \nu_{Su})R_{Su}} \frac{d_{Su}^2}{d_f}. \tag{9}$$

As can be seen from (9), it is not necessary to know the elastic properties of the thin film which are hard to measure in general. It is sufficient to know Young's modulus E_{Su}, Poisson's ratio ν_{Su}, and the radius of curvature R_{Su} of the substrate. The radius of curvature of the substrate is easily measured with a surface profiler.

Thin films with a tensile residual stress tend to peel off the substrate [10], while compressive stress facilitates adhesion. As indicated in Fig. 4 (The figures are not up to scale allowing to recognize small dimensions next to larger ones.), a tensile stress pulls the upper part of the thin film away from the rim generating a bending moment which peels the film off the substrate. Therefore, in the light of good adhesion, compressive stress is desirable in thin films.

The stress has also an influence on the etching under a thin film used as a mask. As shown in Fig. 4, the curling down of a mask with compressive stress reduces the undesired etching under the mask while a tensile stress results in an enhanced sideward etching.

A thin film may also curl due to a *stress gradient*. Such gradients occur often when a thin film is deposited on a substrate. As described above, the mismatch in the crystalline structure of substrate and thin film material results in a strain in the crystals of the thin film generated. When the crystal growth is continued, the mismatch disappears and the natural lattice of the thin film is formed. This results in a residual stress with an absolute value getting smaller with increasing distance from the substrate. Figure 5 shows what happens if a sacrificial layer beneath a thin film is etched away when the film initially was under compressive stress decreasing with the distance from the substrate. The beams in the left part of Fig. 6 are curling up because of a gradient in their residual stress.

The radius R_f of curvature of a curling thin film with thickness d_f and Young's modulus E_f and a difference in stress $\Delta\sigma$ can be calculated with the following equation:

$$R_f = \frac{d_f\,E_f}{\Delta\sigma}. \tag{10}$$

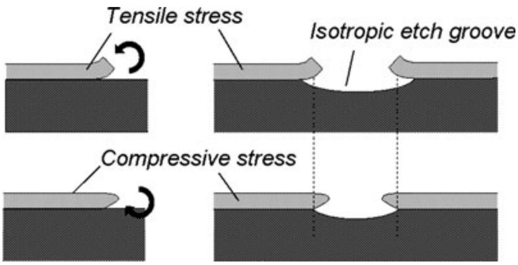


Fig. 4 Thin film on a substrate with tensile and compressive stress, respectively

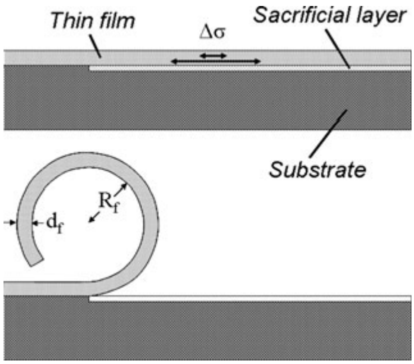


Fig. 5 Thin film on a substrate with a stress gradient after deposition (*top*) and after etching the sacrificial layer (*bottom*)

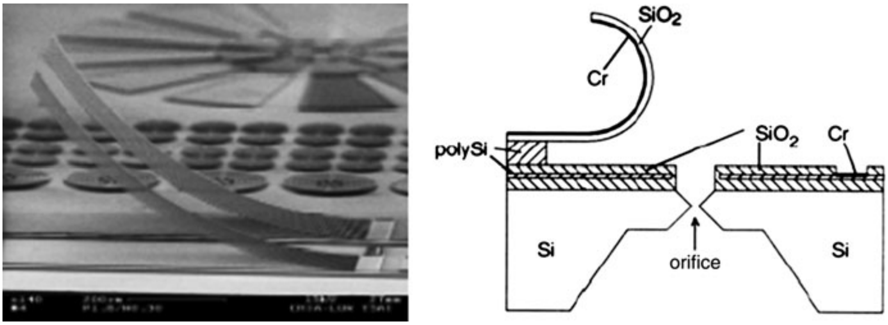


Fig. 6 Thin films bowing up (*left*) [7] and two thin films curling up and down as a design element in a microvalve (*right*) [8]. © [1997] IEEE

Thin films tend to deform membranes as well. If a membrane with tensile stress carries a thin film, it is very unlikely that the thin film shows no stress. Therefore, it has to be expected that bending moments in the film will deform the membrane. The upper part of Fig. 7 shows a thin film with compressive stress on a membrane which has tensile stress, because it is fixed at a frame (frame not shown in Fig. 7). The bending moments produce a deflection of the membrane in the vicinity of the rim of the thin film. Far away from the rim, there is nearly no bending of the membrane neither in the part uncovered with the thin film nor in the covered part.

If the thickness d_f of a thin film is much less than the thickness d_M of the membrane, the following equations can be used to calculate the deflection w of the membrane with a tensile stress σ_M as a function of the distance x from the center of the thin film, its length L_f, and its stress σ_f [11].

The deflection of the part of the membrane covered with the thin film $\left(|x| < \frac{1}{2} L_f\right)$ is described by:

$$w = \frac{1}{2}\frac{\sigma_f d_f}{\sigma_M d_M}(d_f + d_M)\left(1 + \left[\frac{1}{1 + \coth(L_f/(2\lambda_M))} - 1\right]\frac{\cosh(x/\lambda_M)}{\cosh(L_f/(2\lambda_M))}\right) \quad (11)$$

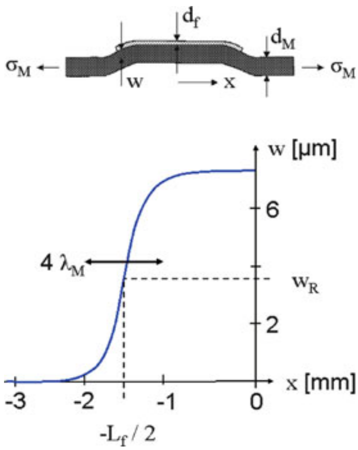


Fig. 7 Thin film on a substrate with compressive stress. *Top*: Schematic drawing; *bottom*: Deflection calculated with (11–14)

and the deflection of the uncovered part $\left(|x|>\frac{1}{2}\,L_f\right)$ is given by:

$$w = w_R\, e^{(-|x|+L_f/2)/\lambda_M} \tag{12}$$

with:

$$w_R = \frac{1}{2}\,\frac{\sigma_f\, d_f}{\sigma_M\, d_M}\,(d_f + d_M)\,\frac{1}{1+\coth\left(L_f/(2\,\lambda_M)\right)}. \tag{13}$$

The parameter λ_M in (11–13) is defined as:

$$\lambda_M = \frac{d_M}{2\,\sqrt{3}}\,\sqrt{\frac{E_M}{\sigma_M(1-\nu_M^2)}}. \tag{14}$$

The lower part of Fig. 7 shows the deflection of a membrane with a thickness of 5 µm, Young's modulus of 120 GPa, Poisson's ratio of 0.3, and a tensile stress of 10 MPa as calculated with (11–14). It is bent by a 1-µm thick thin film with a length of 3 mm. The parameters λ_M and w_R are 234 and 3.6 µm, respectively. As shown in Fig. 7, the bending of the membrane is restricted to a stripe with a width of approximately $4\lambda_M$ and the total deflection is $2w_R$. Note that the total deflection is larger than the thickness of the membrane!

Figure 8 displays the deflection parameter w_R calculated with (13) as a function of the length of the thin film. It is clearly visible that the total deflection due to the stress in a thin film on a membrane may be reduced substantially when the length of a continuous film is designed smaller than λ_M. So, an undesired deflection may be avoided by separating the thin film into parts smaller than λ_M.

A membrane will be bent also, if it is fixed with tensile stress to a frame on one side only and if there is no thin film on the membrane (cf. Fig. 9). The upper part of the membrane is relaxing a little bit by moving from the frame to the free-span membrane. Thus, a bending moment is generated which pushes the membrane down to the side of the frame.

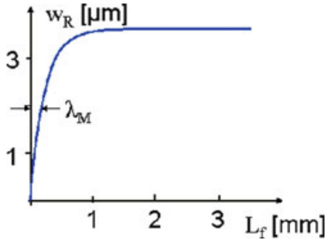


Fig. 8 Deflection parameter w_R calculated with (13)

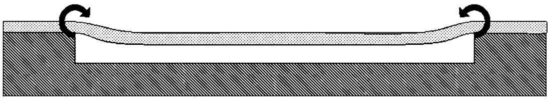


Fig. 9 Deflection of a membrane stretched with tensile stress over a frame

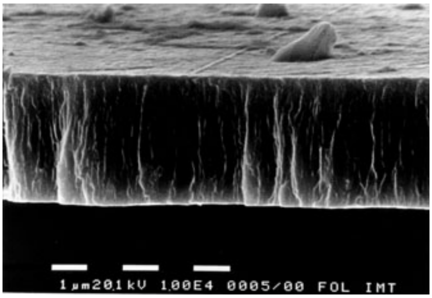


Fig. 10 SEM of a titanium membrane, 2.7 µm in thickness. (Courtesy of Karlsruhe Institute of Technology, KIT)

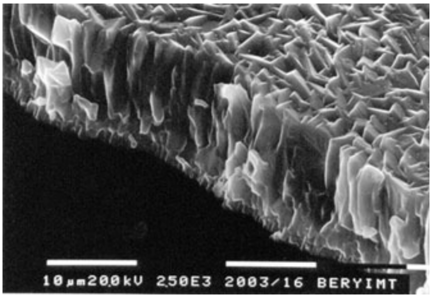


Fig. 11 SEM of a beryllium membrane, 10 µm in thickness. (Courtesy of Karlsruhe Institute of Technology, KIT)

Figures 10 and 11 show breaking edges of membranes sputtered from titanium and beryllium, respectively, as observed with a scanning electron microscope (SEM). It is clearly seen that the *crystalline structure* of these membranes is different. As a consequence, the properties of these membranes are different. Crystal growth during sputtering the beryllium started from the bottom in Fig. 11, and it is seen that at the bottom there are the smallest crystallites. The speed of growth is a function of the orientation of the crystal lattice. Some of the crystallites in the bottom layer are orientated such that they show a quicker growth and they grow over their neighbors. Therefore, the properties of such a thin film become more anisotropic when it is thicker.

In Fig. 10, the crystallites are very small and homogenous. The reason for this is that the crystalline structure was changed by exposing the thin film to high temperatures after sputtering. This has caused *recrystallization*, i.e,. the atoms of the material started to move at elevated temperatures and to change their position from one crystal to the other. This process is lent by the reduction of stress. Thus, the recrystallization continues until a certain stress level is achieved which does not induce further recrystallization.

This became evident in an experiment performed by Flinn [12]. He used silicon wafers on which a thin metal layer had been sputtered. The films showed tensile stress, and, therefore, the wafers were bent. Flinn used the curved thin film as a hollow mirror. The focal length of this mirror was a measure of wafer bending and thin film stress. This way, the stress of the thin film could be measured through the window of an oven and the change of the stress was recorded as a function of temperature. Figure 12 shows the result of this experiment for aluminum films on silicon wafers.

The experiment started at a thin film stress of approximately 100 MPa. Then the temperature was raised and the stress was reduced according to the larger thermal expansion of aluminum compared with silicon. As temperature continued to increase the thin film got compressive stress. At approximately 100°C and a stress of −70 MPa, recrystallization started and the compressive stress no longer changed with raising temperature or even reduced. At 450°C, heating was turned off and cooling down of the sample started. Again the stress was changing according to the difference in thermal expansion of aluminum and silicon until a certain tensile stress was reached and recrystallization reduced further stress enhancement.

A theoretical model of this experiment is shown in Fig. 13: During heating the stress follows the line from *A* to *B*. At *B*, a theoretical line is reached which marks the stress level as a function of temperature at which recrystallization starts. From *B* to *C* the stress follows this line. When cooling starts there is no longer enough stress for recrystallization and the stress follows the difference in thermal expansion until the tensile stress at *D* gets so large that the stress follows another line which marks the start of recrystallization.

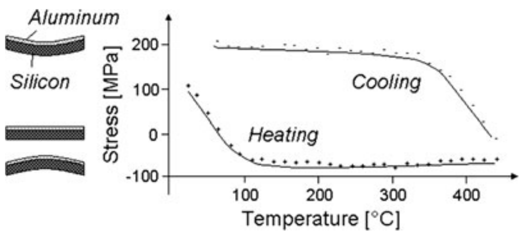


Fig. 12 Thin film stress as a function of heating and cooling (reproduced from [12])

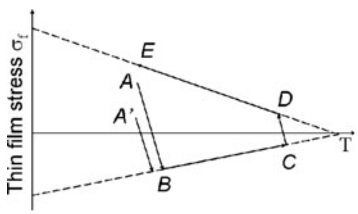


Fig. 13 Theoretical model of the experiment in Fig. 12 [12]

This experiment shows how the stress in thin films can be altered by a temperature cycle. When thin films are sputtered or evaporated onto substrates the conditions differ a bit as a function of the position in the machine. Therefore, thin film stress is not everywhere the same, neither on neighboring substrates in a machine nor on a single substrate. In Fig. 13, it can be seen how the stress of a whole batch may be adjusted to a common level. A thin film starting at A' instead of A will end up at the same stress at E as all the other films with different initial stress.

A stress gradient is reduced or vanishes after temperature cycling. Curling up of the thin films shown in the left part of Fig. 6 (on page 11) might have been avoided with a proper temperature cycle before release from the substrate. The titanium membrane shown in Fig. 10 before separation from the substrate underwent a temperature cycle for 30 min at 450°C. As a result, the membrane stress was changed from compressive to tensile and no more stress gradient was found. The figure shows also the homogeneous crystalline structure generated this way.

The crystalline structure affects other properties of thin films also. One example is the electrical resistance. Usually, conductor metal paths are exposed to a temperature cycle to avoid a later change of the resistance when it may get heated during use.

The strength of thin films is also a function of their crystalline structure. Thin films which have been sputtered or evaporated typically show a larger strength than the casted material. Recrystallization will reduce this strength.

The density of thin films after deposition is approximately 10% less than the density of the same material after casting. Recrystallization increases the density.

Other properties of thin films, which are important for their application, are adhesion to the substrate and other layers, diffusion of molecules and atoms into and through the film, wettening by liquids, chemical reactivity, and thermal conductivity.

Exercises

Problem 1

In Figures E1–E4, you find four typical applications from microtechnique. In these applications, basic elements have been employed which you have got to know in the lecture. Find out which are these basic elements.

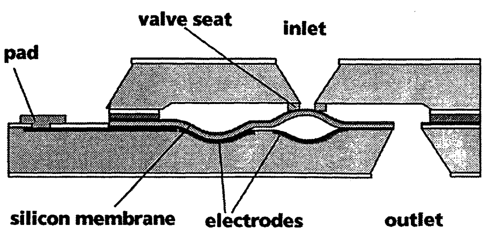


Fig. E1 Schematic setup of a bistable microvalve [13]

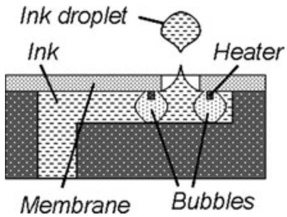


Fig. E2 Cross-section of the nozzle of an ink-jet printer

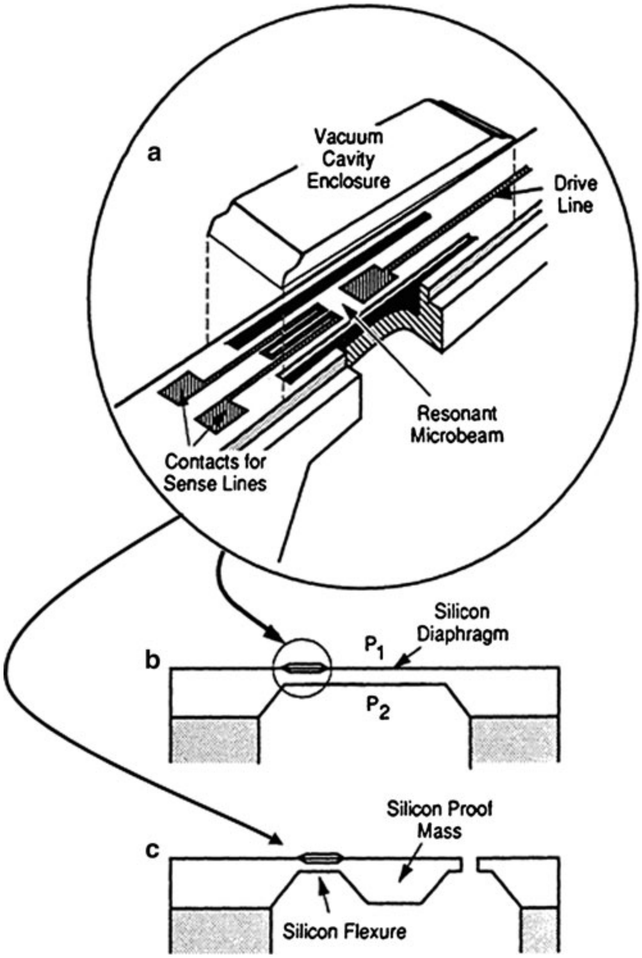


Fig. E3 Schematic setup of a pressure sensor (**b**) and an acceleration sensor (**c**). (Reprinted from [14] with permission from Elsevier)

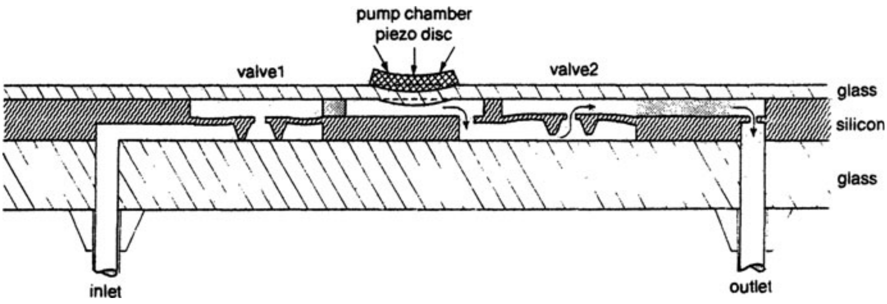


Fig. E4 Silicon micropump [15]

Problem 2

Why is it important to know the mechanical stress of a layer? Which meaning has it for the development of a microtechnical system?

Problem 3

You got to know Stoney's equation in the lecture, which you can use to calculate the stress of a thin film as a function of the radius of curvature. Consider which experimental methods you could use to measure the curvature of the substrate. There are several possibilities.

How could you measure the thickness of a thin layer?

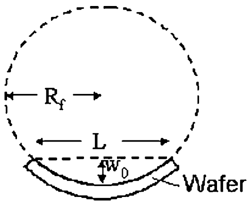
Problem 4

Let us assume that you are able to measure the vertical deflection of the substrate with the method suggested by you. The geometrical situation is approximated by a circle (cf. Fig. E5). If the length of a chord of the circle is L and its maximum distance to the circle is w_0, the radius can be calculated or approximated, respectively, with the following equation:

$$R_f = \frac{w_0}{2} + \frac{L^2}{8\ w_0} \approx \frac{L^2}{8\ w_0}.$$

Figure E6 displays the curvature of a passivated silicon wafer (diameter 100 mm and thickness 0.55 mm) without any metallic layer as measured by Thomas et al.

Fig. E5 Chord of the circle of curvature



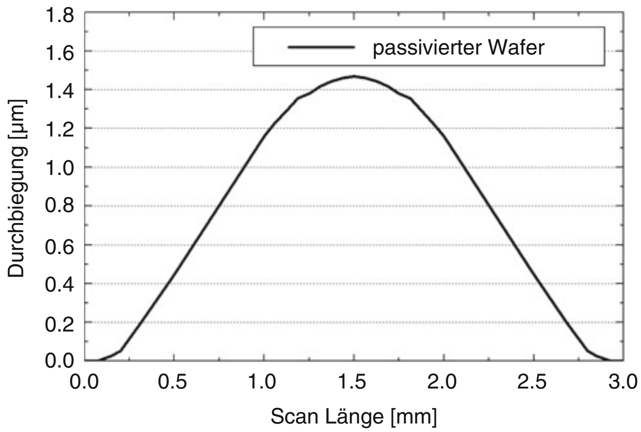


Fig. E6 Curvature of the uncovered passivated silicon wafer [16]. © [1988] IEEE

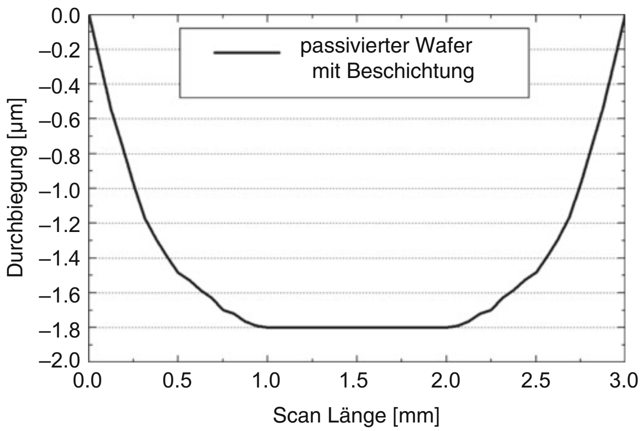


Fig. E7 Curvature of the covered passivated silicon wafer [16]. © [1988] IEEE

[16]. After this measurement, the wafer was sputtered first with 5 nm titanium and then with 500 nm tungsten. The curvature of the covered wafer is shown in Fig. E7.

(a) Obviously, the passivated but uncovered wafer shows some stress already. What is the reason for this curvature?
(b) Please, calculate the (mean) stress in the coating of the silicon wafer.

Hint: Do not get irritated because the curvature is no exact circle. This cannot be expected from a real measurement.

Young's modulus of silicon	133 GPa	Poisson's ratio of silicon	0.28

Problem 5

In surface micromachining, a sacrificial layer in the near of the surface is removed to partly separate movable parts from the substrate. A wafer is first coated with a sacrificial layer from SiO_2 and then with a thin titanium layer. The sacrificial SiO_2 layer is etched away with hydrofluoric acid.

(a) A thin layer may curl after removing its linkage to the substrate. This effect is due to a stress gradient in the layer. Calculate the radius of curvature of titanium films with different thicknesses and a stress difference of 300 MPa. The thicknesses of the titanium films are 10 µm, 5 µm, and 200 nm.

Young's modulus of titanium	120 GPa

(b) You may learn from your calculations that the radii of curled films can become very small. With a modified approach at Max-Planck Institute for rigid body research in Stuttgart so-called nanotubes were generated (cf. Figs. E8 and E9). The stress gradient was generated in this case by deposition of two layers with

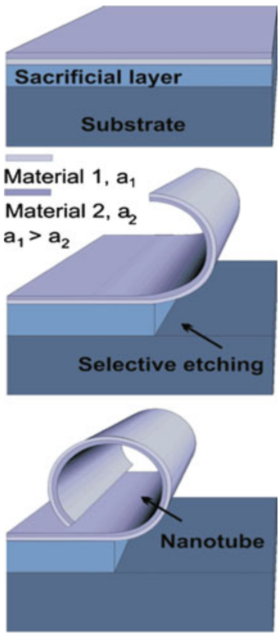


Fig. E8 Schematic drawing of the curling of a nanotube consisting of two thin layers. The upper layer shows a smaller lattice distance than the lower one. Courtesy of [17]

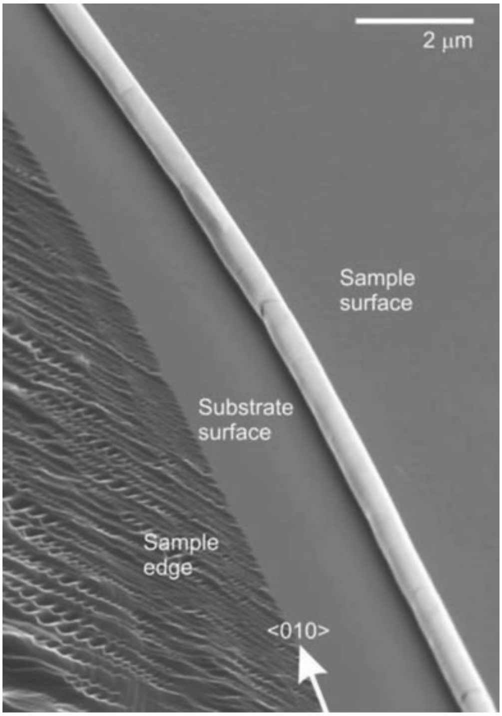


Fig. E9 SEM of a nanotube with a diameter of 530 nm. Courtesy of [17]

a thickness of approximately 60 nm with different stresses. How large was round about the difference in stress of the two layers before the sacrificial layer had been dissolved? The diameter of the nanotube is 530 nm. Assume that the mean Young's modulus of the two layers is 106.5 GPa. Assume that the effective distance of the two layers equals the distance of their centers.