Influence of ion-plasma treatment on residual stress in the microcantilever

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Abstract. The influence of ion-plasma treatment on residual stress in the microcantilever is investigated. The ability of treatment with energy below the sputtering threshold to affect the mechanical stress is shown. It is also demonstrated that a preliminary vacuum thermal annealing of samples reduces the influence of ion bombardment on the residual stress. With the increase of the annealing temperature the effect of ion bombardment disappears.

1. Introduction
Today the most of micromechanical thin-film structures are fabricated using standard technological processes of material deposition, such as vapor deposition or magnetron sputtering. These methods cause the residual mechanical stress in the film [1]. This stress can affect properties and reliability of a microelectromechanical device. Thus, the understanding of the mechanisms of relaxation and generation of stress is very important. The value and type of the residual stress depends on the deposited material and conditions of deposition. In the case of magnetron sputtering it depends on the ion energy, pressure inside the chamber, substrate temperature, etc.

In [2-4] the possibility of ion bombardment to influence the residual stress is shown. The energy of ions was in the order of several keV. Ion bombardment with such an energy leads to the sputtering of the material. It is a negative factor from the practical view. The aim of this work is to investigate the change of the mechanical stress in the microcantilever during the treatment with ions having the energy below the sputtering threshold.

2. Experiment details
The influence of ion-plasma treatment on the mechanical stress in the microcantilevers made of Cr and having the thickness of 500 nm, width from 2 to 10 µm and length from 10 to 100 µm was investigated. Cantilevers were fabricated as follows. At the first step the boron-doped silicon wafer with the diameter of 100 mm and the thickness of 460 µm was thermally oxidized (figure 1, step 1). SiO₂ layer had the thickness of 1 µm. Then the sacrificial polysilicon layer with the thickness of 1 µm (figure 1, step 2) and the chromium layer with the thickness of 0.5 µm (figure 1, step 3) were deposited by magnetron sputtering. The cantilevers were formed by contact photolithography technique and wet etching of Cr (figure 1, steps 4-7). At the last step the release of the cantilever was performed by plasma-chemical etching of the sacrificial layer in SF₆ plasma (figure 1, step 8). Fabricated cantilevers are shown in figure 2. Investigation of the cantilever details and geometrical measurements were performed by SEM Zeiss Supra 40.
Two batches of samples were fabricated. Cantilevers in both batches were bent up after the release. Due to the difference in the deposition conditions the cantilevers from the second batch curled more (curvature $\kappa = 8.5 \times 10^3 \text{ m}^{-1}$) than the cantilevers from the first batch (curvature $\kappa = 5.3 \times 10^3 \text{ m}^{-1}$).

Plasma treatment of the cantilever surface was performed using the radio-frequency high-density low-pressure inductively coupled plasma (RF ICP) [5]. Inductive power of the plasma reactor was 800 W, argon flow was 10 sccm, operating pressure was 0.08 Pa. RF bias power at the substrate was 30, 60 and 100 W, the average ion energy is determined by the self-bias potential. Maximum average energy of Ar$^+$ ions was ~ 50 eV for the 100 W RF bias power. Duration of each treatment was 60 s. The ion current density during the experiment was 6.4 mA/cm$^2$. Stacked ion dose was calculated from the ion current density.

The field of mechanical stress in the microcantilever can be presented as a polynomial [6]:

$$\sigma_{\text{total}} = \sum_{k=0}^{\infty} \sigma_k \left( \frac{z}{h/2} \right)^k,$$

where $z$ is the coordinate across the thickness $h$ of the cantilever, it varies from $-h/2$ to $h/2$ with an origin chosen at the mid-plane of the film. In the first approximation, $\sigma_{\text{total}}$ is the superposition of the constant mean stress $\sigma_0$ and the gradient stress $\sigma_1$, the former being symmetric and the latter anti-symmetric about the mid-plane. The effect of higher-order terms is neglected:

$$\sigma_{\text{total}} \approx \sigma_0 + \sigma_1 \left( \frac{z}{h/2} \right).$$

After the release the constant component of stress leads to a change of the cantilever length $\Delta L$ and gradient of the stress leads to a bending of the cantilever [7, 8]:

$$\sigma_0 = E \varepsilon = E \frac{\Delta L}{L},$$

$$\sigma_1 = \frac{E h}{R} = E h \kappa,$$

where $L$ is the length of the cantilever, $R$ and $\kappa$ are radius of curvature and the curvature of the cantilever respectively, $E$ is the Young's modulus (300 GPa for Cr).

The gradient of mechanical stress $\sigma_1$ was determined after each treatment by the curvature of the cantilever. The curvature was calculated by the cantilever tip deflection from the straight position $\Delta z$ (figure 3) using the following expressions:
3. Results and discussion

Two experiments were performed: in the first one the influence of the preliminary vacuum thermal annealing on the result of ion-plasma treatment was studied, and in the second one the effect of the heat sink on the result of ion-plasma treatment was investigated.

3.1. Effect of the preliminary vacuum thermal annealing on the result of ion-plasma treatment

Samples from the first batch were used for the experiment. Before the step of the cantilever release, the part of the samples was annealed in vacuum during 1 h at the temperature of 300, 450 and 500 °C, and another part of samples was not annealed. Plasma treatment was performed after the release. To avoid the heating of the cantilevers during the treatment the vacuum grease was used which provided better heat removal from the sample to the substrate holder.

The treatment with an average ion energy of 30 eV did not affect the bending of cantilevers (the total dose of ions was $26.3 \times 10^{17} \text{ cm}^{-2}$). The treatment with an average energy of 50 eV affected the bending as follows. This treatment did not affect the curvature of cantilevers which were annealed at 450 °C and 500 °C. But it affected the curvature of cantilevers which were not annealed and were annealed at 300 °C. These cantilevers bent up even more. Results of the treatment of the cantilevers with lateral dimensions $100 \times 10 \text{ µm}^2$ are shown in figure 4. Results obtained with the cantilevers of other dimensions are similar. A high temperature vacuum annealing makes cantilevers immune to the influence of ion-plasma treatment on the residual stress.

The increase of the cantilever curvature during the ion-plasma treatment means an increase of the stress gradient due to the generation of tensile stresses in the surface region of the cantilever (figure 5b).
3.2. Effect of the heat sink and the order of the cantilever release on the result of the treatment

Samples from the second batch were used for the experiment. Treatments were performed in the following regimes: (a) the treatment of the unreleased cantilevers with the heat sink, the average ion energy of 15 eV («cold plasma annealing»); (b) the treatment of the released cantilevers with the heat sink, the average energy of 15 eV; (c) the treatment of the unreleased cantilevers without the heat sink, the average energy of 15 eV; (d) the treatment of the unreleased cantilevers without the heat sink, the average energy of 50 eV («hot plasma annealing»). After the treatments in the regimes (a), (c) and (d) the release of the cantilevers was performed to determine the stress gradient.

The results of the treatment of the cantilevers with lateral dimensions of 100×10 µm² are shown in figure 6. The treatment of the unreleased cantilevers with the heat sink was able to influence the stress even when the average ion energy was 15 eV (figure 6, line (a)). As in the first experiment (figure 4), the curvature of the cantilever increased, which meant the generation of the tensile stress in the surface region and the growth of the stress gradient.

The treatment of the released cantilevers with the ion energy of 15 eV with the heat sink does not affect the bending (figure 6, line (b)). This agrees with the results of the first experiment in which the energy of 30 eV was not enough to affect the stress in the released cantilevers. It means that for the released cantilevers the threshold energy exists. Below this energy the plasma treatment has no influence on the mechanical stress.

The plasma treatment of the unreleased cantilevers with no heat sink gave the opposite result in comparison with the case of the heat sink. The treatment under these conditions led to the straightening of the cantilevers (figure 6, lines (c) and (d)). The absence of the heat sink caused the
heating of the cantilevers approximately to 250 °C. Probably, it had an effect similar to the thermal annealing and reduced the stress gradient (figure 5c).

The occurrence of the tensile stress during the ion bombardment was discussed in several papers [3, 4]. Chromium film bombarded with Ar ions having energy of 110 keV was studied in [3]. Authors attributed the occurrence of tensile stress to the decrease of interatomic distance with the increase of irradiation dose through the formation of defect clusters. Bombardment of Pt/Si cantilevers with ions of noble gases having energy of 0.5–4 keV was investigated in [4]. The occurrence of tensile stress was explained by the accumulation of vacancies due to the escape of interstitial atoms on the surface of the sample. In these studies the ion energy was of the order of a few keV. In our experiment the ions with energies of 15-50 eV were able to change the mechanical stress significantly. But we had the flow of ions three orders of magnitude higher than in mentioned papers, so the energy doses were the same.

![Figure 6. Dependence of the stress gradient in the cantilever on the dose of Ar ions obtained at the different regimes of the treatment.](image)

4. Conclusions

Plasma treatment with the ion energy below the sputtering threshold is able to influence the mechanical stresses in the microcantilever. It can be used to control the shape of the cantilever. Choosing the treatment conditions it is possible to increase or decrease the bending of the cantilever.

Acknowledgments

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