Stress and resistivity controls on *in situ* boron doped LPCVD polysilicon films for high-*Q* MEMS applications[*]

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Abstract: The simultaneous control of residual stress and resistivity of polysilicon thin films by adjusting the deposition parameters and annealing conditions is studied. *In situ* boron doped polysilicon thin films deposited at 520 °C by low pressure chemical vapor deposition (LPCVD) are amorphous with relatively large compressive residual stress and high resistivity. Annealing the amorphous films in a temperature range of 600–800 °C gives polysilicon films nearly zero-stress and relatively low resistivity. The low residual stress and low resistivity make the polysilicon films attractive for potential applications in micro-electro-mechanical-systems (MEMS) devices, especially in high resonance frequency (high-*f*) and high quality factor (high-*Q*) MEMS resonators. In addition, polysilicon thin films deposited at 570 °C and those without the post annealing process have low resistivities of 2–5 mΩ·cm. These reported approaches avoid the high temperature annealing process (> 1000 °C), and the promising properties of these films make them suitable for high-*Q* and high-*f* MEMS devices.

Key words: low pressure chemical vapor deposition; polysilicon; residual stress; film resistivity; annealing; microelectromechanical systems

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1. Introduction

In recent years, micromachined mechanical resonators (*μ* resonators) have attracted intensive attention because of their high frequency, small size, low cost, low power consumption, and on-chip compatibility with integrated circuits (IC). These promising properties make them ideal substitutes for their off-chip counterparts, such as quartz crystal, ceramic, and surface acoustic wave (SAW) resonators, and it is therefore worthwhile exploring their high frequency and radio frequency communication applications, in order to reach the ultimate goal of miniaturizing wireless communication systems[1]. The performance of the *μ* resonators strongly depends on the mechanical and electrical properties of their structural layer materials[1, 2]. Polysilicon thin films are often used as structural layers for *μ* resonators due to their adjustable conductivity, good mechanical properties, and also IC-compatible fabrication processes.

However, polysilicon thin films, which are usually deposited at 600 to 630 °C by low pressure chemical vapor deposition (LPCVD) for a standard IC process, show very high compressive stress (between 300 and 500 MPa)[3]. A high residual stress and stress gradient of polysilicon structural layers could lead to deformation of vibrating elements of *μ* resonators after they are released from the substrate, and such deformation could increase the energy loss and eventually decrease the *Q* values of the *μ* resonators dramatically[2, 4].

Therefore, low-stress structural layers are crucial for the performance and reliability of high-*Q* *μ* resonators.

In addition, for electrostatically-actuated *μ* resonators, not only the residual stress, but also the resistivity of the structural layers should be controlled as small as possible for good electromechanical couplings. In conventional surface-micromachining processes, high temperature annealing (> 1000 °C) is used for this purpose[5], although high temperature processes are not desired in a lot of MEMS device fabrication[6].

This paper systematically investigates the dependence of residual stress and resistivity on the deposition and annealing conditions for LPCVD boron doped polysilicon films. Structural layers with low residual stress and low resistivity are obtained by first depositing silicon amorphous films and subsequently annealing the amorphous films at 600 to 800 °C.

2. Experimental

2-*μ*m thick films of silicon dioxide were deposited by plasma enhanced chemical vapor deposition (PECVD) on (100)-orientated single crystalline silicon wafers. Then polysilicon film deposition was performed in an LPCVD front-injection, side-wall heated rector with 20% silane in argon and 5% diborane in nitrogen as precursors. The deposition temperatures were between 520 and 630 °C and the furnace pressures
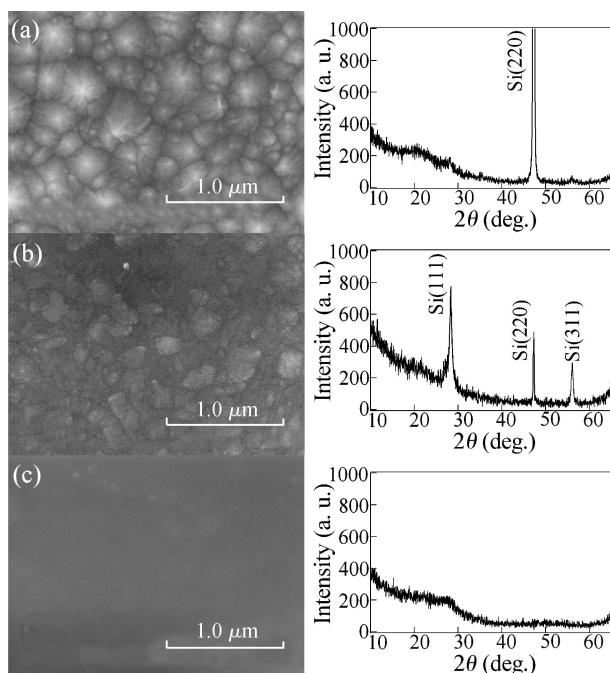
Fig. 1. SEM morphology and XRD spectra of three kinds of as-deposited polysilicon thin films deposited at (a) 570 °C, silane 200 sccm, diborane 10 sccm, (b) 540 °C, silane 200 sccm, diborane 10 sccm, and (c) 520 °C, silane 250 sccm, diborane 10 sccm.

were set between 44 and 94 Pa. The silane-to-diborane mass flow ratio was varied between 8 : 1 and 100 : 1. Annealing was performed at temperatures between 540 and 1050 °C in the furnace with nitrogen gas. In order to avoid possible nitridation and oxidation of the polysilicon film during the annealing process[6], a 1-μm thick PECVD silicon dioxide layer was deposited before annealing, serving as a cap layer to protect the LPCVD layer.

The thickness and surface morphology of the polysilicon films were measured by scanning electron microscopy (SEM). X-ray diffraction (XRD) was employed for film microstructure analysis. The resistivity was determined by the four-probe method. A profilometer (Dektak 8) was used to measure the film residual stress. Wafer curvature was measured before and after polysilicon film deposition, and after each annealing step as well. The residual stress was calculated based on the Stoney equation.

3. Results and discussion

In general, there are three kinds of microstructures of the LPCVD polysilicon thin films: amorphous structures, ellipsoidally shaped fine grains, and (110)-textured columnar grains[7]. The film microstructures depend strongly on deposition conditions and determine the residual stresses of the polysilicon films. Amorphous and columnar films have compressive stresses, while fine-grained films are tensilely stressed.

In our experiment, the *in situ* boron doped films have columnar grains when the deposition temperature is higher than 570 °C, the films consist of fine grains for a deposi-

tion temperature between 540 and 570 °C, and they become purely amorphous as the deposition temperature decreases to 520 °C. The crystallization temperature of the undoped polysilicon is about 580 °C, and diborane doping will decrease the amorphous-crystalline transition temperature[7]. From the SEM image and XRD spectrum in Fig. 1(a), it can be seen that the polycrystallized films deposited at 570 °C possess (110) textured columnar grains with diameters of 200–500 nm. The films deposited at 540 °C consist of mixed amorphous and fine-grained phases, as shown in the SEM image of Fig. 1(b); the XRD diffraction of films with mixed phases are randomly oriented, although modest (311) and (111) textures (compared to randomly oriented powders) are present. The film surface is smooth and the grain sizes are less than 200 nm (Fig. 1(b)). Figure 1(c) shows that the films deposited at 520 °C have randomly-oriented amorphous structures and mirror-like surfaces.

The films with columnar grains (Fig. 1(a)) have high compressive residual stress up to 330 MPa, which could be caused by interstitial atoms located in columnar grain boundaries[8]. During high temperature annealing (1000–1100 °C), the dislocations in the thin films move along the direction of the stress gradient, and the grains coarsen at high temperature; finally the residual stress decreases[9].

The low deposition temperature and high silane-to-diborane mass flow ratio will inhibit the nucleation of silicon and lead to the formation of pure amorphous films, as shown in Fig. 1(c). The compressive stress in the pure amorphous films could result from interstitial impurity atoms (such as hydrogen and oxygen) during deposition[10]. Crystallization of the amorphous phase to the tensilely-stressed fine-grain phase happens during annealing, and this will reduce the total stress of the polysilicon film.

For as-deposited mixed amorphous-polycrystalline films, the residual stresses can be controlled from compressive to tensile. The final stress of the film is determined by the ratio of the tensilely-stressed part to the compressively-stressed part. The tensile stress in the fine-grained portion of the film arises from the volume decrease during crystallization of the as-deposited amorphous silicon film[7]. Although the total stress of the as-deposited mixed films is significantly smaller (–30 to 30 MPa), the stress gradients are great due to the juxtaposition of compressive amorphous silicon on top of tensile fine-grained devitrified polysilicon.

High temperature annealing is often used to release film stress by reducing the defect density. However, this method cannot change compressive stress to tensile stress. To our knowledge, there are two approaches to achieve low tensile stress in polysilicon films. One method is to obtain polysilicon films with low residual stress by adjusting the deposition parameters directly. However, the ratio of the compressively-stressed amorphous portion to the tensilely-stressed fine grain portion in silicon films is very sensitive to the deposition parameters. The residual stress of polysilicon films is hard to control. The second solution is to adjust the annealing
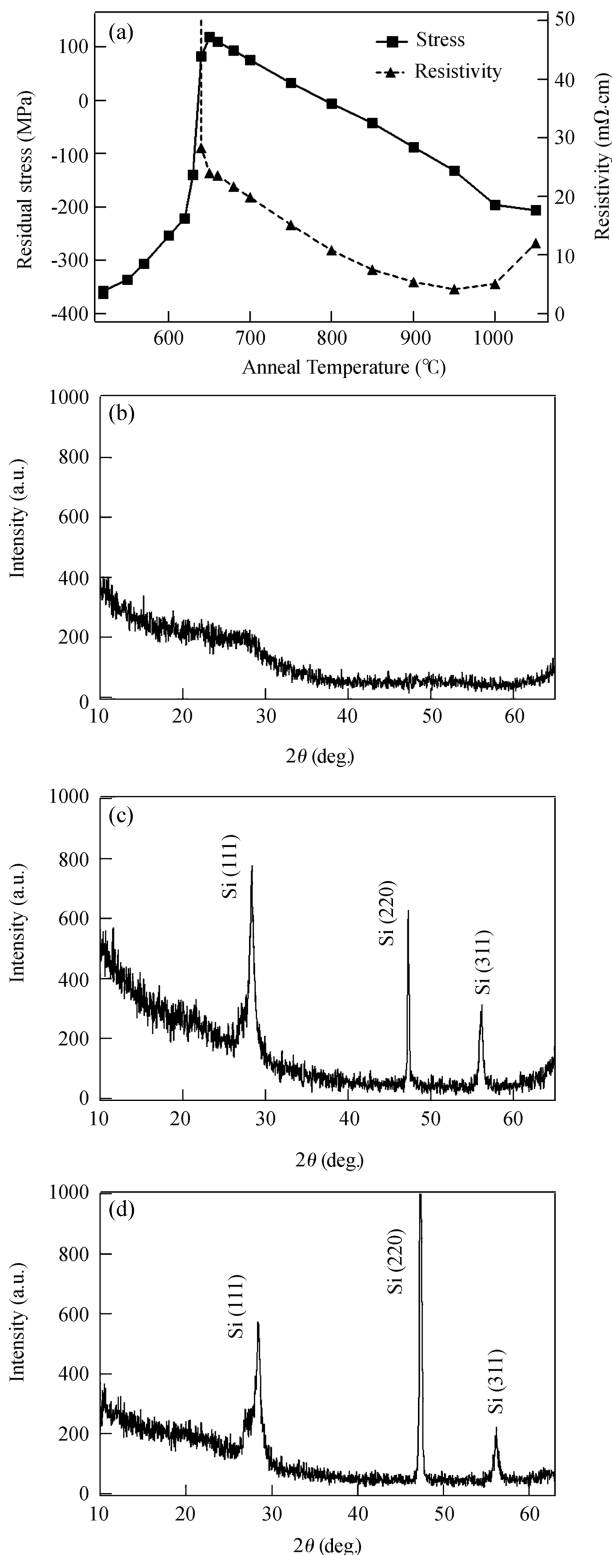
Fig. 2. (a) Residual stresses and resistivity of as-deposited amorphous silicon film and after annealing for 1 h in nitrogen, and XRD spectra of (b) as-deposited amorphous film, (c) after annealing at 800 °C, and (d) after annealing at 1000 °C for 1 h.

conditions for the as-deposited amorphous films, which are crystallized to mixed polycrystalline and fine-grained films with low residual stress and resistivity. In this work, low stress (−30 to 30 MPa) was achieved by annealing the as-deposited amorphous films at ∼ 600 to ∼ 800 °C.

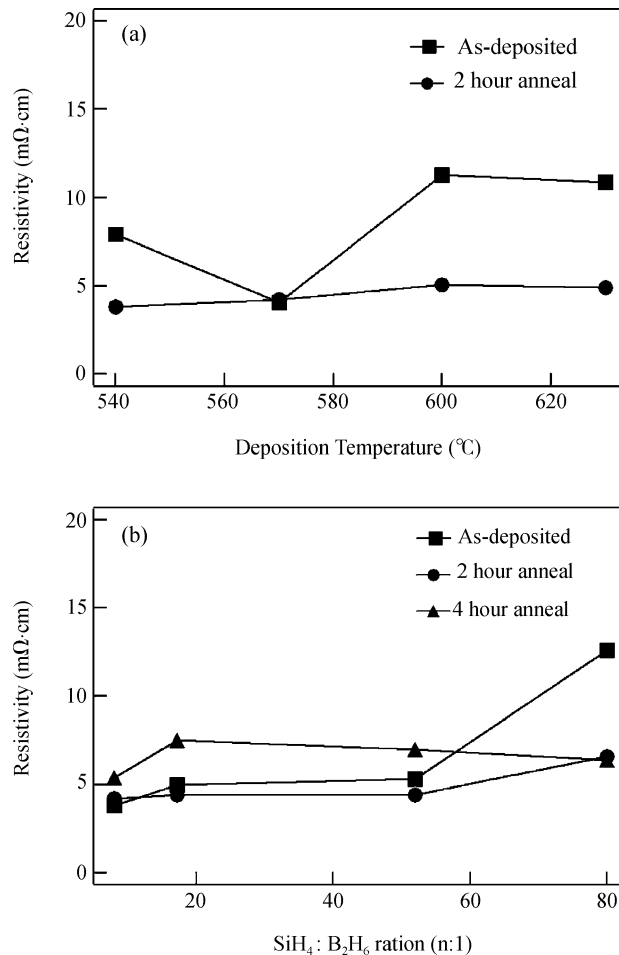Figure 2(a) shows the dependence of the residual stress

Fig. 3. (a) Resistivity of as-deposited and annealed polysilicon films deposited at 540 to 630 °C and annealed for 2 h at 1000 °C. The silane-to-diborane mass flow ratio was 8 : 1. (b) Resistivity of as-deposited and annealed polysilicon films deposited at 570 °C and annealed for 2 h and 4 h at 1000 °C. The silane-to-diborane mass flow ratio was varied from 8 : 1 to 80 : 1.

and resistivity of the as-deposited amorphous silicon films deposited at 520 °C on annealing temperature. The annealing cycle was 1 h. The silane-to-diborane mass flow ratio was 100 : 1. The residual stress changed from compressive (−350 MPa) to tensile (100 MPa) after annealing at 680 °C, and back to compressive (−200 MPa) after annealing at 1050 °C. When the annealing temperature reached 800 °C, the film stress was only −6 MPa. The XRD spectra (Fig. 2(b) to Fig. 2(d)) show that the film microstructures changed from pure amorphous phase to an amorphous and fine-grained mixture, and then displayed columnar grain phases.

Film resistivity is also closely related to film microstructure. The film resistivity sharply decreased when the annealing temperature was higher than 650 °C, while the film microstructure changed from the amorphous phase to the polycrystalline. Overall, the annealing conditions are crucial for achieving low residual stress and resistivity.

The dopant concentration of *in situ* boron doped films is normally high (∼ 10^{20} cm^{-3}), but the film resistivity strongly depends on the film microstructure[11]. Figure 3(a) shows that the resistivity of the as-deposited films varied with deposition
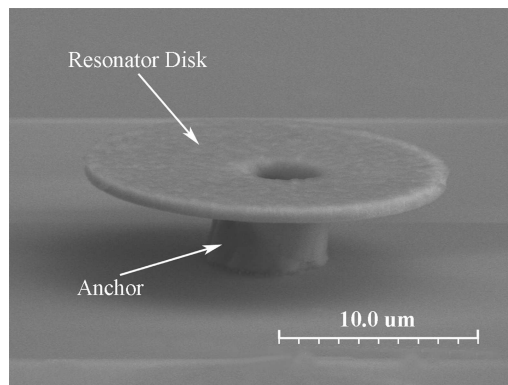
Fig. 4. SEM morphology of a released disk resonator.

temperature. The smallest resistivity value was obtained in the films deposited at 570 °C, and post annealing does not have an obvious effect on film resistivity.

The solid solubility of boron atoms in the polysilicon films increased with film deposition temperature. The effective impurity concentrations of the polysilicon films deposited at 600 and 630 °C were lower than those of the films deposited at 570 °C; therefore, the resistivities of the films deposited at 600 and 630 °C were higher than those of the films deposited at 570 °C. For the films deposited at 540 °C, the high resistivity resulted from the high defect density which traps the carrier. After annealing at 1000 °C for 2 h the films deposited at temperatures higher or lower than 570 °C, their resistivities reached the same level as the films produced at 570 °C. High temperature annealing sharply reduced the film defect density and decreased the film resistivity. The reason for the low resistivity in the polysilicon films deposited at 570 °C is not yet clear, and more studies are needed in the future to clarify this mechanism.

The film microstructure also depends on the reactive gases flow rates. As can be seen from Fig. 3(b), the resistivity of polysilicon films deposited at 570 °C increased from 4.1 to 12.6 mΩ·cm on increasing the silane-to-diborane mass flow ratio from 8 : 1 to 80 : 1. After annealing at 1000 °C for 2 h, the resistivity of the films fabricated with the mass flow ratio from 8 : 1 to 52 : 1 declined to 4.3 mΩ·cm, and for the films produced with the silane-to-diborane mass flow ratio of 80 : 1, they went down to 6.6 mΩ·cm. However, additional annealing at 1000 °C for 2 h resulted in a resistivity increase for all films except those with the ratio of 80 : 1.

The variation of resistivity can be explained by the solubility of boron in polysilicon. Boron doping becomes saturated at a certain silane-to-diborane mass flow ratio. This is the reason that the resistivity of the as-deposited films changes little with silane-to-diborane mass flow ratios less than 52 : 1. Boron doping becomes undersaturated when the silane-to-diborane mass flow ratio is over 52 : 1. During annealing, the dopant impurities were activated, and the resistivity decreased. Nevertheless, excessively long annealing times at high temperatures will result in a resistivity increase, probably due to boron depletion.

Figure 4 presents a scanning electron micrograph of a re-

leased micromechanical disk resonator made of *in situ* boron doped LPCVD polysilicon films deposited at 520 °C and annealed at 800 °C for 1 h. No deformation caused by the residual stress in the structural layer occurred after the resonator disk was released from substrate. This low stress structure layer material guarantees the symmetrical vibration of the disk，decreases the vibrating energy loss and finally increases the Q value of the disk resonator. Measurement of the resonance frequency and Q is ongoing.

4. Conclusion

The residual stress and resistivity of *in situ* boron doped LPCVD polysilicon thin films and their dependence on the deposition and annealing conditions are investigated. A structural layer with low residual stress and low resistivity is obtained by depositing amorphous silicon film and annealing it at 600 to 800 °C. The deposition parameters of LPCVD polysilicon films with good conductivity have also been investigated, and low resistivity values (< 5 mΩ·cm) for these as-deposited polysilicon thin films can be achieved. The approaches reported in this work avoid the use of high temperature processes, and the promising properties of these films make them suitable for high-Q and high-f MEMS devices. Further efforts will be made to systematically optimize the mechanical and electrical properties of polysilicon films in future work.

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