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Stress control of porous silicon films for microelectromechanical systems

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ABSTRACT

Control of stress in porous silicon (PS) through porosity changes was studied using X-ray diffraction rocking curve measurements. The effect of thermal annealing on the stress was also investigated with both X-ray diffraction and radius of curvature measurements. Annealed films could achieve compressive or tensile stress. The effect of annealing was reversed by a short HF dip, except in the case of nitridised samples (annealed in N_2 at temperatures above 500 °C). The effect of hydrogen desorption, oxidation and nitridation, modified via annealing temperature and ambient, was studied to understand the evolution of physical properties and the mechanism of the stress modification. The effect of stress on PS microbeams was studied to determine the influence when PS films are used as the structural layer in a micromachined device. When modelling the effect of stress changes on the order of those observed during thermal annealing, the results indicated that for PS-based microbeams, stress is a significant factor in determining resonant frequency, far more than found in nonporous materials, illustrating the need for accurate control of stress.

1. Introduction

Porous silicon (PS) has drawn significant attention for application in microelectromechanical system (MEMS) and sensing [1,2], due to the large surface area and control of film properties through porosity. Porous silicon based MEMS (PS-MEMS) fabrication requires the films to go through a series of chemical and physical processes which can affect the optical, thermal, electrical and/or mechanical properties of the as-fabricated film. Our group has demonstrated a process based on N_2 annealing at 600 °C which subsequently reduces the effect of oxidation in ambient air, and makes the films compatible with standard CMOS photolithography [3], providing opportunities for complex and scalable MEMS fabrication. By combining N_2 annealing, patterning, electropolishing and repeated HF dips, all-silicon doubly clamped microbeams have been made from laterally uniform porosity PS films, with no other material required for the sacrificial layer [4], demonstrating the potential to create an all-silicon MEMS fabrication process.

However, these processes are likely to induce changes in PS film stress. Previous PS film stress studies focused on either *in situ* ultrahigh vacuum (UHV) [5] or treatment in N_2 below 350 °C [6,7], and thus did not investigate how PS stress evolves at temperatures higher than 475 °C [8] due to continued hydrogen desorption without ultrahigh vacuum conditions. Though the temperature dependence of PS stress and its relationship with hydrogen desorption have been reported [9,10], changes in stress after relatively high temperature annealing in N_2 or after repeated HF exposure, as would be common in a multistep PS-MEMS fabrication process, have not been investigated. Further, the effect of oxidation under ambient conditions is unclear. While stress is known to affect the resonant frequency of non-porous microbeams [11,12], the relative impacts of stress when using low modulus PS films [13,14] is not yet known. Thus the extent of stress changes after typical processing steps must be determined.

In this work, X-ray diffraction (XRD) rocking curve measurements were employed to study the lattice mismatch between the PS layer and silicon substrate, from which the internal stress can be extracted [15]. The relationships between porosity, annealing temperature, oxidation and stress were investigated based on the lattice mismatch measurements. In addition, stress studies based

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on radius of curvature measurements were undertaken, to investigate the stress change for high temperature annealed PS films, which could not be measured via XRD. The internal stress of PS at different stages of processing was subsequently determined to understand the implications of processing steps on the stress, and thus resonant frequency, of PS microbeams. The results show that variation of PS internal stress with porosity, annealing temperature, annealing ambient and HF immersion is extremely complex but repeatable, indicating various pathways to tune the internal stress for PS-MEMS applications. Finally, modelling studies revealed that the variation in stress on the order of that observed in the annealing experiments can alter the resonant frequency in these structures by a factor of up to three, significantly more than is observed in non-porous MEMS structural materials such as Si and Si_3N_4. Thus, changes in stress during fabrication must be allowed for when designing MEMS based on PS films. However, we propose that if carefully calibrated, porosity variation and thermal annealing provide an additional tool for stress tuning in porous silicon.

2. Methods

The wafer material used was moderately doped p-type (100) silicon with resistivity of 0.08–0.12 Ω cm. Room temperature anodisation was performed in a 15% HF/ethanol solution. PS films in this paper were obtained using different current density and anodisation time to form PS layers with the same physical thickness of $t = 2.5 \pm 0.1$ μm, but with a range of porosity from $P = 71\%$ to $P = 87\%$. After fabrication, most samples were annealed in either vacuum or N_2 at atmospheric pressure for 6 min at different temperatures. The temperature of the rapid thermal annealing process was measured using an optical pyrometer at the underside of the susceptor enclosure housing the sample. Subsequently, PS samples were dipped in a 10% HF/ethanol solution for 15 s, which removed oxide and returned them to a Si–H/Si–H_2 surface.

An X-ray diffraction system (Panalytical Empyrean) was used to perform rocking curve measurements of the (400) reflection from the lattice plane parallel to the (100) silicon surface, with a hybrid monochromator, employing Cu $K\alpha_1$ radiation ($\lambda = 1.54052$ Å). The rocking curve measurement provided two Bragg diffraction peaks, a weak one produced by the PS layer and a strong one from the silicon substrate. The angular separation of these peaks provides the lattice parameter variation between the PS and silicon [16].

For samples annealed at high temperature (>320 °C), non-uniform lattice expansion/porosity resulted in the complete loss of the PS peak in the XRD measurements. Thus a second stress measurement method based on radius of curvature measurements was employed. The stress of a film can be expressed using Stoney's equation:

$$\sigma_{ps} = \frac{E_{si}}{6(1-\nu_{si})}\frac{t_{si}^2}{t_{ps}}\Delta\left(\frac{1}{R_1}-\frac{1}{R_2}\right), \quad (1)$$

where E_{si} is Young's modulus of the silicon substrate, and ν_{si} is its Poisson's ratio ($\nu_{si} = 0.26$), t_{si} and t_{ps} are thickness of the silicon and PS layers, which are 280 ± 25 μm and 2.5 ± 0.1 μm, respectively, and R is the radius of curvature for the silicon substrate before (R_1) and after (R_2) formation of PS layer. The radius of curvature was measured in air on the polished backside of the silicon wafer, by a ZYGO NewView™ 6000 profilometer. This avoided any optical interference that may have resulted from the optically transparent thin film. In detail, for this radius of curvature measurement, the PS sample was cut into a rectangular shape (~3 × 15 mm^2) after fabrication, and two marked spots placed along the length of the backside (silicon side) of the wafer piece. The radius of curvature (RadCrv) measurement was undertaken with the profilometer between the two marked spots to minimize any spatial variation in the RadCrv.

The sample was subsequently dipped in a 5% (w/w) KOH solution for approximately 15 s to remove the PS layer, and the radius of curvature measured again, between the same marked spots. The curvature change of Si-only substrate from a 5% KOH dip for 15 s was also investigated and found to introduce <7% uncertainty to the PS film stress. This was not included in the calculation, as it was not significant enough to affect the outcomes of the study.

3. Results and discussion

Using the lattice mismatch and radius of curvature based methods, the effects of various factors on PS film stress were investigated; this will be discussed below along with the corresponding influence on mechanism of stress modification.

3.1. Stress measurements in PS with X-ray diffraction

Rocking curve diffraction patterns for as-fabricated PS layers of different porosity are shown in Fig. 1. With an increase of PS film porosity from 71% to 87%, the PS peak moved further away from the silicon substrate peak, which is consistent with previous studies [17].

Based on the lattice mismatch between the PS film layer and the silicon substrate, the average internal stress of each PS film could be calculated [15], as shown in Eq. (2):

$$\sigma_{ps} = \frac{E_{ps}}{(1+\nu_{ps})}\cdot\frac{\Delta a}{a}, \quad (2)$$

where $\Delta a/a$ is the lattice mismatch between the PS layer and the silicon substrate, E_{ps} is Young's modulus of PS, and ν_{ps} is Poisson's ratio of PS, which is assumed to be a constant ($\nu_{ps} = 0.09$) for the PS samples used in this paper [15,18]. The Young's modulus of PS (E_{ps}) is known to depend on porosity P and is given by $E_{ps} = E_{Si}(1-P)^3$, where E_{Si} is Young's modulus of the silicon substrate (163 GPa) [19]. With XRD measurements, the lattice mismatch could be acquired according to Eq. (3):

$$\frac{\Delta a}{a} = \frac{\Delta\theta}{\tan\theta}, \quad (3)$$

where θ is the Bragg diffraction peak of silicon and $\Delta\theta$ is the peak separation between PS and silicon. Therefore, by using the angle at peak intensity and the angle separation between the two peaks, the average internal stress of each PS film was calculated. The measured

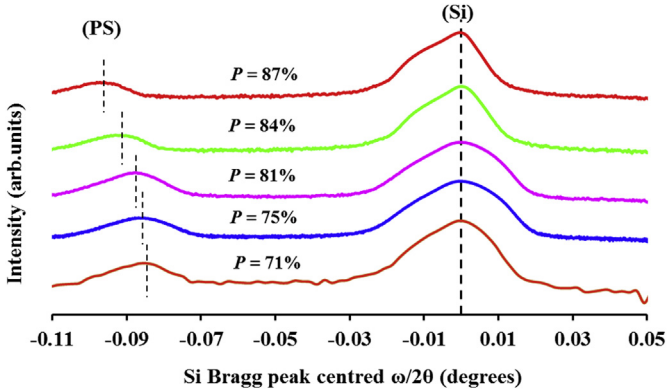

Fig. 1. X-ray rocking curve diffraction patterns for as-fabricated PS layers of different porosity (P).