Molecular beam epitaxial growth and characterization of defects induced by cavitation impacts on polysilicon thin films

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Abstract. Polycrystalline silicon were grown at high temperature in a gas-source molecular beam epitaxy (MBE) and then exposed to cavitation impacts from the backside at different scanning times to introduce compressive stress. The micro-Raman spectroscopy showed that regions of greater structural change due to cavitation impacts experience higher full width at half maximum (FWHM) and that stress was a function of number of scans. AFM was used to analyze the surface morphology of the specimen in the as-grown condition and after exposed to cavitation impacts. The structural features were characterized by X-ray diffraction (XRD) and high resolution transmission electron microscope (HRTEM). HRTEM observations showed that nanoparticles size of grains at the growth/death zone of interface. The dislocations types, twinnings and defects caused by cavitation impacts are discussed.

3. Introduction

As the scaling down and the complexity of MEMs devices increase, stress/strain issues are becoming more and more relevant for the functionality and reliability of the whole device. Thus, it is necessary to specify precisely the structures, interface morphology, the shape and sizes of individual features at the atomic scale and their influence on stress/strain.

Polycrystalline silicon films have been of great interest in integrated circuits due to the electronic properties influenced by the grain boundaries and defects. In this study, the residual stress of polycrystalline silicon grown at high temperature in a gas-source molecular beam epitaxy (MBE) has been characterized using the micro-Raman spectroscopy and the structural features observed by X-ray diffraction (XRD) and transmission electron microscope (TEM). Samples were prepared on CZ-Si(100) p-type substrates coated with 50 nm of SiO2. The film was deposited at 700 ºC and pressure of 4 Torr.

Ghyselen showed that strain engineering for the CMOS applications is done at high temperature [1]. The treatment herein is based on a low-temperature approach by introducing compressive residual stress on PolySi/SiO2 using cavitation impacts. The prime objective is to increase mobility by gettering the polysilicon. Defectivity is a critical parameter in that if not well controlled, unsuitable dislocation will take place due to the strong strain gradient. The authors have successfully established suitable cavitating conditions to cause appropriate dislocation and hence gettering silicon wafer [2].
The poly-Si/SiO₂ sample was then exposed to cavitation impacts from the backside at different scanning times to introduce compressive stress. The FWHM taken from double crystal rocking curve measurements of the [111][022][220][113] diffraction peaks from the polysilicon film were measured to assess the general crystalline quality of the film. The XRD system was not suitable for exact determination of lattice constants and hence no characterization of residual stress could be determined at this point. However, XRD was capable of determining overall composition of the thin film.

The microstructural features have been explored to determine the defects caused by molecular beam epitaxial growth and those associated with cavitation impacts. Thus, HRTEM observations were done to determine the dislocations and nanoparticles size of grains resulting from the processes.

4. Experimental techniques

4.3. Growing polysilicon

Figure 1 shows schematic illustration of the gas-source molecular beam epitaxy system used for growing of poly-Si/SiO₂ samples. The CZ Si(100) substrate wafer was chemically cleaned. The sample was put on the tray and heated to 800 °C for 1 hour to remove native oxide. Subsequently, a 10 nm silicon buffer was deposited, followed by heating at 700 °C for 3 hours in disilane (Si₂H₆) gas at a flow rate of 2.5 sccm [3]. The pressure during growth was 10⁻² Torr.

4.4. Introduction of compressive residual stress

The surface of silicon wafer was masked with a tape and placed onto specimen holding device and then immersed in DI water as shown in Fig. 2. In order to introduce beneficial stress, the test liquid (DI water) was injected onto the surface of the specimen through a nozzle, diameter 0.8 mm, with an upstream and downstream pressure of 2.5 MPa and 0.1 MPa, respectively. The standoff distance \( s_o \), which is defined as the distance between the upstream corner of the nozzle throat and the surface of the specimen under test was 17 mm.

The authors have already established the optimum condition for the qualitative determination of standoff distance \( s_{opt} \) by erosion test [4]. Details of the cavitating jet in submerged condition are found in references [5-9]. Upon leaving the nozzle, a cavitating jet was formed. The collapsing of the cavitation bubbles causes shock wave and/or microjets on the specimen surface thereby causing suitable plastic deformation. The cavitation impacts was controlled by adjusting hydraulic parameters such as injection pressure of the cavitating jet and standoff distance. The cavitating jet was traversed in the \( x \)-direction using an auxiliary leadscrew controlled by a motor thereby allowing uniform exposure on the specimen.
4.5. Surface characterization using atomic force microscopy (AFM)
AFM was used to scan surface topologies of polysilicon film in the as-grown and also treated by cavitation form. Thus, the surface plastic deformation was qualitatively determined at nano-scale as a result of cavitation impact. The intensity of the cavitation bubbles could be determined.

4.6. Structural characterization using high resolution transmission electron microscopy (HRTEM)
For the plan-view TEM observation, the specimen was shaped to 5 mm square using a micro-cutter and then cleaned using ethyl alcohol (CH₃CH₂OH). The sample was fixed to a glass using epoxy resin and then mechanically polished to thickness \( t = 20 \mu m \). The specimen was ion-milled at low angle 12°, 3 kV and argon flow 0.3 cm³ until perforation. For the cross-sectional view TEM observations, the samples were thinned and then glued with deposited films facing each and the ion-milled. The HTREM observations were performed using JEOL JEM-3010 operated at 300 kV.

5. Results

3.1 Surface analysis of poly-Si/SiO₂ using AFM
The surface was characterized using the Environscope AFM equipped with the Nanoscope IV controller, manufactured by Veeco (Digital Instruments). Figure 3 shows the AFM images in the as-grown form 3 (a) and after been exposed to cavitation impacts as shown in 3 (b).

The surface roughness and deformation values per 10 \( \mu m² \) are shown in Table 1. There was a gradual increase on deformation with the number of scans. The surface finish of the as-grown poly-Si/SiO₂ was improved.

|                  | Surface roughness Ra | Deformation nm |
|------------------|----------------------|----------------|
| As-grown         | 3                    | 429            |
| Cavitated at 2 scans | 127                  | 10             |
| Cavitated at 3 scans | 134                  | 23             |
| Cavitated at 4 scans | 153                  | 30             |

Table 1. Surface roughness and deformation on Poly-Si/SiO₂

![Figure 3. AFM images of Poly-Si/SiO₂](image)

![Figure 4. XRD spectrum of Poly-Si/SiO₂ exposed to cavitation impacts.](image)
3.2 XRD Analysis of poly-Si/SiO₂

The poly-Si/SiO₂ sample was then exposed to cavitation impacts from the backside at different scanning times to introduce compressive stress. The full-width at half maximum (FWHM) taken from double crystal rocking curve measurements of the [111][022][220][113] diffraction peaks from the polysilicon film were measured to assess the general crystalline quality of the film. The diffraction at the bulk occurred at 2θ = 47.44 deg and lattice spacing d = 1.914 as shown in Fig. 4. The XRD system was not suitable for exact determination of lattice constants and hence no characterization of residual stress could be determined at this point. However, XRD was capable of determining overall composition of the thin film.

3.3 Microstructural observations using HRTEM

![Figure 5](image1.png)

**Figure 5.** Plan-view TEM observation of Poly-Si/SiO₂ illustrating (a) microtwins and ledge (b) overlying columnar texture.

![Figure 6](image2.png)

**Figure 6.** Cross-sectional view TEM observation of Poly-Si/SiO₂ illustrating (a) near parallel textural columnar structure (N) with and ledge (b) dislocation types.
Figure 5 shows the plan-view TEM observation of the Poly-Si/SiO₂. Parallel microtwins were observed along the grain boundary as well a ledge lying between two adjacent grains. The overlying columns layers shown in (b) were caused by deposition conditions during MBE growth. Edge dislocations were distinct on the columnar crystalline end (see Fig. 5(b)). Cross-sectional view high-resolution transmission electron microscope (HRTEM) showed two distinct microstructural regimes at the low and high end with an intermediate transition region (see Fig. 6(a)). Homogeneous nucleation and growth in the bulk of the films were observed.

Numerous planar twins are observed on the lower end while the polysilicon films have a near parallel textural columnar microstructure (N). The textured grains had rough edges and the death-growth zone was 20 nm. The average intra-grain grain size was 45 nm. Cavitation impacts decreased the gap between two intra-grains. Both homogeneous nucleation and growth in the bulk of the films and heterogeneous nucleation and growth in the film-substrate interface are observed. Deformation streaks (S) and microtwins (T), Lomer-Cottrel locks (L) and screw dislocation (D) were observed on the columnar crystals as shown in Fig. 6(b).

Conclusions
The beneficial defects in polysilicon thin films grown by MBE and exposed to cavitation impacts have been studied. Based on the experimental findings, the following conclusions can be drawn:
1. The defects within polycrystalline silicon are microtwins and dislocations. Cavitation impact is predominantly associated with dislocations. The films show two distinct microstructural regimes at the low and high end with an intermediate transition region. Homogeneous nucleation and growth in the bulk of the films were observed. Both homogeneous nucleation and growth in the bulk of the films and heterogeneous nucleation and growth in the film-substrate interface are observed. Deformation streaks and twins were seen on the large columnar crystals.
2. The death-growth zone at which there is a trade-off in crystal size is approximately 50 nm in length. Surface dislocation loops either vertical or horizontal direction leading to edge and screw-dislocation were also observed.

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