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Heavily boron-doped silicon membranes with enhanced mechanical properties for x-ray mask substrate

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Heavily boron-doped silicon membranes which show substantial improvement in mechanical properties have been fabricated for x-ray mask substrate by eliminating the misfit dislocation from the membrane. The measured surface roughness, fracture strength, and residual tensile stress of the membrane are 20 Å peak to peak, 1.39×10^{10} and 2.7×10^9 dyn/cm², while those of the conventional heavily boron-doped silicon membrane with high density of misfit dislocations are 500 Å peak to peak, 8.27×10^9 and 9.3×10^8 dyn/cm², respectively. The differences between the two membranes are due to misfit dislocation. Young's modulus has been extracted as 1.45×10^{12} dyn/cm² for both membranes. Also, the lattice constant of strain-free membrane, the in-plane lattice constant of the conventional membrane, and the density of extra-half plane contained in the conventional membrane have been extracted as 5.424 Å, 5.426 Å, and 2.3×10^4 /cm, respectively.

X-ray mask substrate which is the most critical aspect of x-ray lithography for feature sizes below 0.25 μm requires very good mechanical properties because it has a very high ratio of area to thickness and very tight distortion tolerance. The required mechanical properties for the substrate are high Young's modulus, high tensile stress, high fracture strength, etc. High Young's modulus is required to minimize in-plane distortions due to substrate-absorber film interactions, while high tensile stress is necessary to minimize out-of-plane distortions.¹ Finally, high fracture strength is essential to reduce the incidence of premature and unpredictable mask substrate fracture.

A heavily boron-doped (p^+) silicon membrane is frequently used as an x-ray mask substrate. However, there are various types of defects in the membrane that may degrade the mechanical properties of the membrane. Among them, misfit dislocation is the dominant defect type generated at the interface of the substrate and p^+ silicon layer.² Thus, one can conjecture that elimination of misfit dislocation may improve the mechanical properties of the membrane. We have already suggested a method to suppress the misfit dislocation in the p^+ silicon layer by surrounding a certain region with undoped region which protects the propagation of misfit dislocation from the wafer edge to the p^+ region inside the undoped region.³

We have fabricated p^+ silicon membranes in misfit dislocation-free region formed by the method described in Ref. 3 (we will call this membrane the proposed membrane) and the mechanical properties of the membrane are experimentally compared with the conventional p^+ silicon membranes. By comparing the properties of the two membranes, one may find whether the proposed membrane is suitable for x-ray mask substrate and the influences of the misfit dislocation to the mechanical properties and lattice parameters of the membranes.

The processes for the fabrication of the membranes are as follows. 4 in., p -type, (100)-orientation Czochralski silicon wafers with the resistivity of 2–10 Ω cm and defect density of 1–2/cm² are prepared. Chemical-vapor deposited (CVD) oxide with 1 μm thickness is deposited and patterned on the top side and the bottom side of the wafer. Boron diffusion is subsequently performed as shown in Fig. 1(a). The diffused regions are classified into two, i.e., one surrounded by the CVD oxide (region A), and the other which is open to the wafer edge (region B). Boron diffusion is carried out at 1080 °C for 2 h. The number of boron atoms diffused into silicon is measured as 2.3×10^{16} /cm² by spreading resistance profile (SRP) analysis. For this diffusion condition misfit dislocations occur in region B, while region A is free of misfit dislocations because propagation of misfit dislocation into region A is prevented by the undoped region under surrounding CVD oxide.³ Therefore, in-plane lattice constant of the p^+ silicon layer in region A remains the same as the lattice constant of the substrate, while that of region B is reduced by insertion of extra-half planes accompanying mis-

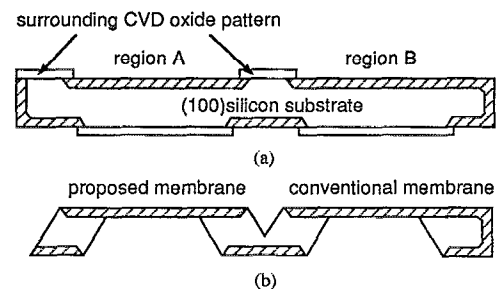


FIG. 1. Process sequence of the fabrication of p^+ silicon membranes (a) is the formation of p^+ silicon layers and (b) is the etching of substrate. Hatched layer is p^+ silicon and region A is surrounded by CVD oxide pattern.

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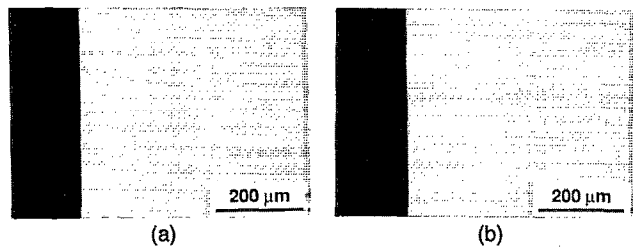


FIG. 2. Photomicrographs of the fabricated membranes (a) is the proposed membrane and (b) is the conventional membrane. Optical microscope with back illumination is used and the dark regions are bulk silicon. There are many textile patterns in the conventional membrane.

fit dislocations (the in-plane means the horizontal plane of the wafer). After boron diffusion, the CVD oxide patterns are removed. Then, the substrate of the wafer is etched away in ethylenediamine-pyrocatechol-water mixture as shown in Fig. 1(b). The proposed membrane is fabricated in region A and the conventional membrane in region B. The measured area and thickness of the fabricated membranes are $5\text{ mm} \times 5\text{ mm}$ and $1.5\ \mu\text{m}$, respectively. The membranes without pin holes, hillocks, and flaws are carefully selected by inspection with optical microscope and the selected 20 samples for both types of membrane are mechanically tested by the blister method⁴ for the extraction of load-deflection characteristics.

Figure 2 shows photomicrographs of the fabricated membranes. There are many textile patterns in the conventional membrane but none in the proposed membrane. The textile patterns are due to the surface roughness and degrade optical transmission characteristics which are an important consideration for optical alignment schemes.⁵ The measured roughness of the etch-stopped surface in the proposed membrane is about $20\ \text{\AA}$ peak to peak which is smaller than that in the conventional membrane which has about $500\ \text{\AA}$ peak-to-peak roughness. Since fatigue resistance improves with smooth surface,⁶ high fatigue resistance of the proposed membrane is expected.

Figure 3 shows typical measured load-deflection curves of the membranes. It can be easily seen that the proposed membrane is deflected less than the conventional membrane for the same applied pressure and that the proposed mem-

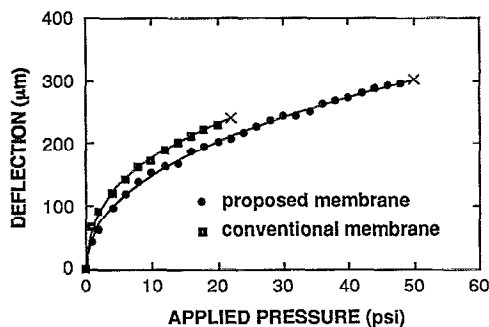


FIG. 3. Measured load-deflection curves of the membranes by blister method. The measured data are fitted by solid lines. Fracture points are represented by marker \times .

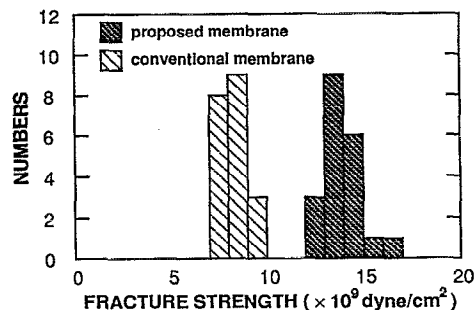


FIG. 4. Measured fracture strength of the membranes. Total numbers are 20 for each of the proposed membrane and the conventional membrane.

brane is fractured at a higher applied pressure than the conventional membrane. For the extraction of Young's moduli, residual stresses, and fracture strengths of the membranes, the measured curves are fitted to Tabata's model⁷ for Poisson's ratio of silicon $\nu=0.28$.⁸ Average Young's modulus of $1.45 \times 10^{12}\text{ dyn/cm}^2$ or average biaxial modulus of $2.02 \times 10^{12}\text{ dyn/cm}^2$ for both membranes, average residual stress (tensile) of $2.7 \times 10^9\text{ dyn/cm}^2$ for the proposed membrane, and average residual stress (tensile) of $9.3 \times 10^8\text{ dyn/cm}^2$ for the conventional membrane have been found from this analysis. Thus, it can be concluded that Young's modulus is not affected by misfit dislocation, while the residual tensile stress is decreased by the insertion of extra-half planes accompanying misfit dislocations. The residual strains of the membranes are directly calculated as $\epsilon_A=1.34 \times 10^{-3}$ for the proposed membrane and $\epsilon_B=0.46 \times 10^{-3}$ for the conventional membrane by division of the residual stresses by the biaxial modulus. Figure 4 shows the histograms of the extracted fracture strengths of the membranes for the 20 samples. The average fracture strength of the proposed membrane is $1.39 \times 10^{10}\text{ dyn/cm}^2$ and its standard deviation is $0.91 \times 10^9\text{ dyn/cm}^2$. The average strength and its standard deviation of the conventional membrane are 8.27 and $0.73 \times 10^9\text{ dyn/cm}^2$, respectively. Since both membranes are fabricated by the same process as shown in Fig. 1 and have the same structure except for the existence of misfit dislocation, the critical stress concentrator in the conventional membrane must originate from misfit dislocation.

Assuming that the membranes are doped uniformly, additional analyses for the membranes are possible. Since in-plane lattice constant a_A of the proposed membrane is the same as that of substrate, the lattice constant a_f of the strain-free p^+ silicon membrane is calculated as $5.424\ \text{\AA}$ by⁹

$$\epsilon_A = (a_A - a_f) / a_f, \quad (1)$$

where $a_A=5.431\ \text{\AA}$ is used. Similarly, in-plane lattice constant a_B of the conventional membrane is calculated as $5.426\ \text{\AA}$ by substituting ϵ_A and a_A in Eq. (1) by ϵ_B and a_B . The value of a_B is similar to the reported data ($5.4263\ \text{\AA}$)¹⁰ by x-ray diffraction for $1.3 \times 10^{20}\text{ atoms/cm}^3$ which is the average boron concentration of the membranes estimated from the SRP data. Since the difference between ϵ_A and ϵ_B is due

to the insertions of the extra-half planes accompanying misfit dislocations, the density of extra-half plane in the conventional membrane can be calculated as¹¹

$$N = (\epsilon_A - \epsilon_B) / b = 2.3 \times 10^4 / \text{cm}, \quad (2)$$

where $b = 3.840 \text{ \AA}$ is the magnitude of Burgers vector of misfit dislocation in p^+ silicon layer.² The density of extra-half plane, i.e., density of misfit dislocation of $2.3 \times 10^4 / \text{cm}$ is very difficult to determine by transmission-electron-microscope analysis because of the relatively large dislocation space (about few thousand \AA). Thus, our experiments are very useful to determine the misfit dislocation density and lattice constants of a p^+ silicon layer.

In conclusion, we have successively fabricated the p^+ silicon membranes for x-ray mask substrate which have better mechanical properties than the conventional p^+ silicon membranes. The membranes show smoother surface, better optical transmission, higher fracture strength, and higher residual tensile stress than the conventional membranes. Using the extracted residual strains, we can determine the lattice constant of the strain-free p^+ silicon membrane, and the in-

plane lattice constant and density of extra-half plane of the conventional membrane.

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