X-Ray Topographic Observation of Strain Generated by Thin Film (TiN) on Silicon Surface

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(Received January 8 , 1994)

SYNOPSIS

The strain in Si substrate induced by locally ion-plated thin film of TiN was observed by X-ray topograph (Lang technique). Circular TiN film was deposited on one side of the Si surface. In all topographs the highest blackness attributed to kinematical diffraction effect occurred at the film edge. Rosette pattern with four-lobes was observed around the film. Blackness as a whole increased with the film thickness. Strain was observed in the depth direction of substrate by limited projection method. When the slit width was narrowed, the kinematical images disappeared, and white images appeared at the film edge. All the contrast disappeared when the TiN film was completely removed in boiling HNO_3. The strain induced by the film deposition was proved to be elastic.

1. INTRODUCTION

Recently TiN, SiO_2 etc. have been used as barrier metal in LSI. TiN film on Si substrate, however, has large compressive stress in it, consequently strain is generated in Si substrate. The causes of stress are, (1)the thermal stress generated by the difference in thermal expansion coefficient between film and substrate, and (2)the stress generated by the growth process of thin film. [1, 2]

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This stress gives significant influences on the electric and electronic property of substrate semiconductor.

X-Ray topographic observations of the strain in Si by the SiO_2 were carried out by several authors, where the shape of oxide film was composed of straight lines.[3, 4, 5] The strain in Si induced by disk-like TiN thin film was studied only by Mikuni et al.[6] It is important to know the extent, the level and the anisotropy of the strain field. In this experimental, this strain was observed by X-ray topograph(Lang technique) and judged whether the strain induced by the TiN film is plastic or elastic.

2. EXPERIMENTAL PROCEDURES

The substrate of Si wafer, parallel (111) surface and 0.5mm thick, was cut out with a fine cutter, as shown in Fig.1. Circular TiN film, 3mm in diameter, was deposited on one side of the Si surface by ion-plating, under the following conditions; N_2 pressure is 5×10^{-4} Torr, RF power is 600W, bias voltage is -200V, temperture of substrate is 773K. The film thicknesses are (a)0.1 μm, (b)0.2 μm, (c)1.0 μm, (d)1.3 μm, (e)2.0 μm, and (f)3.0 μm. Topographs of (20$\bar{2}$) reflection were taken by the Lang method, using MoKα radiation, with the TiN film on beam exit surface faced toward the nuclear photographic plates (EM Type G-2F 50). The photographic density of topograph was measured by use of microphotometer, along the line A-A' through the center of TiN film circle, as shown in Fig.1.

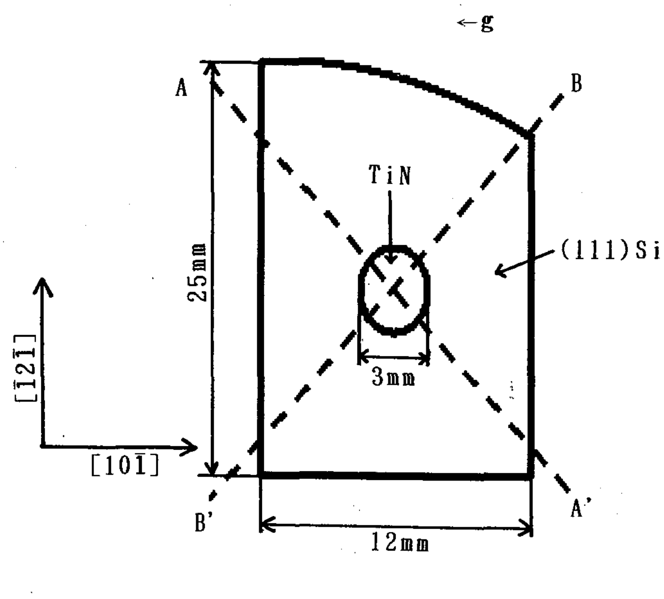

Fig.1 Shape of substrate and position of density mesurement.

Optical geometry is shown schematically in Fig.2. The slits L_3 were adjusted to allow only part of the diffracted X-ray beam to reach the film. The width of the slits L_3 was changed from 0.5mm to 0.1mm by closing from one side to another.

TiN film was dissolved by priority to Si substrate in boiling HNO_3. TiN film was completely removed, but the surface condition was the same as that before this chemical treatment. Dissolved quantity of Si into HNO_3 was measured. When only HNO_3 was boiled without substrate, quantity of Si in HNO_3 was 0.0024 $\mu g/ml$. After the TiN film was removed, quantity of Si was 0.0599 $\mu g/ml$. Therefore the quantity of Si dissolved from the substrate was 0.0575 $\mu g/ml$, corresponding to the substrate thickness about 1.0 μm. The topographs taken after the TiN film was removed were compared with those taken before the removal.

In photographs of this paper, the image is positive and the contrast is opposite to the topograph plates.

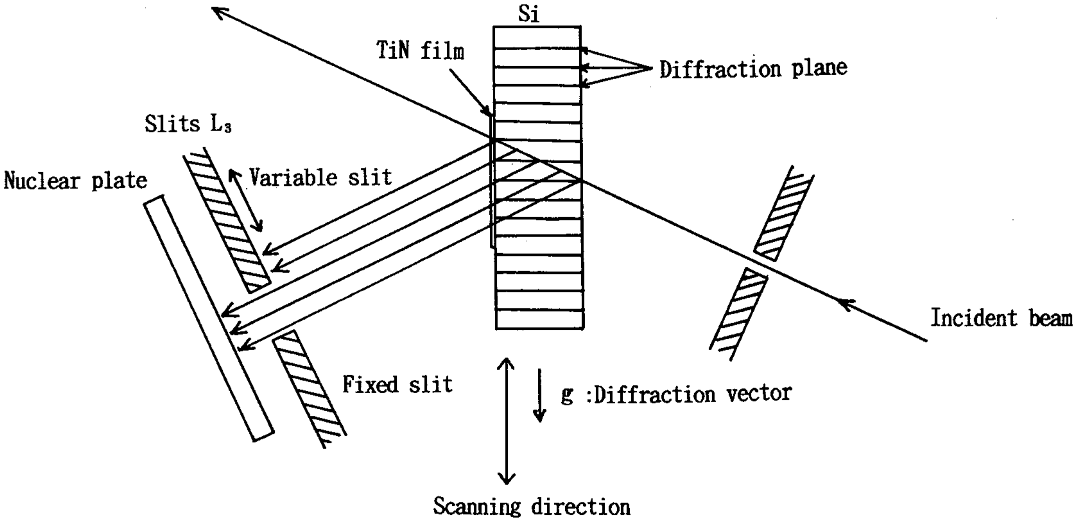

Fig.2 Optical geometory.

1 EXPERIMENTAL RESULTS

Photos 1(a)~(f) are X-ray topographs for different thicknesses of the TiN film. Density curves of these topographs along the line A-A' are shown in Fig.3(a)~(f), respectively. The highest density was observed at the TiN film edge in each topograph. But the contrast disappeared in the $[1\bar{2}1]$ direction, where the strain vector **H** is normal to the diffraction vector **g** .

When the TiN film thickness is 0.1 or 0.2 μm, contrast other than that at the film edge was not observed. When TiN film thickness is more than or equal to 1.0 μm, the radial black images from the film edge appeared. But the image does not occur in $[1\bar{2}1]$ and $[10\bar{1}]$ direction. As a result, the contrast was like a four-lobed rosette pattern. The difference of blackness was observed between orientation A-A' and B-B'. The blackness of orientation A-A' was higher than B-B'. In Fig.3(c) ~(f), peaks of density occurred at the TiN film edges, from which outward gentle shoulders appeared. As the thickness of film increased, the density as a whole increased and the shoulder broadened.

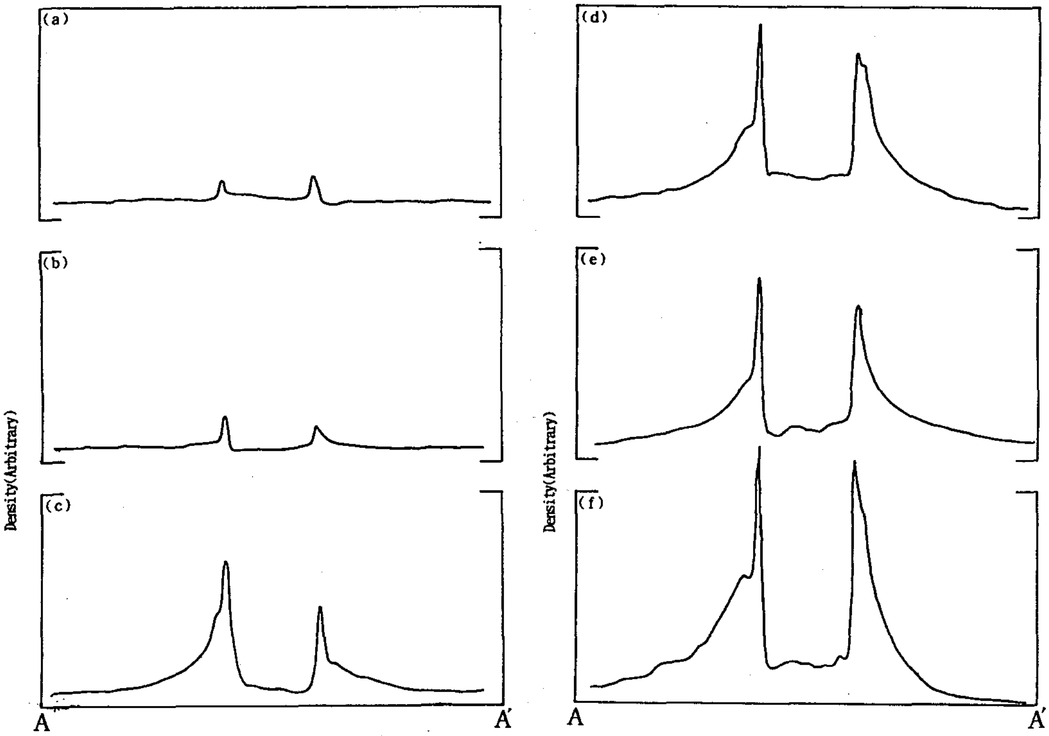

Fig.3 Density curves for the TiN film thickness (a)0.1 μm (b)0.2μm (c)1.0μm (d)1.3 μm (e)2.0μm (f)3.0μm

The density measured at the position 0.3mm distant from the film edge is plotted against the film thickness (Fig.4). As the film thickness increased, the density increased, rapidly until about 0.2μm and then gradually and linearly. This result is somewhat different to the result of Blech and Meieran[7] for the oxide film where the density simply depended linearly on the oxide thickness.

Topographs for the TiN film 1.0μm thick were taken under the limited projection conditions, the width of slits L_3 being 0.5mm, 0.4mm, 0.3mm, 0.25mm, 0.2mm, 0.15mm, and 0.1mm, and are shown in Photo 2(A)~(G). Density curves of these topographs are shown in Fig.5(A)~(G). As the slit width was narrowed, the density decreased as a whole. When the slits L_3 are 0.5~0.25mm, the peak of density occurred at TiN film edge, as in Fig. 3(a)~(f). When the slit width was narrowed to 0.2mm or less, this peak disappeared and a trough appeared at the same position.

After the TiN film was removed under the above condition, topographs were taken. They had no contrast other than the background.

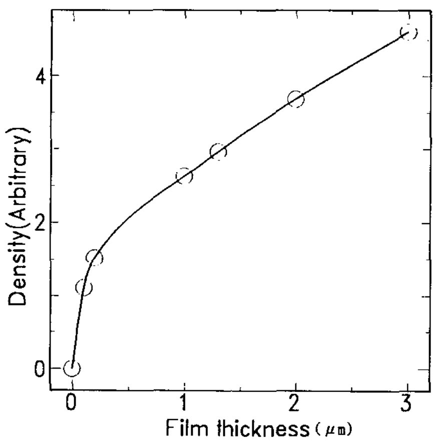

Fig. 4 Density as a function of film thickness

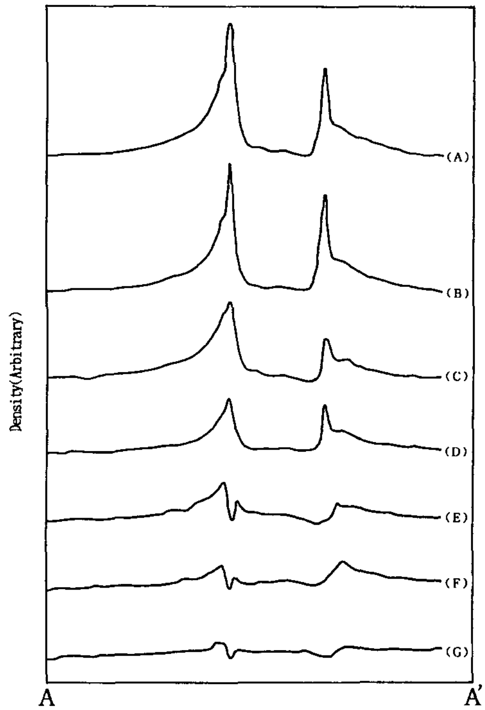

Fig. 5 Density curves for the L_3 slit width(mm) (A)0.5 (B)0.4 (C)0.3 (D)0.25 (E)0.2 (F)0.15 (G)0.1

4. DISCUSSION OF RESULTS

Contrast in topograph is interpreted in terms of the kinematical and dynamical image components. The highest density at film edge in all topographs is attributed to the kinematical diffraction effect. The faint image radiated from the film edge in the topographs and the corresponding shoulders in the density curve, which appeared when the TiN film thickness is more than 1.0 μm (Photo 1(c)~(f), Fig. 3(c)~(f)), may be attributed to the dynamical diffraction effect.

As the film thickness increased, whole density including the peak became higher and the shoulder became broader, as was seen in Figs. 3 and 4. This indicates that the strain generated in the substrate increased and extended with the film thickness.

The slit L_3 was closed from one side (Fig. 2), namely, kinematically diffracted beam was gradually obstructed. As the slit width was changed from 0.5 to 0.25mm, the height of density peak at TiN film edge became lower (Fig. 5). In the kinematical theory of diffraction, when an incident X-ray beam has divergence of a few minutes of arc, the diffracted X-ray beam from defect has divergence of a few minutes of arc. The diffracted divergent X-ray beam was gradually obstructed by narrowing the slits, which reduced the intensity of kinematical diffraction component. When the slit width was narrowed to 0.2mm or less, the kinematical peak completely disappeared, and a trough appeared at the same position. This is able to be explained by means of dynamical theory of diffraction when kinematical images are completely suppressed. If Si substrate is much strained underneath the TiN film edge, the incident X-ray beam does not satisfy Bragg's law in this region. Consequently multiple reflection (dynamical diffraction) does not occur. This results in a lower peak in comparison to the background (a trough). The whole of density became lower as the slit width became narrower. When the slit width was 0.1mm, slight amount of density in comparison to the background remained. Therefore, the strain reached to a depth not less than 0.4mm from the surface.

The purpose of removing the TiN film was to judge whether the strain was elastic or plastic. If the strain was plastic, there should be residual strain after the TiN film was removed from Si and the corresponding contrast should be observed. The result that no contrast was observed after the film was removed, therefore, indicates that the strain was elastic.

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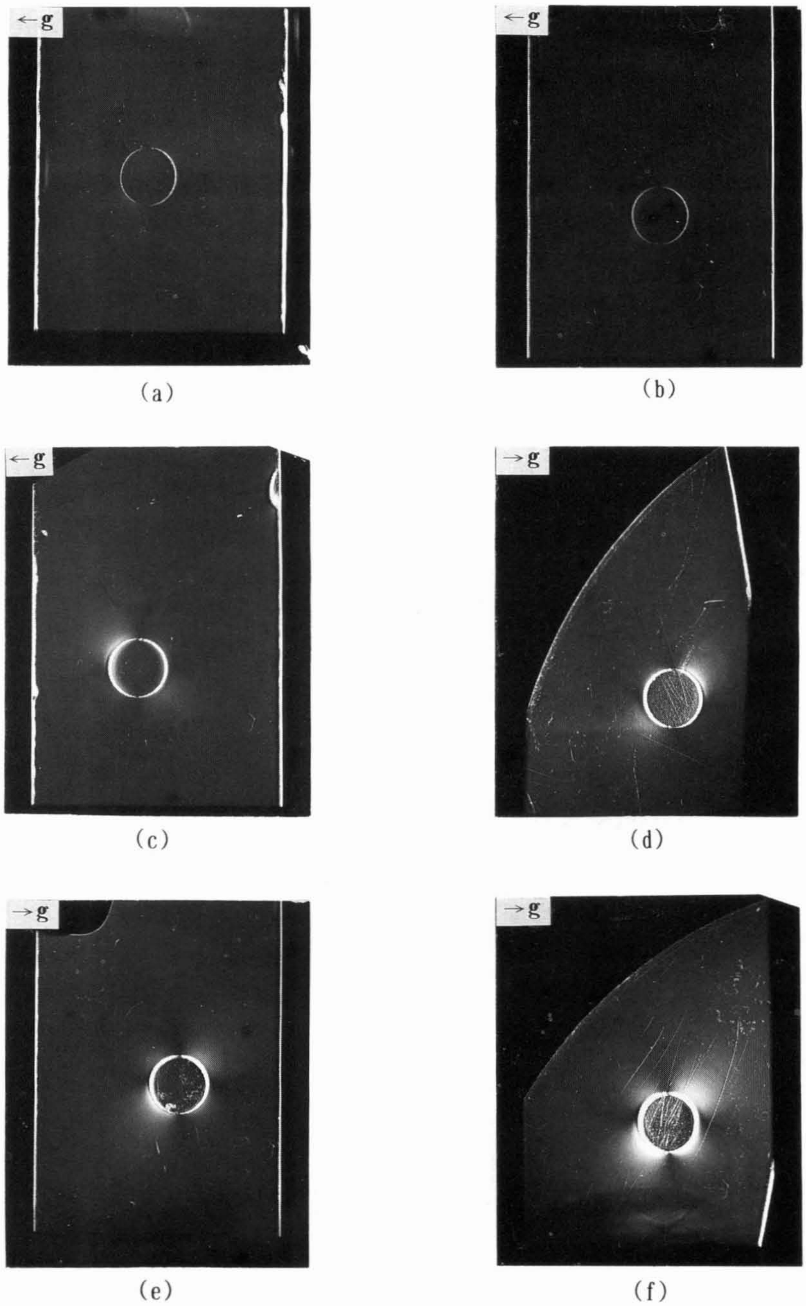

g :Diffraction vector

Photo. 1 Topographs for TiN film thickness
(a)0.1 μm (b)0.2 μm (c)1.0 μm (d)1.3 μm
(e)2.0 μm (f)3.0 μm

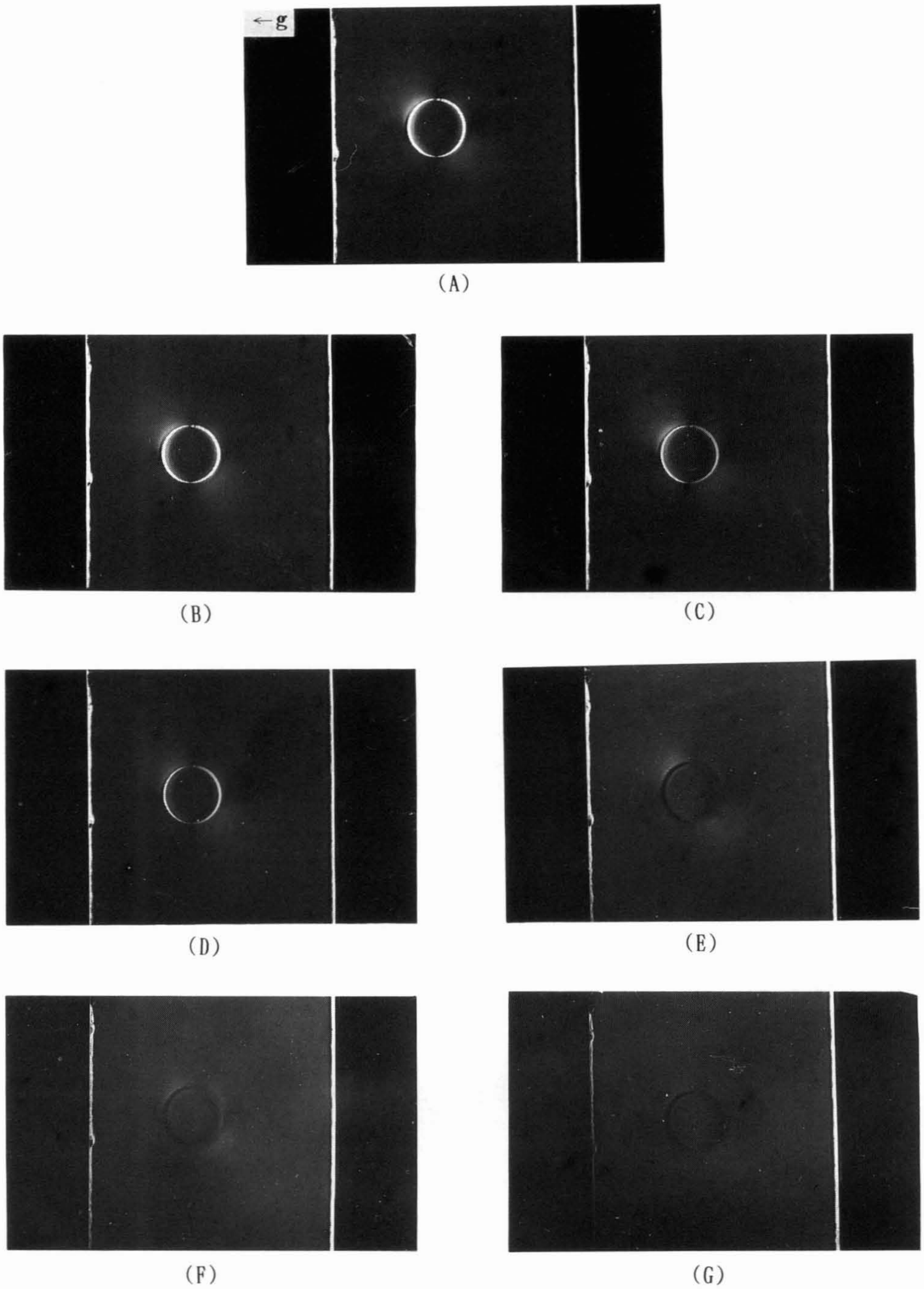

Photo. 2 Topographs (TiN film 1.0 μm thick) for the slit width (A) 0.5mm (B) 0.4mm (C) 0.3mm (D) 0.25mm (E) 0.2mm (F) 0.15mm (G) 0.1mm