Memoirs of the Faculty of Engineering, Okayama University, Vol.25, No. 2, pp. 9 -15, March 1991

X-ray Topographs of Strain Field Induced by Locally Ion-Plated Films on Si Substrates

Masato MIKUNI*, Moritaka HIDA*, Norihide NISHIDA**
Akira SAKAKIBARA* and Masuo YAMADA

(Received February 12 , 1991)

SYNOPSIS

Strains induced in the Si substrates by TiN film were observed with X-ray topography. The image of the sample with TiN film 0.45 μm thick was like that of a dislocation loop observed with transmission electron microscope. The images of the samples with TiN films 1.65, and 1.9 μm thick were different; blackening spreaded in the <112> and <110> direction from the ring contrast in shape of four-lobed rosette pattern. Spreading extended 1.6 times longer than the radius of the ring contrast along the <112> direction. The strain field extended 0.1 μm in depth from the top surface where TiN was plated. From the topographs of bent Si beam, it was found that the blackness was almost proportional to the strain. The strains induced by TiN film locally ion-plated were smaller than those observed previously when TiN was ion-plated on the whole top surface of the substrate. Fine structures were observed in the topographs which could not be explained by the kinematical theory.

1. INTRODUCTION

Studies of the internal stress in thin films of SiO_2, W, TiN and several kinds of metal have been done by many laboratories.(1) Recently TiN came to be

* Department of Mechanical Engineering
** Industrial Technical Center of Okayama Prefecture

used as a "barrier metal" for the LSI. It is well known that a large compressive residual stress occurs in the TiN films made on a substrate by ion-plating, and it was reported that this stress causes significant influences on the electric property of substrate semiconductor. For the TiN films ion-plated on the whole top surface of an Si wafer, measurement of the internal stress in the thin film has been attempted.(2) But for the case of TiN locally ion-plated on the substrate, distribution of the strain in the substrate has not yet been investigated. In the present study the distribution of the strain in the Si substrate was measured by the X-ray topography when circular thin film of TiN was made on the substrate. The strain was evaluated from the blackness of topograph, using the blackness vs. strain relation obtained by the topographic measurement for various know strains.

2.EXPERIMENTAL PROCEDURES

Rectangular pieces of Si plate, of which the dimensions and the orientation are shown in Fig.1, were cut out from a wafer with a fine cutter and used as substrates. Si wafers used in this experiment were dislocation free, n-type, and of <111> direction, on which TiN was ion-plated. Thin films were made under the following conditions; N_2 pressure is $5x10^{-4}$ Torr, RF power is 600W, bias voltage is -200V, temperature is 773K. Film thicknesses are 0.45, 0.95, 1.65 and 1.95μm. Transmission topographs were taken by use of Lang camera (Rigaku A3). A normal X-ray tube was used as a point source of X-rays, 1x1mm^2, and MoKα_1 radiation was used for topography. The ability of resolution of the Lang camera is 29μmx47μm. Two kinds of photographic emulsion, industrial X-ray films (Fuji X-ray film, industrial 150) and nuclear plates (EM Type G-2F 50), were

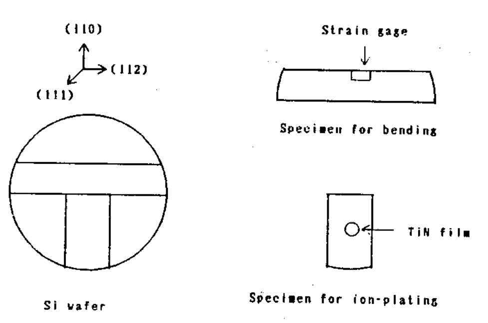

Fig.1 Shape of the sample crystal and crystallographic orientation.

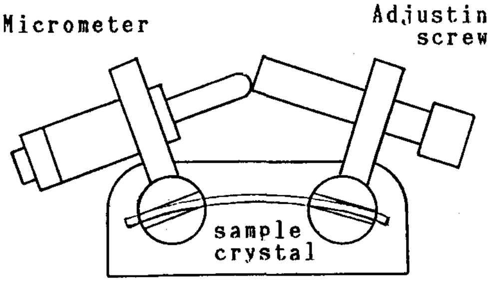

Fig.2 Scheme of the bending jig.

used.

The Si beam was bent by use of a four-point bending jig shown in Fig.2, a modification of the one used by Bonse and Graeff.[3] It can be set at the top of the goniometer head and used with the Lang camera as shown in Fig.3. Four-point bending gives no shear stress in the inner span. Only bending moment acts in the region where X-ray scans. The sample for the bending beam experiment was obtained from the same Si wafer as used for TiN ion-plating. A rectangular Si piece shown in Fig.1, 40x10mm^2, was cut off, long side being parallel to the <112> direction, by use of a fine cutter. A strain gauge was sticked on it and a known strain was given. Then its topograph was taken using (111) as the diffraction plane. The time of exposure was determined considering the proportionality of the blackness of films to the exposure time. The blackness of the topographs was measured by use of a microphotometer (Narumi C Type). The beam size of the microphotometer was 5x100μm.

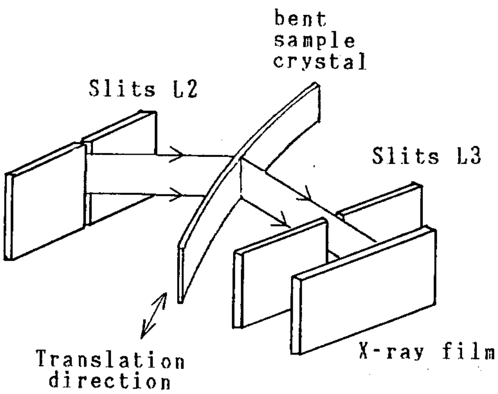

Fig.3 Experimental set up for the topography of the bending beam.

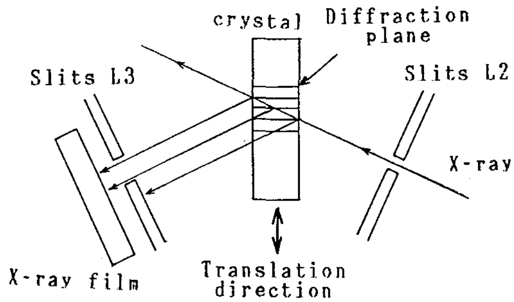

Fig.4 Scheme of the limited projection.

To investigate further the distribution of the strain along the direction of the thickness in the substrate, limited projection method was used where the width of L_3 slit was adjusted for the restriction of diffracted X-ray to be passed, as shown in Fig.4. Thus topographs were taken from a portion of the substrate ranging 1/2 and 1/4 of the whole thickness from the side where TiN was not ion-plated.

3.RESULTS AND DISCUSSION

All topographs of the samples on which TiN was ion-plated have a significant contrast around the TiN films. Along the <112> direction the blackness is the most of all directions, and along the <110> direction it is the least, similar to the transmission electron microscopic image of a dislocation loop. That's why the (111) plane was chosen as the diffraction plane. When we define **g** as a diffraction vector and H as a unit vector which is normal to and pointing away from the film edge, diffracted beam intensity from the region where the value of $\mathbf{g}\cdot\mathrm{H}$ is the largest is enhanced the most of all directions. Thus appears contrast against the gray background which corresponds to the undistorted region and the region where the value of $\mathbf{g}\cdot\mathrm{H}$ is nearly zero. The topograph of the sample of which TiN film thickness is 0.45 μm (Photo 1(a)) shows this situation clearly. As the film thickness is getting thicker, blackness on the ring contrast observed in the topograph increased in the <110> direction and became darker than other undistorted regions. Dark regions spread to the <112> and <110> directions from the ring contrast in the topographs of the samples with TiN film 1.65 and 1.9 μm thick. The spreading extended 1.6times longer than the radius of the ring contrast. But since such blackening did not occur in the <121> directions, the strained region as a whole showed a four-lobed rosette pattern in the topographs shown in Photo 1(c) and (d). This pattern was also observed by Carron and Walford[4], who took the topographs of dislocation stress fields in Si generated by electron beam bombardment.

Density curves obtained with microphotometry are shown in Fig.5. It is clearly shown that blackness near the edge where $\mathbf{g}\cdot\mathrm{H}<0$ is larger than that near the edge where $\mathbf{g}\cdot\mathrm{H}>0$. This result is not corresponding to the result obtained by Blech and Meieran.[5] Fine structures composed of oscillating profiles are observed around the left side peak, while such structures are not

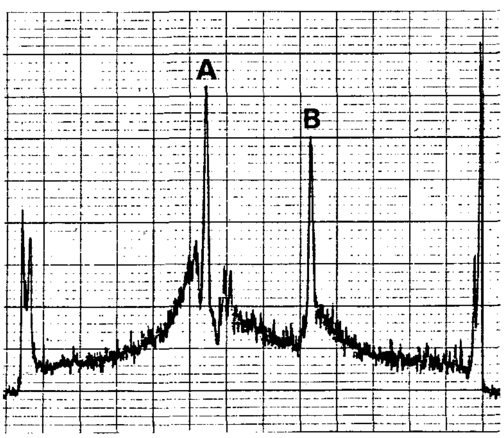

Fig.5 Density curve for the sample with TiN 1.9 μm thick.
A: $\mathbf{g}\cdot\mathrm{H}<0$, B: $\mathbf{g}\cdot\mathrm{H}>0$.

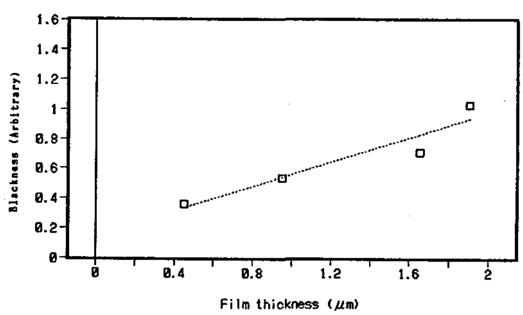

Fig.6 Blackness as a function of TiN film thickness.

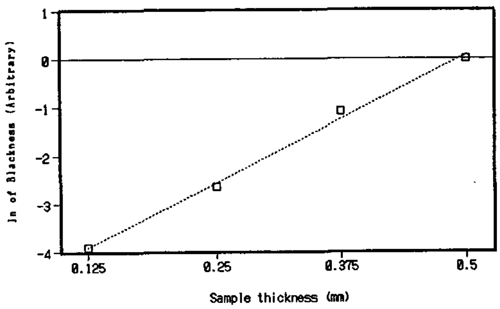

Fig.7 Blackness as a function of limited diffracting thickness.

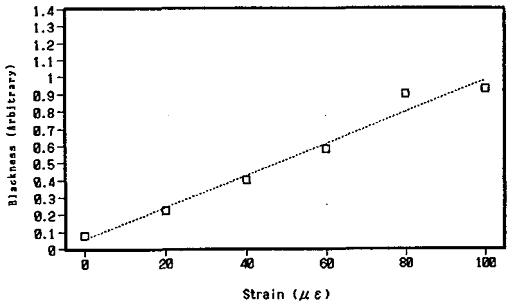

Fig.8 Blackness as a function of bending strain.

observed around the right side peak. This asymmetry cannot be explained by the kinematical theory, and dynamical effect must be taken into account. The relationship between the blackness and the film thickness is shown in Fig.6. It is found that the blackness is getting darker as the film thickness gets thicker.

The relation of the blackness to the distance from the bottom surface where TiN is not plated is shown in Fig.7. The distance where the blackness becomes 1/e that of the whole thickness is 0.4μm. It is considered that the strain field extends to 0.1μm from the side where TiN is plated.

Relationship between the blackness and the strain was obtained from the topographs of an Si beam to which were given known strains, 20μ, 40μ, 60μ, 80μ, 100μ. As the strain was getting larger, the region which miss the Bragg angle appeared from both ends of the bent sample crystal and the image shrinks and becomes darker (Photo 2). The blackness measured was plotted in Fig.8 against the strain. Proportional relationship is found in it. Strains in the Si substrate on which TiN was ion-plated were estimated from the blackness measured and this relationship. Table 1 shows the values of strain thus obtained, together with the values previously obtained by measuring the residual stress in TiN films ion-plated on the whole top surface of Si wafers.[(2)] Present values as a whole are smaller than those previously obtained. This contradiction may

Table 1 Strain in the substrate for various thicknesses of the TiN film

TiN Film Thickness (μm)	0.45	0.95	1.65	1.9
Strain from Blackness(μ)	34	54	71	109
Strain from Displacement*(μ)	43	68	112	124

* Ref (2)

be due to the difference in the quality of the TiN film and to the difference in the area covered by the film. Deformation of the substrate on which TiN is locally ion-plated is considered to be small because the area on which the stress was directly applied by the film is small relative to the case of the substrate of which the whole top surface is covered with the film.

REFERENCES

(1) E. Klokholm and B. S. Berry, J. Electrochem. Soc., 115(1968), 823K.

(2) N. Nishida, H. Kawasaki, K. Honda, and N. Hosokawa, J. Metal Finishing Soc. Japan, 37(1986), 346.

(3) U. Bonse and W. Graeff, Z. Naturforsch., 28a(1973), 558.

(4) G. J. Carron and L. K. Walford, Appl. Phys. Lett., 7(1965), 304.

(5) I. A. Blech and E. S. Meieran, J. Appl. Phys., 38(1967), 2913.

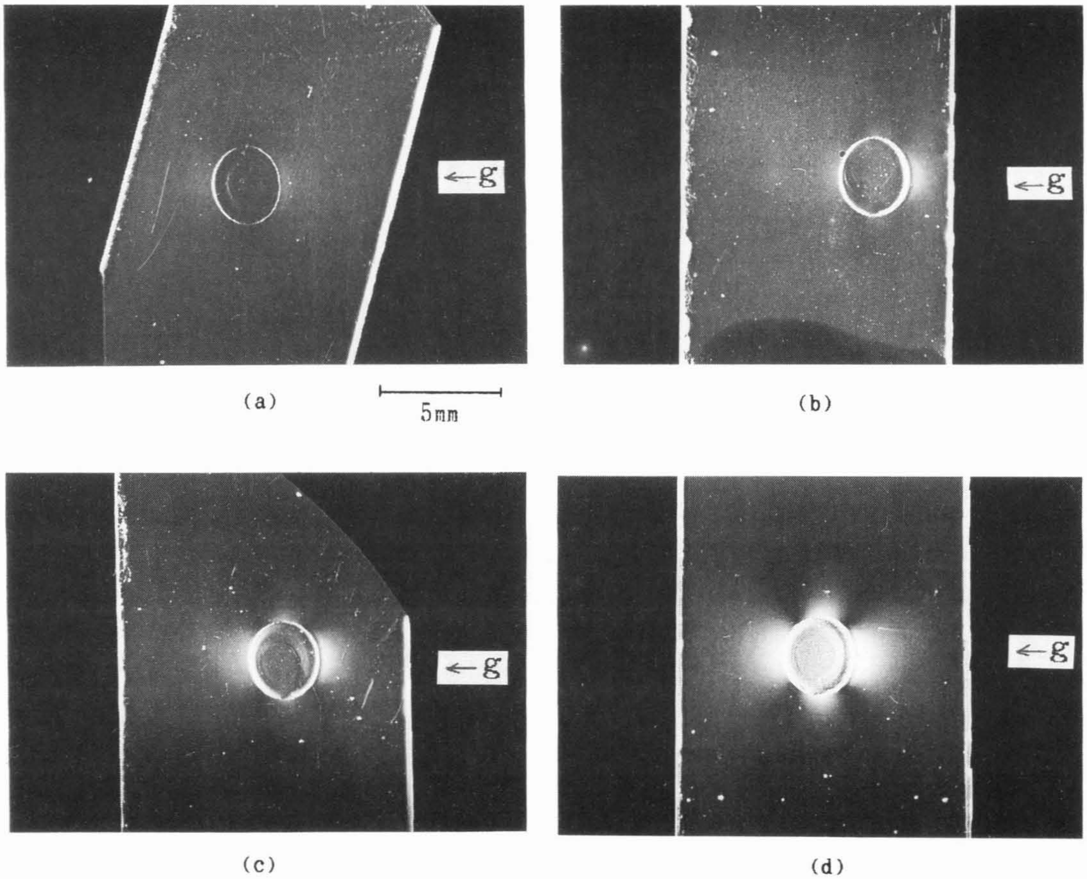

Photo 1 Topographs (positive images) of the samples with TiN films of various thicknesses: (a) 0.45 μm, (b) 0.95 μm, (c) 1.65 μm, (d) 1.9 μm

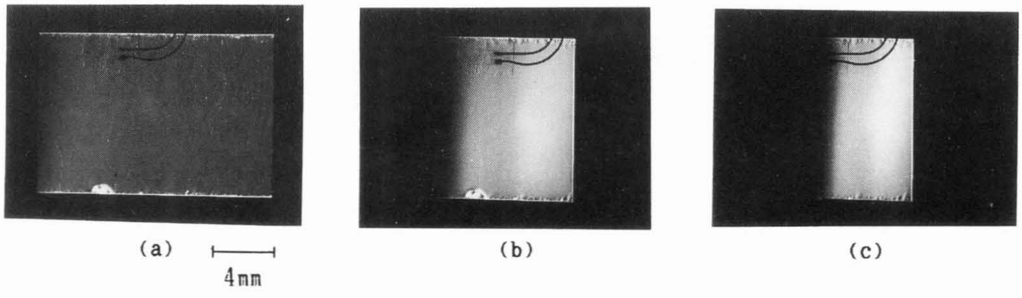

Photo 2 Topographs (positive images) of bent sample crystals which received various strains: (a) 20 μ, (b) 40 μ, (c) 60 μ.