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# Development of an Innovative $5\mu m\phi$ Focused X-ray Beam Energy Dispersive Spectrometer for Analyzing Residual Stresses and Impurities in ULSIs

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A 5.7 $\mu$  m $\phi$  X-ray micro-beam is attained by development of an approximately parabolic inner-wall X-ray guide tube made of glass, and a Li diffused Si X-ray detector with a large area of 200mm<sup>2</sup> is developed for detection of weak X-rays. An energy dispersive spectrometer incorporating these components can simultaneously measure diffraction X-rays for observing local reactions and residual stresses and fluorescent X-rays for analyzing least amount impurity elements at microregions during ULSI processes ( $\leq 900$ °C).

#### 1. INTRODUCTION

Many severe problems have arisen with the shrinking and increasing complexity of elements on an LSI, with the coming of an LSI generation with a sub-micron layout rule as a 1 or 4-mega bit dynamic memory.

Strong residual stress in a gate electrode degrades MOS characteristics . 1). 2) Opening of an Al interconnect is frequently caused by stresses as a "stress-migration" of an Al element.<sup>3)</sup> A heavy metal impurity (contamination) increases leakage current at a shallow pn junction, thus reducing LSI production yield. These many problems awaiting solutions for fabrication of highly reliable ULSIs have stimulated interest in development of instruments for analysing stress, impurity and reaction at a microregion during ULSI fabrication.

X-ray analysis technology is found to have superior accuracy, sensitivity and nondestructive properties to other technology. However, it is difficult to forcus an

<sup>1</sup>Present adress: Faculty of Industrial Science and Technology, Science University of Tokyo, Noda-shi, Chiba, 278, Japan X-ray beam measuring probe to an extremely small diameter in the micrometer level.

In this study, we succeed in forming a 5.7 $\mu$ m diameter focused X-ray beam and developing an innovative micro-X-ray analysis system which can simultaneously measure local stresses, impurities and reactions on ULSIS.

### 2. SPECIAL FEATURE OF SPECTROMETER SYSTEM

The very small diameter X-ray beam in the order of a few microns  $\Phi$  is applied to a micro-region of a ULSI. Then, diffraction X-rays for measuring residual stresses and reactions and fluorescent X-rays for analyzing the least amount impurities are emitted from the micro-region. Their energies and intensities are detected by a solid-state detector (SSD). A diagram of the spectrometer is shown in Fig. 1. It has the following features and performance characteristics.

2. 1 MECHANISM OF FOCUSING X-RAY BEAM AND MONITORING OF X-RAY APPLIED POSITION ON A SAMPLE

An X-ray guide tube (XGT) made of glass is used for focusing X-rays emitted from a commercially available X-ray generator, in the order of a few microns. The inside wall of the XGT has an approximate parabolic shape. The X-rays entering the XGT are totally reflected many times at the surface of the inside wall and finally focused (Fig. 2). The diameter of the outgoing X-ray beam is 5.7  $\mu$  m at 60mm from the XGT outlet. The divergent angle of the beam is 8. 5x10<sup>-5</sup> rad.



sample heating :  $\leq 900^{\circ}C$ 

Fig. 1 Block Diagram of Focused X-ray Beam Spectrometer



Fig. 2 Cross Section of X-ray Guide Tube made of Glass showing Principle of Focusing X-rays

This angle is very small and comparable to that of an X-ray radiated from a syncrotron radiation source (SR) as shown in table 1.

A laser beam is used to monitor the position at which the X-ray beam is applied to a sample. It is introduced onto the sample using a mirror and a convex lens which is a coaxial with the XGT.

# 2. 2 DETECTOR OF VERY WEAK DIFFRACTION AND FLUORESCENT X-RAYS

A Li diffused Si SSD which covers very large area of 200 mm<sup>2</sup> is developed. A signal treatment system consisting of а highly stabilized amplifier circuit and gaussian fitting software are developed. Tn Fig. 1, a linear amplifier and analog-digital converter for signals from an SSD are installed in a constant temperature box to eliminate their characteristic drifts. This detection system has 20 times more sensitivity in weak signal detection and 10 times more accuracy in X-ray energy resolution (0.3eV) than conventional SSD (10eV).

Table	1	Characteristics	of	X-ray	Beam
		Focused by XGT			

	5.7µm ¢		
Beam Diameter	*		
	[10-1~10-3]		
	*		
Intensity	10~1000		
	**		
Divergent	0.8~2.0 (x10 <sup>-*</sup> rad)		
Angle			

%Ratio to X-ray beam formed by pin hole collimator %%Comparable to a divergent angle of an X-ray beam irradiated from a syncrotron radiation source

#### 2. 3 GONIOMETER AND SAMPLE HEATING

A high accuracy goniometer is developed. It has x, y, z axes with  $1 \mu m$  positioning accuracy on 5 inch wafers, and  $\theta$  (goniometer rotation),  $2\theta$  (detector rotation) and tilt axes for measuring X-ray diffraction. A sample stage can be rotated in its plain for analyzing vector components of stresses.

While samples are heated ( $\leq 900$ °C) in various atmospheres of inert gas and air or vacuum ( $1x10^{-5}$ Pa), diffraction X-rays and fluorescent X-rays can be observed by the spectrometer. Thus, local reactions, stresses (strains) and impurities in high temperature LSI processes can be analyzed in situ.

#### 3. APPLICATIONS

In this section, a tungsten interconnect, whose application to ULSIs is being watched with keen interest, is applied to performance eveluation of this developed spectrometer.

#### 3. 1 STRESS MEASUREMENT

The X-ray irradiated from an XGT has the continuous energy characteristic. The lattice constant of a material for measuring a residual stress is determined using Bragg's law.

 $2d\sin\theta = \lambda \tag{1}$  $=hc/E \tag{2}$ 

d:spacing of lattice plain

 $\lambda$ , E: incident X-ray wave length and energy  $\theta$ :Bragg's angle, h:Plank's constant c:light velocity

Two analytical methods for X-ray diffraction can be applied in this system. One is an energy dispersive (E:variable, heta :constant) X-ray diffraction and the other dispersive X-ray diffraction is a wave  $(\theta : variable,$  $\lambda$  :constant). The former is superior for analysis at a micro-region. Because the energy dispersive method measures without mechanical driving, the Xray applying positions on samples can be fixed exactly while measuring. The energy dispersive X-ray analysis is applied to a tungsten gate electrode and interconnect in a 1 Mb D-RAM is produced by way of a trial experiment. The diffraction and fluorescent X-rays are simultaneously detected as shown The horizontal energy axis is ac-Fig. 3. curately calibrated using the fluorescent Xrays of Si and W, and the characteristic Xrays from a Mo target X-ray source. The Bragg's angle  $\theta$  is also determined exactly using (400) diffraction X-ray energy of a single Si crystal in which the spacing of the lattice plain is known. As a result, it is made clear that a tungsten interconnect, after the accomplishment of LSI fabrication, has a high internal tensile stress of 234.5kg/mm<sup>2</sup> and the detection limit of a stress in Si crystal is 0. 1kg/mm<sup>2</sup>.





## 3. 2 IMPURITY ANALYSIS

Total reflection X-ray fluorescence (TRXRF)<sup>5)</sup> can be realized in this spectrometer by making the most of the superior characteristics of the very low divergent Xray micro-beam irradiated from an XGT. This has been noted as an analytical method of least amount elements. A thin tungsten film of 50nm on a SiO<sub>2</sub>(20nm)/Si-substrate is applied to determine the detection limit of least amount elements on this system. When the incident angle of an X-ray beam to a sample is 1° (nearly total reflection condition), the detectable minimum thickness of tungsten is 0.27nm from a W-La peak to background ratio, as shown in Fig. 4.





PI O This is close to a tungsten atomic monolayer and equals about 4.7pg weight of tungsten. On the other hand, the detection limit at a high incident angle  $(45^{\circ})$  is about 10 times thicker ( $\approx$  3nm) than that of a low angle (1°  $\approx$  TRXRF). This high sensitivity in TRXRF results from an increase in signal peak intensity in contrast to a decrease in background X-rays with a lowering of the incident angle of the X-ray beam.

#### 4. SUMMARY

An innovative focused X-ray beam spectrometer is developed for analyzing local residual stresses reactions and least amount impurities at micro-regions in ULSIs. It results from the successes in the following developments. (1) an X-ray guide tube for focusing and strengthening an X-ray be-(2) a Li diffused Si SSD with a very яπ. large solid angle for detection of weak X-(3) highly stabilized amplifiers for ravs. improvement of X-ray energy resolution. The X-ray beam irradiated to a sample is 5.7  $\mu$  m  $\Phi$  and has a low divergent angle, comparable to that from an SR. The minimum analyzing area is reduced to 1/10~ 1/1000 and the irradiation X-ray beam intensity is strengthened to 100 times that of a commercially available X-ray diffractometer with a pin-hole collimator.

The performance comparison of stress analysis instruments for LSIs is shown in table 2. Although micro-ramman spectroscopy can observe stress in the sub-micron region, it does not have general-purpose utilization capability because it can not observe residual stress in a metallic layer. The spectrometer in this work can measure local stress in a Si crystal and a metal layer of a electorde or multi-level interconnect, and furthermore can simultaneously analyze an impurity element. Finally, it should be added that there are very few reports on Xray diffraction measurements for thin films at micro-regions as shown in Fig. 3 even when SR is applied to an X-ray generator.

#### Table 2 Performance Comparison of Stress Analysis Instrument for LSIs

	₩ this work	** A	ЖЖ В
Analysis method	X-ray diffraction	ramman spectroscopy	X-ray diffraction
minimum measuring region	1.0µm¢	0.5~1.0μmφ	1mm~30mm 🗆
detection limit of stress	0.1kg/mm²	1.0~5.0kg/mm²	1.0~5.0kg/mm <sup>2</sup>
applicable material on LSI	metal, Si	Si	metal, Si

★ simultaneously measuring function for local stress-least elements(detectable limit of tungsten: atomic monolayer≒4.7pg)

%% data reported by othor autors

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