

## A New Technique for Quantitative Analysis of Metallic Contamination inside Deep-Submicron-Diameter Holes

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We developed a new quantitative method of analyzing the metallic contamination remaining in deep-submicron-diameter holes with high aspect ratios after cleaning. By using our new method based on atomic absorption spectrometry (AAS), metallic contamination of the order of  $10^{10}$  atoms/cm<sup>2</sup> can be easily analyzed inside 0.1 $\mu$ m-diameter holes with aspect ratios of 10. It was found that the concentration of Fe contamination remaining in such holes after SPM cleaning and DI water rinse was  $2.8 \times 10^{11}$  atoms/cm<sup>2</sup>, which was about 16 times greater than that remaining on the surface. The new analyzing method developed in this study can be effectively used to control metallic contamination in various deep-submicron structures.

### 1. INTRODUCTION

By down scaling very large-scale integrated devices, the dimensions of device fine structures have been decreased to about 0.1 $\mu$ m with aspect ratios of 10. With such fine structures, there is serious concern about that contamination, induced by dry etching or ion implantation, even after cleaning. With metallic contamination, degradation of device characteristics such as leakage current increase easily occurs. In order to ensure the high reliability of device characteristics, it is necessary to achieve low contamination levels after cleaning.

To find a suitable cleaning process for fine structures, a method of examining contamination inside the structures is needed. The conventional technology for metallic contamination analysis, however, has not sufficiently satisfied such a requirement. For example, an analysis method with high sensitivity, such as total reflection x-ray fluorescence (TXRF), or inductively coupled plasma mass spectrometry (ICP-MS), cannot be applied for analyzing the inside of deep-submicron structures. Methods which can be used for analyzing structures, such as field emission Auger electron spectroscopy (FE-AES), do not have a sensitivity of less than  $10^{14}$  atoms/cm<sup>2</sup>.

In the present work, we propose new analysis technology for metallic contamination in fine structures having dimensions of 0.1 $\mu$ m with aspect ratios of 10. This method is based on conventional atomic absorption spectrometry (AAS). Even though the technique is simple,

detection sensitivity is in the order of  $10^{10}$  atoms/cm<sup>2</sup>. Such convenience and high performance is suitable for the practical investigation of contamination control and cleaning processes of ULSI devices.

### 2. EXPERIMENT

The sample structure is schematically shown in Fig.1. Holes of 1 $\mu$ m-depth were fabricated in high temperature oxide (HTO) films all over the wafers. Two types of samples with 0.1 $\mu$ m and 0.2 $\mu$ m-hole diameter, were prepared. The hole area ratios were about 1.2% for the 0.1 $\mu$ m-diameter samples (a1), and about 4.9% for the 0.2 $\mu$ m-diameter ones (a2).

Figure 2 shows the experimental procedure for the quantitative analysis of metallic contamination in submicron structures; for example, Fe contamination inside a hole structure. Both types of SiO<sub>2</sub> films with holes (a) and without holes (b) were treated simultaneously. First the samples were intentionally contaminated with Fe by dipping in an APM (NH<sub>4</sub>OH / H<sub>2</sub>O<sub>2</sub> / H<sub>2</sub>O) solution containing 1 ppm Fe for 10 minutes at room temperature. Next the contaminated samples were cleaned by an SPM (H<sub>2</sub>SO<sub>4</sub> / H<sub>2</sub>O<sub>2</sub>) solution for 10 minutes and were rinsed in DI water for 4 minutes. After rinsing, the SiO<sub>2</sub> films of samples (a) and (b) were completely etched off by a DHF solution. The amount of Fe in the etching solution was analyzed by AAS.

The measured amounts of metallic contamination, in this experiment the amount of Fe, are to be treated as follows. The total amount of Fe contamination on the sample (a),

with holes, can be expressed as;

$A$  = the amount of Fe remaining on  $\text{SiO}_2$  (film surface + hole-side-walls + hole-bottoms) + the amount of Fe in  $\text{SiO}_2$  film.

The total amount of Fe contamination on sample (b), without holes, can be expressed as;

$B$  = the amount of Fe remaining on  $\text{SiO}_2$  (film-surface) + the amount of Fe in  $\text{SiO}_2$  film.

Therefore, the amount of Fe remaining inside the holes (hole-side-walls + hole-bottoms) is given by

$C \approx A - [B \times \{1 - \text{hole area ratio on the sample (a) surface}\}]$ .

The difference in  $\text{SiO}_2$  film volume between samples (a) and (b), due to the volume of holes in (a), is negligibly small.

### 3. RESULTS

Figure 3 shows the total amount of Fe contamination on wafers measured by AAS for samples (a1) with 0.1 $\mu\text{m}$ -diameter holes, (a2) with 0.2 $\mu\text{m}$ -diameter holes, sample (b) without holes, and a reference sample (a1 without Fe contamination). The Fe concentration inside the holes (hole-side-walls + hole bottoms) calculated by using the results from Fig. 3 and Equation C is shown in Fig. 4. As the hole diameter decreases, the remaining Fe concentration increases larger, although the surface area inside the holes decreases. The Fe concentration remaining inside the 0.2 $\mu\text{m}$ -diameter holes was  $5.1 \times 10^{10}$  atoms/ $\text{cm}^2$  and was about 3 times greater than that remaining on the film surface. In samples with 0.1 $\mu\text{m}$

holes, the Fe concentration remaining in the holes was  $2.8 \times 10^{11}$  atoms/ $\text{cm}^2$  and was about 16 times greater than that on the surface. The results show the concentration of Fe contamination, which still remains inside the holes after SPM cleaning and DI water rinse, drastically increases with decreasing hole diameter.

### 4. CONCLUSION

The present work established a new method of analyzing metallic contamination inside submicron structures with a high aspect ratio. This method can be applied to structures of less than 0.1 $\mu\text{m}$ , and with aspect ratios higher than 10. The minimum area ratio required for analysis is about 1%, which is sufficient for analyzing next-generation devices such as 256M bit or 1G bit DRAMs with a minimum hole area of about 2%. Applying the method for deep-submicron holes, the contamination of Fe remaining on hole-side-walls and hole-bottoms after cleaning was found to drastically increase with decreasing hole diameter and to reach about  $3 \times 10^{11}$  atoms/ $\text{cm}^2$  in 0.1 $\mu\text{m}$ -diameter holes. The new method investigated in this work and the results obtained can be effectively applied to device failure analysis and the development of new cleaning processes.

### Acknowledgment

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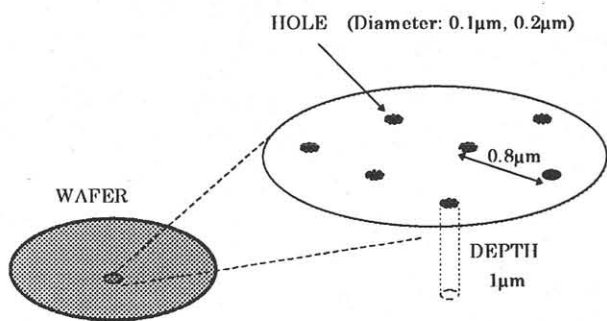


Fig.1 Structure of samples.

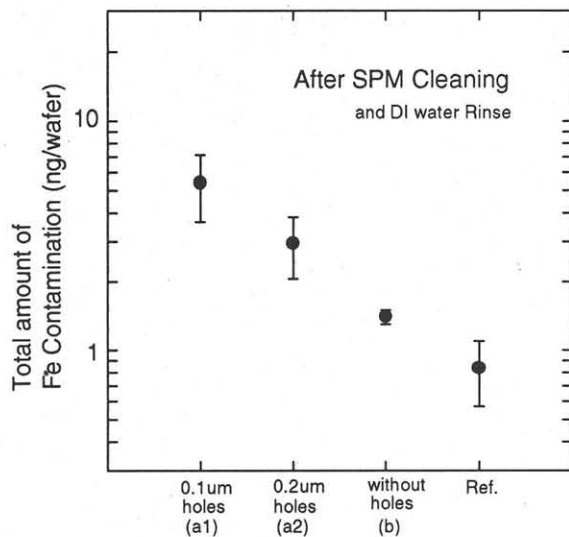


Fig. 3 The amount of Fe measured by AAS for the sample (a1), with 0.1 μm-diameter holes, sample (a2), with 0.2 μm-diameter holes, sample (b), without holes, and a reference (sample (a1) without Fe contamination.)

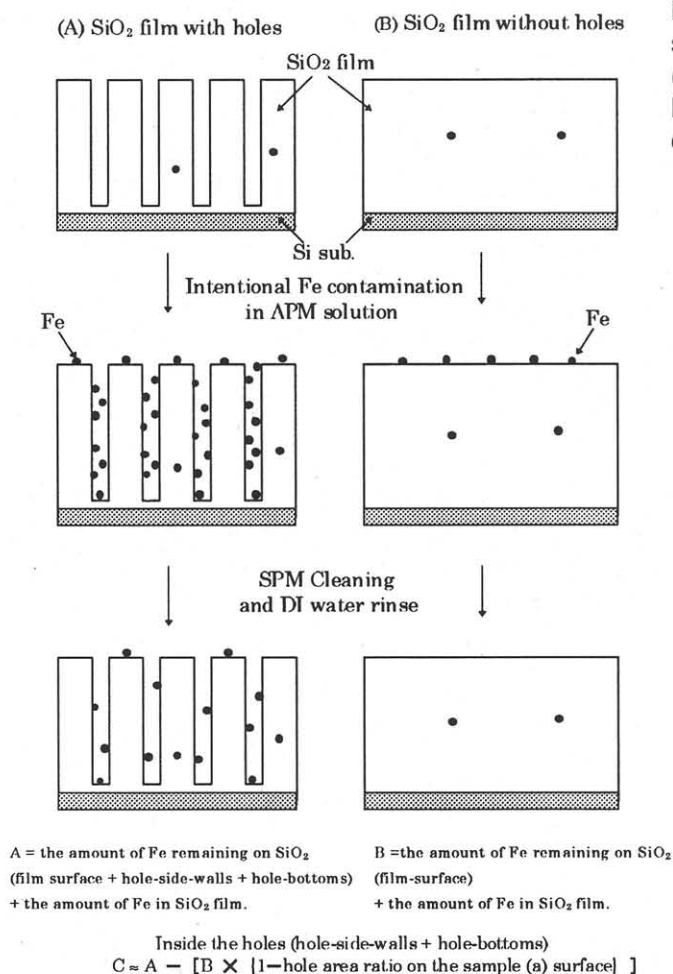


Fig. 2 Method for quantitative analysis of metallic contamination inside submicron structures. Fe contamination in holes is shown as an example.

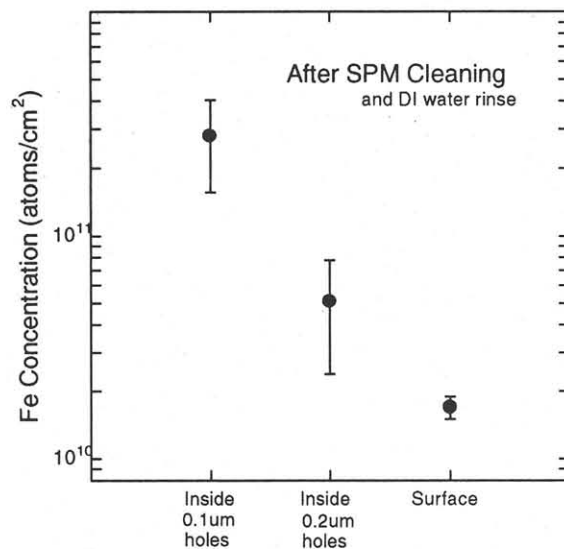


Fig. 4 Fe concentration inside holes: on hole-side-walls and hole-bottoms.