

Analysis Technology of Silicon Wafers

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Abstract

Development of analysis technologies for silicon wafers has been urged due to the increasing demands for wafers with higher quality levels. This paper describes the analysis technologies for metals, organic compounds and particle contamination on silicon wafers. Metal impurities analyses using atomic absorption spectrophotometry, inductively coupled plasma - mass spectrometry, and total reflection X-ray fluorescence spectrometry are described together with the new wafer pretreatment and standard sample preparation methods developed. Adsorption behavior of organic compounds is studied by using the atmospheric pressure ionization mass spectrometry - thermal desorption spectroscopy. Some examples of particle analysis by scanning electron microscope in combination with automatic surface particle mapper are also described.

1. Introduction

Manufacturing of silicon devices having finer design rules has required higher quality levels of substrate Si-wafers and enhanced cleanliness of their surface. Advancement of analysis technologies has been urged for the progress of cleaning technologies, since wafer surface cleanliness can only be certified when reliable evaluation techniques are available. The recent wafer diameter expansion and the increasing numbers of elements to be analyzed are also additional reasons for developing analysis technologies.

This paper discusses the latest wafer analysis technologies for metals, organic compounds and particles.

2. Analysis of Metals

2.1 Atomic absorption spectrophotometry (AAS)

2.1.1 Outline

AAS is one of the first methods applied to the quantitative analysis of contaminant metals on wafer surfaces. Since the AAS is a solution analysis method, it requires a pretreatment of the wafer to dissolve the surface contaminant metals in an acid solution. Usually, the vapor phase decomposition (VPD) method is applied for the pretreatment step¹⁾. During a typical VPD process, the wafer surface becomes hydrophobic after decomposition of the oxide films on the

surface by HF gas at room temperature. Then, the whole wafer surface is scanned with less than 1mL droplet of ultra pure water or diluted HF to obtain a concentrated solution of the contaminant metals. However, the VPD method has the disadvantage of not recovering Cu sufficiently²⁾ because of its lower ionization tendency than Si. Cu ion once dissolved in the droplet solution precipitates back onto the Si wafer-surface.

As Cu represents one of the most problematic contaminant metals in the semiconductor manufacturing process, the "HF+HNO₃ dew etching"³⁾ has been developed as a new method for recovering contaminants on the wafer surface with high Cu recovery.

2.1.2 Experimental

Fig. 1 schematically shows an outline of the HF+HNO₃ dew etching method. A 3:97 mixture of 38% HF and 68% HNO₃ was heated in a PTFE vessel to generate the mixed acid vapor, and a sample of wafer surface was exposed to this vapor. Therefore, uniformly distributed dew of the mixed acid vapor formed on the wafer surface. The dew were recovered by blowing high purity nitrogen gas with an air gun onto the wafer surface, and the recovered liquid was directly injected into a flame-less atomic absorption spectrophotometer Z5100 PC (Perkin Elmer) for analysis. The heating patterns used were those the equipment manufacturer recommended. Diluted so-

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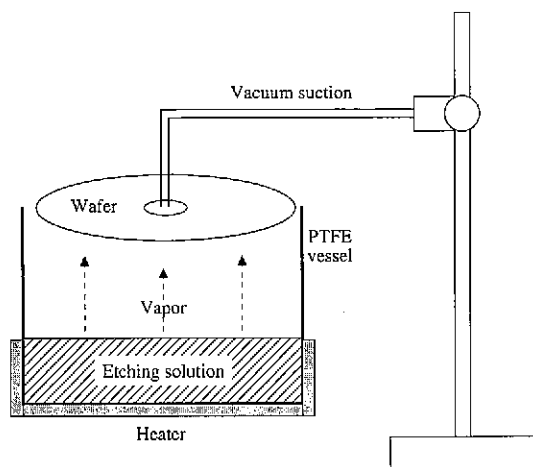


Fig. 1 Schematic view of HF+HNO₃ dew etching method

lutions of certified metal standard solutions for chemical analysis were used for calibration. Their matrix was adjusted to that of the recovered liquid.

This method is expected to achieve a high Cu recovery due to the high oxidation potential of nitric acid. The background caused by impurities in the chemicals is also expected to decrease by the use of dew, as it means the chemicals are distilled "in-situ".

2.1.3 Results and discussion

The conventional VPD method dissolves only the surface oxide layer and cannot recover contaminants in the surface of Si layer where the devices are actually formed. Meanwhile, under the present method, a surface layer of Si is etched to some extent due to the use of a mixture of nitric acid and hydrofluoric acid, which has etching capability of Si. The Si etching extent was measured from the difference between the wafer weight before and after the pretreatment, and it was confirmed that a surface layer was etched roughly by 0.4 μm in thickness. Thus, this method can dissolve the surface till the depth of the device formation layer and will be more suitable for the quality control assurance of Si wafer manufacturing, rather than the VPD method.

Next, silicon wafers contaminated with different concentrations of Cu were analyzed by both the present and the conventional VPD methods to confirm the Cu recovery. The results are shown in Fig. 2. As expected, the present method achieved higher determination values than the VPD method, suggesting high recovery rate. Samples contaminated with Cu in the order of 10^{12} atoms/cm² were repeatedly treated by this method to check the recovery rate. The result showed that more than 90% of Cu was recovered in the first etching with the HF+HNO₃ vapor.

Table 1 shows the lower limits of detection of the main metals determined by this method, expressed as triplicates of respective standard deviations obtained through repetitive measurements of blank solution. A high sensitivity analysis in the order of 10^9 atoms/cm² has been achieved.

As the results showed, the HF+HNO₃ dew etching method proved to be an efficient technique in enhancing Cu recovery rate and analysis depth while maintaining the advantage of the conventional VPD method. The present method has actually been applied to wafer quality control and also in some other manufacturing activities at NSC Electron Corp.

2.2 Inductively coupled plasma mass spectrometry (ICP-MS)

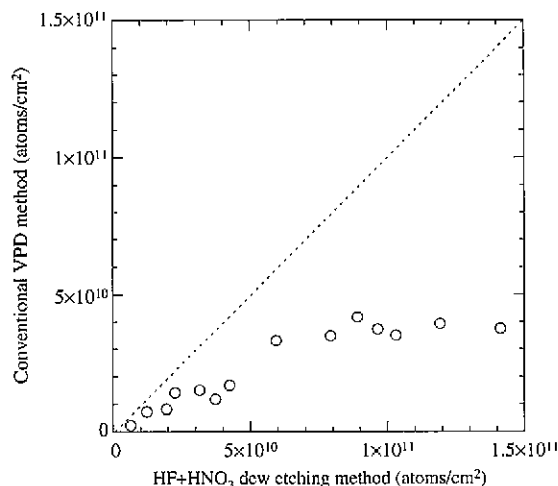


Fig. 2 Comparison of Cu analyses by HF+HNO₃ dew etching method and conventional VPD method

2.2.1 Outline

The ICP-MS is a high sensitivity analysis method for multi-element determination with the capability of analyzing sample solutions directly and it has been widely applied to evaluate pure water, chemical solutions, etc. in the semiconductor industry⁴⁾. Recently, the ICP-MS method has been used in determining trace impurities on Si-wafer surfaces or in its raw material, poly-silicon, after the development of analytical pretreatment methods like decomposition and dissolution for those materials^{5,6)}.

The sample is usually introduced to the ICP-MS by a concentric or a Babington nebulizer, where the sample flow rate is 0.5mL to 2mL/min requiring 1mL to 5mL of sample solution during one analysis operation. Those nebulizers are unsuitable for small volume sample as in the order of several tens of μL . Hence, some sample introduction systems such as electro-thermal vaporization (ETV)^{7,8)}, micro-concentric nebulizer (MCN)⁹⁾ and direct injection nebulizer (DIN)^{10,11)} have been developed to deal with small volume samples.

Hereafter is reported an analysis method for small volume samples, where a micro-nebulizer is combined with a micro-suction method to minimize sample introduction volume by controlling sample suction time.

2.2.2 Experimental

Ultra-pure nitric acid AA-10 (Tama Chemicals) and pure water from a MilliQ ultra-pure water system (Millipore) were used. Calibration standards were prepared by dilution of AAS standard solution (Kanto Kagaku) with 0.1% nitric acid.

The instrument and operation conditions are listed in Table 2. A SPQ8000A1 ICP-MS coupled to an AT-600 micro-suction system (Seiko Instrument Inc.) was used. A MCN-100M2 micro-concentric nebulizer (Cetac) was used for sample introduction into the plasma. The sample flow rate was controlled to 0.05 mL/min by a peristaltic pump.

Fe has been selected for the basic study of the method because it

is an important element for evaluating wafer cleanliness as it can easily be adsorbed on the wafer surface oxide layer. Besides, some of the main device defects are caused by formation of oxidation-induced stacking fault (OSF) or formation of precipitates through diffusion into the wafer interior during a thermal process in the LSI manufacturing.

Table 2 ICP-MS and operation conditions

ICP-MS	SEIKO SPQ8000A1
Micro-suction system	SEIKO AT600
Nebulizer	CETAC MCN-100M2
Sampling depth	8 mm
RF power	1.0 kW
Plasma gas flow rate	15.5 L/min
Auxiliary gas flow rate	0.8 L/min
Nebulizer gas flow rate	1.1 L/min
Chamber-gas flow rate	0.35 L/min
Sample flow rate	0.05 mL/min
Sample solution	0.1 wt% HNO ₃

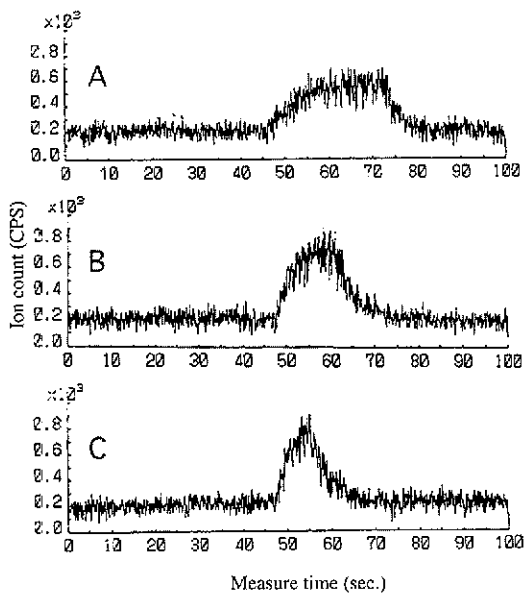


Fig. 3 Signal profile of 100 pg/mL Fe

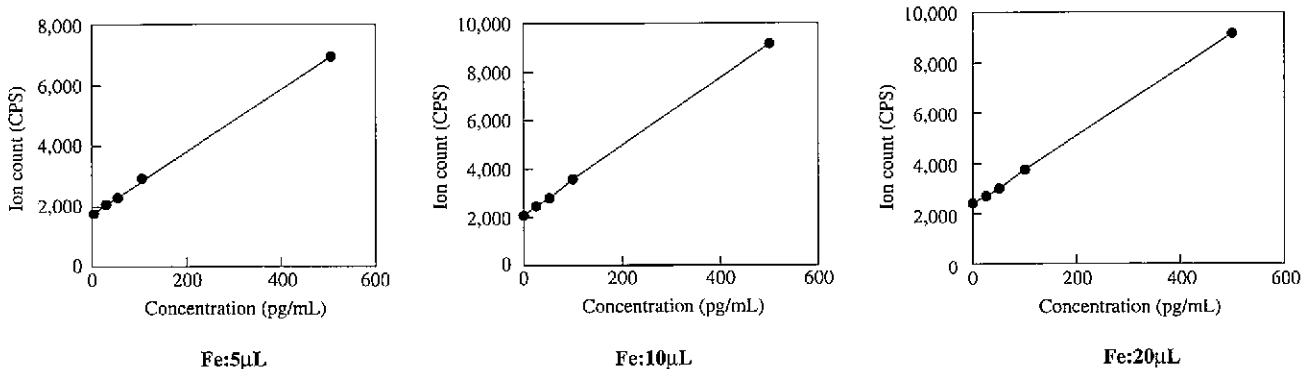


Fig. 4 Calibration curves (single element)

In the micro-suction method, changes in the sample suction time can control the sample introduction volume. The sample volume introduced was controlled to 5, 10 and 20µL by adjusting the sample suction time at 6, 12 and 24 sec., respectively. Fig. 3 shows the 100pg/mL Fe signal profiles obtained for each sample volume introduced by the micro-suction method. Increase in the time range of the obtained ion signals is observed according to the increase of the sample volume introduced. The measurement conditions and the signal integrating time for each sample volume introduced are shown in Table 3.

2.2.3 Results and discussion

Fig. 4 shows the calibration curves obtained for Fe when the sample volume was adjusted to 5, 10 and 20µL. Very good straight lines were obtained for each sample volume.

Relative standard deviation (%) of the signal intensity obtained through 10 measurements of blank solution is shown Fig. 5. Higher precision is observed with the increase of the sample volume introduced.

Table 4 summarizes the lower limits of detection calculated as triplicates of the standard deviation of the blank solution measurements. Fe can be measured to the order of several pg/mL in a single element determination with a sample volume ranging 5 to 20µL. Converting into the absolute amount of Fe present in a sample solution, for instance, the lower limit of detection will be less than 50 fg of Fe when the sample volume is 5µL.

Next, relying on the capability of ICP-MS in determining multi-element, small volume samples were tested for that purpose. Some highly contaminant key elements during the manufacturing process of LSIs were selected for testing. They were Na among alkali metals, Al among amphoteric metals, Fe, Ni and Cu among transition

Table 3 Measurement conditions (single element)

Element	Fe		
Mass	56 amu		
Dwell time	50 ms		
Channel width	3 channels		
Measure time	90 s		
Trg. delay time	5 s		
Sample introduction volume	5 µL	10 µL	20 µL
Integrate from	45 s		
Area	25 s	30 s	40 s
Sample suction time	6 s	12 s	24 s

elements.

The multi-element determination was conducted adjusting the

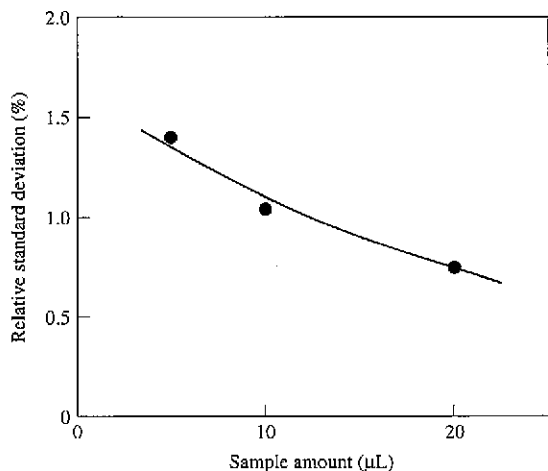


Fig. 5 Relative standard deviation of signal intensity in blank measurement

Table 4 Lower limits of detection (single element)

Sample introduction volume	Fe
20 μL	3.9 pg/mL
	78 fg*
10 μL	4.4 pg/mL
	44 fg*
5 μL	6.7 pg/mL
	34 fg*

*Absolute amount

sample volume to 5, 10 and 20μL. Measurement conditions and the signal integration time are summarized in Table 5 and the calibration curves obtained in a sample volume of 5μL are shown in Fig. 6. Very good straight lines were obtained for all the tested elements.

Table 6 shows the lower limits of detection of this present method, calculated as triplicates of the standard deviation of the blank solution measurements. The lower limits of detection of each tested element are less than 20 pg/mL in terms of solution concentration. Converting into the absolute amount of tested elements present in a sample solution, for example, the lower limits of detection will be less than 100 fg for all tested elements when the sample volume is 5μL.

Further, the present method was applied to Si-wafer analysis. The samples were 8" Si-wafers intentionally contaminated by the spin-coat method. 100μL of 0.1 wt% nitric acid solution was dripped onto a Si-wafer and the liquid on the surface recovered with a micro-pipette was analyzed by the micro-suction ICP-MS. The area of recovery was 3 cm². The result is shown in Table 7, together with the analysis result of the entire wafer surface by the HF+HNO₃ dew etch-

Table 5 Measurement conditions (5-elements determination)

Element	Na	Al	Fe	Ni	Cu
Mass (amu)	23	27	56	58	63
Dwell time	50 ms				
Channel width	3 channels				
Measure time	90 s				
Trg. delay time	5 s				
Sample introduction volume	5 μL	10 μL	20 μL		
Integrate from	45 s				
Area	25 s	30 s	40 s		
Sample suction time	6 s	12 s	24 s		

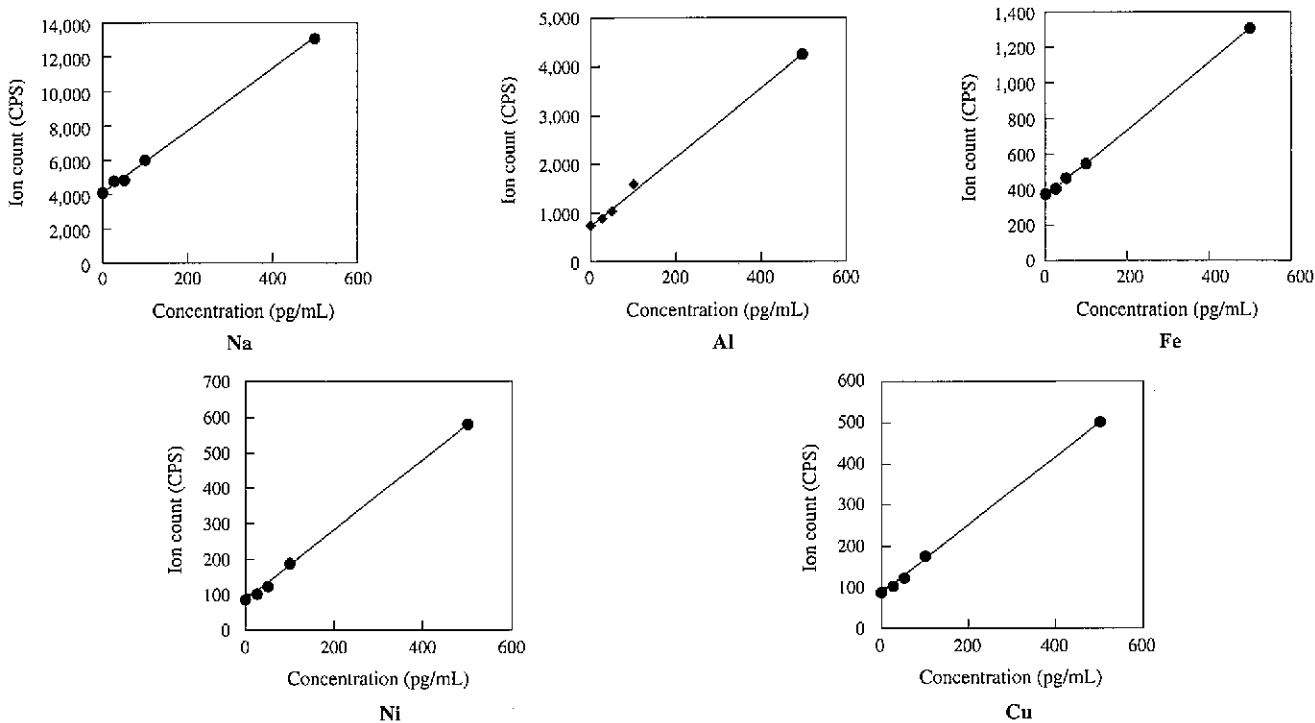


Fig. 6 Calibration curves (5-elements determination)

Table 6 Lower limits of detection (5-elements determination) (pg/mL)

Sample introduction volume	Element				
	Na	Al	Fe	Ni	Cu
20 μL	10.1	3.8	9.4	2.5	5.4
	202 fg*	77 fg*	189 fg*	49 fg*	107 fg*
10 μL	14.1	10.6	10.8	8.9	6.0
	141 fg*	107 fg*	108 fg*	89 fg*	60 fg*
5 μL	15.8	15.1	12.5	11.1	10.8
	79 fg*	75 fg*	63 fg*	55 fg*	54 fg*

*Absolute amount

Table 7 Wafer analysis results (×10¹⁰atoms/cm²)

	Na	Al	Fe	Ni	Cu
AAS*	4.4	3.6	3.9	3.7	0.5
ICP-MS**	4.4	3.7	4.1	3.2	0.5

* HF+HNO₃ dew etching-AAS method

** Micro-suction ICP-MS method (area of recovery: 3 cm²)

ing-AAS method. The results of both methods agree very well with each other. Hence, the present method has showed effectiveness for local analysis of Si-wafer surface since it requires only 100μL of recovered liquid for obtaining information of a 3 cm² recovery area. Besides the wafer surface analysis, wafer interior can also be analyzed by changing the composition of the dripping solution.

Therefore, feasibility in analysis of very small, 5 to 20μL, sample volumes has been accomplished by the micro-suction ICP-MS method employing a micro-nebulizer. This method has also offered an opportunity to determine multi-element in small sample volumes.

2.3 Total-reflection X-ray fluorescence spectrometry (TXRF)

2.3.1 Outline

TXRF is a variety of fluorescent X-ray spectrometry whose major difference from the AAS or the ICP-MS methods, described before, is that it is a non-destructive analysis method. Fig. 7 shows the principle of the method. When an X-ray beam is irradiated to a mirror-polished wafer surface at a low glancing angle (0.10 deg.), major portions are reflected by total reflection but minor portions of them penetrate to a depth of several nanometers from the surface, exciting the elements in the area and causing the emission of fluorescent X-ray. The emitted fluorescent X-ray is detected by a solid state detector which is established perpendicular to the sample surface. This method has the advantages of not requiring pretreatment, being a

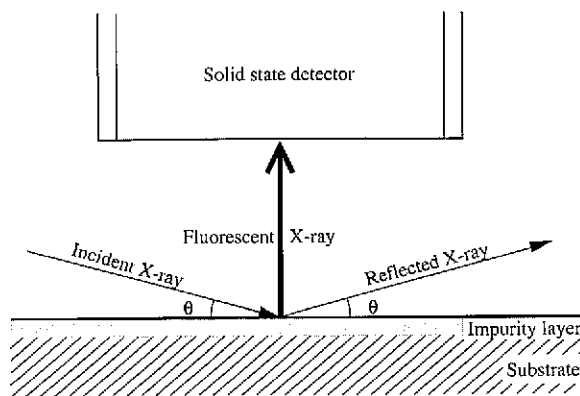


Fig. 7 Principle of TXRF method

non-destructive analysis, quick (roughly 10 min/point) and capable of mapping.

Despite the advantages of the TXRF, it presents some problems in quantitative analysis inevitably derived from its principle. The most serious among them is the one related to the depth profile of the analyte elements: difference in the intensity of the fluorescent X-ray emitted by an analyte element is observed with different depth profile of the element, even when the total amount is the same. In other words, when the amount of the analyte element is the same and the measurement conditions are perfectly identical, the intensity of the fluorescent X-ray is stronger when the analyte element exists near the surface, where the X-ray excitation is higher, and the intensity becomes weaker when the analyte element exists far from the surface. This phenomenon can clearly be observed in the profile of the angle scan (glancing angle dependence of fluorescent-X ray intensity).

Fig. 8 shows theoretical calculations of the angle scan profiles for the same amount of analyte element, assuming different profiles along the depth. Here, we can see that a difference of some nanometers in depth largely affects the fluorescent X-ray intensity. Therefore, conformity of the depth profile between a standard sample and an unknown sample is extremely important in quantitative analysis by TXRF¹²⁾.

However, this point was not considered during the early stages of the TXRF development and each analyst conducted determinations with different standard samples which have different depth profiles. This fact caused much confusion in the differences of determination values between TXRF and other analytical methods (AAS, etc.). Hence, the "immersion in alkaline hydrogen peroxide solution (IAP) method" was developed as a new standard sample preparation method capable of easily creating identical depth profile^{13, 14)}.

2.3.2 Experimental

The IAP is a method to have a metal ion adsorbed into the wafer surface by immersing a wafer in a mixture of ammonia and hydrogen peroxide (standard cleaning solution-1, SC-1) wherein a certain amount of metal ion is intentionally added. The metal ion is adsorbed only into a SiO₂ film approximately 1 nm in thickness formed on the

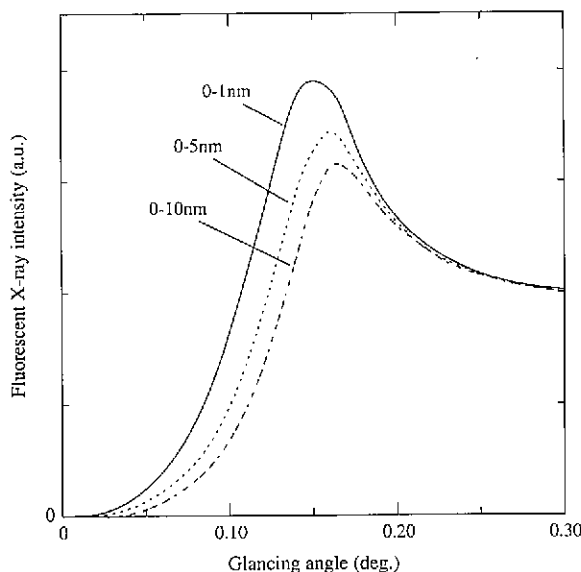


Fig. 8 Calculations of angle scan profiles for different depth profiles for the same total amount of element

wafer surface¹⁵⁾, and thus the depth of the adsorbed metal becomes automatically constant even with different metals in different amounts.

Wafer surfaces prepared by the IAP method were analyzed using the TRXF method to evaluate the forms of contamination and homogeneity of the adsorbed elements. Reference standard samples were prepared by the IAP, conventional spin-coat and micro-drop methods to compare the determination values by the TRXF method based on these respective standard samples. The sample wafers were randomly picked out from a commercial production line for the evaluation. A TXRF device, SYSTEM3726 of Rigaku Industrial Corp., was used under a condition of an excitation X-ray of W-L β (9.67 keV) and applied voltage and current of 30 kV and 400 mA, respectively.

2.3.3 Results and discussion
The depth profiles of 7 metal elements at the surfaces of the samples prepared by the IAP method were evaluated by the TXRF angle scan measurement. The result is shown in Fig. 9. The profiles are in good agreement with each other, showing that the same depth profile is obtained with different elements.

Table 8 shows a result of the TXRF examination of areal concentration dispersion of the wafers intentionally contaminated with Fe, Ni and Zn by the IAP method. Good areal homogeneity is seen here, demonstrating uniform adsorption.

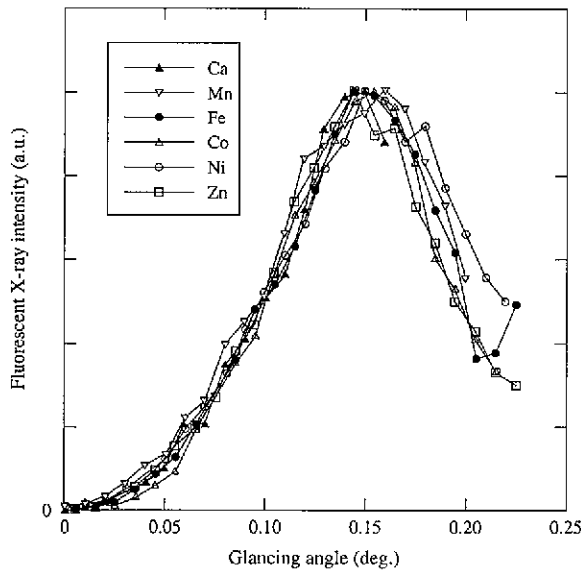


Fig. 9 Angle scan of several metals adsorbed by IAP method

Table 8 Homogeneity of metal concentration on wafer surface adsorbed by IAP method

Element	Concentration (atoms/cm ²)	Relative standard deviation (%)
Fe	9.0×10 ¹¹	7.7
	4.5×10 ¹²	3.5
	1.7×10 ¹³	4.0
Ni	3.5×10 ¹¹	12.3
	3.3×10 ¹²	19.6
	1.0×10 ¹³	4.3
Zn	7.3×10 ¹¹	9.3
	3.0×10 ¹²	3.4
	6.0×10 ¹²	4.8

Table 9 Comparison of unknown sample determination based on different standard samples (×10¹⁰atoms/cm²)

Method	AAS	TXRF		
		IAP	Spin-coat	Micro-drop
Standard sample				
SampleA (Fe)	16	20	6	3
SampleB (Fe)	8	6	3	2
SampleA (Zn)	24	25	16	9
SampleB (Zn)	3	4	2	1

The result of the comparative test based on different reference standard samples is shown in Table 9. The deviations from the AAS determination values were large when based on the reference standard samples prepared by spin-coat and micro-drop methods, whereas the determination values based on IAP reference standard samples agreed well with the AAS values. This is because the depth profiles of the standard samples could not be controlled by the former two methods and thus the profiles were different between the standard samples and the unknown samples, while reliable calibration curves were obtained in the case of the latter method since the depth profile was the same between the standard samples and the unknown samples. Thus, it has been verified that the TXRF method offers accurate analysis of wafer surface contamination when standard samples are prepared by the IAP method.

3. Analysis of Organic Compounds

3.1 Outline

3.1.1 Background

Metal elements and particles are well known as the Si-wafer contaminants causing serious influences on the silicon devices quality, and recently, chemical contamination is also attracting attention. The chemical contamination results from molecular substances suspended in the air adhering onto the Si-wafer surface. Those substances include organic compounds, ions (acids and alkalis), dopants (phosphorus, boron) and moisture. Many researches have been conducted specially on organic compounds contamination. Cases where device characteristics are seriously affected by deterioration of gate oxide films when organic compounds adhered onto the Si-wafer surface have been reported¹⁶⁻²⁰⁾.

Hereafter, an effective analysis method for organic compounds, the atmospheric pressure ionization mass spectrometer-thermal desorption spectroscopy (APIMS-TDS) is described with some results obtained by this method.

3.1.2 Atmospheric pressure ionization mass spectrometer - thermal desorption spectroscopy (APIMS-TDS)

An APIMS-TDS consists of an APIMS part and a TDS part. The APIMS part is widely used for detecting impurities in semiconductor gases and can measure those impurities in the order of ppt by introducing the sample gas directly at the atmospheric pressure. The TDS part can desorb organic compounds and H₂O adsorbed on the wafer surface by heating the wafer at the atmospheric pressure. In the APIMS part, a cation type detector is used to determine organic compounds and H₂O while an anion type detector is used for determining chloride, sulfate and other ions. Thus, the APIMS-TDS configuration is capable of determining organic compounds and H₂O on the wafer surface at the same time. The lower limit of detection of organic compounds is in the order of 10¹² molecules/cm².

3.2 Experimental

Fig. 10 schematically shows an outline of the APIMS-TDS. It

consists of UG-400 (APIMS part) and UG-21 (TDS part) of Hitachi Tokyo Electronics. High purity argon gas was used as the carrier gas, and the samples were heated from room temperature to 800°C at a heating rate of 20°C/min. The TDS can heat a wafer up to 8" in diameter without dividing it and measure either of the wafer surfaces separately. Sample determinations were conducted after the removal of organic compounds and H₂O, adhered onto the surfaces of the quartz chamber and pipes, by heating.

3.3 Results and discussion

3.3.1 Adhesion behavior of organic matters on silicon wafer surfaces caused by plastic cases

In order to investigate the degassing behavior of the plastic additives, Si-wafers were stored in polypropylene boxes intentionally added with a plastic additive for a prescribed period and organic compounds adhered onto the wafer surface were determined by the APIMS-TDS. The plastic additive used was an antioxidant for polypropylene, n-octadecyl-3-(3,5-di-t-butyl-4-hydroxyphenyl) propionate (OBHP).

First of all, a study was conducted regarding the desorption behavior of the additive (OBHP) from the Si-wafer surface. OBHP was dissolved in acetone and the solution was applied onto a wafer surface, previously treated to remove any organic compounds. Next, a study was conducted regarding the adhesion behavior of organic compounds. Sample wafers were stored in the polypropylene boxes added with OBHP for 2 years. The APIMS-TDS was used for determinations on both studies and the results are shown in Fig. 11.

The wafer with direct OBHP application showed desorption of molecules having mass number higher than 120 besides those having mass numbers lower than 120. It is supposed that C_nH_{2n-1} seen in aliphatic hydrocarbons are responsible for the peaks in the high-mass number range. However, the wafer stored in the polypropylene boxes added with OBHP for 2 years showed many peaks in the mass number range lower than 120 but no peaks were observed in the higher

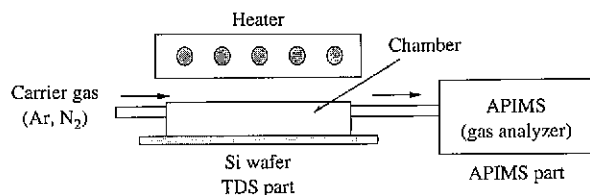


Fig. 10 APIMS-TDS equipment

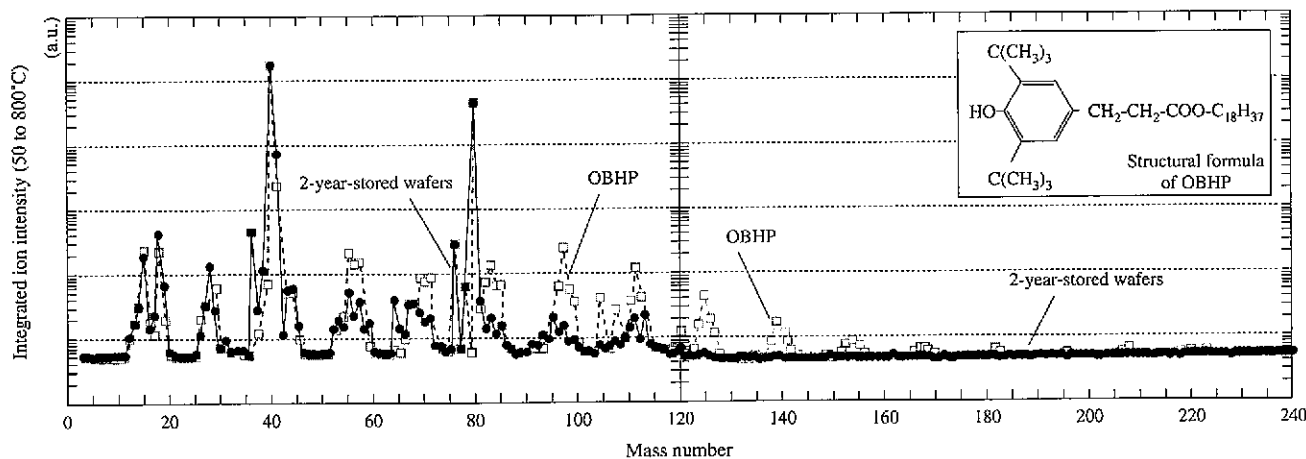


Fig. 11 Desorption spectrum of OBHP-applied wafers and 2-year-stored wafers

mass number range.

Hence, a study was conducted to examine the historical changes of low molecular weight substances and high molecular weight substances. As seen in Fig. 12, desorption of low molecular weight substances (mass number 55, 67) began to start immediately after the wafers were stored in the boxes and it increased with the lapse of time, while high molecular weight substances (mass number 95, 111) showed desorption only after elapsing approximately 100 days. A substance having yet a higher mass number (140) showed desorption after elapsing 700 days.

From the above results, it is suspected that OBHP used as additive does not volatilize as the high molecular weight original form, but decomposes into low molecular weight substances by aging or thermal cracking during the box formation process and adhere onto the wafer surfaces after volatilization inside the box.

The APIMS-TDS is effective for determining chemical contaminants on the Si-wafer surfaces and is expected to become more important analysis method with increasing demands for higher integration and higher quality semiconductor devices.

4. Analysis of Particles

4.1 Outline

Particles on the wafer surfaces have to be minimized since they are responsible for pattern failure and film defects. It has been considered that particles approximately 1/5 to 1/3 of the design rule in

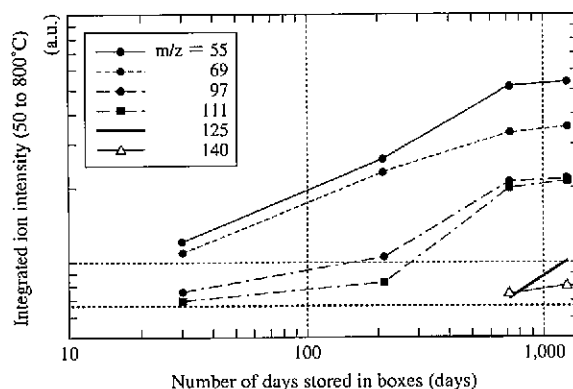


Fig. 12 Relationship between stored time and desorption of organic compounds-derived mass numbers

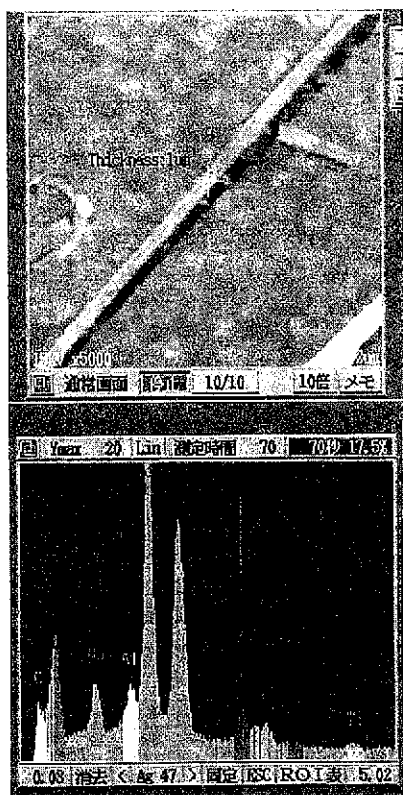


Fig. 15 Glass fiber HEPA filter

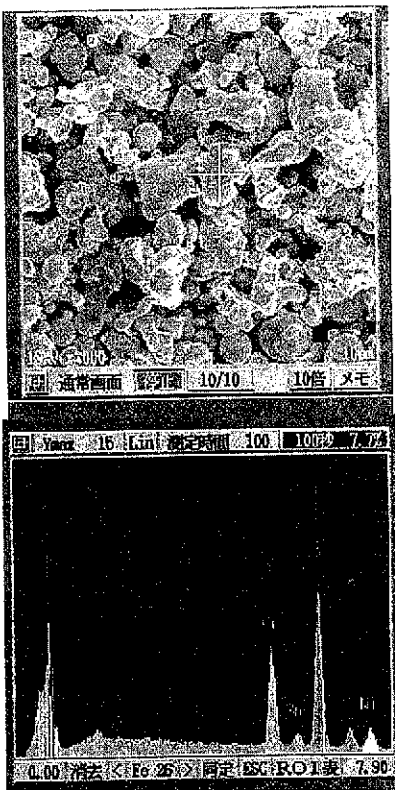


Fig. 16 Stainless steel

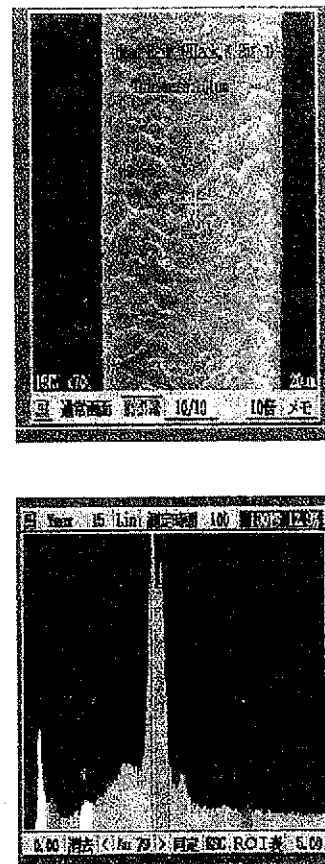


Fig. 17 Hair

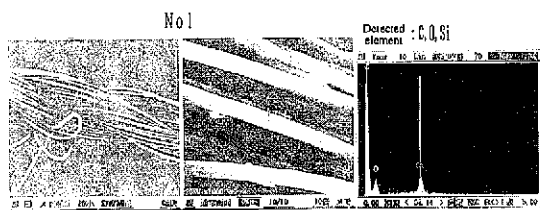


Fig. 20 Hemp fiber



Fig. 21 Cotton fiber

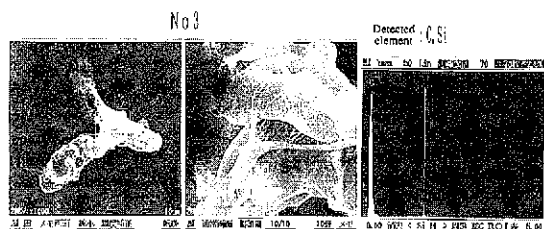


Fig. 22 Contaminant from wafer case

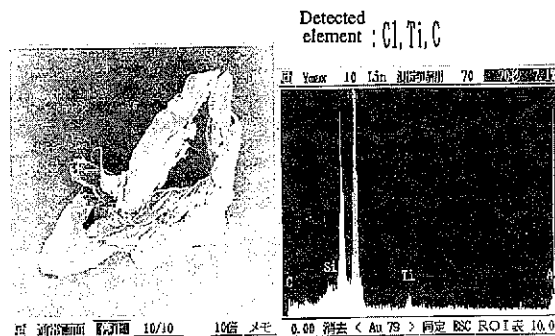
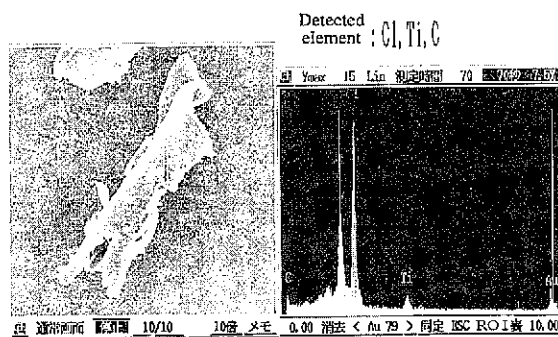


Fig. 23 Contaminant from PVC bath

ticles or, often, as compound particles where these are combined. In a routine particle checking, the amount of particles can be measured but their origins are difficult to identify. Appropriate countermeasures can only be taken when the origins of the particles are found out through analysis to identify what the particle materials are. A database built up with investigation of known substances is of great importance for the particle analysis. In our actual particle analysis, identifications of problematic particles are accomplished by comparing their analysis results with the database regarding shape (SEM image) and composition (EDX spectrum) of the several known substances.

Although a mini-environment has been proposed, basically, it is still necessary to maintain the utmost clean environment, during the wafer transposition, cleaning or handling steps in the manufacturing process. Furthermore, identification of the particles will be the key to enhance cleanliness of the wafer surface since the ways to achieve higher cleanliness is different for each kind of particle.

5. Conclusion

Analysis technologies of metals, organic compounds and particles contamination on Si-wafer surface were discussed in this paper. The advancement of analysis technologies has contributed toward determinations of impurities that conventional methods cannot detect and it has also clarified that those impurities affect seriously the quality of the Si-wafers. Hence, analysis technologies are expected to develop further as demands for higher cleanliness wafers are predicted to increase.

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