Coupling of an electrodialyzer with inductively coupled plasma mass spectrometry for the on-line determination of trace impurities in silicon wafers after surface metal extraction

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ABSTRACT

Understanding the properties that determine the distribution and behavior of trace impurities in Si wafers is critical to defining and controlling the performance, reliability, and yields of integrated microelectronic devices. It remains, however, an intrinsically difficult task to determine trace impurities in Si because of the minute concentrations and extremely high levels of matrix involved. In this study, we used an electrodialyzer for the simultaneous on-line removal of the silicate and acid matrices through the neutralization of the excessive hydrogen ion and selectively separation of acid and silicate ions by the combination of electrode reaction as a source of hydroxide ions with the anion exchange membrane separation. To retain the analyte ions in the sample stream, we found that the presence of moderate amounts of nitric acid and hydrazine were necessary to improve the retention efficiency, not only for Zn^{2+}, Ni^{2+}, Cu^{2+}, and Co^{2+} ions but also for CrO_4^{2-} ion. Under the optimized conditions, the interference that resulted from the sample matrix was suppressed significantly to provide satisfactory analytical signals. The precision of this method was ca. 5% when we used an electrodialyzer equipped with an anion exchange membrane to remove the sample matrix prior to performing inductively coupled plasma mass spectrometry (ICP-MS); the good agreement between the data obtained using our proposed method and those obtained using a batchwise wet chemical technique confirmed its accuracy. Our method permits the determination of Zn, Ni, Cu, Co, and Cr in Si wafers at detection limits within the range from 2.2×10^{15} to 9.0×10^{15} atoms cm^{-3}.

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1. Introduction

When manufacturing semiconductor devices, surface metallic contamination can result in process variation and defects, such as poor coverage, vacancies, voids, leakage, shorts, and overburdens. With the increasing integration density and decreasing line widths of modern integrated circuits (ICs), trace metal contamination is becoming a detrimental factor that may lead to the faulty operation of such devices [1,2]. Consequently, greater control over contamination during the fabrication processes involved in device manufacturing is becoming increasingly crucial because of the growing complexity and miniaturization of ICs. To ensure the high electrical performance of semiconductor devices and, thereby, develop competitive Si circuit technologies, it will be essential to develop new techniques for trace impurity analysis, including those that determine the concentrations of impurities and those that clarify the sources of metallic contamination during fabrication.

A variety of direct instrumental methods, including auger electron spectroscopy [3], secondary ion mass spectrometry [4], and total reflectance X-ray fluorescence (TXRF) [5], can be used to measure the levels of metallic contaminants on Si wafers. According to Krushevska et al. [6], it can be difficult to determine trace impurities in Si wafers using these methods because of inadequate analytical sensitivity, poor quantitation, and the fact that only limited amounts of analytes located at specific points can be obtained during each measurement. Rather than using direct instrumental methods, vapor phase decomposition (VPD) and droplet surface etching (DSE) in conjunction with inductively coupled plasma mass spectrometry (ICP-MS) or TXRF have become the most prevalent methods for the determination of surface metallic contamination in the native or thermal oxide layers of Si wafers, due to their superior analytical sensitivities and lower running costs [7–9]. When monitoring and characterizing metal impurities on the surfaces of Si wafers, mixtures of HF and HNO_3 are frequently employed to dissolve the samples prior to instrumental determination [10]. Because the Si concentration in the resulting solutions generally

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Table 1
Si- and F-based polyatomic interference on Co, Ni, Cu, and Zn.

Element	Abundance, %	Possible polyatomic interference
^{64}Zn	48.9	^{64}Ni, $^{36}Ar^{28}Si$
^{66}Zn	27.8	$^{38}Ar^{28}Si$, $^{28}Si^{19}F_2$
^{58}Ni	67.8	$^{40}Ar^{18}O$, $^{40}Ar^{17}O^1H$, $^{40}Ar^{16}O^1H_2$
^{60}Ni	26.2	$^{28}Si^{16}O^{16}O$
^{63}Cu	69.1	$^{28}Si^{16}O^{19}F$
^{59}Co	100	$^{40}Ar^{19}F$, $^{40}Ar^{18}O^1H$

Table 2
Operating conditions for the electrodialyzer/ICP-MS hyphenation system.

ICP-MS	
ICP-MS spectrometer	Perkin-Elmer Sciex Elan 5000
Nebulizer	Microconcentric (MCN-100)
Sample uptake rate	$100\,\mu L\,min^{-1}$
Gas flow rates	
Plasma	$15\,L\,min^{-1}$
Auxiliary	$0.8\,L\,min^{-1}$
Nebulizer	$0.8\,L\,min^{-1}$
RF forward power	1000 W
Scanning mode	Peak hop
Sweeps/reading	1
Readings/replicate	3
Dwell time/sweep	300 ms
Number of replicates	7
Electrodialyzer	
Electrodialyzer	Dionex CSRN-I
Flow rate of sample solution	$0.1\,mL\,min^{-1}$
Stripping solution	Pure water
Flow rate of stripping solution	$5\,L\,min^{-1}$

range from 10 to 2000 mg L^{-1} [11], two potential sources of physical interferences in the sample matrix – dissolved Si and concentrated acids – might limit the accuracy of instrumental measurement [12,13].

Although ICP-MS has been used widely for multi-element trace and ultra-trace analysis in a variety of sample types, the occurrence of spectral and non-spectral interference can lead to inaccurate results when a complicated matrix is being analyzed. Table 1 summarizes the isotopes of interest and their most severe polyatomic interferences for sample solutions obtained from native and thermal oxide layers on Si wafers. In addition to spectral interference, a sample containing significant amounts of solids and/or chemical matrix can also incur reversible and irreversible non-spectral interference during ICP-MS measurement [14]. Until recently, the main tools used to eliminate the spectral interference arising from the matrix were sector field ICP-MS [15], cold plasma ICP-MS [6], and reaction/collision cell ICP-MS [11,16]. In light of the costliness of magnetic sector ICP-MS, quadrupole ICP-MS is more widely used in both industrial applications and academic research. According to Greg and Debrah [17], a combination of cold and normal plasma – even with a nebulizer optimized for small samples – may not be the best approach for analyzing VPD extracts because of severe non-spectral interference, poor analytical sensitivity associated with the cooler plasma temperature, and inferior elemental coverage. In addition, as demonstrated by Takahasi and Youno [11], even though the reaction/collision cell can effectively remove plasma- and matrix-based polyatomic interference, incomplete recovery of the analytes can also be related to non-spectral interference. Therefore, multi-elemental analyses of samples containing high levels of Si and other chemical compounds require the development of appropriate on-line separation procedures to improve the analytical reliability of the characterization of trace metal contaminants in Si wafers.

To minimize the matrix effect associated with the Si matrix in ICP-MS measurements and to eliminate the need to add large quantities of boric acid to complex the fluoride ions, both off-line [18,19] and on-line [17] procedures have been developed for the removal of HF and the Si matrix as volatile SiF$_4$. Although conventional hotplate heating methods have been employed broadly for the removal of SiF$_4$ from the samples, they are often time consuming and susceptible to contamination. Consequently, Greg and Debrah [17] developed an on-line system, combining a membrane desolvator with ICP-MS, to determine trace impurities in VPD samples. According to Krushevska, however, elements that can form volatile fluorides might be lost during the desolvation step [6].

Special attention has been paid recently to membrane separation techniques, mainly because they are solvent-free procedures exhibiting effective separation capability [20]. Electrodialysis is an electrically driven membrane separation process that is capable of separating selected ions from aqueous solutions. In previous studies [21–24], we used an on-line electrodialyzer equipped with a cation-permeable membrane to selectively separate salts and alkaline matrices prior to ICP-MS determination. Our aim in this present study was to develop an on-line analytical technique employing an electrodialyzer equipped with an anion-permeable membrane to on-line separate dissolved SiO$_4^-$, NO$_3^-$, and F$^-$ matrix ions from

the analyte ions prior to ICP-MS determination. This approach also minimized the interference in the ICP-MS resulting from the presence of SiO$_4^-$ and F$^-$ ions in the sample solution. As a result, this proposed on-line system allows the determination of surface metallic contamination in the thermal oxide layers of Si wafers.

2. Experimental

2.1. Apparatus

The electrodialyzer/ICP-MS hyphenated system is depicted in Fig. 1. The connections and conduits were polytetrafluoroethylene (PTFE) connecting tubes (1.0 mm i.d.). Pump tubing (20 cm long × 0.8 mm i.d.) was employed to propel the samples. An electrodialyzer equipped with an anion exchange membrane (Dionex CSRN-I, CA, USA) was installed upstream of the ICP-MS to remove matrix anions. Pure water was used as the stripping solution to generate hydroxide ions, which could move from the cathode chamber into the sample chamber to neutralize sample hydronium ions. The inductively coupled plasma mass spectrometer was an Elan Model 5000 system (Perkin-Elmer Sciex, Thornhill, Ontario, Canada). A microconcentric nebulizer (MCN-100, Cetac Technologies, Omaha, NE, USA) was fitted to a Scott-type Ryton double-pass spray chamber (Perkin-Elmer, parts number N8120124) to introduce the effluents from the electrodialyzer into the ICP-MS. Levels of ^{52}Cr, ^{64}Zn, ^{58}Ni, ^{63}Cu, and ^{59}Co were measured according to their abundances. The instrumental operating conditions providing optimal sensitivity are listed in Table 2.

2.2. Reagents and containers

Solutions of HNO$_3$ (60 wt%), HF (48 wt%), and H$_2$O$_2$ (31 wt%) of high purity (Merck Ultrapur, Merck, Darmstadt, Germany) were used throughout. Hydrazine sulfate was obtained from Riedel-de Haen (Seelze, Germany). High-purity water was obtained using deionization and double-distillation techniques. Stock solutions (1000 mg L^{-1}) of the analyte species were also purchased from E. Merck Company (Darmstadt, Germany). The working aqueous standards were prepared afresh daily using pure water; the PTFE containers were cleaned through immersion overnight in conc. HNO$_3$.
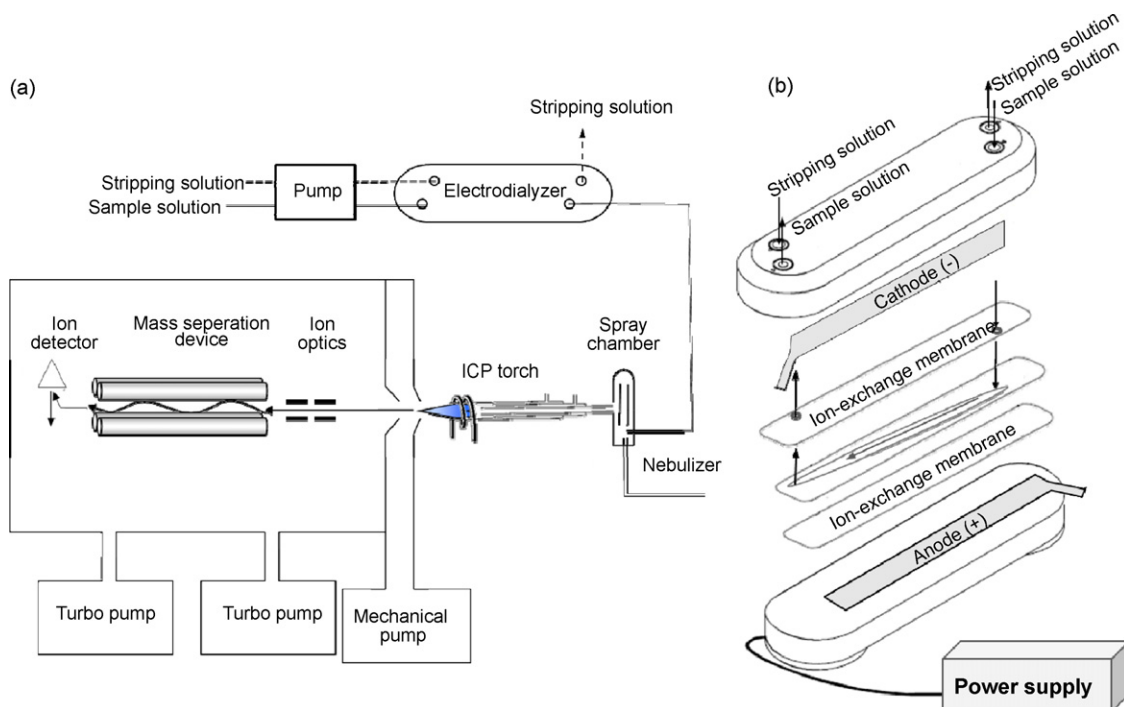
Fig. 1. Schematic representation of (a) the on-line electrodialyzer/ICP-MS hyphenated system and (b) the configuration of used electrodialyzer.

2.3. Preparation of intentionally contaminated wafers

Samples were single polished (100) p-type (boron-doped) wafers having a diameter of 135.89 mm. To obtain a new Si surface, the native oxide layer on the Si wafer was first washed away using 2% (v/v) HF solution prior to growth of a new oxide layer over the entire wafer through thermal dry oxidation. A 1.0% (v/v) HNO_3 solution containing 200 mg L^{-1} metallic contaminants of interest (Cr, No, Co, Cu and Zn) was transferred onto the wafer surface using a micropipette. To ensure homogeneous distribution of metallic contaminants on the wafer surface, the wafer was divided into 236 districts (0.8×0.8 cm^2); an aliquot of the solution described above (10 μL) was pipetted onto each district and evaporated under infrared light. The oxide layers were then formed at different temperatures under an O_2 atmosphere for 12 h. Based on scanning electron microscopy (SEM) data, the thickness of the thermal oxide layers obtained at annealing temperatures of 800, 900, and 1000 °C were 203, 355, and 531 Å.

2.4. Preparation of sample solution

A mixture of 2% (v/v) HF and 3% (v/v) H_2O_2 (1.5 mL) was placed onto a Si wafer. After dissolution of the surface oxide layer, a pipette was used to collect the remaining solution from the surface of the resulting hydrophobic Si wafer. To transform the dissolved Cr species to Cr(III) and to adjust the acid concentration to a suitable level, 1% (w/v) hydrazine sulfate (0.03 mL) was added to the resulting solution and then 10% (v/v) HNO_3 solution was added to dilute the sample to 3 mL.

3. Results and discussion

3.1. Removal of matrix ions

In this study, we used pure water as a stripping solution to exchange SiO_4^-, F^-, and NO_3^- ions for OH^- and to neutralize excess

H_3O^+ in the sample solutions. To optimize the separation efficiency, we evaluated the removal efficiency of these matrix ions as a function of the flow rate of the sample stream at an electrolytic current of 50 mA. Fig. 2 presents the variation in the removal efficiencies of the matrix ions in a solution containing 1500 mg L^{-1} silicate, 1% (v/v) HF, and 5% (v/v) HNO_3 at various sample flow rates. The removal efficiencies of each tested matrix ion increased upon decreasing the sample flow rate; for a flow rate of the stripping solution of 5 mL min^{-1}, almost 100% of the matrix ions were removed when the flow rate of the sample stream was 0.1 mL min^{-1}. To avoid any possible interference resulting from residual matrix ions in the sample stream, we employed these flow rates in our subsequent experiments.
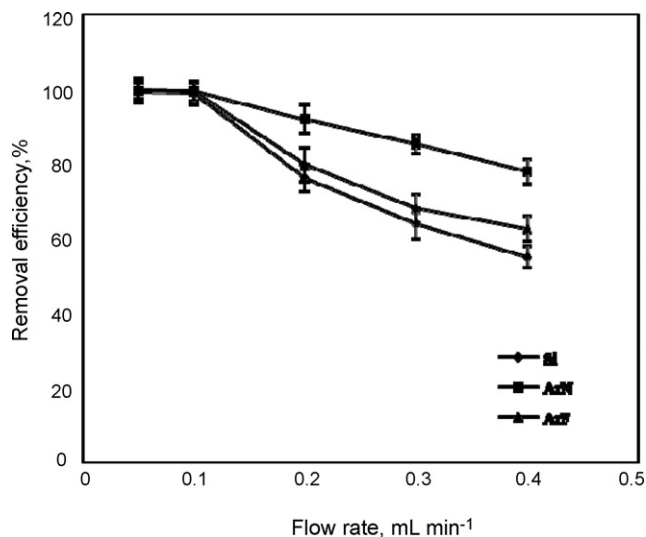

Fig. 2. Variations in the signal intensities of matrix ions at various sample flow rates ($n = 3$).
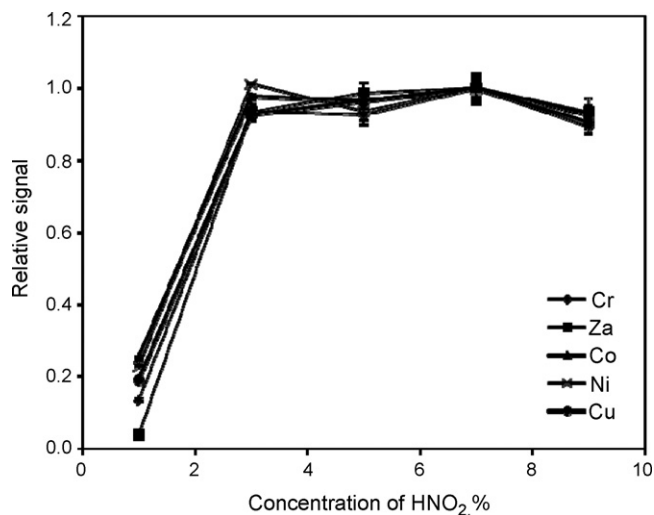
Fig. 3. Effect of the concentration of HNO_3 on the signal intensities of the analyte ions. The relative signal intensity was calculated as the ratio of the signals of tested element to their maximum intensities ($n = 3$).

Fig. 4. Effect of the amount of N_2H_4 on the recovery of Cr(VI) ions ($n = 3$).

3.2. Recovery of analyte ions

In principle, our removal of anionic species from the sample stream using electrodialysis equipment and an anion exchange membrane was based on the exchange of target ions for OH^- ions. According to our observations, the pH of a sample solution can increase to greater than 8 when a solution containing $1500\,mg\,L^{-1}$ of silicate, 1% (v/v) HF, and 5% (v/v) HNO_3 is passed through the electrodialyzer. Unfortunately, Fig. 3 reveals that inadequate retention of the analyte ions occurred after passing this sample solution through the electrodialyzer. We suspect that the loss of analyte cations was related to (i) the association of the quaternary ammonium groups with OH^- ions in the membrane [22] when the pH of the sample stream was greater than 8, (ii) a reduction in the selectivity of the membrane and (iii) the formation of insoluble metal hydroxides. Accordingly, to minimize the loss of analyte cations, we added higher concentrations of HNO_3 to the sample solutions to improve the retention efficiencies of the analyte cations. The percentage recoveries of the tested elements increased upon increasing the concentration of HNO_3 in the sample solution, presumably as a result of the improved selectivity of the ion exchange membrane upon increasing the degree of dissociation of the $RN^+(CH_3)_3OH^-$ species. For HNO_3 concentrations in excess of 3%, the recoveries of some of the analyte ions became quantitative. We conclude, therefore, that a mixture of 1% (v/v) HF and 5% (v/v) HNO_3 should be used to dissolve the thermal oxide layer on Si wafers to maintain the analyte ions during the electrodialysis process.

Because we used 3% (v/v) H_2O_2, 1% (v/v) HF, and 5% (v/v) HNO_3 to dissolve the oxide layer on the Si wafer, we expected, according to Kotaś et al. [25] that Cr(VI), which exists as CrO_4^{2-}, would become the main species in the resulting solution. Fig. 4 suggests that a relatively large fraction of Cr(VI) escaped from the solution through the anion exchange membrane, providing very poor recovery, when the sample solution was poured into the electrodialyzer. To avoid the loss of Cr during the electrodialysis process, we added hydrazine as a reducing agent to convert Cr(VI) to Cr(III), which exists as a cationic species, prior to measurement. To determine the percentage conversion of Cr(VI) to Cr(III), we poured the resulting solutions into the electrodialyzer and measured the residual concentration of Cr in the effluent. Fig. 4 displays the effect of the concentration of hydrazine on the percentage conversion of Cr(VI); it was readily and completely converted into Cr(III) within 30 min when the con-
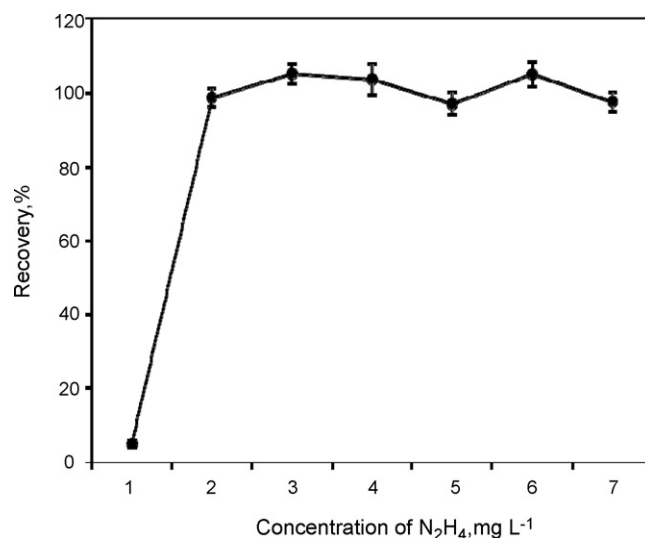
centration of hydrazine was greater than $2\,mg\,L^{-1}$. For the sake of safety, in subsequent experiments we used $10\,mg\,L^{-1}$ of hydrazine to reduce Cr(VI) to Cr(III) prior to determination.

3.3. Reduction of spectral interference

As revealed in Table 1, most of the isotopes of interest suffer from different degrees of Si- and/or F-based polyatomic interference when original sample solutions are analyzed directly using ICP-MS. In this present study, we aimed to analyze metallic impurities in the solution containing Si, HNO_3, and HF using an electrodialyzer/ICP-MS hyphenated system in which almost all of the SiO_3^{2-}, NO_3^-, and F^- matrix ions can be removed prior to ICP-MS determination. To ensure the accuracy of the ICP-MS measurement, we measured the $^{64}Zn/^{66}Zn$, $^{58}Ni/^{60}Ni$, and $^{63}Cu/^{65}Cu$ isotope ratios directly (Fig. 5); we found that they differed from the theoretical values as a result of additive interference caused by the appearance of polyatomic ions. It is obvious that our proposed electrodialysis procedure resulted in very low matrix ion concentrations remaining in the solutions, allowing more accurate isotope ratios to be obtained. In other words, the interference resulting from the sample matrix was significantly reduced when using our proposed on-line electrodialysis device.
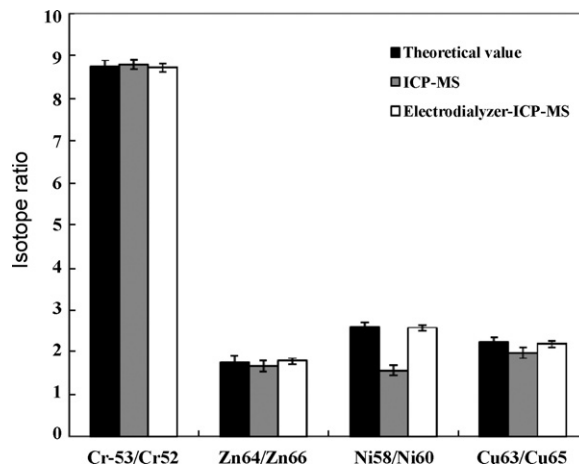

Fig. 5. Variations in the isotope ratios of various analytes measured using different methods ($n = 3$).

Table 3
Analytical characteristics of the proposed electrodialyzer/ICP-MS hyphenation system.

Element	MDL				Measured concentration, µg L^{-1}		Spike recovery, %	Stability, %[a]
	µg L^{-1} *	atoms cm^{-3} *	ng L^{-1} #	atoms cm^{-3} #	ED/ICP-MS[b]	Hot plate[c]		
^{52}Cr	0.41	8.3×10^{15}	3.66	6.6×10^{10}	10.77 ± 0.74	10.94 ± 0.39	103	1.4
^{64}Zn	0.44	8.9×10^{15}	–	–	10.76 ± 0.15	9.91 ± 0.09	99	3.1
^{59}Co	0.10	2.2×10^{15}	0.81	1.3×10^{10}	10.51 ± 0.35	9.56 ± 0.19	98	1.6
^{60}Ni	0.45	9.0×10^{15}	5.48	8.7×10^{10}	11.06 ± 0.73	9.29 ± 0.01	98	1.7
^{63}Cu	0.33	6.7×10^{15}	1.05	1.6×10^{10}	11.09 ± 0.19	10.98 ± 0.90	101	1.8

[a] [Analyte] = 20 µg L^{-1}; 10 replicates.
[b] Electrodialyzer/ICP-MS; n = 5.
[c] Hot plate + ICP-MS; n = 3.
* ICP-MS = Perkin-Elmer Elan 5000.
# ICP-MS = Agilent 7500A.

3.4. Analytical features and application

To demonstrate the applicability of our proposed on-line system, we determined the concentrations of Cr, Zn, Co, Ni, and Cu in the thermal oxide layers of Si wafers. To do so, we interfaced an electrodialyzer equipped with anion exchange membrane to the ICP-MS (as depicted in Fig. 1 and described in Section 2) and added hydrazine to reduce Cr(VI) to Cr(III) prior to analysis. In this case, the time interval between the electrodialysis process and the ICP-MS measurement was only 3 min.

To measure the long-term stability of our proposed on-line method – e.g., if an extended monitoring process were required to collect the data – we repeatedly analyzed a simulated sample containing 20.0 µg L^{-1} of analyte ions. Table 3 indicates that the precision of measurements conducted over a period of 5 h was within 5% RSD, with no temporal decrease in the sensitivity or deterioration in the precision. In other words, any build-up of salts in the system was prevented effectively when this on-line electrodialysis process was applied to eliminate the sample matrix.

In view of the extremely low concentrations of metal ions in the extractants of Si wafers and the limited amount of sample collected after the extraction, it was vitally important to have strict control over the blank value. In this study, all of the reagents used, including the acids, H$_2$O$_2$, and hydrazine, were of ultrahigh-purity grade. We determined the blank by following the established process of this on-line system, using a solution containing 1500 mg L^{-1} of silicate, 1% (v/v) HF, and 5% (v/v) HNO$_3$ as the blank sample. The method detection limits (MDLs) were estimated based on three times the standard deviation of the signal of the blank sample (n = 7). Table 3 reveals that the MDLs for the elements tested were all in the range from 2.2×10^{15} to 9.0×10^{15} atoms cm^{-3}.

Because no certified values were available for the analyte contents in Si wafers, we checked the accuracy of our proposed on-line method by spiking simulated sample solutions containing 1500 mg L^{-1} of silicate, 1% (v/v) HF, and 5% (v/v) HNO$_3$ and comparing the results with those obtained using a conventional hotplate sample preparation method; Table 3 reveals that the results were similar. The good analytical performance of our proposed method, evaluated in terms of the detection limit, accuracy, precision, and stability, suggests that this electrodialyzer/ICP-MS hyphenated system is suitable for the determination of trace impurities in the small volumes of solutions obtained from dissolution of the native and thermal oxide layers of Si wafers.

3.5. Changes in trace impurities in oxide layers at various annealing temperatures

The high diffusivity and solubility of these metal impurities in Si allow their rapid diffusion into the bulk Si wafer [26,27]. There are numerous reports of metal gettering techniques being employed to minimize metallic contamination on the surface of Si wafers by
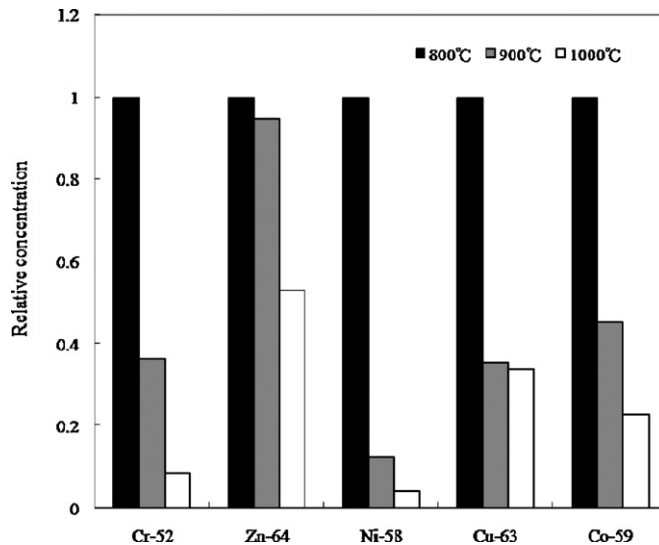
Fig. 6. Effect of heating temperature on the surface concentrations of metallic impurities.

creating gettering sinks at the wafer back surface or at a distance just below the active device zone of the wafer [28]. Most gettering procedures include a high temperature annealing cycle. To evaluate the effectiveness of the gettering procedure, it is necessary to determine the levels of metallic contamination after high temperature annealing [28,29]. Therefore, we further examined the applicability of our established electrodialyzer/ICP-MS system through the determination of residual metal concentrations in oxide layers obtained after annealing at various temperatures. Using the methodology described above, Fig. 6 illustrates that the levels of the Cr, Zn, Co, Ni, and Cu contaminants in the oxide layer of the Si wafer decreased upon increasing the annealing temperature. This finding is important for further study and optimization of the gettering process. Because we could observe significant differences in the concentrations of these analytes after high temperature annealing, our proposed method appears to be applicable to other studies of the changes in the concentrations of metallic impurities in the oxide layers of Si wafers.

4. Conclusion

The novel and simple on-line electrodialyzer/ICP-MS method reported herein is suitable for the routine determination of ultratrace metallic elements in both the native and thermal oxide layers of Si wafers, after dissolving these layers in a mixture of 3% (v/v) H$_2$O$_2$, 1% (v/v) HF, and 5% (v/v) HNO$_3$. By optimizing the instrument measurement protocol, our developed method allows the determination of at least five elements, including such critical semi-

conductor elements as Cr, Zn, Co, Ni, and Cu, in the dissolved solutions of the silicon oxide layer. A distinct feature of this technique is that almost all of the SiO_3^{2-}, NO_3^-, and F^- matrix ions are removed by the electrodialyzer so that they never reach the ICP-MS system. Therefore, the results are not subject to any spectral or non-spectral interference caused by the complex matrix in the dissolved solutions, allowing detection limits of 2.2×10^{15} to 9.0×10^{15} atoms cm^{-3} to be achieved on a 135.89-mm wafer. In addition, quantification of trace metal impurities in the oxide layers of Si wafers can be conducted with satisfactory reliability, even when performing surface metal extraction on wafers having oxide layers up to 2600 Å thick.

Acknowledgment

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