Investigation on Ga contamination due to Dual Beam FIB for In-line Use

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Abstract

We have investigated for Ga contamination issue of a wafer served for analysis by a dual beam FIB. The Ga lateral distribution around ion milled spots was analyzed by TXRF and TRXPS methods. The both results indicated that the most of deposited Ga species distribute within the area of 10 to 20 mm ϕ, and also that Ga contamination level around milled spots was found to be 2 to 10 $\times 10^{10}$atoms/cm^2 for Ga dosages of 3 to 113 min.nA. These results did not indicate that Ga concentration and injected dosage are much to do with each other. ICP-MS analysis, where Ga distribution were supposed to be in the area of 10 to 20 mm ϕ, gave 10 to 100 times larger values than those obtained by the mentioned two method. Oppositely, Ga concentration increases against dosage. This is because ICP-MS detected Ga species not only at the surface but also those implanted under the surface. For wafers which have been unloaded from the chamber after only SEM observation without ion milling, Ga contamination was found to be below the ICP-MS and also TRXPS detection levels.

1. Introduction

In a LSI device fabrication, the wafer surface cleanness is one of the important factors related with production yield and reliability of products. It is required that contamination must be less than 10^9 atoms/cm^2 order, the detection limit by total reflection X-ray fluorescence spectroscopy (TXRF).

A dual beam focussed ion beam (FIB) system, which is the FIB integrated with scanning electron microscopy (SEM) function, has become one of the important tools for the yield management business. In its typical application, defects are initially observed with SEM, and a part of them are cut with the FIB to expose their cross sections and then the internal structures are reviewed with SEM and/or scanning ion microscopy (SIM). When the dual beam system is used in a production line, one must pay attention to the contamination due to the primary ion source, namely Ga contamination, and also that due to the sputtered species of device constituent atoms. It is very concerned whether the wafer can be returned back to a production line or not after the analysis. Regarding the Ga contamination, the following two cases should be taken into account: 1) the ion milled wafer, 2) the SEM observed wafer without milling. We have investigated for the Ga contamination issue in relation to the dual beam system operation.

2. Experimental

The level of Ga contamination for the wafers after being milled by a dual beam FIB, JFS-9815 (JEOL) [1], has been investigated using the following three methods: total reflection X-ray photoelectron spectroscopy (TRXPS) [2], TXRF and inductively coupled plasma mass spectroscopy (ICP-MS). For milling in the specimen preparation, the 30 kV Ga ions were used under the area scan (the ion currents were approximately 1 nA and 8.5 nA, and the area size was approximately 25 μm x 25 μm). TRXPS measurements were done with JPS-9000MC (JEOL) using AlKα X-rays. The power used for generating X-rays was 300 W (10 kV, 30 mA). The X-ray beam size on a sample surface was restricted to 5 mm (vertical) by 3 mm (horizontal). The TXRF measurement for each case was achieved with different apparatus, but the acceleration voltage and emission current for generating X-rays were kept commonly at 40 kV and 40 mA, respectively, and also the X-ray beam size on the specimen surface was set commonly 10 mm ϕ. The ICP-MS measurement was carried out with SPQ-9000 (SEIKO instruments) for the

Table 1 The summary of Ga contamination levels obtained by TRXPS, TXRF and ICP-MS for various experimental cases. For milling in preparation, the 30kV Ga ions were used. The concentration by ICP-MS were derived based on an estimated distribution area.

Experimental Case		Ga ion dosage min.nA	Detected Ga atoms $x10^{10}$atoms	Electron beam irradiation condition kV, min, nA	Ga concentration over 10mmϕ area $x10^{10}$atoms/cm^2	Ga concentration over 3×5mm^2□ area $x10^{10}$atoms/cm^2	Ga concentration over 20mmϕ area $x10^{10}$atoms/cm^2	Ga concentration over 180mmϕ area $x10^{10}$atoms/cm^2	Method
Milled Wafer	Case 1	*3 **	—	—	—	5	—	—	TRXPS
	Case 2	*14 †*	—	—	—	2	—	—	〃
	Case 3	*113 †*	—	—	—	4	—	—	〃
	Case 4	*30 **	—	—	10	—	—	—	TXRF
	Case 5	*9 **	—	—	7	—	—	—	〃
	Case 6	*20 **	—	—	6	—	—	—	〃
	Case 7	*27 †*	—	—	370	—	—	—	〃
	Case 8	*71 †*	1,100	—	280	—	70	—	ICP-MS
	Case 9	*567 †*	5,100	—	1,300	—	325	—	〃
Non milled Wafer	Case 10	—	<4.3	*3kV,100min,1nA*	<5.5	—	<1.4	<0.02	ICP-MS
	Case 11	—	<4.3	*3kV,100min,1nA*	<5.5	—	<1.4	<0.02	〃
	Case 12	—	<4.3	—	<5.5	—	<1.4	<0.02	〃
	Case 13	—	<4.3	—	<5.5	—	<1.4	<0.02	〃
	Case 14	—	—	*5kV,10min,1nA*	—	<1	—	—	TRXPS
	Case 15	—	—	*5kV,10min,1nA*	—	<1	—	—	〃

※The primary currents were 1nA for * and 8.5nA for †.

condensed chemical etching solution, which etched out Ga species. A water solution of complex acid of HNO_3 and HF was used as the chemical etcher.

3. Results and Discussion

The values of Ga concentration for the different cases have been summarized in Table 1 with the experimental conditions.

3.1 Milled Wafer

The three specimens injected with different Ga dosages (3, 14 and 113 min.nA at 30 kV) were analyzed by TRXPS and the results are listed as the case 1 to case 3 in Table 1. The detected Ga concentration level was 2 to 5 $x10^{10}$atoms/cm^2 and is not related with the dosage in this range.

The similar measurement was also carried out by TXRF and the results are listed as the case 4 to case 7 in Table 1. The detected Ga concentration was 6 to 10 x 10^{10}atoms/cm^2 except for the case 7, in which the derived value is almost of 2 order higher (3.7 x 10^{12} atoms/cm^2). At the moment we cannot give a good reason for this big difference. Among the data except for the case 7, it is noticed that the detected Ga levels and the injected dosages do not much relate each other.

Lateral distribution

The Ga lateral distribution was investigated by TXRF for the milled spot and its neighboring area. The result is shown in Fig. 1. The measurement was carried out at 10 mm intervals in each direction. The Ga of amount 7 $x10^{10}$atoms/cm^2 was detected in the area of 10 mmϕ including the milled point. It fell down to 1.3 $x10^{10}$atoms/cm^2 at the position of 10 mm away in the x direction and 10 mm away in the y direction from the milled point.

The spatial resolution of TXRF is limited to 10 mm, while that for TRXPS is a little better than it, namely 3 mm. Fig. 2 shows the Ga distribution obtained by TRXPS along the line running the two milled spots separated 13 mm away each other. Although the accuracy is not enough, the obtained profile indicates that the most of Ga atoms were distributed within the area of 10 mmϕ to 20 mmϕ.

ICP-MS measurement

The Ga contamination for the two wafers of 200 mmϕ, on each of which totally five spots were milled but with different ion dosages (71 min.nA and 567 min.nA for the total five spots, respectively, under 30 kV and 8.5 nA) were

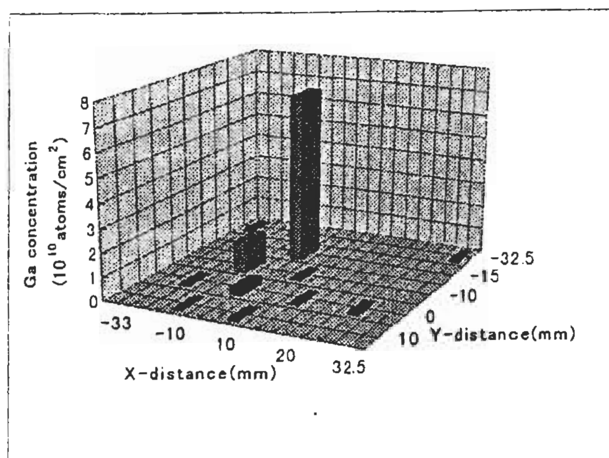

Fig.1 The Ga lateral distribution around the ion milled point obtained by TXRF analysis.

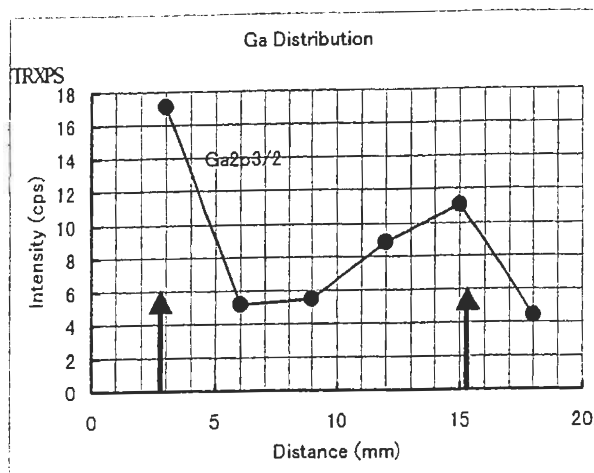

Fig.2 The Ga2p3/2 line profile by TRXPS along a line running over the two ion milled spots separated 13mm away each other. The ion milled positions are indicated by the allows.

analyzed. A thin layer of the wafer surface except the wafer edge of 10 mm width were etched out by the water solution of complex acid of $HNO_3$and HF. The etching solution including Ga were condensed and served for ICP-MS analysis. Thus, the data will give an amount of Ga existing on the total surface area of certain thickness. The results were, as shown in Table 1, $1.1x10^{13}$ atoms/ wafer for 71 min.nA (case 8) and $5.1x10^{13}$ atoms/wafer for 567 min.nA (case 9), respectively. Taking into account the results by the TXRF and TRXPS analyses for the lateral extent of Ga contamination, we suppose the contaminated area is limited to be in 10 to 20 mmϕ. If it were 10 mm ϕ, 1.1 $x10^{13}$ atoms/wafer is converted to 280 $x10^{10}$ atoms/cm^2 and 5.1 $x10^{13}$atoms/wafer is converted to 1300 $x10^{10}$atoms/cm^2 , respectively. If it were 20 mmϕ, through the similar conversion, they are 70 $x10^{10}$ atoms/cm^2 and 325 $x10^{10}$ atoms/cm^2, respectively. These values are larger than those obtained by TXRF and TRXPS analyses. Also, oppositely from the results by TXRF and TRXPS analyses, the ICP-MS results indicate that Ga concentration increases against dosage. It can be explained as that ICP-MS detects Ga species not only from the surface but also under the surface, where implanted species exist, but the other two methods detecting only from the surface area.

Chemical state

The TRXPS brings us not only quantitative information but also chemical state information. Fig. 3 shows the spectra for the experiment case 1 to 3. Under the lower dosages, Ga in elemental state was richer than that in oxide state, but under the higher dosages the situation became inverse. We cannot interpret for this phenomenon at the moment, but only point out that TRXPS brings us chemical information for such a trace element. Finally it is noticed that the peak position in the bottom spectrum is shifted from other two. It seems to be due to the shift of specimen position.

3.2 Non-Milled Wafer

The Ga contamination issue was investigated for the wafers, which have been unloaded from the chamber after only SEM observation without ion milling. Before this test, the instrument had been used for ion milling a number of times.

The ICP-MS analysis was conducted for the following four cases: 1) Electron beam irradiation for 100 minutes under 3 kV and 1 nA but no ion beam irradiation (by a mechanical shutter), 2) Electron beam irradiation under the same condition but no ion beam irradiation (by an electrical beam blanking), 3) Keeping in the chamber for 6 hours without electron and ion beam irradiation (by the mechanical shutter) and 4) Keeping in the chamber for 6 hours without electron and ion beam irradiation (by the electrical beam blanking) . It is noticed that the mechanical blanking shut off both ion beam and neutral beam but the electrical beam blanking allows to pass neutral species.

The results are listed in Table 1 (in turn, case 10 to 13). The Ga has not been detected for any cases by ICP-MS analysis. The detection limit

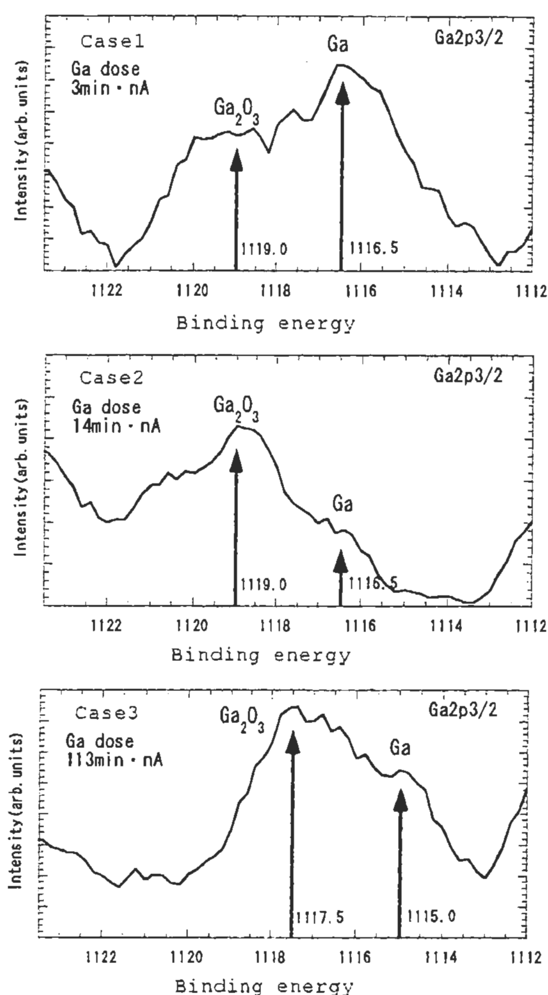

Fig.3 The Ga2p3/2 peaks obtained by TRXPS at ion milled spots. When lower Ga dosage, elemental Ga is stronger, but when higher dosages oxidized Ga gets stronger.

by this method is $4.3x10^{10}$ atoms. If the entire surface were uniformly contaminated, the possible concentration must be less than 0.02 x 10^{10} atoms/cm^2. However, the beam irradiation area seems to have more possibility for contamination because neutral species may come to the specimen surface. Also, since the distance between the specimen analysis area and the instrumental objective lens surface where it might be contaminated are close each other, Ga deposition from there due to the back scattered electron stimulation may occur. If the contaminated area were localized to be in 20 mmϕ, it is less than $1.4x10^{10}$ atoms/cm^2. Also, it is less than $5.5x10^{10}$ atoms/cm^2 for 10 mmϕ area. This result could not give an enough answer to the question, namely for the possibility of localized contamination of low level.

To investigate further, we have conducted TRXPS analysis. We have used in advance the dual beam system for ion milling (created a hole of volume 10x10x10μm^3 on aluminum) and loaded Si wafers of the size 20 mm x 20 mm after 10 minutes later. The two specimens were exposed in the chamber atmosphere: the one (Case14) was set on the stage at horizontal with electron beam irradiation for 10 minutes under 5 kV and 1 nA, the other (Case15) set at 60° tilted with electron beam irradiation under the same condition. It was found by TRXPS analysis that the Ga levels were less than $1x10^{10}$atoms/cm^2 for the both cases.

4. Summary

The Ga contamination issue for the wafer after an analysis by a dual beam FIB system was investigated.

1) The Ga lateral distribution around the ion-milled spots was investigated by TXRF and TRXPS methods. The both results indicate that the most of Ga distributes within the area of 10 to 20 mmϕ.

2) The Ga contamination level around the milled spots was found to be 2 to 10 x 10^{10} atoms/cm^2 for 3 to 113 min.nA dosages by TRXPS and TXRF analyses. It did not indicate that Ga concentration and injected dosage are much to do with each other.

3) The ICP-MS analysis, where Ga distribution were supposed to be in the area of 10 to 20 mm ϕ, gave 10 to 100 times larger values than those by the other two surface analysis methods. The Ga concentration oppositely increased against Ga dosage. This is because ICP-MS analysis detected Ga species not only at the surface but also those implanted inside the bulk.

The Ga contamination for the wafers that have been unloaded from the chamber after a review and the followings were found.

4) The Ga contamination was below the ICP-MS detection level.

5) The Ga contamination was below the TRXPS detection level.

5. References

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[2] J. Kawai, S. Hayakawa, Y. Kitajima, K. Maeda and Y. Goshi, J. Electron. Spectrosc. Relat. Phenom. 76, 313 (1995)