A First Look at a New Approach to On-line ICP-MS Measurement

On-line measurement of chemical baths located within a manufacturing site has several advantages over the traditional method of sample gathering. Imagine a situation where a chemical contamination is suspected in a chemical bath located within a semiconductor manufacturing site. To check for this contamination using ICP-MS technology located in a laboratory would require several time consuming steps. First, a laboratory person must be called by a person working at the chemical bath. The laboratory person would need to gown up, approach the chemical bath to collect the sample, remove the gown to exit the manufacturing site, then carry the sample to the laboratory for analysis. This could easily result in an hour or more elapsing before the sample results could be sent back to the person at the chemical bath. The time elapsed could be very costly in either down time or in poor product quality. A system that would continuously monitor the chemical in the bath and keep the operator informed of any contaminations that may be present could significantly improve productivity.

Several technical challenges exist in the design of an on-line monitoring system. If the on-line system is to be added to an existing manufacturing facility, it is most likely that the ICP-MS will need to be located some distance from the chemical bath(s) – possibly up to thirty meters. It is important that the sample be transferred quickly and without loss of the analytes to be measured. The ICP-MS must be one that provides a high degree of accuracy and productivity with a minimal amount of operator intervention. Three companies, Micron Technology, Inc. (Boise, ID), Elemental Scientific Inc. [ESI] - (Omaha, NE), and PerkinElmer (Shelton, CT), have worked together to develop an on-line system that takes these challenges into account. By combining the Remote Aerosol Transfer System ® from ESI with the ELAN® DRC II and adapting the system so that it would be suitable to place within a Micron Technology facility, the possibility of on-line monitoring for three cleaning chemicals commonly used in the traditional RCA cleaning procedure was investigated. This informal article will take a first look at a system designed to provide such an ICP-MS on-line monitoring of chemical baths within a semiconductor manufacturing facility.

Note: At the writing of this article, on-line systems have not been installed in customer locations. All test data provided in this article were obtained at PerkinElmer-SCIEX and ESI facilities.

Accuracy and productivity from the ICP-MS

The ELAN DRC II ICP-MS used in this system incorporates dynamic reaction cell technology to remove a wide variety of interferences. Such effective removal of interferences provides a high level of accuracy even when measuring at the part per trillion (ppt) level. To demonstrate how effectively the ELAN DRC II can remove sample based interferences, we will look at the systems ability to accurately measure semiconductor elements in a challenging sample type - 20% HCl.

Several chlorine based interferences can make the analysis of HCl by ICP-MS problematic. In Figures 1 and 2, we can view how effectively these interferences are removed by the dynamic reaction cell. On the right side of each of the figures, we see a line that is nearly horizontal even though the concentration of the analyte is increasing. A quick glance at the scale will disclose that the counts per second are quite high in both examples. The horizontal line is the result of a high background caused by ClO in Figure 1 and Cl_2 in Figure 2. In both examples, the readings were taken without the use of the dynamic reaction cell. Now a small flow of ammonia gas is introduced into the dynamic reaction cell. In each figure, the graph on the left shows the resulting reduction in background signal. Now we are aable to measure ^{51}V in Figure 1 and ^{74}Ge completely free of background interference. This is the technology

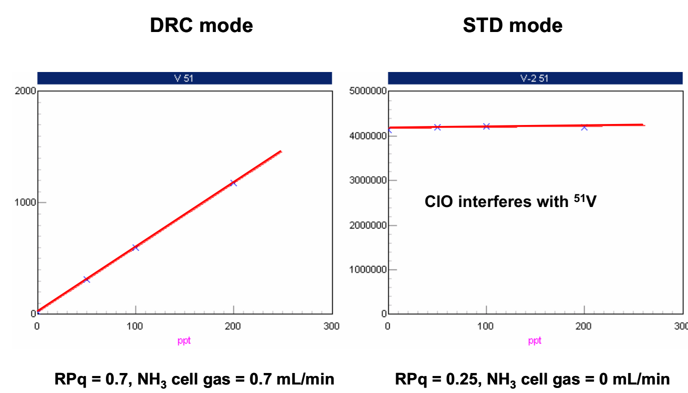

Figure 1 The removal of the ClO inteference on ^{51}V

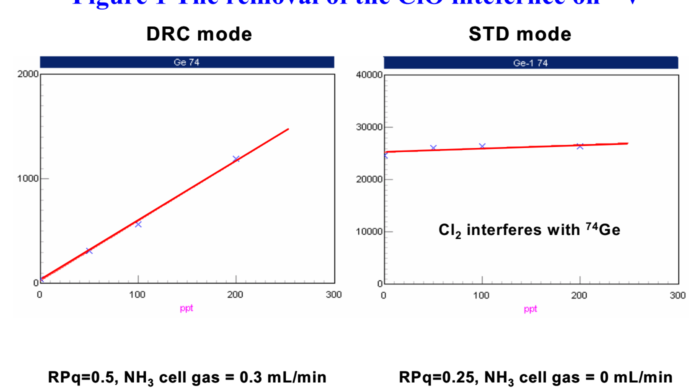

Figure 2 The removal of Cl_2 interference on ^{74}Ge

20% HCl quantitative results

Element	m/z	Conc (ppt)	DL (ppt)	25 ppt rec.(%) with MSA	Element	m/z	Conc (ppt)	DL (ppt)	25 ppt rec.(%) with MSA
B	11	13.7	5.1	101	Cu	63	36.7	3.4	102
Na	23	0.4	0.5	99	Zn	64	4.0	2.0	97
Mg	24	0.5	0.9	95	Ga	69	1.2	0.9	90
Al	27	1.8	1.8	112	Ge	74	5.0	1.9	96
K	39	32.2	11	112	As	75	30.0	14.1	115
Ca	40	3.7	1.1	113	AsO	91	13.2	3.0	104
Ti	48	6.8	2.5	106	Mo	98	3.5	1.6	97
V	51	2.6	1.3	101	Cd	114	0.6	0.7	102
Cr	52	7.3	2.9	95	Sn	120	11.2	1.9	98
Mn	55	5.9	1.0	101	Sb	121	13.7	9.9	92
Fe	56	10.1	1.4	99	W	184	1.0	0.9	96
Co	59	0.8	0.9	100	Au	197	0.9	0.8	99
Ni	60	4.3	2.4	105	Pb	208	0.2	0.2	102

Figure 3 Detection limits and recoveries in 20% HCl (elements typed in brown are elements analyzed in DRC mode) DL was calculated based on 20% HCl blank

hat assures a high level of accuracy for important semi-conductor elements even when the analysis is made in a challenging sample.

Productivity is provided through a unique method for calibrating the instrument and exceptional instrument stability. Although it might go against what you learned as good technique, it is possible to analyze several different materials using nitric acid based external standards. Using such a technique, the ELAN DRC II can be calibrated just once for the analysis of several different chemicals including HF, SC-1, SC-2, and others commonly used chemicals. Due to several design features including a thermostated quadrupole power supply and an externally mounted sample introduction system, and a high quality nebulizer, the ELAN DRC II displays unequalled long term stability. In a practical sense, this requires that a single external calibration routine (requiring about ten minutes) need be performed only once every four hours. The result is that the ELAN DRC II is available to analyze samples 95% of the time.

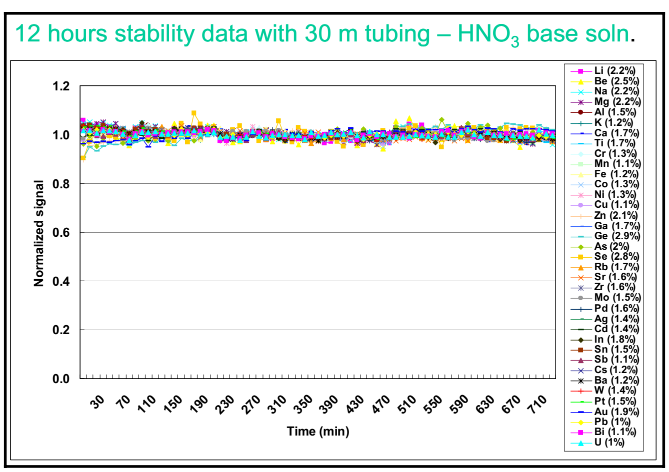

Figure 4 ELAN DRC II long term stability

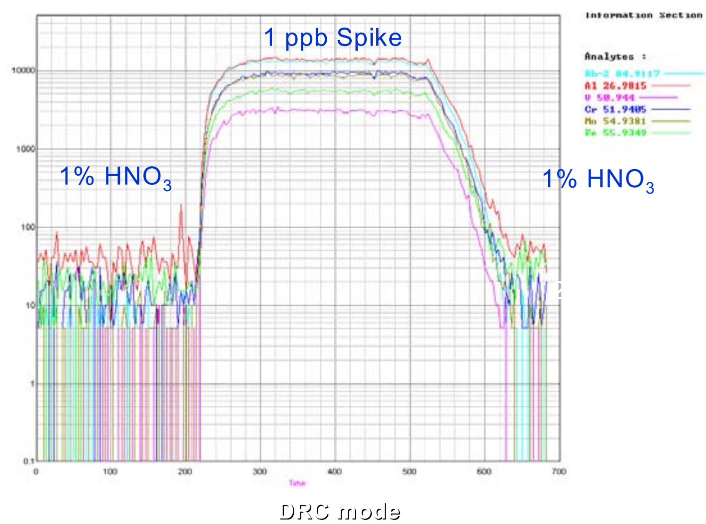

Figure 5 A 1 ppb spike transferred over a 30 meter transfer tube (total time elapsed = 700 seconds)

Transfer of sample

Transporting a sample over a distance of 30 meters quickly and efficiently is a daunting task. Previous on-line systems attempted to transfer the sample from the bath to the ICP-MS as a liquid. At least two difficulties were encountered with such a design. Since the ICP-MS system requires only 1 ml/minute of sample, the sample could take many minutes to be transported from the chemical bath to the ICP-MS. Overflow cups could accelerate the process, however, they result in a major waste of the sample. Additionally, several important elements are absorbed by the walls of the transfer tubing which impacts the accuracy of measurement. Unlike previous systems, the design discussed in this article converts the sample to a fine

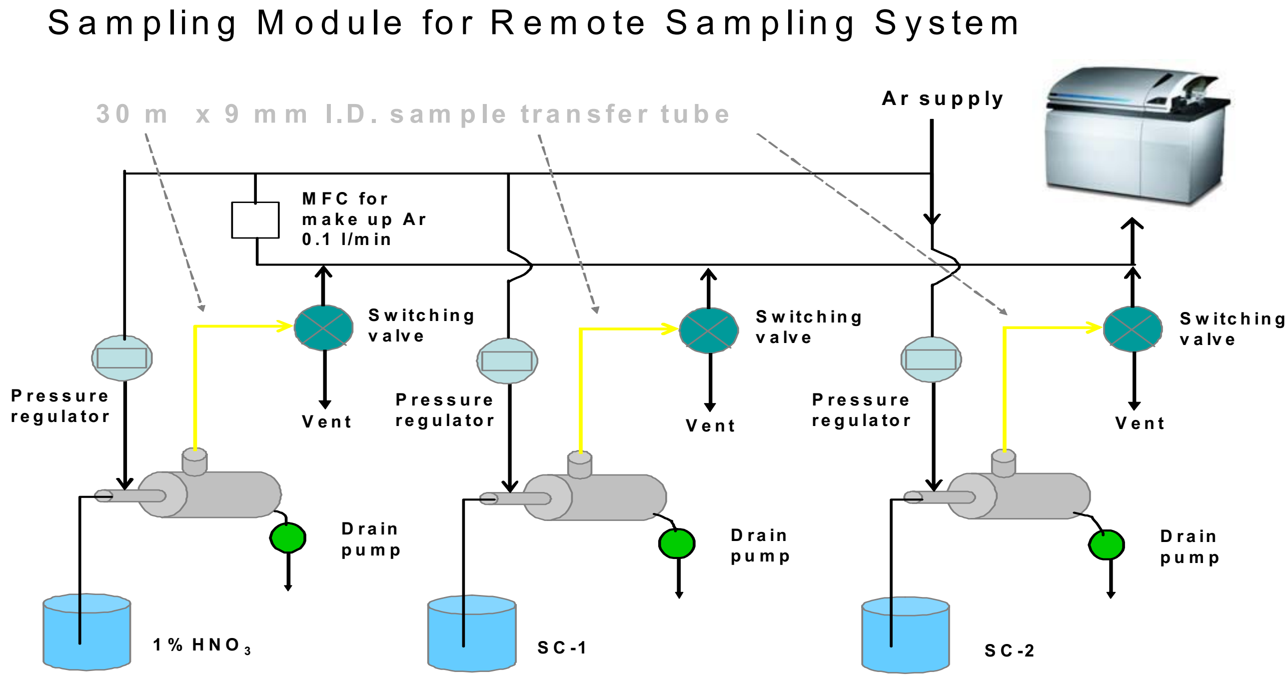

Figure 6 Schematic of Remote Sampling System

aerosol at the chemical bath and transfers the aerosol to the ICP-MS in a flow of argon. The sample is transferred from the chemical bath to the ICP-MS quickly and using the Remote Aerosol Transfer System ® from ESI. Figure 5 (previous page) shows the signal generated by a 1 ppb spike transferred over the 30 meter transfer tube. The return to baseline occurs within two minutes of the signal. Extensive tests performed in our laboratories have shown that all semiconductor elements are transferred efficiently over the 30 meter transfer tube.

Measuring from multiple baths

By combining multiple Remote Aerosol Transfer Systems® with the appropriate pressure regulators and switching valves, it is conceivable that three (or more) baths could be monitored using a single ELAN DRC II ICP-MS. At the rate of six measurements per hour, the system shown in Figure 6 would be capable of providing two readings per hour for each of the three chemical baths monitored. Contaminations introduced into the chemical bath through operator error, failing components in the bath, or other sources would be identified within thirty minutes of the event. A problem can identified and corrected before it results in either manufacturing downtime or production of inferior product.

Conclusion

An on-line ICP-MS system offers great potential for increased productivity and improved product quality in the semiconductor manufacturing environment. The use of Remote Aerosol Transfer System® results in the rapid transfer of the sample to the ICP-MS in a short period of time with high retention of the elements to be measured. Interference removal and long term stability demonstrated by the ELAN DRC II make it the ideal ICP-MS to be used in such a demanding application.

To learn more about the on-line ICP-MS system, please contact Dave Armstrong at adavid.armstrong@perkinelmer.com