Consistent Hydrogen Chloride Purity Delivery through the use of a Built-in Cylinder Purifier (MegaBIP® HCl)

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Abstract

Oxygen contamination from process gases used in silicon wafer etching can result in defects that may promote polycrystalline rather than single crystal growth of subsequent layers and slow down devices by occupying sites in the structure and limiting free flow of electrons. One of the primary sources of oxygen atom contamination is moisture (H_2O).

Many specialty gas vendors offer hydrogen chloride (HCl) with moisture specifications of 1-2 parts-per-million (ppm). HCl, a liquefied gas, undergoes a partitioning phenomenon as the product is withdrawn, whereby impurities such as moisture collect in the liquid phase. This causes the outlet gas to have variable impurity concentrations during its usage and for a large release of H_2O if the customer runs the cylinder to liquid dry. Since the end-user desires low, consistent moisture levels, external purifiers are often installed in the process lines to maintain low parts-per-million levels. External purifiers require large capital outlays and often off-gas or fail during processing , which frequently results in a haze on the wafer.

Air Products and Chemicals, Inc. (APCI), has developed and now offers MegaBIP® HCl--a new high purity HCl product that contains a built-in purifier and filter within the cylinder and removes the output uncertainty observed in traditional HCl cylinders. The MegaBIP® HCl product produces a consistent, low parts-per-billion (ppb) moisture concentration stream of HCl over the entire lifetime of the cylinder. The partitioning of moisture still occurs; however, the built-in purifier removes the moisture generated by this phenomenon and enables the cylinder to be used beyond the liquid-dry point with the same product quality as a full cylinder. Consequently, reduced moisture concentrations lead to less corrosion in the gas delivery system and process chamber, resulting in less process downtime, as well as improved consistency and quality of the wafers.

Analytical testing of MegaBIP® HCl using Cavity Ring-down Spectroscopy (CRDS), a developmental analytical tool for determining low ppb_v moisture in next generation electronic specialty gases, was performed to demonstrate the low parts-per-billion moisture level and consistency of MegaBIP® HCl. Additionally, actual product testing of silicon wafers, including high temperature wafer etching, low temperature selective epitaxial growth (SEG), and low temperature Silicon-Germanium alloy (Si:Ge) processing, was performed in conjunction with Lawrence Semiconductor Research Laboratories, Inc. (LSRL). Both VLSI and MegaBIP® HCl were tested, concluding that the MegaBIP® HCl product can be used successfully without an external point-of-use purifier to yield lower impurity contamination in a process.

Introduction

The consistent delivery of high-purity process gases from cylinder sources represents one of the biggest impurity control challenges in semiconductor processing. If not properly controlled, impurities such as metals, moisture, atmospherics, and particles can seriously interfere with many wafer processes, leading to inconsistent results and adverse effects on device performance and yields. Successful efforts have been made in recent years to control all of these contaminants as industry roadmaps have demanded ever-decreasing impurity levels as device sizes are reduced. (1)

This need for higher purity processing gases processes like Si:Ge, etc., is continually leading to more stringent product specifications for various specialty gases. One of the most critical impurities in terms of semiconductor device performance is moisture. It is also one of the most difficult impurities to minimize due to its ubiquity and innate chemical properties. The degree of difficulty of measuring trace levels of moisture in corrosive gases is also quite a challenge.

Bulk inert gases such as nitrogen can achieve sub-parts-per-billion levels of all impurities through purification, and analytical techniques exist to confirm this purity. It is a different challenge, however, with cylinder process gases, since these gases are often corrosive, toxic, and more difficult to handle and analyze. (2) These process gases also have an immediate effect since they directly participate in layer and device chemistries. In spite of this, several semiconductor companies are now pushing for lower moisture specifications on corrosive gases used for silicon wafer processing.

One consequence of oxygen incorporation from moisture in this process is crystal defects that optically manifest themselves as haze in the crystal. More significantly, the presence of oxygen slows down the performance of the devices by slowing down electron transfer rate within the semiconductor. In addition to electrical and structural defects, the presence of moisture in these gases also leads to corrosion of the gas handling equipment. This often leads to unplanned failures and further capital expenditures. Particles generated from corroded surfaces can then incorporate into the devices and have an adverse impact on the performance.

Although an exact moisture specification has not been formally set, evidence from epitaxial processes that use HCl suggests that moisture specifications in the low ppb_v range are desirable. To address the need for corrosive gases with lower impurity specifications, Air Products and Chemicals, Inc., developed and now offers the MegaBIP® HCl cylinder technology with the specifications listed in Table 1. This cylinder technology is unique since it includes a built-in-purifier (BIP) and filter within the liquefied gas cylinder, thus producing consistent purity HCl during the complete use of the product in the cylinder.

Table 1. MegaBIP® HCl impurity specifications.

Impurity	Specification (ppmv)
CO_2	2.0
CO	1.0
CH_4	1.0
N_2	2.0
O_2/Ar	1.0
H_2O	0.2
Fe	0.5 ppm_w
Other Metals	1.0 ppm_w

MegaBIP® HCl Design

Figure 1 shows how filling the cylinder through a separate pathway, called the BIP bypass, controls gas quality. When the standard customer valve is used, gas is removed through the purifier, providing gas with moisture levels below 200 ppb_v directly from the cylinder. The MegaBIP® cylinder also contains a check valve so there is no possibility of back contamination compromising the purifier. In addition, with the purifier completely sealed within the cylinder, it is immune to atmospheric leaks.

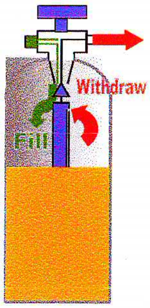

Figure 1. Schematic of the MegaBIP® HCl design.

By operating at a higher pressure within the cylinder, the efficiency of purification is enhanced over ambient pressure operation. Unlike conventional low-pressure external purifiers, the high-pressure manifold upstream of the process regulator in MegaBIP® systems is also protected, since the purifier is already delivering purified gas directly from the cylinder. This is important in corrosive gas systems where piping is thermodynamically more vulnerable to corrosion at higher pressure than in standard systems, especially when stainless steel tubing and components are frequently used. (1) Also, HCl regulators often fail due to the Joule-Thomson (JT) effect of moisture collecting on delivery side, and the MegaBIP® HCl low ppb_v moisture delivery aids in extending the lifetime of such components.

High purity components are used within the MegaBIP® HCl design to maintain the consistent purity cylinder output. This is achieved through the use of electropolished stainless steel and Hastelloy materials, a proprietary tied-diaphragm valving design to minimize contamination, and the incorporation of 30 Å filter, to name a few.

As it has been stated, the most significant impact of the MegaBIP® HCl technology is the consistency of low-level moisture in the gas emerging directly from the cylinder. As shown in the schematic in Figure 2, the moisture level remains below 200 ppb_v to the end of the cylinder, allowing the use of the cylinder to a liquid dry state. Because of this fact, it has been possible to eliminate costly external purifiers with substantial savings in operational costs, with no detriment to process and product performance over a substantial period.

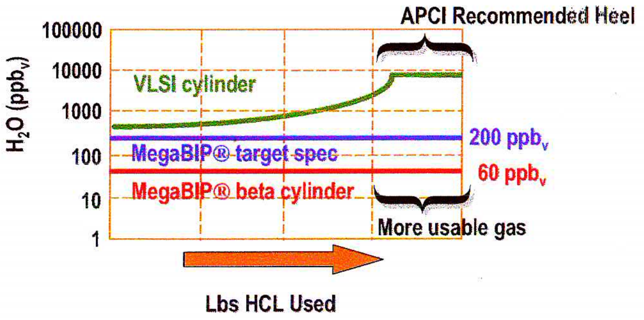

Figure 2. Advantage of MegaBIP® HCl over VLSI HCl for usage of cylinder.

Trace Detection of Critical Impurities in MegaBIP® HCl

In order to meet the 200 ppb_v moisture specification for the MegaBIP® HCl product, an analyzer is needed that has a detection limit on the order of 20 ppb_v or less and a fast response time. Cavity ring-down spectroscopy (CRDS) (3-6) is a commercially available technique that shows great potential of measuring moisture at single ppb_v levels in a variety next generation electronic specialty gases including corrosive gases such as HCl. Although CRDS is an absorption-based technique, the CRDS technique offers advantages over conventional absorption spectroscopy, which include increased sensitivity, speed of response, a wide dynamic range, and freedom from calibrations. Moreover, CRDS is a non-destructive measurement technique that is chemically specific with an effective path length of ~10^5 meters for superior signal to noise.

The cavity ring-down analyzer is comprised of a sample cell that serves as an optical cavity, a pulsed laser source and a photodetector (Figure 3). The cell consists of two mirrors forming a stable optical cavity approximately one meter long.

The mirrors are highly reflective, typically with reflectivities (R) of 0.9999 or greater. Light from a pulsed laser source is sent to the cavity, and a small but stable fraction ($\sim10^{-5}$) of the light energy enters the cavity. This light bounces back and forth between the mirrors and on each pass $\sim10^{-5}$ of that light is transmitted through one of the end mirrors and is detected with a photodetector in the form of an exponentially decaying pulse train, called the ring-down time. Changes in the ring-down time, as compared to a reference sample (which is a measurement taken off-peak of the moisture line), is proportional to the amount of moisture present in the sample, and thus removes the need for an external calibration.

A Tiger Optics model MTO-1001-H_2O CRDS analyzer with a continuous wave near-IR diode laser was used for the determination of trace moisture in MegaBIP® HCl. The MTO-1001-H_2O CRDS analyzer operates slightly above atmospheric pressure (~5 psig) using a pressure regulator before the sample cell. It incorporates a four-valve inlet manifold for multiple gas stream sampling. Each inlet has a respective bypass outlet to maintain a flow through the sample lines while the inlet valve is not open. This maintains a representative sample of the gas being measured right at the inlet of the instrument.

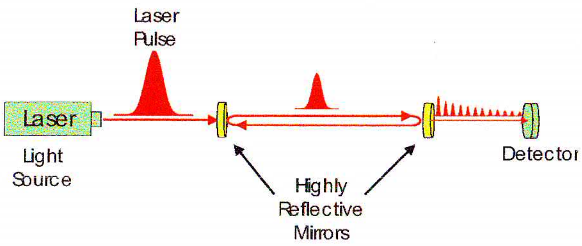

Figure 3. Schematic representation of the cavity ring-down spectroscopy (CRDS) technique.

The MTO-1001-H_2O determines moisture concentration in corrosive gases by monitoring the intensity of the 1.3925 μm combined band rotational line of water. HCl has no interfering absorption features in this region. The MTO-1001-H_2O CRDS also comes precalibrated with a broadening coefficient for HCl. Since the CRDS is independent of laser and detector intensity fluctuations it requires no further calibration as long as the τ_{empty} (i.e. off peak moisture measurement) is checked periodically to ensure that it is constant.

The linearity of the CRDS response to moisture in HCl was tested through standard additions of moisture to HCl using an in-house built moisture generator containing calibrated moisture permeation tubes. The sampling and dynamic dilution system, which includes the moisture generator, for this experiment is presented in Figure 4. Moisture is added directly to the corrosive gas stream by opening a valve on the output of the moisture generator. Since the moisture generator produces wetted nitrogen that mixes with HCl, the HCl in the manifold is diluted to 98.2% at the maximum moisture concentration challenge for this study. The manifold also utilizes an external corrosive gas purifier with a bypass for a zero gas (baseline) measurement.

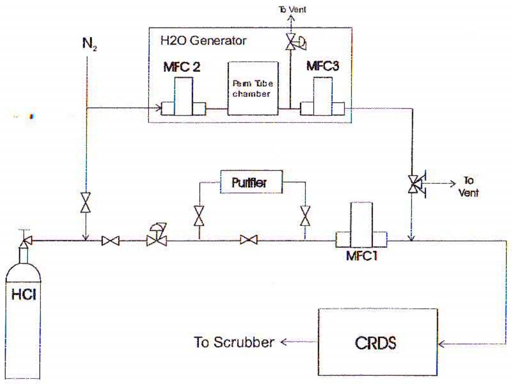

Figure 4. Schematic diagram of the CRDS sampling and dynamic dilution system.

The MTO-1001-H_2O CRDS exhibits good linearity for moisture concentrations between 0 and 95 ppb$_v$ in HCl. Figure 5 shows the MTO-1001-H_2O response to step changes of moisture in HCl of 94.5, 48.4, 23.4, 9.84, and 0 ppb$_v$, respectively. The fluctuations at the 94.5 ppb$_v$ moisture level in Figure 5 are due to the dynamic dilution manifold and not the CRDS. In addition to this manifold effect, there is also an effect caused by room temperature fluctuations. Neither issue affects the overall method detection limit (MDL) of the measurement.

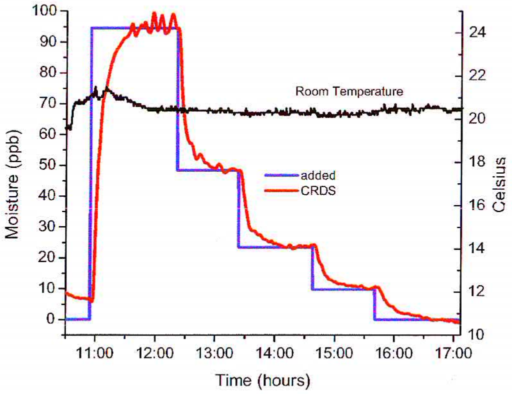

Figure 5. MTO-1001-H_2O CRDS response to moisture challenges in HCl from 0 to 94.5 ppb$_v$.

A correlation coefficient of 0.9994 was calculated for the data shown in Figure 6 using ordinary least squares fitting (OLS). Each data point represents an average of 30 to 50 data points for each concentration. The data used in Figure 6 comes after the moisture step had achieved a steady state (Figure 5). A moisture in HCl MDL of 1.2 ppb$_v$ (OLS)/ 0.5 ppb$_v$ weighted least squares (WLS) was calculated from the data in Figure 6.

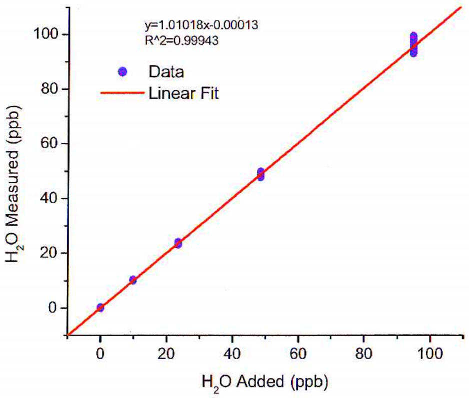

Figure 6. Observed linearity of CRDS for moisture in HCl.

After validating the CRDS for moisture measurements in HCl, moisture determinations were made for numerous MegaBIP® HCl product cylinders. Table 2 shows the actual moisture measurements for recently analyzed cylinders. To date, all of the MegaBIP® HCl product cylinders analyzed for moisture using the CRDS technique have passed the 200 ppb$_v$ moisture specification.

Table 2. Moisture determinations in a sampling of MegaBIP® HCl product cylinders.

MegaBIP® HCl Cylinders	ppb$_v$ H_2O (CRDS)
Cylinder 1	154
Cylinder 2	160
Cylinder 3	180
Cylinder 4	114
Cylinder 5	118
Cylinder 6	159
Cylinder 7	157
Cylinder 8	161
Cylinder 9	141

The true test of the MegaBIP® HCl product is how well the BIP purifier performs when a cylinder is at or near liquid dry. Figure 7 shows the CRDS measurement of moisture in the heel of a MegaBIP® HCl cylinder both using the MegaBIP® HCl purifier and in the BIP bypass mode. As it can be seen from the data, the moisture is concentrated in the heel and is approaching the limit of quantitation for the CRDS. However, when the product is measured through the MegaBIP® purifier, it sufficiently passes the 200 ppb$_v$ specification.

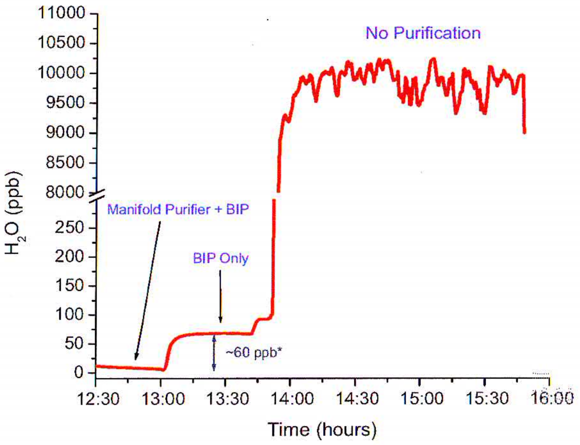

Figure 7. Moisture measurements from the heel of a MegaBIP® HCl cylinder.

Another measure of the performance of the MegaBIP® HCl internal purifier is the CRDS moisture measurement after refills of the cylinder. Typically, cylinders are not used to a complete liquid dry state. Instead, a small liquid heel of a few pounds of HCl remains upon which new HCl material is added. Since the liquid heel is concentrated with moisture, the top-filling procedure actually creates a situation where the fill material increases in moisture concentration on each fill. Table 3 shows the moisture data for three consecutive fills of a MegaBIP® HCl cylinder. As it can be seen in the table, the MegaBIP® HCl internal purifier is consistently below 200 ppb$_v$ for each of the fills.

Table 3. Moisture determinations after a MegaBIP® HCl cylinder has been filled three times.

No. of Cylinder Fills	ppb$_v$ H_2O (CRDS)
Fill 1	167
Fill 2	187
Fill 3	163

Comparison between a full cylinder and a heel could indicate whether or not the internal purifier of the MegaBIP® HCl cylinder is adding to total the impurity level of the product. Table 4 shows a comparison of trace metals and atmospherics from the same cylinder when it was full and with a few pounds of product remaining. Atmospheric contaminants (CO_2, CO, CH_4, N_2, O_2+Ar) are analyzed with a Gow-Mac Series 590 gas chromatograph and trace metals are analyzed with an Agilent Model 4500 and Model 7500 ICP-MS. The overall level of impurities appear to be lower in the heel of the cylinder, indicating that the MegaBIP® HCl internal purifier is not adding impurities to product. Moreover, if impurities, such as trace metals, concentrate in the heel of a cylinder, the design of the MegaBIP® purifier is performing adequately for the removal of these impurities.

Table 4. Trace metal and atmospheric impurities in the same MegaBIP® HCl cylinder when full and with product heel. All values are in ppb (ppb_w for metals, ppb_v for atmospherics)

Impurity (ppb)	Full Cylinder	Heel Cylinder	Impurity (ppb)	Full Cylinder	Heel Cylinder
CO_2	< 500	830	**Al**	28	10
CO	< 20	< 20	**Ca**	26	25
CH_4	< 20	< 20	**Cr**	< 20	< 20
N_2	< 30	< 30	**Mn**	< 5	< 5
O_2/Ar	< 20	< 20	**Ni**	< 5	< 5
Other Metals	< 270	< 110	**Cu**	< 5	< 5
Fe	< 10	< 10	**Zn**	29	15
Mg	< 5	< 5	**Cd**	< 1	< 1

Silicon Wafer Processing Tests

In order to better understand how the low moisture levels of MegaBIP® HCl can better serve the semiconductor industry, Lawrence Semiconductor Research Laboratories, Inc. (LSRL, Tempe, AZ), an Epitaxial services company, tested APCI's MegaBIP® HCl in several silicon wafer processes. These processes included high temperature wafer etching, low temperature selective epitaxial growth (SEG), and low temperature Silicon-Germanium alloy (Si:Ge) processing. These processes are particularly sensitive to oxygen contamination, which primarily comes from water, and to a lesser extent, from carbon dioxide in HCl. The wafers were examined visually for haze, using TXRF for metallic contamination, and using SIMS for oxygen contamination.

Both APCI MegaBIP® and VLSI grade of HCl were tested at Lawrence Semiconductor Research Laboratories, Inc. The primary difference between the two products is the moisture specification (1 ppm_v for VLSI, 200 ppb_v for MegaBIP® HCl). A new pigtail was installed on the gas cabinet purge panel to ensure that no pigtail degradation would affect the experiments. However, the remaining components of the delivery system could not be replaced or inspected. All in-line purifiers were also isolated or removed before commencing this study.

Visual Inspection of the Wafers

The wafers processed using MegaBIP® HCl did not show signs of hazing that is typically indicative of high oxygen contamination—without a point of use purifier. This hazing effect is often seen when a VLSI or Electronic grade cylinder is used in conjunction with an in-line purifier that is almost exhausted or when moisture or other contaminants begin desorbing from the media in the purifier.

High Temperature Wafer Etch

Wafers were baked at 1150°C in hydrogen and then exposed to 750 sccm of HCl to remove a controlled amount of silicon. These wafers were analyzed for metal contamination using Total Reflection X-ray Fluorescence (TXRF) at Charles Evans & Associates (Evans Analytical Group, Sunnyvale, CA).

Two control wafers (baseline), one VLSI grade HCl wafer, and two MegaBIP® HCl wafers (full cylinder, heel cylinder) were created for metallic contamination testing. Table 5 presents the result of TXRF on those wafers. While it is true that the MegaBIP® HCl analyses demonstrated nickel and iron concentrations above the detection limit of TXRF, the concentrations have been shown to be within the existing process capability. Historical data was gathered for the epitaxial growth process on several tools and evaluation of this data is most indicative of a mixed data set that followed a Weibull distribution. Analysis of the distribution of the process capability was well above the LOD of the TXRF, and the two MegaBIP® HCl analyses fall within this capability. Interestingly, this is also indicative of a random occurrence in the process. This might be expected, for instance, if the system is shedding particles intermittently. Moreover, this data is inconsistent with trace metals analysis of the MegaBIP® HCl product in full and heel cylinders (Table 4). Thus, the TXRF analysis for metals contamination was inconclusive for iron and nickel. Looking at the data in Table 5, there were no statistical differences between VLSI and MegaBIP® for the other species measured by TXRF.

Table 5. TXRF data for wafers produced during high temperature processing with VLSI and MegaBIP® HCl.

TXRF Results for Control Wafers†

Position	S	Cl	K	Ca	Ti	Cr	Mn	Fe	Ni	Cu	Zn
Center	530±40	125±11	<30	<5	<1.4	<0.8	<0.6	<0.5	<0.4	<0.4	<0.5
30,-30	580±40	124±11	<45	<5	<1.5	<0.7	<0.6	<0.5	<0.4	<0.4	<0.6
-30,30	230±20	90±10	<30	<5	<1.5	<0.7	<0.6	<0.5	<0.4	<0.4	<0.6

Position	S	Cl	K	Ca	Ti	Cr	Mn	Fe	Ni	Cu	Zn
Center	390±30	64±8	<25	<5	<1.4	<0.7	<0.6	<0.5	<0.4	<0.4	<0.5
30,-30	650±40	117±11	<50	<5	<1.5	<0.8	<0.7	<0.5	<0.4	<0.4	<0.6
-30,30	430±30	90±9	<30	<5	<1.4	<0.7	<0.6	<0.5	<0.4	<0.4	<0.6

† units of 10^{10} atoms/cm^2

TXRF Results for High-Temperature Processing with VLSI HCl†

Position	S	Cl	K	Ca	Ti	Cr	Mn	Fe	Ni	Cu	Zn
Center	600±40	490±30	<40	<5	<1.5	<0.7	<0.6	<0.5	<0.6	<0.4	<0.6
30,-30	810±50	2500±150	<50	<5	<2.2	<0.8	<0.6	<0.5*	<0.4	<0.4	<0.5
-30,30	390±30	780±50	<35	<5	<1.4	<0.6	<0.6	<0.5	<0.4	<0.4	<0.5

† units of 10^{10} atoms/cm^2

*may be present near detection limit

TXRF Results for High-Temperature Processing with MegaBIP® HCl, Full Cylinder†

Position	S	Cl	K	Ca	Ti	Cr	Mn	Fe	Ni	Cu	Zn
Center	770±50	670±40	<45	<5	<1.5	<0.8	<0.6	<0.5	1.8±0.3	<0.4	<0.5
30,-30	1130±70	4000±200	<45	<5	<3	<0.8	<0.6	<0.5	<0.4	<0.4	<0.7
-30,30	540±40	420±30	<35	<5	<1.4	<0.7	<0.6	<0.5	<0.4	<0.4	<0.5

† units of 10^{10} atoms/cm^2

TXRF Results for High-Temperature Processing with MegaBIP® HCl, Heel of Cylinder†

Position	S	Cl	K	Ca	Ti	Cr	Mn	Fe	Ni	Cu	Zn
Center	700±50	850±50	<30	<5	<1.4	<0.7	<0.6	<0.5	0.8±0.2	<0.4	<0.5
30,-30	1180±70	4700±300	<50	<5	<4	<0.8	<0.7	<0.5	<0.4	<0.4	<0.6
-30,30	720±50	5900±400	<35	<5	<1.6	<0.7	<0.6	0.7±0.3	<0.4	<0.4	<0.8

† units of 10^{10} atoms/cm^2

Low Temperature SEG and Si:Ge Process

Two different low temperature studies were performed to see the effect of oxygen incorporation into the wafer for the MegaBIP® and VLSI grade of HCl. A low temperature selective epitaxial growth process used a small flow of HCl as part of the deposition gases, while the low temperature Silicon-Germanium alloy process had a chamber clean sequence using HCl. These wafers were analyzed for oxygen contamination using Secondary Ion Mass Spectrometry (SIMS). The germane for the Si:Ge process had a moisture specification of < 1 ppmv.

The low temperature SEG and Si:Ge processes were chosen since the sensitivity to oxygen would be greater, given the absence of baseline experiments. Instead of a baseline, the reactor process temperature was adjusted to yield detectable quantities of oxygen in the layer with a lesser quality grade HCl. In principle, the oxygen level would decrease to a non-detect with higher purity HCl.

As shown in Figure 8 (SEG) and Figure 9 (Si:Ge), the atomic oxygen concentration profiles acquired using SIMS confirms the absence of oxygen in the layers. The SIMS results show a slight preference toward the MegaBIP® HCl product; however, it is difficult to discern the actual difference of these runs from those using VLSI HCl because the SIMS values are near the detection limit. It is interesting to note the interfacial spike in Figure 9 for the VLSI product. Features such as these have been documented in the past on systems containing point-of-use purifiers for gas purification, although the cause of this spike may also be related to surface preparation.

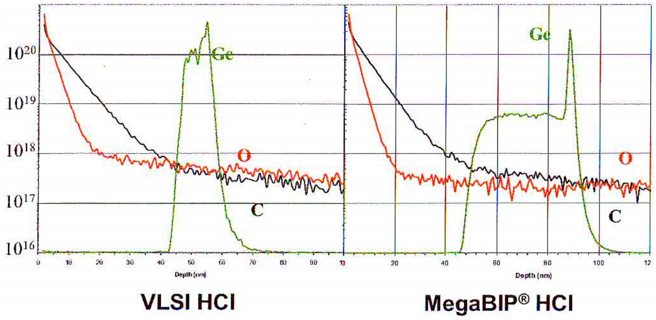

Figure 8. SIMS analysis of low temperature SEG for VLSI and MegaBIP® HCl.

In future efforts, a baseline design could be implemented whereby the process temperature is adjusted to achieve measurable oxygen concentrations using VLSI product. The process temperature should be held constant at this value to demonstrate the MegaBIP® HCl capability. This process could also be used to determine if the limiting factor in SEG or Si:Ge processing is moisture in not only the HCl product but also the germane product. If the latter is true, efforts could be directed to reduce the germane moisture concentration to below 1 ppmv.

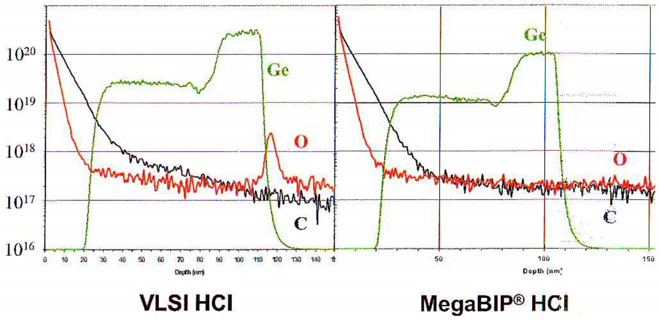

Figure 9. . SIMS analysis of low temperature Si:Ge epitaxy for VLSI and MegaBIP® HCl.

Conclusions

Although advances in conventional technology have enabled significantly improved purity levels in electronics specialty gases to be achieved on a consistent basis, a new cylinder technology based on built-in purification offers the prospect of further driving the state-of-the-art in achieving improved purity and consistency in these gases.

Several key features of the Air Products and Chemicals, Inc. MegaBIP® HCl product were tested and verified as being of value to semiconductor industry, most notably the consistency of the 200 ppb_v moisture specification as determined using CRDS. Additionally, testing of the inter-cylinder and intra-cylinder variations also demonstrated consistency with this low moisture specification, particularly when using cylinders to a liquid dry state. Finally, silicon wafer processing experiments conducted in conjunction with Lawrence Semiconductor Research Laboratories, Inc., concluded that the MegaBIP® HCl product can be used successfully without an external point-of-use purifier to yield lower impurity contamination in a process.

As a commercialized technology, Air Products and Chemicals, Inc., MegaBIP® HCl benefits the end user in the semiconductor industry by improving efficiency, lowering costs, and extending delivery system lifetimes. Use of MegaBIP® HCl enables maximum use of cylinder contents, decreasing the number of cylinder change-outs and extending product utilization. Since Air Products manages both the product and purifier, purifier maintenance issues are essentially eliminated for the end user. Moreover, the end user has capital and maintenance savings since use of MegaBIP® HCl reduces metal particles generated by corrosion, eliminates in-line purifier costs, and reduces gas cabinet component replacement (i.e. regulators and pigtail) costs. A final benefit of the use of MegaBIP® HCl can be improved quality and yields of epitaxial films for the end user

Acknowledgments

MegaBIP® is a registered US trademark of Air Products and Chemicals, Inc.

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