APPLICATION NOTE

LD15-02

Measurement of part per billion Ar and N_2 in oxygen for semiconductor industry

▲ MultiDetek-2

The oxidation of silicon is a common and frequent step in the manufacture of integrated circuits (IC).The semiconductor industry requires the production of Ultra High Purity oxygen for the wafer production.

LDETEK SOLUTION:

The oxygen pipeline purity that goes to the semiconductor industry must be properly measured to ensure that there is no contaminant in it. It is critical and challenging to measure the ppb content of argon and nitrogen impurities in oxygen. The conventional technique used for such application is with a heated Oxy-Trap system combined with HID or conventional PED. Such technique requires a complex chromatography system with periodic Oxy-Trap regeneration with hydrogen. The operations of such system require a lot of maintenance and specialist interventions on a routine basis. The limitation of the lifetime of the trap reduces the continuous operation. Depending on the system condition, the Oxy-Trap has to be regenerated more or less once a week.

Our solution consists of eliminating all the consumables and ensuring continuous operation without maintenance and specialist intervention. Moreover, LDetek can also offers an all in one solution using the MultiDeteks-2 combined with the PlasmaDetek-E for monitoring the CO, CO_2 and some hydrocarbons which are critical impurities to measure for semiconductor gases.

* Patent pending

▲ PlasmaDetek-E

Measuring Ar in oxygen :

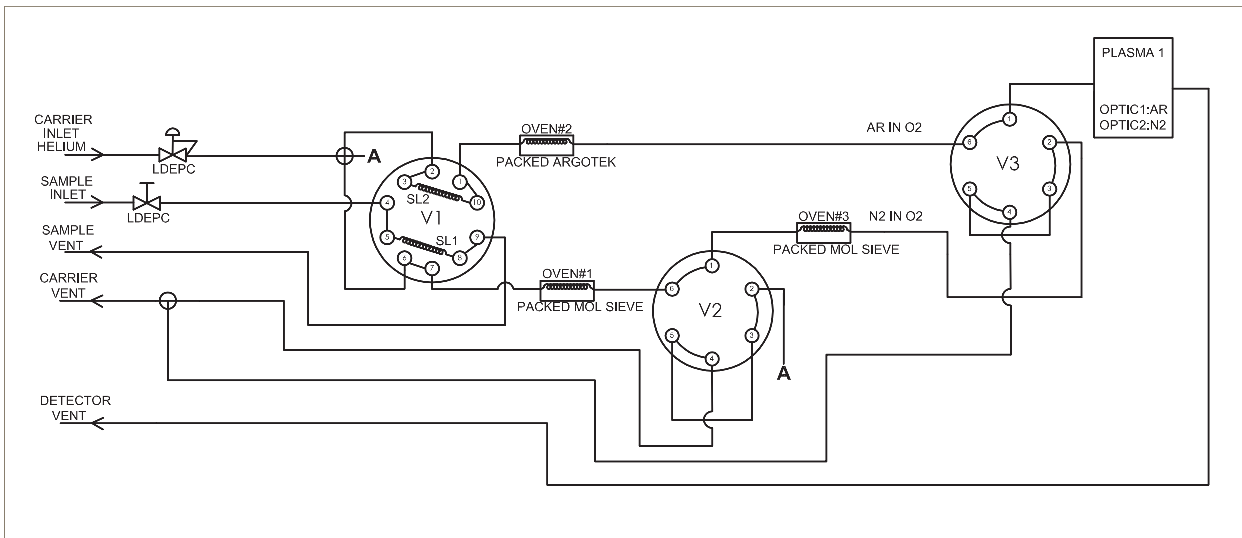
A first channel using a straight 10 port double injection diaphragm valve V1 combined with the LDetek unique ArgoTek packed column to separate part per billion argon from pure oxygen at an isothermal temperature of 45° Celsius with a carrier flow rate of 20-30sccm. The helium is used as carrier gas source. There is no need of cryogenic or Oxy-Trap system as commonly used. As simple as a basic injection through our ArgoTek packed column to conduct the accurate analysis of argon in oxygen.

Measuring N_2 in oxygen :

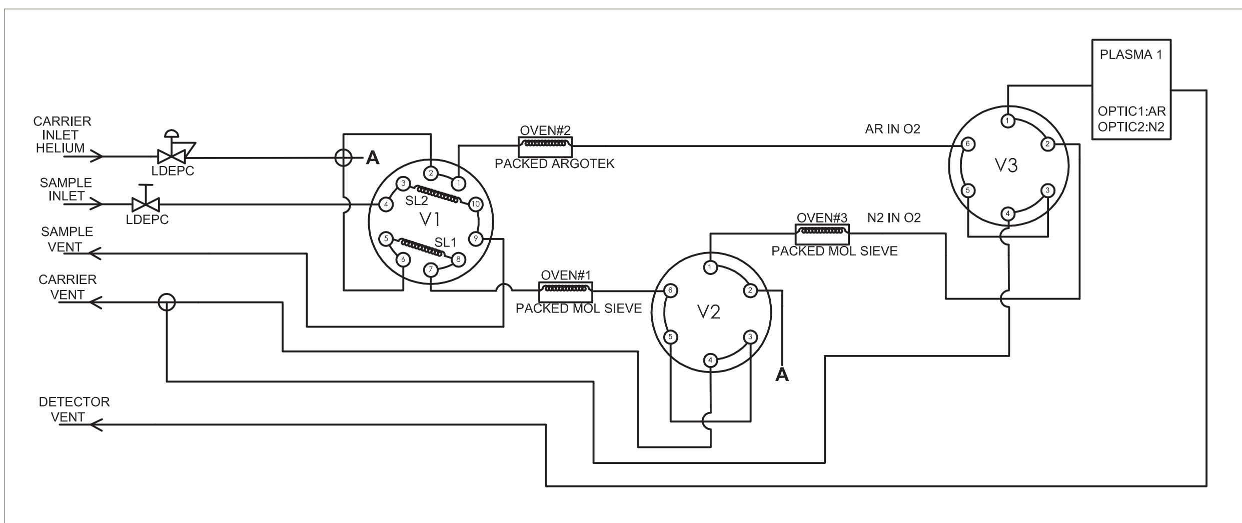
A second channel still using the same straight diaphragm injection valve V1 is used to perform the analysis of N_2 in oxygen with two packed molecular sieve columns combined with a Heartcut diaphragm valve V2. One more time, this configuration allows the measurement of N_2 in oxygen without the use of a complex Oxy-Trap system. Both channels are selected accordingly at the right time with the selection diaphragm valve V3.

PlasmaDetek-E configuration for measuring Ar & N_2 in oxygen :

The PlasmaDetek-E used for this system has a selective configuration to block the interference coming from the oxygen matrix. A specific optic circuit having its wavelength for measuring selectively the argon is mounted in the PlasmaDetek-E. Another specific optic circuit also having its wavelength for selectively measuring the N_2 is in place. The appropriate optic circuit is automatically selected at the proper elution time to allow peak integration. The selectivity of the PlasmaDetek-2 for the Ar-N_2 in oxygen gives the ability to have an easy to use system allowing quick analysis time even at ultra low concentration.



MultiDetek-2 configuration diagram #1 » **V1 position OFF: Filling loop #1 and injecting loop #2**
V2 position OFF: Catch N_2 peak from the first Packed Mol Sieve in Oven #1 to the second Packed Mol Sieve in Oven #2
V3 position OFF: Select channel #2 for Ar in O_2 to the PED



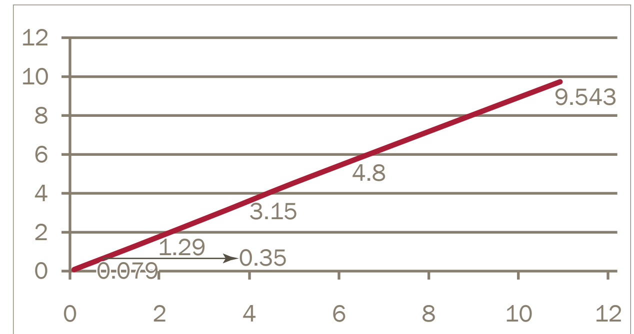
MultiDetek-2 configuration diagram #2 » **V1 position ON: Injecting loop #1 and filling loop #2**
V2 position ON: Venting oxygen out of the system
V3 position ON: Select channel #1 for N_2 in O_2 to the PED

RESULTS:

Linearity on a 0-10ppm range

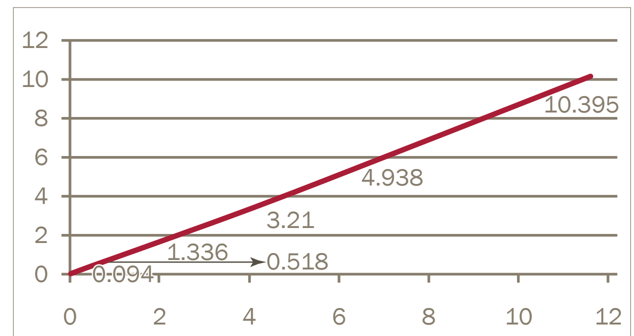
Argon impurity

DILUTED GAS (PPM)	CALCULATED VALUE (PPM)
0.081	0.079
0.35	0.31
1.3	1.29
3.13	3.15
4.73	4.8
9.6	9.543

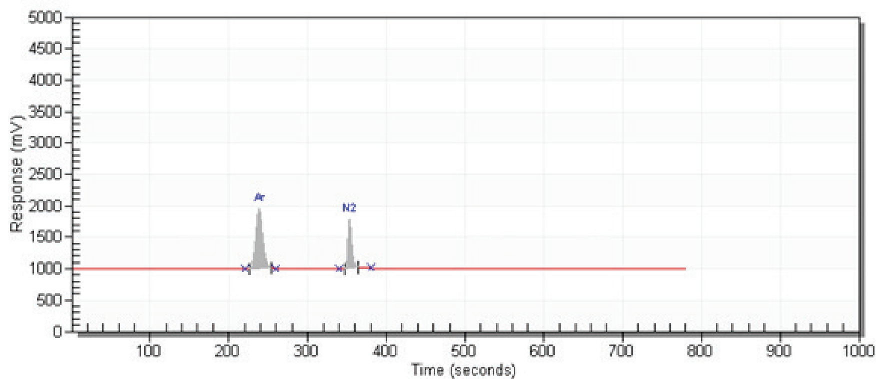


Nitrogen impurity

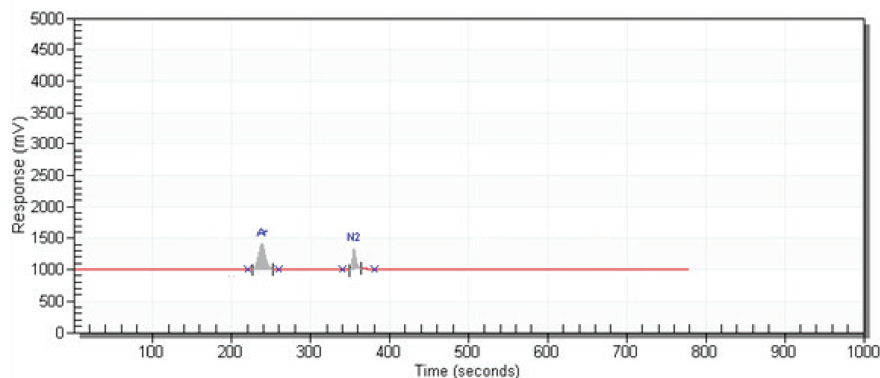
DILUTED GAS (PPM)	CALCULATED VALUE (PPM)
0.097	0.094
0.53	0.518
1.4	1.336
3.36	3.21
5.07	4.938
10.3	10.395



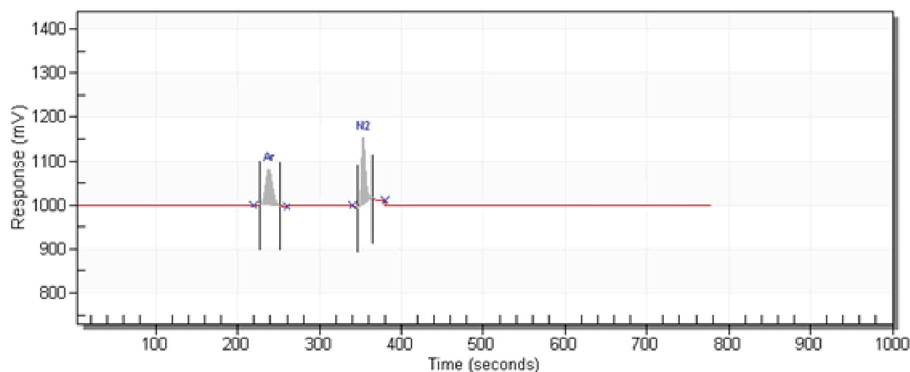
LOW PPB CHROMATOGRAM EXAMPLES:



Chromatogram of 61ppb Ar & 77ppb N_2 in pure oxygen



Chromatogram of 30ppb Ar & 38ppb N_2 in pure oxygen



Chromatogram of 3ppb Ar & 3.8ppb N_2 in pure oxygen

LDL calculation

COMPONENT	CONCENTRATION	PEAK HEIGHT	NOISE	LDL (3X NOISE)
Ar	3ppb	95 mV	1.5 mV	0.1ppb
N_2	3.8ppb	156 mV	2.7 mV	0.2ppb

Note: other LDL could be obtained with different injection volume and chromatographic condition.

CONCLUSION:

With its user friendly interface and the simple configuration of this compact MultiDetek-2 GC, it is easy to use the instrument as a process control analyser that is a perfect fit for this semiconductor application. The enhanced sensitivity and selectivity of the PlasmaDetek-E allows extreme low limit of detection. Combined with the LDetek exclusive ArgoTek column, it makes this system perfectly suitable for semiconductor industry.

Where innovation leads to success

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