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In-line NDIR Gas Monitor for Precursor and Impurity Monitoring HORIBA's Gas Monitoring Solution for Semiconductor Process



Toru Shimizu

*Application Engineering Dept.
HORIBA Taiwan, Inc.
toru.shimizu@horiba.com*

Precursor delivery by bubbling method faces technical challenges on the production stability of ALD/CVD. An in-line NDIR monitor was designed to monitor a commercially available precursor and one of its impurities. The behaviors of the precursor and the impurity including the effect of idle time were observed for two different canisters. This article shows that monitoring precursors with in-line NDIR monitors may help to improve production yields by ensuring the precursor status between canisters and before starting the process.

Introduction

In recent semiconductor processes, more and more variety of precursors are used for ALD/CVD processes to deposit various thin films. Most of these precursors are liquid or solid materials. The bubbling method, which is the combination of a canister and carrier gas, is widely used to deliver these liquid/solid precursors.

However, from the aspect of delivery stability, the bubbling method is not necessarily an appropriate method of vaporization. There are several factors that affect the vapor delivery stability, like temperature of precursor, operating pressure, carrier gas flow rate, amount of residual precursor in canister, etc. Furthermore, impurities or fragments of precursor contained in the canister may affect to film quality or growth rate of film.

NDIR (Non-Dispersive Infrared) is one of the techniques to continuously measure various gas concentration, including precursor vapor, in real time. In this article, several behaviors of a precursor and its impurity measured by HORIBA in-line NDIR gas monitor are reported.

Experiment

The experimental setup is shown in Fig. 1. We prepared a commercially available precursor adopted in the semiconductor manufacturing process as a target precursor. Ar is used as a carrier gas and its flow rate is controlled by a mass flow controller (MFC). The canister is under the ambient condition with no means of temperature control. The NDIR gas monitor, shown in Fig. 2., is designed by HORIBA to detect this precursor vapor and one of its impurities.

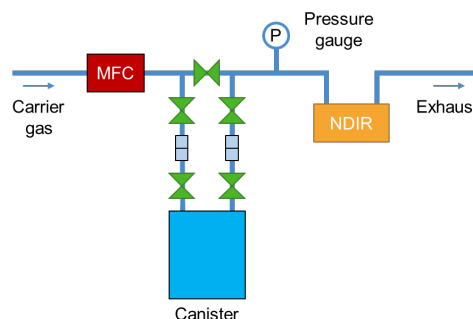


Figure 1 Experimental setup

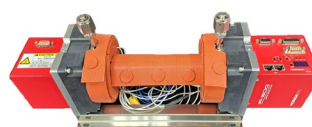


Figure 2 NDIR gas monitor

Result and discussion

Fig. 3 shows NDIR signals observed with the carrier gas of Ar 300 SCCM. The vertical axis indicates a signal level of NDIR output, which corresponds to the partial pressure of the measured gases (precursor vapor and its impurity).

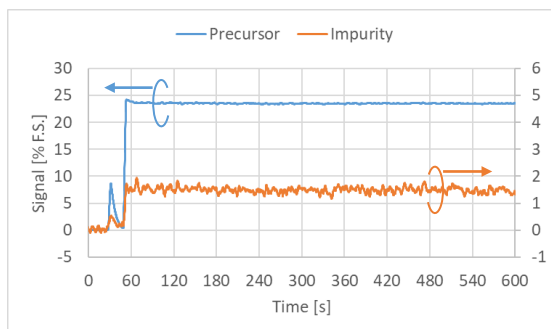
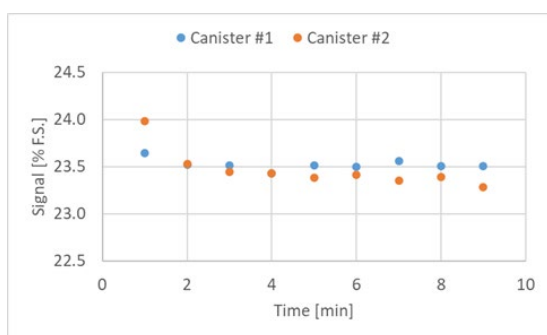
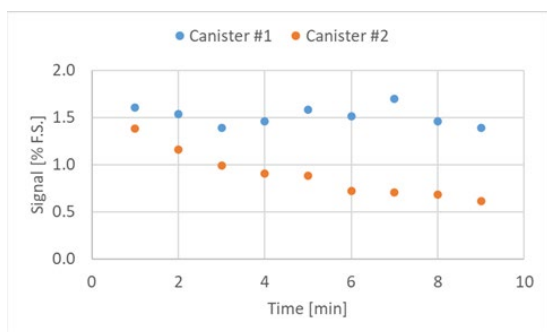


Figure 3 NDIR signal with Ar 300 SCCM carrier flow

We performed the data acquisition with two different canister which contains the same precursor under the same operating condition. Fig. 4(a) shows a time trend of the precursor for about 10 minutes. Fig 4(b) shows a time trend for the impurity.



(a) Signal of precursor vapor



(b) Signal of impurity

Figure 4 Comparison result of two canisters

The two canisters showed almost the same trend for the precursor signal. A slight decreasing trend was observed for the first 3 minutes. It is well known that evaporation causes localized heat loss because of energy needed for evaporation. The local temperature of the liquid interface might be lowered and not reach equilibrium at the beginning of the test after long idle, like the first 3 minutes in Fig. 4(a).

The impurity signal showed different behaviors for the canister #1 and #2. During the test period of 10 minutes, the impurity signal of the canister #1 maintained a relatively higher level while the canister #2 decreased with time. This behavior can be explained as follows: The head space of the canister #2 contained a relatively higher concentration of the impurity before the bubbling process. The carrier gas, which flows into the canister, purged the head space and the gas containing a high level of impurity was replaced by new-coming carrier gas gradually. On the other hand, the precursor liquid of the canister #1 may contain more impurity than the canister #2. As a result, the impurity signal maintained a high level even after the gas replacement of 10 minutes.

The Fig. 5 shows another data of the impurity of the canister #2. This data was acquired after a longer idle period of 2 months. In this idle period, the canister was just stored at normal room temperature (around 22 deg. C) without any gas flow.

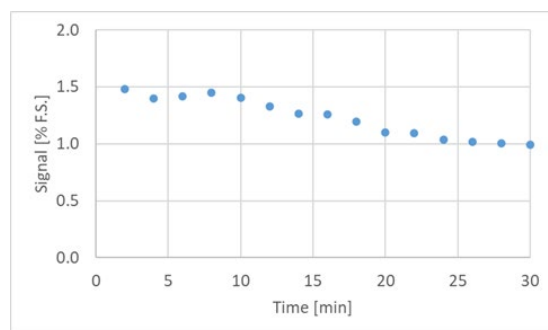


Figure 5 Impurity signal after idle of 2 months

The signal of impurity has returned to the higher level of 1.5% F.S. at the beginning of the flow. It gradually decreased to 1.0% F.S. during this 30-minute test. Compared to the result of Fig 4(b), it took a much longer time to reduce the impurity and cannot reach the lower impurity level as seen in Fig 4(b). Therefore, the storage period, although under the common temperature condition, may affect the quality, especially the impurity amount, for the precursor.

Conclusion

This article presented the data of several behaviors of a commercially available precursor, that is for advanced semiconductor processes, and its impurity situation detected by HORIBA in-line NDIR gas monitor.

According to the miniaturization of semiconductor process and request of device performance in term of power consumption and overall device computing performance, the device design and process window is becoming more and more challenging. Therefore, new materials and new precursors are used to achieve these challenging tasks. This new precursor handling also has many challenges, such as stable vapor delivery and minimization of impurity, and optimization of process condition which impact the film quality and final device performance.

NDIR gas monitors are useful for continuous monitoring of precursor vapor concentration before starting the process and during the process. It also allows to confirm that concentration of impurity is low enough to start the process. HORIBA in-line NDIR gas monitors are one of the tools that enable reliable precursor delivery and improve yields in semiconductor device production.

Related product

Vapor Concentration Monitor IR-300 is designed for monitoring vapor concentration of precursors especially used in MOCVD processes. The MOCVD process is widely used in the manufacture of LEDs, optical devices and other components. IR-300 can be used for other ALD/CVD processes which use liquid or sold precursor delivery with bubbling method.

The IR-300 Series measures and reports the precursor concentration in real time giving the user the various benefits like improvement device yield, preventing wafer scrap, and efficient use of precursor.

[Vapor Concentration Monitor IR-300 - HORIBA](#)



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Date : December 11 - 13, 2024

Venue : Tokyo Big Sight

e-Readout Editorial Office:

Research & Development Division, HORIBA, Ltd.

2, Miyahigashi-cho, Kisshoin, Minami-ku, Kyoto 601-8510, Japan

E-mail : readout@horiba.co.jp

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