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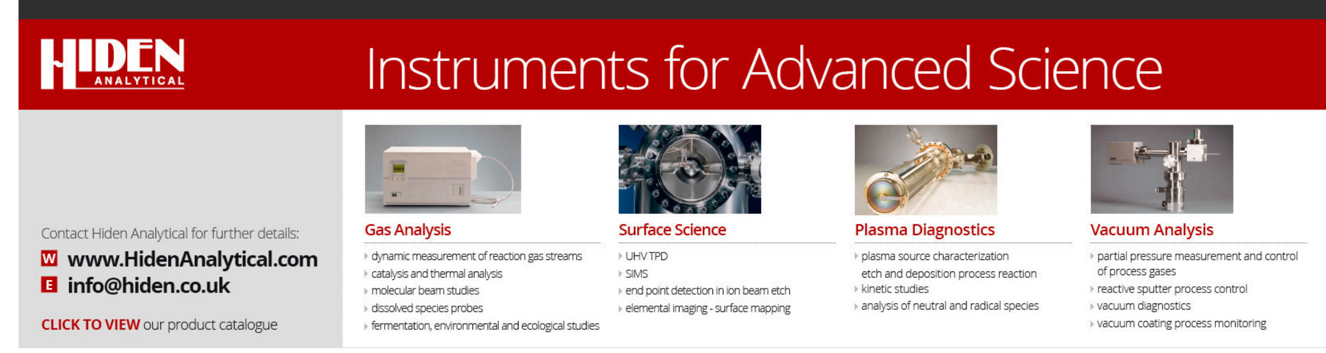

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Real time quantitative diagnostic technique for measuring chemical vapor deposition precursors

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This study proposes an accurate method of monitoring precursor consumption in chemical vapor deposition (CVD) systems. Since precursor costs are significant, finding an efficient method to monitor precursor consumption is necessary. One example is the use of noncontact and inexpensive ultrasonic sensors for determining the liquid level in a container. In this study, sensors based on ultrasonic techniques have been developed for monitoring the precursor consumption in a CVD system. Moreover, the prototype sensors developed in this study can be useful in the field of semiconductors. © *2005 American Vacuum Society.* [DOI: 10.1116/1.1913681]

I. INTRODUCTION

With semiconductors continuously being reduced to nano dimensions, the issues regarding the high aspect ratio and the efficiency of the chemical vapor deposition (CVD) process are also becoming increasingly important.[1–3] In particular, research and development have been extended to the metal organic CVD (MOCVD) process using organic metal compounds as precursors.[4,5] One of the critical factors in this process is ensuring a stable supply of precursors into the chamber. The efficient replenishment of precursors in the reservoir at the appropriate moment is important. There are no proper methods, however, to accurately diagnose or determine the exact time for replenishment. While buoyancy sensors are generally used, the sensors to be inserted into the precursors cause some problems, such as corrosion or cleaning difficulties, and very complicated construction, which may cause problems, such as gas leakage. Meanwhile, if the process is implemented without proper monitoring of the depletion of precursors in the semiconductor line, the thin films will not properly form on the wafer, and a number of particles may be generated. Such problems seriously affect the quality and amount of the yield. On the other hand, changing the precursor while a substantial amount is still present may entail significant economic losses. Therefore, the accurate measurement of the precursor residue in the reservoir and its efficient replenishment at the appropriate time will be very useful in the CVD process. For an accurate measurement of the precursor residue in a CVD system, this study introduces an ultrasonic noncontact sensor system, which can be safely used even with the toxic CVD process. Moreover, the system is less expensive compared to other noncontact methods, such as electrical, magnetic, and optical systems.[6,7] For the application of this ultrasonic monitoring device in a CVD system, the transducer is first installed at the bottom of the reservoir to generate ultrasonic waves. Next, the pulse is reflected against the liquid–gas interface, and returns to the transducer at the bottom. At this point, the amount of time it takes for the pulse to return to the transducer is easily translated as data on the position or level of the liquid.[8]

This test examines the efficiency of the proposed ultrasonic system for monitoring the amount of liquid precursor consumption in the CVD system. As a result, it is concluded that the application of an ultrasonic sensor system in semiconductor fabrication will have substantial potential for the improvement of the processes involved, and may also spell economic gains for the industry.

II. EXPERIMENT

Figure 1 illustrates the equipment designed to measure the liquid level in the precursor. The combined transmitting–receiving sensor (diameter=6.35 mm, Technisonic Co.) is installed at the bottom of the reservoir. The pulse (5 MHz) is emitted through the transmitter, and returns after being reflected from the surface of the liquid. Then, the receiver detects the pulse, and the waveform can be observed through the oscilloscope. The interval between the transmission and reception of the ultrasonic pulse is used to determine the liquid level of the CVD precursor. The liquid level (d) in the reservoir is described below:

$$d = r\left(\left[\tfrac{1}{2}\right]t\right), \tag{1}$$

where v is the ultrasonic pulse rate and t is the round-trip time of the pulse. For calculating the liquid level (d), the ultrasonic pulse rate (which is a value unique to each type of precursor) in the precursor used is first acquired. Next, the round-trip time (t) in each condition is calculated, and the value d is then acquired.[9,10]

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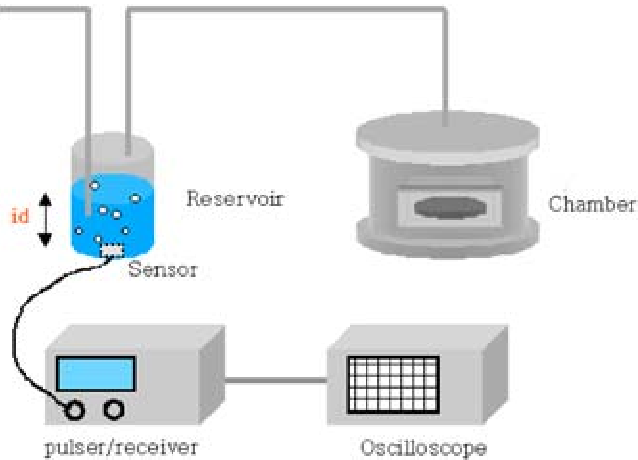

FIG. 1. Precursor consumption measuring equipment setup using ultrasonic pulse.

The precursor used in this test is tetrakis-ethoxy-silane (TEOS), which is the most commonly used precursor in the CVD SiO_2 process.[11]

III. RESULTS AND DISCUSSION

To determine the liquid level (d) of the precursor, it is necessary to calculate the ultrasonic pulse rate for each type of precursor. Then, the difference of the distance (d) from the liquid's surface is fixed to get the ultrasonic pulse rate in a liquid material. The ultrasonic pulse is then transmitted twice. The ultrasonic pulse rate (r) is then calculated using the interval of round-trip time (t) in accordance with formula (1).[9] When the ultrasonic pulse rate (r) of the TEOS was calculated, as described above, the liquid level (d) was fixed, with a gap of 2 cm. Next, the ultrasonic pulse was transmitted twice, and each round-trip time (t) was then measured. For the liquid level with the 2 cm difference, the round-trip time (t) gap of the ultrasonic pulse was 39 μs. In accordance with formula (1), the ultrasonic pulse rate $(r)=1024$ m/s (Fig. 2). Figure 3 describes the test that calculated the liquid level (d) using the round-trip time (t) after the ultrasonic

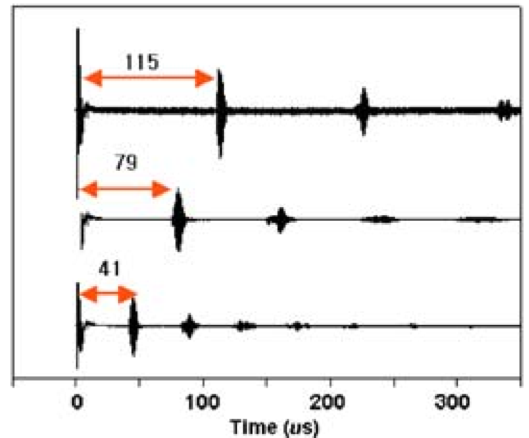

FIG. 3. Liquid level measurement of TEOS using ultrasonic pulse.

pulses were transmitted through different liquid levels (2, 4, and 6 cm). The round-trip times (t) were 41, 79, and 115 s for liquid levels 2, 4, and 6 cm, respectively. When these values were substituted in formula (1) along with the rate (1024 m/s) obtained above, the liquid levels were 2.1, 3.8, and 5.9 cm, respectively. The results are nearly the same in the actual liquid levels and allowance limits. Figure 4 compares the actual values and measured values with the results above. It is found that the measured values in the TEOS are almost equivalent to the actual values. The results, therefore, verify that the ultrasonic sensor system can be used as the monitoring device for the TEOS, the precursor for semiconductors. The possibility that the system can also be used with MOCVD will be further examined through tests using several organic metal compounds. Moreover, for field applications in semiconductor fabrication plants, we designed and manufactured the measuring equipment which is weighing only a few kilograms, portable and easy to use (Fig. 5). The pulser–receiver function as well as the microprocessor (200 MHz) were installed in the equipment in such a way that the distance (t) between the measured waveforms (the round trip time) can be automatically calculated to the liquid level (d), which can then be monitored in real time. The waveforms

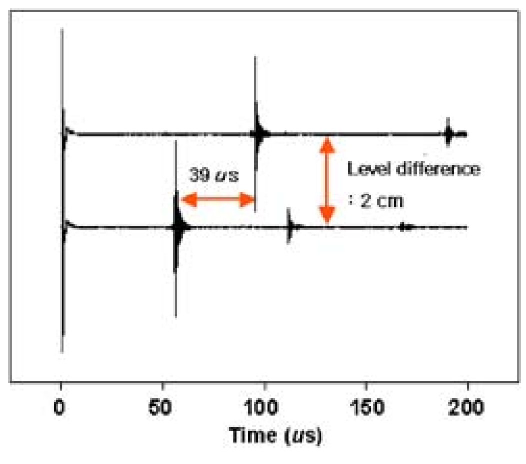

FIG. 2. Measurement of velocity of ultrasonic pulse in TEOS.

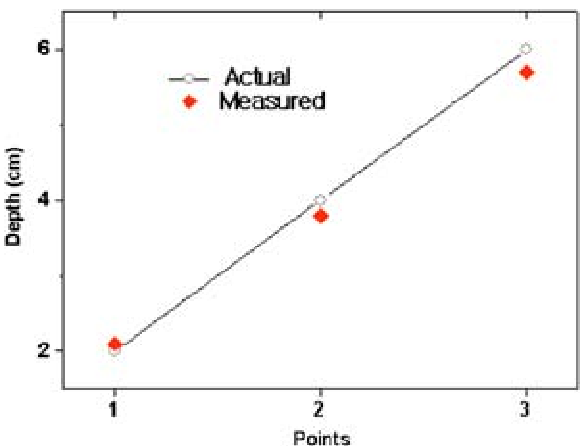

FIG. 4. Actual liquid level vs measured liquid level.

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FIG. 5. Manufactured equipment for measuring precursor consumption for field application.

and numerical data (liquid level) are simultaneously displayed on the screen. Furthermore, the data on the ultrasonic pulse rate for each type of precursor can be preprogrammed. With such features, this equipment is designed to be used in a variety of CVD processes. This precursor consumption monitoring method and equipment can prove to be very useful in actual semiconductor fields.

IV. CONCLUSION

In this study, an equipment to measure the residual amount of precursors in a container, and a measuring method for the CVD process, is proposed. More specifically, this study deals with a method to measure the residual amount of precursor for the accurate measurement of the liquid level of remaining precursors in a container by analyzing the signals generated when the ultrasonic pulses are transmitted and received during the CVD process. Thus, by measuring the liquid level of the precursor in real time, the ultrasonic sensor system can prevent damages caused by unmonitored depletion of precursors. Accordingly, this system can significantly reduce possible defects in products. Since accurate time for replenishment of the precursor can now be determined, the system can significantly extend the life cycle of the precursor.

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