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Improvement of flow and pressure controls in diffusion-tube humidity generator: Performance evaluation of trace-moisture generation using cavity ring-down spectroscopy

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Abstract

A system of flow and pressure controls for a magnetic suspension balance/diffusion-tube humidity generator (MSB/DTG) has been developed to realize a humidity standard in the trace-moisture region. The system essentially consists of a pressure regulator combined with a piezo valve, and adopts a simple stepwise change in the total flow rate. It has been demonstrated that the uncertainty of evaporation rates is greatly reduced by applying buoyancy correction to mass data measured with the MSB. Using the system and buoyancy-corrected data, the negative effect on the MSB/DTG observed upon changing the total flow rate become negligible. The relative standard uncertainty of total flow rates is evaluated using mass flow meters composed of critical flow Venturi nozzles to be less than or equal to 0.22%. The uncertainty of humidity generation has been significantly reduced by improvements introduced in this work. A comparison between humidity generated with the DTG and humidity measured with a moisture analyzer (MA) based on cavity ring-down spectroscopy is presented in the amount-of-substance fraction range between 10 nmol/mol and 250 nmol/mol. The results of the comparison confirm that the DTG is capable of producing a trace-moisture gas down to approximately 10 nmol/mol, and of easily varying and controlling the amount-of-substance fraction of water using the system. The sources of bias observed in the MA reading are attributable mainly to systematic errors in the temperature of the absorption cell and absorption cross section used to calculate the water concentration with the MA.

Keywords: Humidity standard; Diffusion tube; Magnetic suspension balance; Trace moisture; Sensor calibration; Critical flow Venturi nozzle

1. Introduction

The measurement of trace impurities in process gases has become increasingly important in the past decade in semiconductor industries, because it has been recognized that impurities play a critical role in the yield and product quality of semiconductor devices. Water is a common impurity, and the level of control currently required is considered to be better than 1 μmol/mol in amount-of-substance fraction [1]. Various sensors have been developed to detect such a small amount of water vapor [2], and many of them are commercially available and commonly used in the fields of science and industry. However, the accurate measurement of trace moisture is not straightforward. A major reason behind this is the lack of suitable humidity standards for the calibration of sensors; the periodic calibration of sensors is needed to achieve reliable measurement.

In our previous study [3], it was demonstrated that a diffusion-tube humidity generator (DTG) is a suitable and reliable humidity standard in the trace-moisture region. The DTG generates humidity by mixing water vapor evaporated from a diffusion cell [3] with dry gas. Therefore, to establish a primary humidity standard with this method, it is necessary to measure and control the evaporation rate of the water and the flow rate of the gas. In the previous study, we developed a system for the real-time mass measurement of the evaporated water using a magnetic suspension balance (MSB) combined with the DTG. Furthermore, using a moisture analyzer (MA) based on cavity ring-down spectroscopy [4–6], it was shown that the stable generation of water evaporation is realized by precisely controlling temperature and pressure.

In this work, we focus on the measurement and control of flow rate. The water concentration of the humid gas generated

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with the DTG can be varied by adjusting the total flow rate. This was performed using a mass flow controller (MFC) by adjusting the flow rate of the gas passing through the bypass line of the MSB/DTG. When the total flow rate is changed, we must not disturb the flow to the generation chamber of the MSB/DTG because of the need to maintain the weighing stability and sensitivity of the MSB. We must also maintain the pressure inside the chamber because of the need to realize a stable and constant evaporation rate. This pressure change also produces a change in the buoyancy acting on the diffusion cell, which would be a serious problem in real-time mass measurement with the MSB. In fact, disturbances in the flow and pressure were observed upon changing the total flow rate, and they brought about the problems mentioned above. To solve these problems, we have developed a system of flow and pressure controls using a pressure regulator combined with a piezo valve, and by adopting a stepwise change in the total flow rate.

The measurement of the total flow rate is a fundamental issue for determining the water concentration of humid gas generated with the DTG. In the present study, the rate was measured using mass flow meters (MFMs) composed of critical flow Venturi nozzles, which are often used as a transfer standard among national metrology institutes [7]. The uncertainty of the total flow rate has also been evaluated with the MFMs.

A comparison between humidity determined with the evaporation and flow rates and humidity measured with the MA is presented. The sources of the bias observed in the MA reading are discussed.

2. Experimental

2.1. Experimental setup

Fig. 1 shows a schematic of the experimental setup used in the present study. Two thermal mass flow controllers (Stec, SEC-F440M), referred to as MFC_1 and MFC_2, were used to control the flow of dry nitrogen (N_2) gas. The full scales of MFC_1 and MFC_2 were 20 L/min and 5 L/min, respectively; the flow rates used in this paper correspond to those measured under the standard conditions of 101.325 kPa and 0 °C. The total flow rate of the system was controlled with MFC_1 in the range between 1 L/min and 20 L/min. A portion of the flow, at a rate of 0.10 L/min, was introduced to the inlet of a generation chamber using MFC_2, and the rest of the flow was bypassed. The flow to the bypass line was choked at the inlet of the line using an orifice to maintain sufficient pressure upstream of MFC_2 so that MFC_2 performed properly. A diffusion cell was suspended inside the chamber. The water vapor from the diffusion cell was diluted with the dry N_2 gas coming from the inlet. Humid gas obtained from the outlet of the chamber was mixed again with the bypassed flow. A mixing device (Noritake, Static mixer) was inserted after the connection point to achieve homogeneous mixing. This mixed flow was divided into two flows as below. One flow was introduced to a pressure regulator (PR) to control the pressure inside the chamber. The other flow was introduced to an MA based on cavity ring-down spectroscopy (Tiger Optics, MTO-1000) to monitor the amount-of-substance fraction of water in the trace-moisture gas generated. The flow to the MA was controlled with the built-in MFC of the MA. The MA probes the peak intensity of an absorption line of H_2O using a near-infrared diode laser. The deviation of the laser frequency from the peak is an uncertainty of the measurement. We confirmed that the uncertainty due to this effect in this work was negligible. The two flows were combined again after passing through the PR and MA. The flow rate of this combined flow was measured using one of two MFMs (Hirai, MR series), denoted MFM_1 and MFM_2, respectively. MFM_1 was used for flow rates greater than or equal to 10 L/min, and MFM_2 for those less than 10 L/min. The MFM consisted mainly of a temperature sensor, two pressure sensors, and a critical flow Venturi nozzle [8–10]. The Venturi nozzle produced a constant flow rate at its throat. Therefore, with a known cross-sectional area of the throat, the flow rate was calculated using the temperature and pressure of the gas measured upstream of the nozzle. The effect of leaks in the pipework on the measurement of flow rate was checked in advance and found to be negligible.

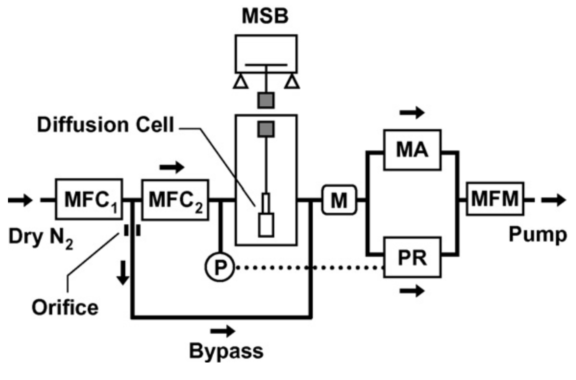

Fig. 1. Schematic of the experimental setup used in this work. MFC denotes the thermal mass flow controller, P is the pressure gauge, MSB is the magnetic suspension balance, M is the mixing device, PR is the pressure regulator, MA is the moisture analyzer based on cavity ring-down spectroscopy, and MFM is the mass flow meter composed of a critical flow Venturi nozzle.

Evaporation rates were measured as the mass-change rates of the diffusion cell using the MSB. The mass data were collected every 1 min. The zero-point correction and calibration of the MSB were performed every 10 min and every 30 min, respectively. The details of the diffusion cell and the MSB were described in our previous paper [3]. The temperature of the chamber was maintained at 25 °C by monitoring the temperature with a platinum resistance thermometer (PRT). Atmospheric pressure was measured with a digital manometer (Yokogawa, MT210). Temperature and relative humidity near the MSB were measured with a PRT and a humidity sensor (Sato Keiryoki, SK-L200TH), respectively. The data of pressure, temperature, humidity, and flow rate were collected every 1 min using a personal computer.

2.2. Flow and pressure controls

A piezo actuator valve (Horiba Stec, PV-2000) combined with a valve controller (Horiba Stec, PCU-2100) was used to control the pressure inside the chamber at a constant value by