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Development of humidity standard in trace-moisture region: Characteristics of humidity generation of diffusion tube humidity generator

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Abstract

The characteristics of humidity generation of a diffusion tube humidity generator (DTG) have been studied by the real-time mass measurement of evaporating water using a magnetic suspension balance and by humidity measurement using a moisture analyzer based on cavity ring-down spectroscopy in the amount-of-substance fraction range of water between 20 nmol/mol and 600 nmol/mol. The observed mass-change rates of a diffusion cell due to water evaporation agree well with the evaporation rates calculated on the basis of Fick's law of diffusion. The stable generation of trace-moisture in nitrogen gas is realized by precisely controlling temperature and pressure. The relative standard uncertainty of the stability of trace-moisture generation of the DTG is 0.006 (0.6%). The effect of temperature on the stability of humidity generation of the DTG is smaller than that of a frost-point generator.

Keywords: Humidity standard; Diffusion tube; Trace-moisture; Sensor calibration

1. Introduction

The sensing of trace-moisture in gases, with an amount-of-substance fraction less than 1 μmol/mol, has become increasingly important in the recent decade in semiconductor industries, because it has been recognized that even such a small amount of water vapor plays a critical role in the yield and product quality of semiconductor devices [1]. Various sensors have been developed to measure the water concentration in the trace-moisture region [2]. They are commonly used not only in industrial fields but also in scientific fields. 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 the sensors; the periodical calibration of sensors is indispensable in achieving reliable measurement. Humidity generators utilizing the saturated vapor of ice maintained at a constant temperature, such as frost-point generators, have the capability of generating humidity with an accurate and stable value. In fact, they have been selected as national humidity standards in many countries because of their reliability. However, these generators normally are expensive and require considerable skills and knowledge on their proper operation, particularly in the trace-moisture region.

Diffusion tube humidity generators (DTGs) offer a relatively simple and useful method of realizing a humidity standard in the trace-moisture region. The principle of this method is based on molecular diffusion. Hence, the evaporation rate of a diffusion tube can easily be estimated using the diffusion theory (Fick's law of diffusion). Moreover, this theory indicates that the method should be capable of generating trace-moisture with a stable value by controlling precisely the temperature and pressure in the system. However, its practical applicability to trace-moisture generation has not yet been quantitatively examined in detail.

In this work, we have experimentally studied the characteristics of humidity generation of a DTG being developed at the National Institute of Advanced Industrial Science and Technology (AIST). The temperature and pressure dependences of evaporation rates of diffusion tubes have been investigated by the real-time mass measurement of evaporating water using a magnetic suspension balance (MSB) and by humidity measurement using a moisture analyzer (MA) based on cavity ring-down spec-

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troscopy. A comparison between the evaporation rates observed and those calculated on the basis of the diffusion theory is presented. The stability of trace-moisture generation of the DTG is reported. The effect of temperature on the stability of humidity generation is discussed in comparison with that of a frost-point generator.

2. Experimental

Fig. 1 shows a schematic diagram of the experimental setup used in the present study. The flow of dry nitrogen (N_2) gas, dried by a purifier (Saes Getters, Monotorr PS4-MT3-N-1), was controlled with a thermal mass flow controller (Stec, SEC-F440M) at a flow rate of 2.00 L/min. Flow rates used in this paper correspond to those measured under the standard conditions of 101.325 kPa and 0 °C. A portion of the flow with a rate of 0.50 L/min was introduced to the inlet of a generation chamber made of stainless steel, and the rest of the flow was bypassed. The generation chamber was attached to the bottom of the MSB (Rubotherm) with a common vacuum flange to form a closed system with the MSB. The chamber was cylindrical, and its length and outer diameter were approximately 180 mm and 30 mm, respectively. A diffusion cell, described in detail below, 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. Stainless-steel tubes with an outer diameter of 6.35 mm (0.25″) were used for the pipework. The inner surfaces of the tubes and chamber were electrochemically polished (Fujikin, UP treatment). The temperature of the chamber was maintained at 25 °C or 55 °C with a temperature controller (Cell System, TDC-1130) by monitoring the temperature with a platinum resistance thermometer (PRT) mounted on the chamber. Using another PRT, we measured the temperature difference between the PRT mounted and the inside of the chamber in advance. The difference was considered in estimating the temperature of water in the diffusion cell suspended in the chamber. The absolute pressure inside the chamber was controlled with a pressure regulator (Bellofram, TYPE70BP) at 150–450 kPa, and was measured with a pressure gauge (Yokogawa, FP-101A). The temperature and pressure were recorded every 1 min using a personal computer. The mean values of the standard deviations of the temperature and pressure in this work were 0.02 K and 0.36 kPa, respectively. The MSB consisted of a magnetic suspension coupler (MSC) and an analytical balance (Mettler Toledo, AT21) with a weighing readability of 1 μg. The measuring load in the MSC was magnetically suspended without contact with the analytical balance using a permanent magnet and an electromagnet. The voltage on the electromagnet was controlled to maintain the measuring load at a constant vertical position using position sensors. The measuring load had a hook at its bottom for hanging the diffusion tube. The surface inside the MSC was covered with electropolished stainless steel. The MSB was capable of load decoupling, allowing the balance to perform zero-point correction and calibration with internal reference weights any time. In this work, the zero-point correction and calibration were performed every 10 min and every 2 h, respectively. Evaporation rates were measured as the mass-change rates of the diffusion cell using the MSB. The control of the MSB and the acquisition of the mass data were carried out using the computer mentioned above with a software program provided by the manufacturer. The mass data were collected every 1 min. The amount-of-substance fraction of water in the trace-moisture gas generated by the system was monitored with a commercially available MA based on cavity ring-down spectroscopy (Tiger Optics, MTO-1000). Using the MA, we also estimated the amount-of-substance fraction of residual moisture in the dry gas in this work to be below 1 nmol/mol.

The diffusion cell consisted of a diffusion tube (Valco Instruments) and a small vessel, as shown in Fig. 2. They were connected by ferrule-type fittings, and could be disconnected for water filling. The diffusion tube and vessel were made of

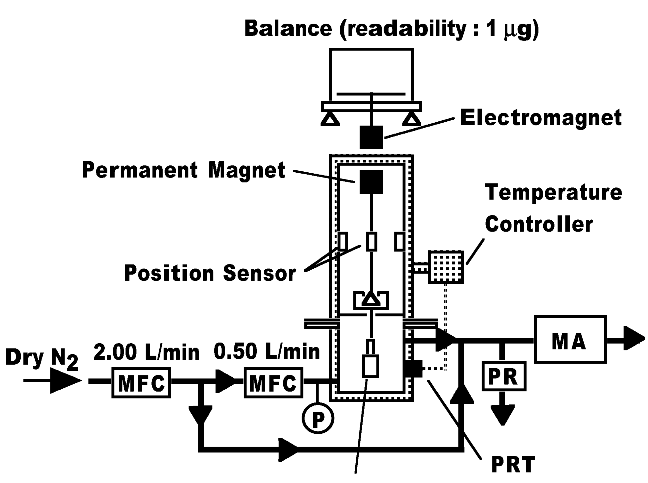

Fig. 1. Schematic diagram of the experimental setup used in this work. MFC stands for the mass flow controller, P is the pressure gauge, PR is the pressure regulator, PRT is the platinum resistance thermometer, and MA is the moisture analyzer based on cavity ring-down spectroscopy.

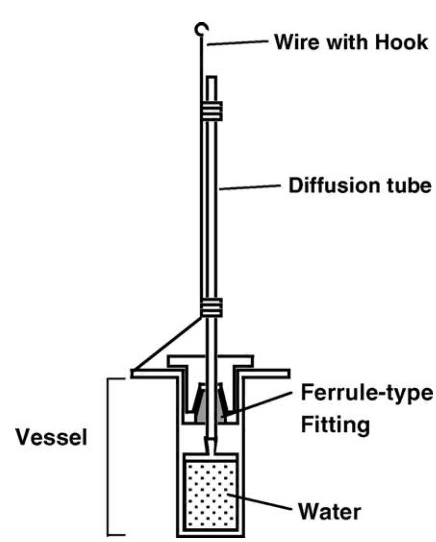

Fig. 2. Schematic diagram of the diffusion cell.