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# Moisture Adsorption Desorption Characteristics of Stainless Steel Tubing Measured by Ball Surface Acoustic Wave Trace Moisture Analyzer

# 球状弾性表面波微量水分計により計測されたステンレス配管の水分吸脱着特性

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## 1. Introduction

In the production of semiconductor devices, the management of trace moisture (<1ppmv) in highly pure gases is important[1], where the adsorption desorption (AD) of water from tubing cannot be ignored. Inner surface treatment of stainless steel tube was significantly improved by ‘ultra clean technology’[2,3], proposed by Ohmi et. al. AD characteristics of basic parts such as straight and coil tubes was evaluated by air pressure plasma ion mass spectroscopy (APIMS)[1], that can measure ppbv moisture quantitatively, and used for quality assuarance of commercial production.

However, although many manifold tubing systems were developed using such parts, almost all was not inspected because of expensive immovable APIMS and on-site built-in manifold systems. Recently, cavity ringdown sepctroscopy (CRDS)[1], with the sensitivity comparable to APIMS and mobility, becomes commercially available, but the application to low flowrate (typically less than 1L/min) system was difficult.

In this situation, we found the ball surface acoustic wave (SAW) sensor coated by sol-gel silica film with similar sensitivity and ten times fast response compared to CRDS under 0.1L/min [4], developed burst waveform under sampling circuit for practical use of two-frequency-measurement [5,6], and fabricated a prototype of portable trace moisture analyzer (TMA) [7]. In this study, we aim to show the applicability of ball SAW TMA to measuring AD characteristics.

## 2. Principle

Schematic diagram of moisture adsorption by stainless steel tube is shown in **Fig.1**. First, the tube is completely dried using dry $N_2$ gas (<1ppbv)[Fig.1(a)]. Next, wet $N_2$ gas with standard concentration (e.g. 1ppmv) is injected. All water molecules are adsorbed on inner surface at early stage with dry gas outflowed [Fig.1(b)]. As the adsorption approaches the saturation, intact wet gas begins to be outflowed. The adsorption characteristic is evaluated by the time lag from the injection to the state shown by Fig.1(c). The desorption characteristic is evaluated by the decay curve of water concentration after switching the state from Fig.1(c) to Fig.1(a).

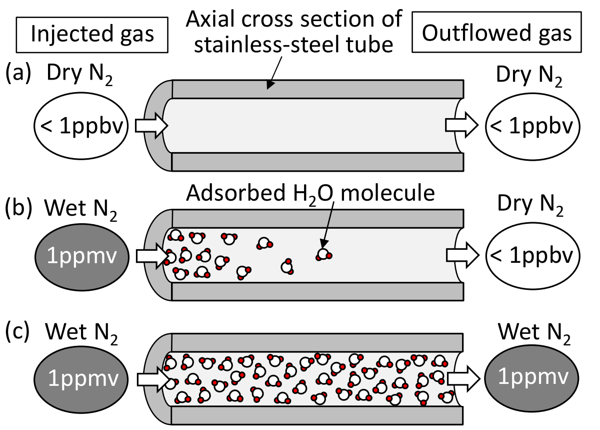


Fig. 1 Moisture adsorption by stainless steel tube. (a) Completely dry tube. (b) Early stage of wet gas injection into (a), outflowing dry gas. (c) Final stage outflowing intact wet gas.

## 3. Measurement

Schematic diagram of the measurement system is shown in **Fig.2**. Dry gas, purified by molecular sieve filter, or wet gas, generated by diffusion tube method [8], was instantly changed using rotary valve [Fig.2(a)], where the flowrate was 0.1L/min. The sample tubes were fabricated by commercially available SUS316L tube with 1/4 in. o.d. and 100mm length welded by VCR grounds (28mm), leading to line velocity of 360mm/s. Inner surface treatments were bright annealing (BA), electro polishing (EP), and electro chemical buffing (ECB)[**Fig.3**], where the specifications of maximum roughness $R_y$ were less than 3 μm, 0.7μm, and 0.1μm, respectively. In addition, the sample only VCR ground was prepared to subtract the lag by the injection tube (1/16 in. o.d., 500mm, BA). The waveforms of sol-gel SiOx coated quartz harmonic device were recorded using BUS circuit at every 3s with 1000 times averaging, and was analyzed by developed method[6], and plotted relative delay time change as the sensor output.

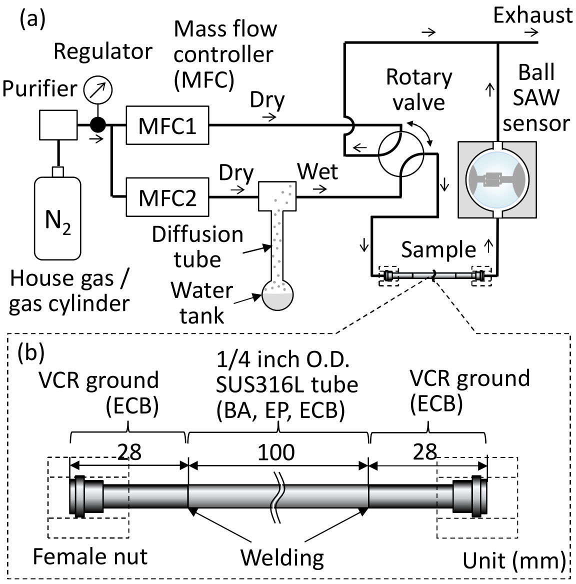

Fig.2 Measurement system. (a) Block diagram. (b) Schematics of tube sample.

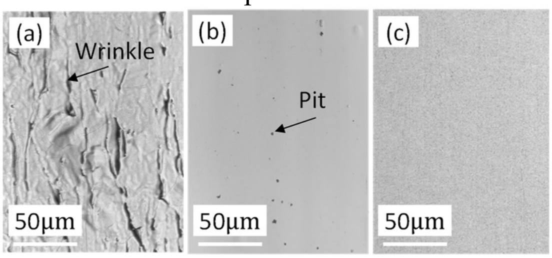

Fig.3 Observation of inner surface by laser microscope showing reflection intensity. (a)BA, (b)EP, and (c) ECB.

## 4. Result

Variation of sensor output by surface treatment due to switching dry gas to wet gas is shown in **Fig.4**, where negative response represents water concentration increase. $t_d$ is the lag due to the injection tube and $t_s$ is the moisture transit time through the sample. As $R_y$ decreased, $t_s$ decreased. There was significant difference in the adsorption characteristic between EP and ECB treatments. Results of switching wet gas to dry gas are shown in **Fig.5**. Although each sensor started to respond at the same time [Fig.5(a)], the water concentration decayed earlier as $R_y$ decreased. Logarithmic-scale plot is shown in Fig. 5(b), originating the decay curve from switching time. It was clear that the response was proportional to the power of -1/2, suggesting the concentration decrease inversely proportional to time [4].

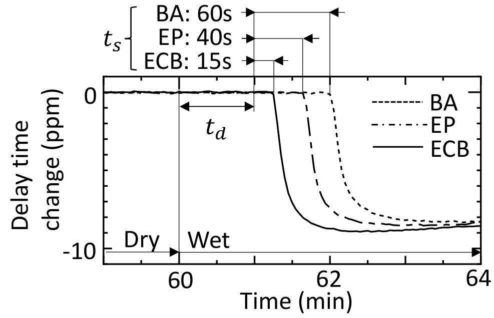

Fig.4 Variation in moisture transit time through 100mm tube by surface treatment.

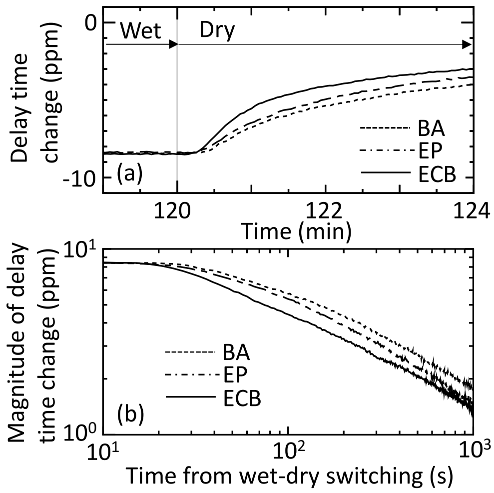

Fig.5 Variation of desorption characteristic by surface treatment. (a) sensor output. (b) logarithmic plot from wet-dry switching.

## 5. Conclusion

The variation of AD characteristics by inner surface treatment of stainless steel tube only with 100mm length was detected using sensitive and fast ball SAW TMA.

**Acknowledgment**

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