Fundamental study of nondestructive testing of inner surface of stainless-steel tubings by ball SAW trace moisture sensor

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1. Introduction

Water molecule is the most difficult substance to desorb from the electro-polished (EP) surface of stainless-steel tube.[1)] The authors have studied inverse gas chromatography (IGC) using a ball surface acoustic wave (SAW) sensor as a detector and trace moisture (TM) as a probe to inspect the condition of highly pure gas tubing system.[2-4)] The shortening of the measurement interval less than 1 s was attained by reducing the accumulation number and the record length of a burst undersampling (BUS) waveform and it was applied for the samples with bending-induced (BI) morphology.[4)] The passing-through (PT) time of the probe was shorter as the bending was severer. However, it was not sure whether initial condition was sufficiently comparable among the experiments only by 10 h drying at room temperature. In this study, the baking prior to the IGC measurement was performed to accelerate water desorption and the affection of welding and BI morphology was studied.

2. Experiment

Figure 1 shows the schematics of the samples. Sample A was EP tube of austenitic stainless steel (SUS 316L, 500mm in length, 6.35 mm in O. D.). Sample B was rolled one radially strained by 4% (0.25 mm). Sample C was 10-times welded one cut by 40 mm and welded in Ar flow.

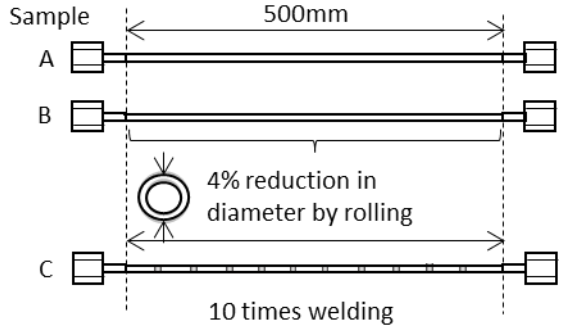

Fig. 1 Samples of the stainless-steel tubing with damaged inner surface of EP treatment. (a) raw [A], (b) radially rolled [B], and (c) welded [C] tube samples.

Figure 2 shows the diagram of typical sequence of thermal desorption and TM injection (24 h), where the flow rate of N_2 was 100 sccm. Four experiments were successively repeated. Firstly, the sample was baked at 143℃ for 6 h (21600 s) by 1.6 ppbv flow (thermal desorption). The sample was cooled down to room temperature (about 24℃) within 1.5 h. Next, the sample was wetted by 40 ppbv for 3 h and was dried by 1.6 ppbv until next day (IGC measurement).

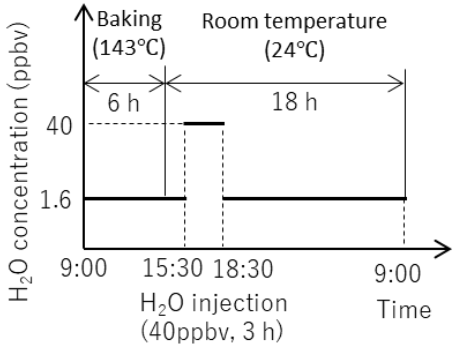

Fig. 2 Diagram of sequence of thermal desorption and TM injection.

The ball SAW sensor was fabricated with quartz ball with 3.3 mm in diameter and coated with sol-gel SiOx film,[5)] where the fundamental frequency of 80 MHz and its harmonic frequencies were generated. In BUS measurement, excitation was the composite burst waveform of 80 MHz and 240 MHz, and the frequency components of 10 MHz and 20 MHz were generated by undersampling at 100 MHz. 30-points waveform (23.5 μs – 23.8 μs) was obtained from the 7th roundtrip signal and the delay time was determined at each frequency by wavelet transform.[4)] The arrival of TM brought negative delay time change since the stiffness of SiOx film was increased. The reference value of the delay time was selected at that just before TM injection.

3. Results

3.1 Thermal desorption at 143℃

Figure 3 shows the results of the thermal desorption, where solid, dashed, chained, and dotted, curves represent the 1st, 2nd, 3rd, and 4th experiments, respectively. The inset shows the magnification of initial steep response. In the 1st experiment of sample A, significant moisture was desorbed by 400 s and

the desorption was gradually decreased. After the 2nd experiment, overall desorption was significantly reduced but each behavior was similar. Although the 1st response in sample A was converged, those in samples B and C were not converged, suggesting the existence of adsorbed H_2O even under 143℃. It is judged that the water desorption was greater in the order of samples A, B, and C from the minimum of the desorption peak. Two local minima were commonly observed at 250 s and 400 s.

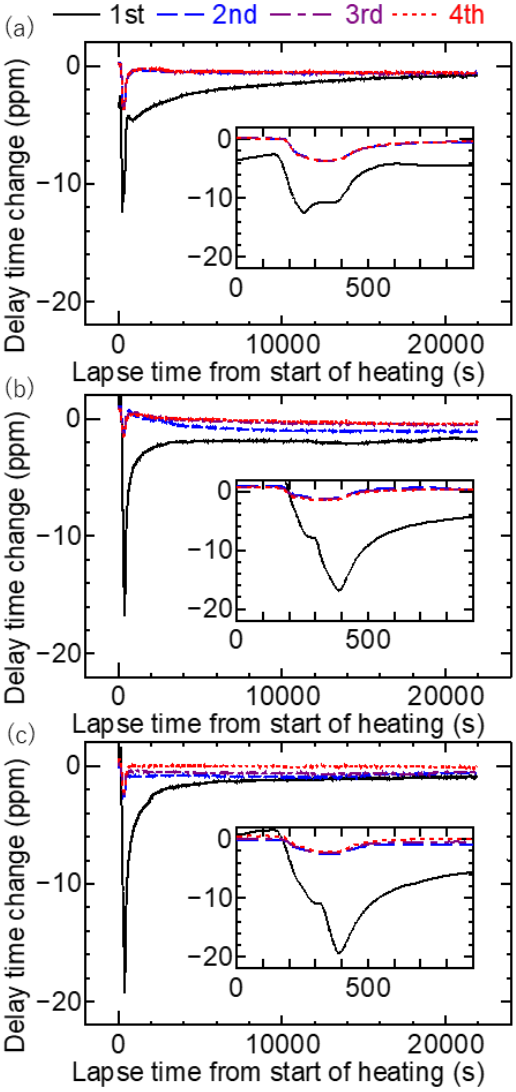

Fig. 3 Thermal desorption at 143℃ with 1.6 ppbv N_2 flow showing samples (a) A, (b) B, and (c) C. Each inset represents the magnification of initial response.

3.2 TM passage (40ppbv)

Figure 4 shows the PT behavior of 40 ppbv probe at room temperature, where solid, dotted, and chained curves represent the results of samples A, B, and C, respectively. PT time in sample B and C became shorter than that in sample A and the difference became clear as the thermal desorption repeated. It may be caused by the decrease of strong adsorption site due to the increase of H_2O molecule density on metal surface, accompanying the increase of weak adsorption site by hydrogen-bonding.[6)]

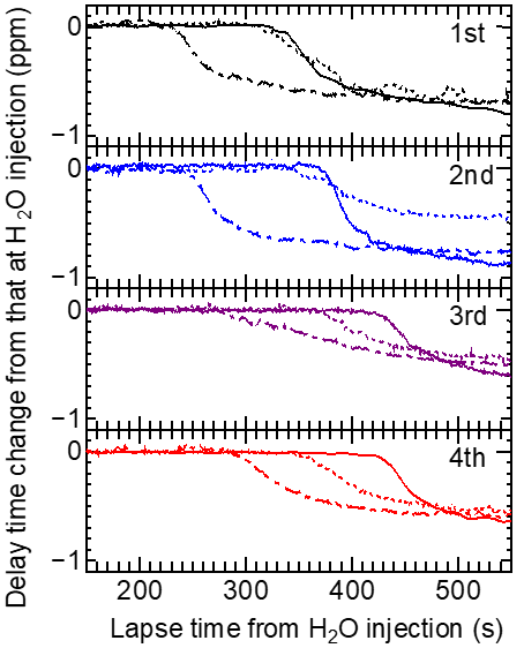

Fig. 4 Variation in TM passage by successive thermal desorption, where solid, dotted, and chained curves represent the results of samples A, B, and C, respectively.

4. Summary

The increase of water adsorption was verified by generating BI and weld morphology in EP surface. PT time of TM probe may be a clue for judging the inner surface condition.

Acknowledgment

This work was supported by KAKENHI 19H02148.

References

1. K. L. Siefering, and W. H. Whitlock, J. Vac. Sci. Technol. A **12**, 2685 (1994).
2. T. Tsuji, S. Akao, T. Oizumi, N. Takeda, Y. Tsukahara, K. Yamanaka: Jpn. J. Appl. Phys. **56**, 07JC03 (2017).
3. T. Tsuji, T. Oizumi, N. Takeda, S. Akao, Y. Tsukahara, K. Yamanaka: Jpn. J. Appl. Phys. **54**, 07HD13 (2015).
4. T. Tsuji, H. Fukushi, T. Oizumi, N. Takeda, T. Iwaya, S. Akao, Y. Tsukahara, K. Yamanaka, Y. Ohara, T. Mihara: Proc. Ultrason. Electron. **42**, 3J2-3 (2021).
5. S. Hagihara, T. Tsuji, T. Oizumi, N. Takeda, S. Akao, T. Ohgi, K. Takayaangi, T. Yangisawa, N. Nakaso, Y. Tsukahara, K. Yamanaka: Jpn. J. Appl. Phys. **53**, 07KD08 (2014).
6. P. A. Thiel and T. E. Madey: Surf. Sci. Rep. **7**, 211 (1987).