Cleaning of Aluminium Alloy Chambers with Ozonized Water

C K Chan¹, G Y Hsiung¹, C C Chang¹, Rouge Chen¹, C Y Yang¹, C L Chen¹
H P Hsueh¹, S N Hsu¹, Ivan Liu², J R Chen¹,²
1. National Synchrotron Radiation Research Center, Hsinchu 30076, Taiwan
2. Department of Biomedical Engineering and Environmental Sciences, National Tsing Hua University, Hsinchu 30013, Taiwan
E-mail: ckchan@nsrrc.org.tw

Abstract. In the fabrication of aluminium vacuum chambers of the Taiwan Photon Source (TPS), a cleaning treatment with ozonized water followed machining in an ethanol environment. After cleaning with ozonized water, aluminium samples were analyzed according to Auger electron spectroscopy (AES) and measurement of the thermal outgassing rate and photon-stimulated desorption (PSD). The results showed that cleaning with ozonized water has a superior performance. A thermal outgassing rate $q_\text{eff} \sim 6.4 \times 10^{-12} \text{Pa} \cdot \text{m/s}$ after baking and a photon-desorption yield $\eta \sim 2 \times 10^{-5}$ molecules/photon at an accumulated beam dose $3 \times 10^{21}$ photons/cm² were obtained.

1. Introduction
In the past Freon was used to clean the aluminum bending chambers after the process of ethanol machining for the Taiwan Light source (TLS), but it is well known that such chlorofluorocarbons deplete the ozone layer in the stratosphere; according to the Montreal Protocol, use of these substances was to be phased out by year 2000. At the National Synchrotron Radiation Research Center (NSRRC), a project to construct a third-generation synchrotron source – Taiwan Photon Source – is developing. It is thus necessary to create an environmentally acceptable method of cleaning the TPS vacuum systems. Momose et al. used ozone to clean ultrahigh vacuum materials; surface analysis with AES and x-ray photoelectron spectroscopy showed that ozone effectively removes carbon from the metal surface and the rear surface layer especially for the materials aluminium and copper [1]. Asano et al. applied ozonized ultrapure water for a final rinse to recover the degraded performance and to suppress the field emission due to exposure to air of the superconducting RF cavities of KEK B-Factory [2]. Both their studies reveal that the cleaning effect on the treated surface can be retained more than four days, which is important to avoid contamination for the vacuum chambers to be installed after cleaning. Here we describe the characteristics of aluminium samples with a treatment of ethanol machining and cleaning with ozonized water, and report the measurement of the thermal outgassing rate of a 2 m-long aluminium chamber after cleaning with ozonized water.

2. Experiments
To prepare ozonized water, ozone produced with a ozonizer was conducted into deionized (DI) water to attain a concentration $\sim 6.7$ ppm, into which the aluminium samples were subsequently immersed at $24 ^\circ\text{C}$ for 30 min. Figure 1 shows the system for the measurement of the thermal outgassing rate, which was divided into three major parts – the pumping chamber, the sample chamber and the orifice.
chamber. A turbomolecular pump (TMP, 400 L/s) was installed beneath the pumping chamber and backed with another TMP (70 L/s) and a dry pump in order to increase the compression ratio of the pumping system. Two extractor ion gauges were arranged to measure the pressure of the orifice chamber and the sample chamber respectively. An orifice built inside the orifice chamber, providing a \( N_2 \)-equivalent conductance \( \sim 0.8 \, \text{L/s at 20}^\circ \text{C} \), was used to measure the outgassing rate of aluminium samples in the sample chamber by the throughput method; the sample chamber itself can alternatively be a specimen. To measure the rate of PSD, we used a dedicated beam line BL-19B at NSRRC [3]; the energy of the electron beam is 1.5 GeV and the critical energy of the synchrotron radiation is 2.14 keV. The synchrotron radiation was collimated to 6 mm high and 50 mm wide on the sample at a distance 7.5 m from the beam source. The design of PSD samples with cooling channels makes the temperature rise < 0.5 \(^\circ\)C during SR exposure to decrease outgassing from the thermal effect. Furthermore, a silicon pipe installed 3 mm before the sample and a bias voltage 100 V applied to the samples during exposure can decrease the PSD contributed from the surrounding chamber wall. The experimental station with the PSD sample was baked at 150 \(^\circ\)C for 24 h before illumination with synchrotron radiation. For the AES analysis, the treated samples were stored in a dry box with a humidity control (RH < 30 %) for 12 h and then sent to the NTHU Instrument Center for analysis.

![3D drawing of the system for the measurement of the thermal outgassing rate](image)

**Figure 1**: 3D drawing of the system for the measurement of the thermal outgassing rate

![Auger spectra for aluminium samples after treatments A (a), B (b) and C (c)](image)

**Figure 2**: Auger spectra for aluminium samples after treatments A (a), B (b) and C (c)

### 3. Results and discussion

The materials and the surface treatments of aluminium samples are listed in table I. To evaluate these treatments we adopted the measurement of the thermal outgassing rate and PSD, and AES analysis, and describe the results in the following sections.

| Materials | Surface treatment |
|-----------|-------------------|
| A         | A6063 Oil machining and chemical cleaning\(^a\) |
| B         | A6063 Ethanol machining and ozonized water cleaning |
| C         | A6063 Ethanol machining only |

\(^a\) Immersion in NaOH (45 g/L) at 45 \(^\circ\)C for 2 min, in a bubble bath in DI water for 10 min, in acidic solution (50 % HNO\(_3\) + 3 % HF) for 2 min, in a bubble bath again in DI water for 10 min, in DI water (< 5 \( \mu \text{S cm}^{-1} \)) for 2 min for ultrasonic rinsing, then drying with pure nitrogen (99.9999 %)
3.1. Auger electron spectroscopy

Auger electron spectra of aluminium samples, according to each treatment listed in table I, are shown in figure 2. Comparison of these spectra shows that carbon signals were detected on samples after treatments A and C, but not after treatment B; the surface carbon on aluminium is hence effectively cleaned with ozonized water.

3.2. Measurement of the thermal outgassing rate

Thirty aluminium alloy A6063 specimens (dimensions 150 mm × 70 mm × 5 mm) were fabricated with treatments A, B and C and placed into the sample chamber to measure the outgassing rate, \( q \). The outgassing rate of the sample chamber \( q_s \) was measured before each experiment; the thermal outgassing rate of the samples \( q_t \) is thus derived on subtracting \( q_s \) from \( q_t \). According to our experimental procedure, the sample chambers with aluminium samples were evacuated at 20 °C for 24 h, baked at 120 °C for 24 h, and then further pumped at 20 °C for 24 h. The \( q_t \) for the various treatments are shown in figure 3. In the first 10 h pumping, the thermal outgassing rate of samples with treatment B, \( q_{10}(B) \approx 7.1 \times 10^{-7} \text{Pa} \cdot \text{m} / \text{s} \), was worse than that with treatment A, \( q_{10}(A) \approx 1.3 \times 10^{-7} \text{Pa} \cdot \text{m} / \text{s} \), and treatment C, \( q_{10}(C) \approx 4.6 \times 10^{-7} \text{Pa} \cdot \text{m} / \text{s} \). However \( q_t(B) \) measured after 24 h from baking out had a superior performance \( q_{72}(B) \approx 6.4 \times 10^{-12} \text{Pa} \cdot \text{m} / \text{s} \), compared to \( q_{72}(A) \approx 1.2 \times 10^{-10} \text{Pa} \cdot \text{m} / \text{s} \) and \( q_{72}(C) \approx 1.8 \times 10^{-11} \text{Pa} \cdot \text{m} / \text{s} \).

![Figure 3](image)

*Figure 3*: Curves of rate of outgassing vs. time for aluminium samples with treatments A (a), B (b) and C (c) during 24 h pumping at 20 °C, 24 h baking at 120 °C and after baking.

3.3. Measurement of the thermal outgassing rate of the 2m-long aluminium chamber

An aluminium alloy A6061 vacuum chamber (length 2 m) was manufactured to test whether ethanol machining combined with cleaning with ozonized water is applicable to a large vacuum system. This chamber as the bending chamber was formed from two pieces of aluminium plate, which were shaped by ethanol machining and then immersed in ozonized water for 30 min. In this work, the concentration of ozonized water attained only 2 ppm, limited by the capacity of the ozonizer. The chamber (total inner surface area 5879 cm²) was assembled using tungsten-inert-gas (TIG) welding. The experimental procedure resembled that for the measurement of the thermal outgassing rate in section 3.2 except that...
the chamber was baked at 150°C. The test results show that \( q_{10} = 5.7 \times 10^{-7} \text{ Pa} \cdot \text{m/s} \), but \( q_{72} \) decreases to \( \sim 4.4 \times 10^{-11} \text{ Pa} \cdot \text{m/s} \) after baking.

3.4. Measurement of photon-stimulated desorption

Figure 4 shows the photon desorption yield as a function of the accumulated beam dose for each treatment. At an early stage (beam dose \( \sim 1 \times 10^{18} \) photons/cm\(^2\)), the photon desorption yield \( \eta_B \) of treatment B was one tenth that of the other treatments. After attaining an accumulated beam dose \( 3 \times 10^{21} \) photons/cm\(^2\), a photon desorption yield \( \eta_B \sim 2 \times 10^{-5} \) molecules/ photon was attained. Figure 5 presents the PSD molecules from aluminium samples after cleaning with ozonized water; the contributions were mainly from H\(_2\), CO, CO\(_2\), H\(_2\)O and CH\(_4\).

**Figure 4**: Photon desorption yield vs. beam dose for aluminium samples according to three treatments.

**Figure 5**: PSD molecules vs. beam dose for aluminium samples after cleaning with ozonized water

4. Summary

According to our investigation of a treatment using ozonized water to clean aluminium materials, the results indicate that ozonized water is effective for cleaning aluminium materials based on experimental data for the measurement of the thermal outgassing rate and PSD, and for AES analysis. An aluminium alloy chamber (length 2 m) was made to test this process; the thermal outgassing rate of this chamber is \( q_{10} \sim 5.7 \times 10^{-7} \text{ Pa} \cdot \text{m/s} \) before baking and \( q_{72} \sim 4.4 \times 10^{-11} \text{ Pa} \cdot \text{m/s} \) after baking.

References

[1] T. Momose, Y. Maeda, K. Asano and H. Ishimaru 1995 *J. Vac. Sci. Technol.* A **13**(3) 515

[2] K. Asano, T. Furuya, S. Mitsunobu, T. Tajima and T. Takahashi 1996 Stable Performance of 508-MHz Superconducting RF Cavities for KEK B-Factory KEK Preprint 95-191

[3] G. Y. Hsiung, K. Y. Young, Y. J. Hsu and J. R. Chen 2001 *J. Vac. Sci. Technol.* A **19**(4) 1657