Titanium Materials for UHV/XHV Systems

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Abstract- **Outgassing properties and surface profiles of titanium materials have been investigated in this paper. Titanium materials showed excellent outgassing rates below 10^{-12} Pams^{-1} after baking process. These values are two orders of magnitude smaller than that for standard vacuum materials under the same pre-baking condition. Outgassing rates of the pure titanium of JIS grade 2, titanium alloy, KS100, are about 1/4 of that for a stainless steel without baking process. Titanium materials are applicable for the vacuum chamber and vacuum components. The vacuum equipment made of the titanium material has excellent vacuum performance, e.g., very quick evacuation and UHV/XHV pressures are easily obtainable.**

I. INTRODUCTION

A reduction in the outgassing rate of vacuum materials is important for the ultrahigh vacuum (UHV) and extremely high vacuum (XHV) systems. Standard vacuum materials, such as stainless steels and aluminum alloys, have outgassing rates of the order from 10^{-9} to 10^{-10} Pams^{-1} under the conventional pre-treatments such as electrolytic or chemical polishing and the baking process with temperature below 500 K. In order to reduce the outgassing rate for standard materials, the special treatments and surface modifications, e.g. vacuum-firing processes with high temperatures, oxidization and thin film coating, are necessary. These pre-treatment, however, seem not to be applicable to vacuum systems for practical use.

Recent years, titanium has been paid attention as a high vacuum material because of lightweight, non-magnetism, a low Yong's modulus, and a small thermal expansion coefficient, and vacuum characteristics of titanium materials have been reported so far. In the reports, the outgassing rate of chemically polished pure titanium (JIS grade 2) is clarified to be less than 1/5 of that for electrolytic polished stainless steel[1,2]. An oxidation treatment of material surface under the Ar/O_2 atmosphere with 200 ^{o}C, was found to be effective to improve outgassing rate for some kinds of titanium materials such as pure titanium (JIS grade 2), Ti-3Al-2.5V and etc.[3,4]. Authors clarified that a certain titanium alloy (KS100 made by KOBE STEEL, LTD) shows a very low outgassing rate of 6×10^{-13} Pams^{-1} after a baking process using a modified orifice method which can accurately measure an outgassing rate with very low detection limit of the order of 10^{-13} Pams^{-1}[5]. In this paper, outgassing properties and surface conditions were discussed for three kinds of titanium materials, pure titanium materials JIS grade 2, grade 4, and titanium alloy, KS100. And a vacuum chamber and components were fabricated utilizing titanium materials, which showed excellent outgassing properties.

II. EXPERIMET

Samples

JIS grade 2, pure titanium material, is composed of 99.5 % of pure Ti, and its hardness is 150 Hv. JIS grade 4 is also composed of 99 % of pure Ti, and its hardness is 250 Hv. KS100, titanium alloy, is composed of 0.35 wt% of oxygen, 0.35 wt% of Fe, and 99.3 wt% of Ti, and its typical hardness is 250 Hv. Unpolished basis metal, mechano-chemically polished and chemically polished titanium materials were prepared as reference.

Surface analysis

The surface morphologies of the samples were measured by atomic force microscopy (AFM), and the surface roughness (Ra) were estimated by AFM image in a 10 μm^2 area. In order to measure the surface oxide layer thickness, depth profiles were analyzed by auger electron spectrometer (AES) with Ar etching gas, and transmitting electron microscope (TEM) images were measured.

Outgassing rate

A modified orifice method, switching between two pumping paths (SPP) as shown in Fig. 1[6], was employed for the measurements of the outgassing rate in nitrogen equivalent. When the valve V_1 is opened and V_2 is closed, named sample test, the pressure, P_{U1}, of upstream chamber is affected by the total outgas from the samples per unit time, Q_S (Pam^{3}s^{-1}), and that from the gauge and the upstream chamber, Q_{U}. On the other hand, when the valve V_1 is closed and V_2 is opened, named blank test, the pressure, P_{U2}, is affected by Q_U.

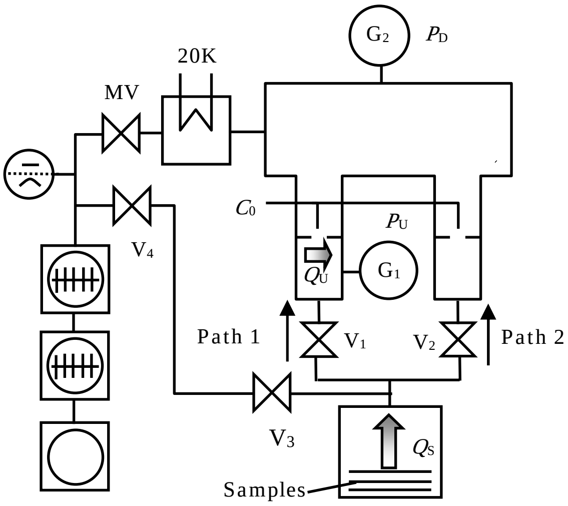

Fig. 1 Schematic diagram of the apparatus employing the SPP method for the measurement of outgassing rate.

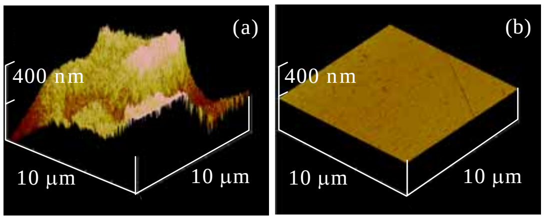

Fig. 2 AFM images basis metal (Ti_{KS}(BS)) (a) and mechano-chemically polished titanium alloy Ti_{KS}(MCP) (b) of KS100.

Table 1 Surface roughness and thickness of surface oxide layer of mechano-chemically polished titanium materials and electrolytic polished stainless steel

	Surface Roughness (Ra)	Thickness of Surface Oxide Layer
Ti_{G2}(MCP)	2.56 nm	11 nm
Ti_{G4}(MCP)	4.18 nm	—
Ti_{KS}(MCP)	0.7 nm	5 nm
SUS(EP)	2.60 nm	3 nm [7]

Since the down stream pressures P_{D1} and P_{D2} for both sample test and blank test are equivalent, the outgas Qs from the samples is expressed by [6]

$$Q_S = C_0 \,(P_{U1} - P_{U2}). \qquad (1)$$

Thus outgassing rate of the sample, $q = Q_S / A$ (Pams^{-1}) can be accurately estimated, because this method eliminates the outgases of gauges and chamber walls. Here, A (m^2) is the surface area of samples.

In the measurement system, the upstream and downstream pressures, P_U and P_D, attained to 10^{-9} Pa and below using a bakeable cryopump and those values were measured by an extractor gauge which can measure the pressure of 10^{-10} Pa range. The orifice conductance C_0 was set to 6.1x10^{-3} m^{3}s^{-1} and the sample area was 8.7×10^{-1} m^2 (sample size is 60 mm$\times$60 mm $\times$1mm^t and the number of sample is 120) Then the detection limit of the outgassing rate is 7×10^{-13} Pams^{-1}. An employed sample chamber was made of titanium alloy with 1.0×10^{-1} m^2 of inner area. The chamber was pre-baked at the temperature of 423 K for 20 hours to initialize chamber condition. And the chamber was baked at 423 K for 20 hours after 0.5 hour of air exposure. And then the outgassing properties were measured. The outgassing properties for non-baked samples were also estimated after pre-baking process.

III. Results and discussions

Surface analysis and Outgassing properties

Figure 2 shows typical AFM images in a 10 µm^2 area of basis metal (Ti_{KS}(BS)) and mechano-chemically polished titanium alloy (Ti_{KS}(MCP)) of KS100. The surface of Ti_{KS}(BS) is very rough, while Ti_{KS}(MCP) has a smooth surface. The values of surface roughness (Ra) of Ti_{KS}(BS) and Ti_{KS}(MCP) are estimated to be 95.1 and 0.7 nm, respectively. Smooth surface was similarly obtained in pure titanium materials both JIS grade 2 and grade 4 by mechano-chemically polishing. Table 1 shows the values of surface roughness (Ra) of mechano-chemically polished pure titanium materials, JIS grade 2 ((Ti_{G2}(MCP)) and JIS grade 4 ((Ti_{G4}(MCP)), and Ti_{KS}(MCP), and that of electrolytic polished stainless steel (SUS(EP)). In the pure titanium materials, the value of Ra of Ti_{G2}(MCP) is equal to that of SUS(EP), while Ra of Ti_{G4}(MCP) is 1.5 times that of SUS(EP). Many micron size halls were actually observed on the surface of Ti_{G4}(MCP). It is feared that this micro halls brings the increase of outgassing rate in Ti_{G4}(MCP). The surface of Ti_{KS}(MCP) is the smoothest in the three kinds of titanium materials. It is considered that this originates the fact that the grain size of Ti_{KS}(MCP) is smaller than those of pure titanium materials.

Composition of the material surface was analyzed by AES depth profile for the titanium material, which showed that the composition of titanium materials changes from TiO_2 to TiO with increasing the depth and finally only titanium exists at the depth deeper than 10 nm from material surface. TiO_2 layer is confirmed to be an amorphous by the TEM observations. Thickness of surface oxide layer of the titanium materials, which are estimated by AES depth profiles and TEM images, are thinner than about 10 nm(see Table 1). The thickness of surface oxide layer for titanium material, however, are thicker than that for SUS(EP). Obtained thickness of surface oxide layer for titanium materials are comparable to that reported by Morimoto *et al*.[4], which shows low outgassing rate.

Figure 3 shows the time dependence of the outgassing rates for the titanium materials and SUS(CP) without baking process. The outgassing characteristic for SUS(CP) is shown to be equal to SUS(EP) [7]. And the outgassing rate of Ti_{G4}(MCP) is almost the same as that of SUS(CP). The Ti_{G2}(MCP) and Ti_{KS}(MCP) showed 1/4 of the outgassing rates comparing to SUS(CP). This indicates that Ti_{G2}(MCP) and Ti_{KS}(MCP) are the most

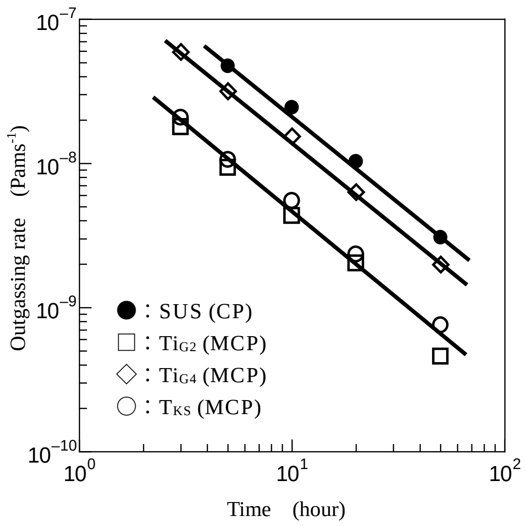

Fig. 3 Time dependence of the outgassing rates without baking of the titanium materials Ti_{G2} (MCP), Ti_{G4}(MCP) and T_{KS} (MCP), and chemically polished stainless steel SUS(CP).

Table 2 Outgassing rates after baking at 423K ×20 hr of the titanium materials Ti_{G2} (MCP), Ti_{G4}(MCP) and T_{KS} (MCP), and chemically polished stainless steel SUS(CP).

	Ti_{G2} (MCP)	Ti_{G4}(MCP)	Ti_{KS} (MCP)	SUS(CP)
Outgassing Rate (Pams^{-1})	7×10^{-13}	2.1×10^{-13}	6×10^{-13}	2.0×10^{-10}

preferable vacuum materials among the three titanium materials discussed in this study.

Table 2 shows the Outgassing rates of the titanium materials and chemically polished stainless steel after the baking process at 423 K for 20 hours. Titanium materials have very low outgassing rates in the range from 10^{-12} to 10^{-13} Pams^{-1}. Outgassing rate of 7×10^{-13} Pams^{-1} for Ti_{G2}(MCP) and 6×10^{-13} Pams^{-1} for T_{KS}(MCP) correspond to a detection limit for measurement condition. Discrepancy for the measured outgassing rates for Ti_{G2}(MCP) and T_{KS}(MCP) is due to sample size. The outgassing rates of titamium materials was found to be two orders magnitude smaller than that of SUS(CP) under the same pre-baking process. This translates that the titanium material is a suitable material for UHV/ XHV apparatus.

Chemical polishing for titanium

The authors have developed a chemical polishing for a titanium material as a low outgassung material[8]. Table 3 shows Ra measured by AFM in a 10 μm^2 and 1 μm^2 area, and the thickness of the surface oxide layer of chemically polished pure titanium of JIS grade 2 (Ti_{G2}(CP)) and Ti_{G2}(MCP) as a reference. Ra for Ti_{G2}(CP) scanned in 10 μm area, is 10 times larger than that for Ti_{G2}(MCP). On the other hand, the Ra for Ti_{G2}(CP) comparable to that for Ti_{G2}(MCP) in the case of small scanning area of 1 μm^2. The surface oxide layer of Ti_{G2}(CP) is thinner than 10 nm. Ti_{G2}(CP) possesses the excellent outgassing properties comparable to Ti_{G2}(MCP) despite non-baking, which corresponds to

Table 3 Surface roughness and thickness of surface oxide layer of chemically polished pure titanium material JIS grade 2 (Ti_{G2}(CP)) and reference sample of Ti_{G2}(MCP)

	Surface Roughness (Ra)	Thickness of Surface Oxide Layer
Ti_{G2}(CP)	25.0 nm (10 μm^2)	7 nm
	1.80 nm (1 μm^2)	
Ti_{G2}(MCP)	2.56 nm (10 μm^2)	11 nm
	1.03 nm (1 μm^2)	

1/4 of outgassing rate for Ti_{G2}(CP). Furthermore, Ti_{G2}(CP) after baking at 423 K for 20 hours showed the outgassing rate as small as 7×10^{-13} Pams^{-1}.

Development of vacuum chamber and components

A vacuum chamber and vacuum components for UHV/XHV systems are fabricated using titanium materials. The mechanical properties of the titanium materials are similar to that of a stainless steel. Titanium, however, has difficulty in welding, because the melting point is higher than 1800 K and titanium is easy to oxidize at the high temperature. In order to avoid these difficulties, the atmospheric shielding of the welding was employed, and titanium material was successfully welded without the oxidation scale as shown in Fig. 4.

A durability of the knife edge for the ConFlat(CF) flange made of titanium materials was evaluated. Figure 5 shows the image of knife edges of CF flanges, (a) virgin flange, (b) pure titanium of JIS grade 2, and (c) KS100. Figures 5(b) and 5(c) are knife edges after 50 times tightening repetition. The knife edge of the CF flange made of the pure titanium of JIS grade2 was worn out. On the other hand, the knife edge made of KS100 showed durable property. KS100, hard titanium alloy, and JIS grade 4, pure titanium, are considered to be suitable for CF flange.

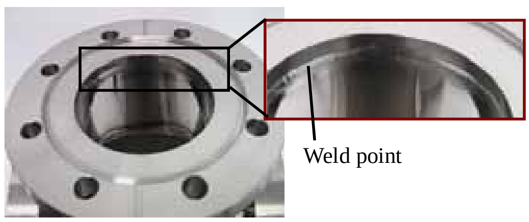

Fig. 4 Photograph of welded vacuum flange and pipe made of the titanium material.

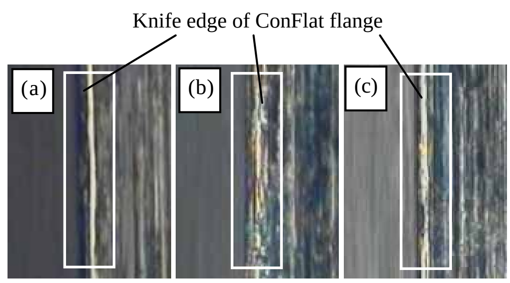

Fig. 5 Images for virgin knife edge of CF114 flange (a), and knife edge of the pure titanium of JIS grade 2 (b) and KS100 (c) after 50 times tightening repetition.

Fig.6 Fabricated titanium vacuum chamber and vacuum components[9].

Figure 6 shows the fabricated titanium vacuum chamber and vacuum components. UHV pressure was quickly obtained for the fabricated vacuum chamber and components without baking process even though a vacuum pump with low flow rate was employed.

IV. SUMMARY

Outgassing properties and surface characteristic of three kinds of titanium materials, pure titanium materials of JIS grade 2, JIS grade 4, and titanium alloy of KS100 were evaluated in terms of the effects of polishing method. In the titanium materials with the smooth surface and surface oxide layer thinner than about 10 nm, are obtained by mechno-chemical polishing and chemical polishing. These surface conditions provided an excellent outgasing property. Outgassing rates of these samples are lower than 10^{-12} $Pams^{-1}$ through baking process, and the outgassing rates of these pure titanium of JIS grade 2 and titanium alloy of KS100 without baking treatment are about 1/4 of that of stainless steel. These two titanium materials are good candidates for the vacuum material.

Vacuum chamber and components have fabricated for UHV/XHV systems using titanium materials. The welding process without the oxidation scale was achieved by the atmospheric shielding. Hard titanium material is preferable for a material used as the knife edge of the CF flange. Vacuum equipment was fabricated using titanium materials, which showed excellent vacuum properties for UHV/XHV systems.

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