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# Application of Titanium Materials to Vacuum Chambers and Components

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**Abstract.** This paper describes the outgassing properties of titanium materials, and development of vacuum chambers and components for practical UHV/XHV systems. The mechano-chemically polished and the chemically polished titanium materials have a smooth surface and a thin ( $\leq 10$  nm) oxide surface layer, which showed extremely low outgassing rate below  $10^{-12}$  Pams<sup>-1</sup> after baking process. In order to fabricate practical vacuum systems welding, metallizing and brazing processes were optimized, and complex shaped vacuum chambers and various vacuum components such as a bellows, valve, electric feedthrough and ceramic duct with titanium sleeve were fabricated. Sufficient mechanical properties and durability were obtained for practical use.

## 1. Introduction

A reduction in the outgassing rate of vacuum materials is important for the ultrahigh vacuum (UHV) and the extremely high vacuum (XHV) systems. Stainless steels and aluminum alloys have outgassing rates of the order from  $10^{-9}$  to  $10^{-10}$  Pams<sup>-1</sup> 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 additional surface finishing, e.g. vacuum-firing [1,2], oxidization [3,4] and thin film coating [5, 6], and use of a very thin wall [7] are necessary.

A titanium material is well known as an evaporated and a non-evaporated getter material for the UHV/XHV pumps. However, titanium materials have been hardly applied to vacuum chambers and components except for some parts of a magnetron ionization gauge [8] until 1980's, since titanium was not so machinable comparing to the stainless steels or aluminium alloys, and had difficulty in welding. The outgassing property for a titanium alloy similar to those for standard vacuum materials was reported [9], although the outgassing property for the titanium material was not intensively studied in the past.

After 1990's, researches on the vacuum characteristics of titanium materials and development of vacuum chambers and components have began. The vacuum characteristics have been reported that the outgassing rate of the chemically polished pure titanium (JIS grade 2) was less than 1/5 of that for the electrolytic polished stainless steel [10,11].

The authors have commenced intensive research and developments for titanium materials after year 2000. Some kinds of surface finishing were developed and evaluated, in which very low outgassing properties were clarified [12-14]. And the titanium materials were successfully applied to vacuum chambers and components for practical vacuum systems such as UHV systems for fabrication of nano-devices and the proton beam accelerator in the Japan Proton Accelerator Research Complex (J-PARC) [15,16].

In this paper, the outgassing properties and the surface finishing were mainly discussed for pure titanium materials JIS grade 2. And development of vacuum chambers and components utilizing some kinds of titanium materials were described.

## 2. Physical Properties of Titanium

Table 1 shows the physical properties of pure titanium (JIS grade 2, purity 99.7 %), stainless steel (SUS304, Cr: 18 wt% Ni: 8 wt%) and aluminium alloy (A5052, Mg: 2.8 wt%). The features of the titanium are as follows: (1)

lightweight and high hardness are desirable for a chamber and a transfer system; (2) low-Z number is advantageous for installation and maintenance under high-radiation level, compared to stainless steel; (3) low Young's modulus is suitable for a bellows component; (4) low thermal expansion coefficient is advantageous for a heating component and renders titanium less prone to develop leaks during bakeout; (6) non-magnetic property is suitable for a duct and the component used in the strong magnetic field; (7) the high corrosion resistance, since the surface oxide layer is a chemically stable. Therefore, titanium is considered to be preferable for the vacuum applications.

## 3. Surface Finishing and Outgassing Property

### 3.1. Surface Finishing

The surface polishing is beneficial to vacuum materials, because a smooth and passive surface influences the outgassing property. For stainless steels and aluminium alloys, various surface polishing such as the electrolytic polishing and the chemical polishing have been developed so far, and the low outgassing property was obtained. On the other hand, the surface polishing for titanium materials were hardly evaluated. Although the mechano-chemical polishing was found to be effective to improve the outgassing rate, this polishing method is not easily applied to complex shaped chambers and components.

In this study, the chemical polishing effects for the titanium material were evaluated. Chemical polishing using a nitric acid solution was found to be the most suitable for the pure titanium material among the tested polishing solutions, since a smooth surface and a thin and uniform surface layer were obtained. Since the cleaning process is important, the surface finishing which combines the chemical polishing and the precision cleaning was developed. Here, the precision cleaning is composed of a degreasing, a nitric acid soaking and a pure water soaking processes.

Table 1 Physical properties of titanium, stainless steel and aluminium alloy

	Titanium (JIS - 2)	Stainless Steel (SUS304)	Aluminium Alloy (A5052)
Atomic Number	22	Fe: 26 Cr: 24 Ni: 28	13
Specific Gravity ( $10^3 \text{ kg} \cdot \text{m}^{-3}$ )	4.5	7.9	2.7
Hardness (Hv)	~ 150	~ 190	~ 60
Young's Modulus (G Pa)	106	200	69
Thermal Expansion Coefficient ( $10^{-6} \text{K}^{-1}$ )	8.4	17	24
Thermal Conductivity ( $\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ )	17	16	137
Specific Heat ( $\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$ )	520	500	900
Electric Resistance ( $10^{-9} \Omega \text{m}$ )	480	720	49
Magnetism	Non- magnetic	Little- magnetic	Non- Magnetic
Cost (US\$) ( $1\text{m} \times 1\text{m} \times 1\text{mm}$ )	658	134	77

### 3.2. Surface Analysis and Outgassing Property

#### 3.2.1. Experimental Procedure

Pure titanium of JIS grade 2 was employed as sample material. Unpolished basis metal Ti ( $Ti_{(BM)}$ ), buffed Ti ( $Ti_{(BP)}$ ), mechano-chemically polished Ti ( $Ti_{(MCP)}$ ) and chemically polished Ti ( $Ti_{(CP)}$ ) were prepared. The sample size for surface analysis was 10 mm×10 mm×1 mm. The surface roughness ( $R_a$ ) was estimated by an atomic force microscopy (AFM) in 10  $\mu\text{m}^2$  and 1  $\mu\text{m}^2$  area. In order to measure the surface oxide layer thickness, depth profiles were analyzed by an auger electron spectrometer (AES) with Ar etching gas, and transmitting electron microscope (TEM) images were observed. The distribution of hydrogen atoms around the surface layer was analyzed by a time of flight - secondary ion mass spectrometry (TOF-SIMS). The outgassing rate was estimated by a modified orifice method switching between two pumping paths (SPP) [17]. The detection limit of the system was  $7 \times 10^{-13}$  Pams<sup>-1</sup> since the upstream and downstream pressures attained to  $10^{-9}$  Pa and below, the orifice conductance  $C_0$  was set to  $6.1 \times 10^{-3}$  m<sup>3</sup>·s<sup>-1</sup> and the sample area was  $8.9 \times 10^{-1}$  m<sup>2</sup> [13]. The outgassing rates were measured under the condition of non-baking or after baking at 393K for 20 hours.

#### 3.2.2. Results and Discussions

Table 2 shows the  $R_a$  and thickness of surface oxide layer for  $Ti_{(BM)}$ ,  $Ti_{(BP)}$ ,  $Ti_{(MCP)}$  and  $Ti_{(CP)}$ . In the macroscopic area of 10  $\mu\text{m}^2$ ,  $R_a$  for  $Ti_{(CP)}$  is small comparing to those of  $Ti_{(BM)}$  and  $Ti_{(BP)}$ , while it is 10 times larger than that of  $Ti_{(MCP)}$ . However, the surface of  $Ti_{(CP)}$  in microscopic area of 1  $\mu\text{m}^2$  is as smooth as that of  $Ti_{(MCP)}$ . This implies the difference in  $R_a$  is due to the macroscopic undulation.  $R_a$  for  $Ti_{(CP)}$  is comparable to that for  $Ti_{(MCP)}$  in microscopic area. Since the amount of the adsorption gas is influenced by the microscopic roughness, the surfaces of  $Ti_{(CP)}$  and  $Ti_{(MCP)}$  are considered to be smooth enough.

The thickness of the surface oxide layer in  $Ti_{(CP)}$  is smaller than those of the other polished samples as seen in Table 2, where the thickness was estimated by the half maximum value of the AES depth profile of oxygen. The surface oxide layer is confirmed to be an amorphous by the cross sectional TEM observation. The thin oxide surface layer is considered to act as barrier for bulk gas, mainly hydrogen, diffusion, resulting in low outgassing.

Table 3 shows the outgassing rates measured at 5 and 50 hours after the pumping without baking process, and those with the baking process.  $Ti_{(CP)}$  and  $Ti_{(MCP)}$  without baking showed 1/5 of the outgassing rate comparing to the chemically polished stainless steel ( $SUS_{(CP)}$ ), and the outgassing rate of  $Ti_{(BP)}$  is comparable to that for  $SUS_{(CP)}$ . The outgassing rates for  $Ti_{(CP)}$  and  $Ti_{(MCP)}$  with baking process reach to  $7 \times 10^{-13}$  Pams<sup>-1</sup>, which is two orders magnitude smaller than that of  $SUS_{(CP)}$  under the same pre-baking process. This means that the chemical polishing and the mechano-chemical polishing are suitable as the surface treatment for titanium materials.

Table 2 Surface roughness and thickness of surface oxide layer of polished pure titanium

	Surface Roughness ( $R_a$ )		Thickness of Surface Oxide Layer (nm)
	10 $\mu\text{m}^2$ range (nm)	1.0 $\mu\text{m}^2$ range (nm)	
$Ti_{(BM)}$	108	-	13
$Ti_{(BP)}$	44	-	10
$Ti_{(MCP)}$	2.5	1.03	11
$Ti_{(CP)}$	25	1.80	8

Table 3 Outgassing rates for polished titanium and chemical polished stainless steel

	Non-Baking (Pams <sup>-1</sup> )		With Baking (Pams <sup>-1</sup> )
	5 h	50 h	
$Ti_{(BP)}$	$3.3 \times 10^{-8}$	$2.8 \times 10^{-9}$	-
$Ti_{(MCP)}$	$9.3 \times 10^{-9}$	$4.6 \times 10^{-10}$	$7 \times 10^{-13}$
$Ti_{(CP)}$	$7.0 \times 10^{-9}$	$5.1 \times 10^{-10}$	$7 \times 10^{-13}$
$SUS_{(CP)}$	$4.7 \times 10^{-8}$	$3.1 \times 10^{-9}$	$1.0 \times 10^{-10}$

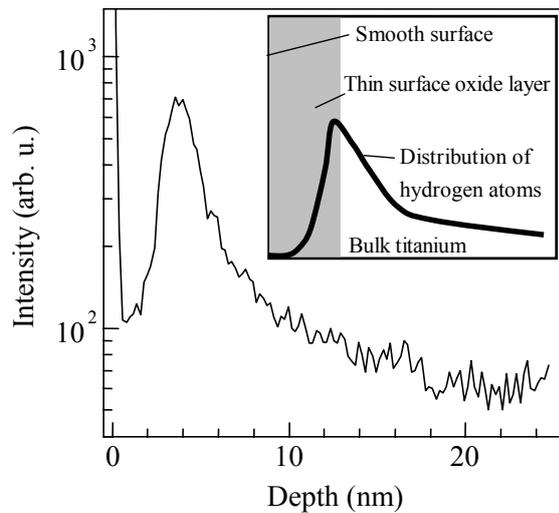


Fig. 1 Depth profile for the negative hydrogen ions (H) in  $Ti_{(CP)}$  and schematic diagram for the low outgassing titanium material (insert figure).

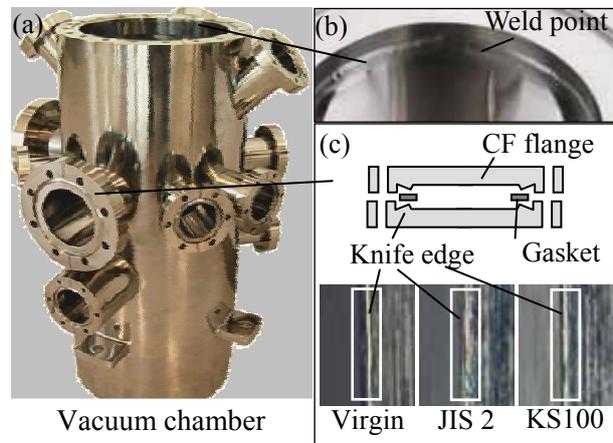


Fig. 2 (a) Vacuum chamber made of titanium material, (b) weld point between CF flange and pipe, (c) diagram of CF flange, and images of knife edge of virgin CF flange and knife edge of the pure titanium of JIS grade 2 and KS100 after 50 times tightening.

Figure 1 shows the TOF-SIMS depth profile of negative hydrogen ions (H) in  $Ti_{(CP)}$ . Here the depth was estimated by an etching rate of  $SiO_2$ . The hydrogen atoms localize at the boundary between the surface oxide layer and the bulk titanium. This region is considered to prevent the hydrogen diffusion, which brings the excellent outgassing property. In addition, the thin oxide surface layer is considered to be important for the lower outgassing because the thick oxide layer has many defects and dislocations which adsorb and dissolve gasses. Consequently smooth surface, thin oxide surface layer and barrier region preventing the bulk hydrogen diffusion give the extremely low outgassing rate for the titanium materials.

#### 4. Development of Vacuum Chambers and Components

##### 4.1 Vacuum Chambers and Components

Vacuum chambers and components for the UHV/XHV systems were fabricated using titanium materials as shown in Figs. 2 and 3. The machinability of the titanium materials is similar to that of the stainless steels. Titanium, however, has difficulty in welding, because the melting point is higher than 1800 K and titanium is easy to oxidize. In order to avoid this problem, the titanium was welded using an atmospheric shield in a semi-clean room, and the titanium material was successfully welded without the oxidation scale as shown in Fig. 2 (b). Thus, the complex vacuum chamber with many ConFlat (CF) flanges can be fabricated as shown in Fig. 2 (a).

A durability of the knife edge for the CF flange made of the titanium materials was evaluated. Figure 2 (c) shows the top view of the knife edges of CF flanges, virgin flange (left), pure titanium of JIS grade 2 (middle) and a hard titanium alloy, KS100 (right) which is product of KOBE Steel, Ltd.. Middle and right images of Fig. 2(c) are knife edges after 50 times tightening. The knife edge of the CF flange made of the pure titanium of JIS grade 2 was worn out, and vacuum leak occurred. On the other hand, the knife edge made of KS100 showed durable property, and vacuum leak did not occur. Therefore, hard titanium alloy of KS100 (Vickers hardness, 250 Hv), is considered to be suitable for CF flange.

A titanium bellows is expected to have a low stiffness, because 106 GPa of Young's modulus for titanium is the almost half of stainless steel. Mechanical properties for a hydro-formed bellows made of titanium were evaluated. The stiffness per convolution was  $25 \text{ Nmm}^{-1}$  which is 64 % of that for

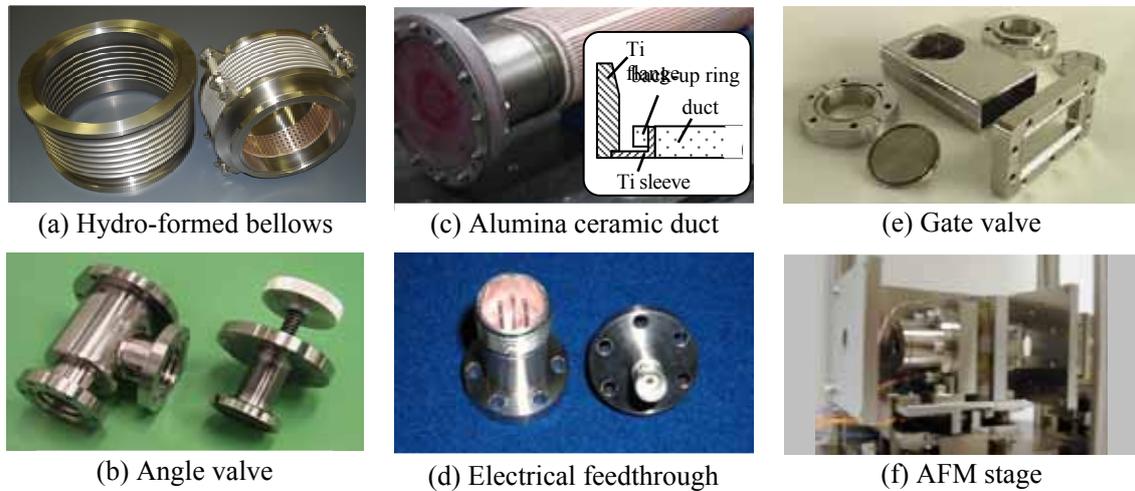


Fig. 3 Vacuum components made of titanium materials.

stainless steels. The displacement endurance was similar to that of stainless steel. Many hydro-formed titanium bellows as shown in Fig. 3 (a) were installed in the 3- and 50-GeV synchrotron and in the beam-transport line in J-PARC. A welded bellows made of titanium was also fabricated, and the durability was verified. The bellows was installed in an angle valve as shown in Fig. 3 (b).

Alumina ceramic duct jointed with Ti sleeve (see Fig. 3 (c)) and an electrical feedthrough (see Fig. 3 (d)) were fabricated by the metallizing (Mo-Mn) and the brazing (Ag-Cu). Rupture strength for a joint part of the alumina ceramic and the titanium was obtained to be greater than 100 MPa which is enough for practical use. The alumina ceramic duct was used in a rapidly varying magnetic field of 3-GeV synchrotron. The electric feedthrough was installed in a Kundsens cell for a molecular beam epitaxy (MBE) system and in an UHV-AFM system. In addition other vacuum components, e.g. a gate valve (see Fig. 3 (e)), an AFM stage (see Fig. 3 (f)) and a magnetic transporter were fabricated and installed in UHV-MBE and AFM systems.

#### 4.2 Vacuum Characteristics of UHV Systems

Figure 4 shows the pump-down curve of the MBE system (main chamber:  $\phi$  200 mm  $\times$  440 mm, inner area:  $3.6 \times 10^{-1}$  m<sup>2</sup>) made of the titanium material measured from the beginning of the evacuation using the turbo-molecular pump (pumping speed:  $250 \times 10^{-3}$  m<sup>3</sup>/s, base pressure:  $2 \times 10^{-8}$  Pa). The pressure showed  $1 \times 10^{-6}$  Pa at 3 hours after the pumping. The pressure showed  $5 \times 10^{-8}$  Pa at 12 hours after the pumping through the modest baking process at 393 K for 6 hours, and the ultimate pressure at 25 hours later was  $2 \times 10^{-8}$  Pa which corresponds to the base pressure of the vacuum pump. Thus, UHV pressure is quickly obtained for the vacuum system without / with baking process. Moreover simple test chamber ( $\phi$  250 mm  $\times$  420 mm, inner area:  $4.2 \times 10^{-1}$  m<sup>2</sup>) reached to  $10^{-10}$  Pa evacuated by an ion pump.

Figure 5 shows the pressure rise measured by a B-A gauge during the build up measurement of the

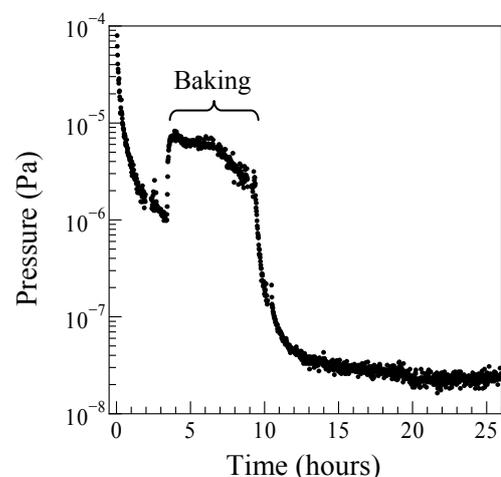


Fig. 4 Pump-down curve of MBE system composed of titanium material.

simple test chamber ( $\phi$  250 mm  $\times$  420 mm, inner area  $4.3 \times 10^{-1}$  m<sup>2</sup>) composed of titanium material. The test chamber retained the pressure lower than  $1 \times 10^{-6}$  Pa for the first 6 months, and the pressure rose to  $8 \times 10^{-5}$  Pa at 12 months later. The outgassing rate is estimated to be  $4 \times 10^{-13}$  Pams<sup>-1</sup> at the rapid increasing region after 8 months. This is close to  $7 \times 10^{-13}$  Pams<sup>-1</sup> obtained by SPP method. This indicates that the titanium materials have excellent outgassing property.

### 5. Summary

This paper described the outgassing property of the titanium materials, and development of the vacuum chambers and components for the practical UHV/XHV systems. The mechano-chemical polishing and the chemical polishing give titanium the smooth surface and the surface oxide layer thinner than 10 nm which provides excellent outgassing property. Outgassing rates are lower than  $10^{-12}$  Pams<sup>-1</sup> through baking process, and the outgassing rate without baking process is about 1/5 of that for the stainless steel.

Vacuum chambers and components have been fabricated for UHV/XHV systems utilizing the titanium materials. The titanium was successfully welded by the atmospheric shielding without formation of the oxidation scale. Hard titanium material was found to be preferable for the knife edge of the CF flange. Vacuum equipments were also fabricated using titanium materials, which showed excellent vacuum property.

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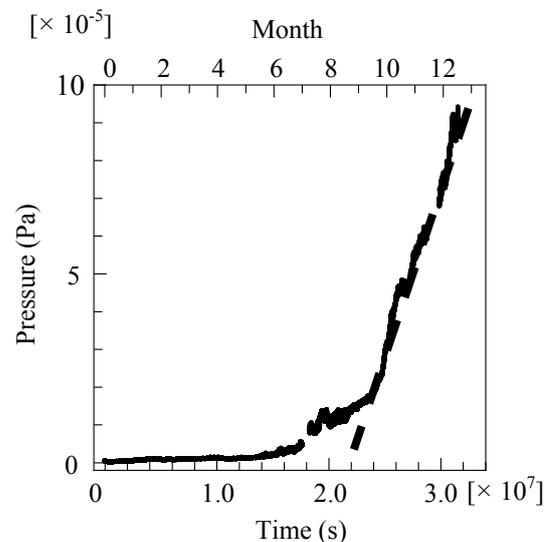


Fig. 5 Pressure rise during the build up measurement of test chamber made of titanium material.