# UTILIZATION OF $Cr_2O_3$ PASSIVATION PROCESS ON STAINLESS-STEEL CHAMBER FOR XHV ENVIRONMENT*

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*Abstract*

Forming of chromium oxides using the optimised vacuum thermal oxidation process has been conducted with a stainless-steel chamber for the extreme high vacuum (XHV) applications. With the previous chemical cleaning and a moderate bake-out in vacuum at 150 °C, the passive $Cr_2O_3$ thin film was made using the oxygen partial pressure of $1\times10^{-9}$ Torr at a chamber temperature of 450 °C. The passive $Cr_2O_3$ chamber appeared XHV performance with the base pressure in the range of $10^{-12}$ Torr, in which a stabilized degree of XHV conditions has been sustained in room temperature. The experimental investigations of the pump-down characteristics of the passive stainless-steel chamber will be presented, followed by a discussion of $Cr_2O_3$ surface atomic compositions and the practicability of $Cr_2O_3$-treated chamber.

## INTRODUCTION

Minimisation of outgassing is the most important problem in reaching an extremely high vacuum (XHV) of vacuum chamber as well as in the improvement of vacuum pumps and measuring instruments [1]. Stainless steels are the most commonly used materials for vacuum chambers due to their excellent corrosion resistance and good mechanical fabrication characteristics. During manufacturing and air exposure, oxides are still formed and containments are adsorbed on the surface of stainless steels. Furthermore, when exposed to air the surface is vulnerable to sorption of gases such as water because of a strong affinity of naturally produced passive oxide layer. This disadvantage of stainless steel against sorption within the near surface region has been a trouble in unbaked vacuum chamber.

Recently the vacuum thermal oxidation method, which is based on the concepts of selective oxidation of chromium at low oxygen partial pressures has utilized for practical application of dense surface oxide film on stainless steels [2]. In a well-controlled UHV environment, the annealing temperature of 450 °C under oxygen partial pressure of $1\times10^{-9}$ Torr was found to be adequate in the $Cr_2O_3$ film formation process of stainless steel. It has been reported [3] that if the porous oxide layer has been replaced by a dense chromium oxide film on the surface, the outgassing features of the surface of stainless steel will be more improved. Moreover, it has been studied that the outgassing rate of an oxidized chamber is about 100 times lower than that of an untreated one. Furthermore, it is demonstrated that surface constituents are changed with oxygen treatments and $Cr_2O_3$ is predominant with a small amount of $Fe_2O_3$ and Mn [4]. However, the cost effective utilization of a dense $Cr_2O_3$ stainless steel chamber is still a challenge for the XHV chamber manufacturers. In the present study the optimised passivation process is applied to find a cost effective pump-down characteristics of XHV stainless steel chamber. An attempt is also made to investigate the most effective passivation process in terms of the compositions of the surface oxide film.

## EXPERIMENTAL

The stainless steel vacuum chamber was prepared with the processes shown in Fig. 1. The law material of the chamber is 304LSS. After the cutting and welding, the surface of the chamber was treated with mechanical polishing, degreasing and electropolishing.

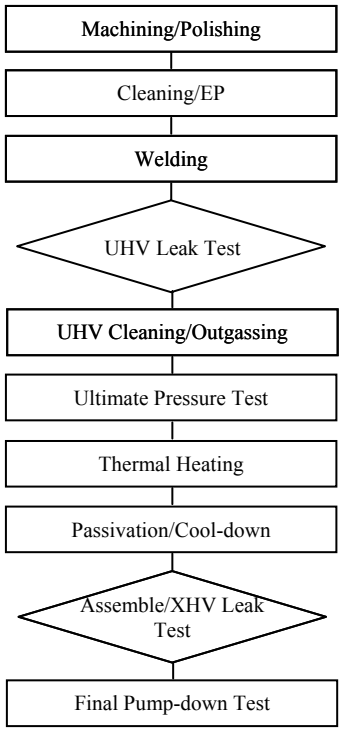


Figure 1: Fabrication flow of XHV vacuum chamber

In order to investigate the surface properties of clean 304LSS, the substrate samples have been made after each polishing processes. Each sample is a small slab size of $10\times10$ mm$^2$ with 1 mm thickness. The XPS analysis (Al$K_\alpha$ radiation) was performed for the samples. We also examined the surface properties of the sample treated by passive chromium oxide film using the atomic force microscope (AFM). After the deoxidation cleaning, the

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outgassing from the chamber was conducted at 150 °C for 24 hours and maintained the UHV pressure of $10^{-10}$ Torr. The XHV apparatus utilized in this work is shown in Fig. 2. The system consists of the main baking furnace system and roughing pumping system with diffusion pump, ion pump and cryopump. The bake-out furnace has a workable dimension of 2200 mm in height with a diameter of 1000 mm, and is to be controlled into the chamber temperature within ±1.5 °C at a maximum of 1200 °C. The vacuum inside bake-out furnace is also maintained with about $5\times10^{-7}$ Torr at 700 °C. The configuration of the vacuum chamber used for the passivation process is 8″ six-way stainless steel chamber, commercially produced. The pumping system for XHV chamber during the passivation consists of cryopump (EBARA ICP300 UQ) and 240 l/s ion pump for the bake-out.

Figure 2: Photo of XHV chamber of 8″ six-way installed in the bake-out furnace

The baking at 450 °C [4,5] was sustained for 24 hours, and the $Cr_2O_3$ film formation was simultaneously conducted with an oxygen partial pressure of $10^{-9}$ Torr. For the temperature uniformity of the chamber, the temperature of bake-out furnace is automatically controlled with high precision temperature sensors placed around the six-way chamber. The pump down characteristics of XHV chamber was measured with an extractor gauge (Leybold, gauge head; IE514, controller; IM520, sensitivity; 6.6, limit; $1\text{-}2\times10^{-12}$ Torr), installed at the symmetric position of upper part of the chamber.

## RESULTS AND DISCUSSION

### *Surface characteristics*

The surface atomic compositions and bonding states of the slab samples prepared by machine polishing and electropolising are shown in Fig. 3. The depth profiles of signal intensify $Fe2p_{3/2}$, $Cr2p_{3/2}$, O1s for machine polishing and electropolishing. For the polished samples, the signal intensity of $Fe2p_{3/2}$ core level gradually increased with the ion etching time. In the XPS spectra for Fe, a larger broad peak ranging from 706 to 711 eV was observed to be high in the top surface. The binding energies of Fe, FeO and $Fe_2O_3$ have been clarified 707, 709 and 710.9 eV, respectively [6]. The results show that the mixture composition of Fe, FeO and $Fe_2O_3$ is constituted on the polished surface. For the state of chromium, the binding energy of $Cr2p_{3/2}$ core level was found in the range of 576.9 to 574.4 eV for the sample. It is understood that the Cr compound layer, such as $Cr_2O_3$ was formed on the surface of each polished sample. As indicated in other study [2], when exposed to air the surface is vulnerable to sorption of gases of water, providing a reservoir for water into the porous surface oxide layers within the near-surface region. It has also pointed out that if the porous oxide layer of stainless steels is replaced by a dense chromium oxide film on the surface by using vaccum thermal oxidation method, the outgassing characteristics of the stainless steels will be improved.

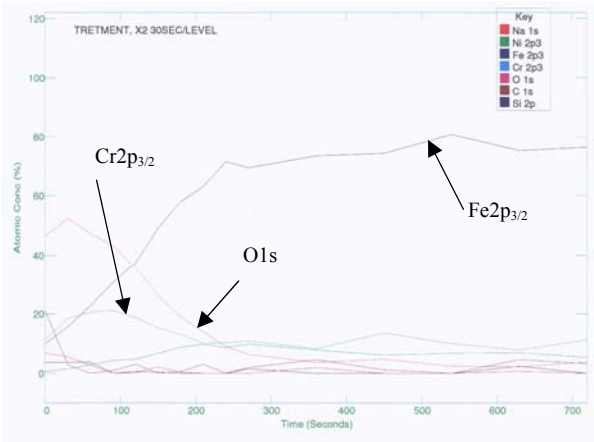
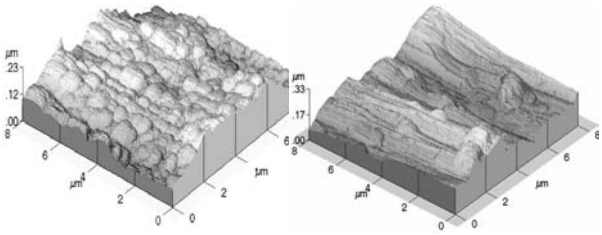



Figure 3: An example of depth profiles of XPS signal intensity for the electropolished sample ($Fe2p_{3/2}$, $Cr2p_{3/2}$, O1s)

In addition to the compositional changes of the oxide film, it has great possibility to vary the surface morphology of the film due to different growth rate of chromium oxide film depending on the oxygen partial pressure. Figure 4 gives the examples of AFM images of samples. The images show stripe pattern, which are considered to originate from the fabrication process under a rolling mill and cutting process. The image of the sample deposited with oxygen partial pressure of $\geq1\times10^{-6}$ Torr (Fig. 4a) show very distinct grains, but the images of the sample treated with oxygen partial pressure of $\geq1\times10^{-9}$ Torr (Fig. 4b) show vague grain boundaries. In this analysis, the stainless steel vacuum chamber has great inherent roughness originating from the fabrication process. It can be inferred that the dense oxidation in the oxygen partial pressure of $\geq1\times10^{-9}$ Torr can smooth out the surface of the oxide film atomically.



(a) (b)

Figure 4: AFM images of the samples based on the oxygen partial pressure; (a) $\geq 1\times10^{-6}$ Torr, (b) $\leq 1\times10^{-9}$ Torr

### *Pump-down characteristics*

The pump-don curves of the passive $Cr_2O_3$ XHV stainless steel six-way chamber were shown in Fig. 5, with a comparison of only baked state of the vacuum chamber. Here, the pump-down curves were obtained after the evacuation conditions, such as the initial pressure from TMP pumping systems are followed by the degassing conditions of extractor gauge at a pressure of $2\times10^{-8}$ Torr. The measurement of pressures in the $10^{-12}$ Torr range relies exclusively on the degassing of extractor gauge. The pressure decreases very slowly with time for 24 hours from starting of the evacuation. The system is allowed to continue pumping and the pressure reaches into $8\times10^{-12}$ Torr with as long as several hours, remaining the same for 30 days during the test with a small fluctuation in XHV ranges. And the measured pressure in the range of $5\times10^{-12}$ Torr is quite closely reproduced upon baking the passive chamber, so that there is no prominent difficulties in reproducing XHV condition for the commercially available stainless steel vacuum chamber.

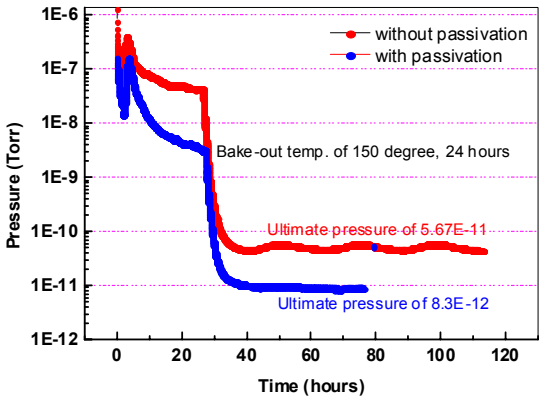



Figure 5: Pump-down curves of the XHV chamber made from stainless steel with the passive $Cr_2O_3$ treatment

Through the mass spectra analysis during the pump-down test, the major gas species in the pressure of $8\times10^{-12}$ Torr are only $H_2$ and atomic oxygen ion, to be thought as the emission from the operation RGA or extractor gauges. It is indicated that the XHV pressure in this experiment is determined by the desorption rate of hydrogen diffused from the chamber analysing equipments, such as the extractor gauge and mass spectrometer.

## CONCLUSION

The extreme high vacuum (XHV) was easily obtained with a forming of chromium oxides using the optimised vacuum thermal oxidation process. In order to make clear the gas desorption properties of the passive chamber, the surface chemical compositions, bonding state and topography of the polished and $Cr_2O_3$ treated samples were investigated. For the dense $Cr_2O_3$ film, a smooth surface and the decrease of gas desorption were observed, The passive $Cr_2O_3$ chamber appeared XHV performance with the base pressure in the range of $10^{-12}$ Torr, in which a stabilized degree of XHV condition has been sustained with less evacuation time comparing to that of no passive chamber under the same evacuation scenario. For the cost effective commercial production of XHV chamber, it will be of importance that the optimised passivation process with bake-out, evacuation procedures and pump-down are to be carefully harmonized.

## ACKNOWLEDGEMENTS

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