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Vacuum Characteristic of a Chamber Made of Mild Steel

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The base pressure and outgassing rate of a mild steel chamber were measured and compared to those of a stainless steel chamber. A combined sputter-ion and non-evaporable getter pump with a nominal pumping speed of 490 *l/s* generated the base pressure of 2.7×10^{-11} mbar in the mild steel chamber and 1.2×10^{-10} mbar in the stainless steel chamber. The rate-of-rise measurements show that the mild steel has an extremely low outgassing rate of 2.6×10^{-13} mbar l s^{-1} cm^{-2}, which is about one-order of magnitude smaller than the outgassing rate of the stainless steels. Vacuum annealing of the mild steel at 850^{o}C reduced the outgassing rate further to 8.8×10^{-14} mbar l s^{-1} cm^{-2}, which was comparable to the outgassing rate of a heat treated stainless steel for extreme-high vacuum use.

Keywords : Outgassing rate, Mild steel, UHV

I. Introduction

Mild steel normally refers to a ferritic steel which contains carbon less than 0.3% and is used for a general purpose structural steel. Because the steel is a soft magnetic material, it is widely used for shielding sensitive experimental apparatuses from stray magnetic field in high vacuum. For instance, electron microscopes employ nickel plated mild steel for constructing their specimen vacuum chambers in which the electron beam propagates and interacts with specimens; presence of stray magnetic field deteriorates proper propagation of the electron beam, degrading the resolution of the electron microscope [1].

The mild steel has been known for a long time that its outgassing rate during roughing down as well as after *in-situ* bakeout is so high, the material is not suitable for ultra-high vacuum (UHV) applications [2,3]. As a consequence, one has to find an expensive ferritic material when a stray magnetic field is of concern in UHV. For example, Kamiya et al. selected Permalloy and SUS 430 (ferritic stainless steel), after careful screening of the suitable materials, to prevent electron beam from external magnetic field in an particle accelerator [4].

In this article, we demonstrate that the thermal outgassing rate of a mild steel is not as high as we think. Surprisingly it is much smaller than that of stainless steels, the most common UHV materials. The base pressure of a vacuum chamber made of the mild steel was 2.7×10^{-11} mbar, and its outgassing rate was $\sim3\times10^{-13}$ mbar l s^{-1} cm^{-2}, which indicates the mild steel is even appropriate for XHV use.

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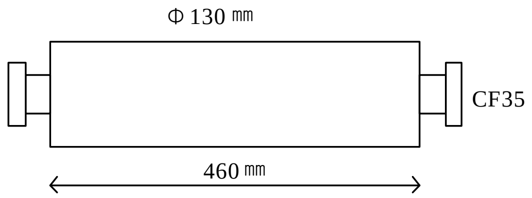
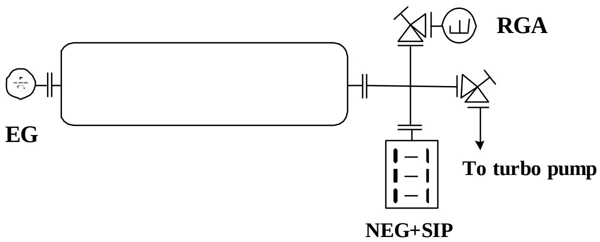
Table 1. Chemical compositions of the AISI 1020 mild steel.

	C	Si	Mn	P	S	Ni	Cr	Cu	B	As	Fe
AISI 1020 (S20C)	0.2	0.21	0.42	0.021	0.007	0.03	0.08	0.03	0.002	0.01	Balance



(a)



(b)

Figure 1. (a) Schematic design sheet of the mild steel and stainless steel test chamber (b) schematic of the experimental configuration for measuring the base pressure of the test chambers.

Vacuum annealing of the mild steel at 850°C reduced the outgassing rate further.

II. Experimental

A mild steel round bar (AISI 1020, carbon concentration <0.2%,) was machined into a long bored cylinder with inner diameter of 130 mm, two circular plates with two DN35 ConFlat flanges. Table 1 gives the chemical compositions of the mild steel. The circular mild steel plates were directly welded on both ends of the cylinder. The thickness of all parts was 10 mm, which was much thicker than usual stainless steel chambers in order to supply enough magnetic shielding effect. The total surface area is about 2,400 cm^2 and the volume is about 7 liters. A stainless steel 304 chamber of the same configuration with thickness of 3.3 mm was made for comparison. Fig. 1(a) shows the overall configuration of the test chambers. All parts were degreased using alkaline detergents, rinsed with de-ionized water, welded, and helium leak checked prior to the tests.

Firstly, we measured the base pressures of the thick mild steel chamber and the stainless steel chamber, which were mounted on a pumping system composed of a small stainless steel four-way cross chamber, a sputter-ion pump with a nominal pumping speed of 60 *l/s*, a non-evaporable getter (NEG) pump (SAES getters, WP950) with a nominal pumping speed of 430 *l/s*, and an all-metal right angle valve for roughing. The base pressure of the pumping system itself had been measured to be 2.8×10^{-11} mbar before the installation of the sample chamber. The pressure was measured using an extractor ionization gauge (Leybold, IM540). The experimental configuration is given in Fig. 1(b). The base pressure of the stainless steel 304 chamber was also measured for comparison.

The rate-of-rise (RoR) method [5] was used to measure the outgassing rate, because when the hydrogen is outgassed from the steel vacuum chamber after bakeout, the pressure increases linearly over a long time period. We can assume that the re-adsorption is zero, if the outgassing rate is constant over a long period. Under the assumption, we can measure the intrinsic hydrogen outgassing rate (q),

$$q = (V/A)(\mathrm{d}P/\mathrm{d}t) \text{ mbar } l \text{ s}^{-1} \text{ cm}^{-2},$$

where V is the volume of the test chamber, A is the geometrical surface area of the chamber, and $\mathrm{d}P/\mathrm{d}t$ is the measured rate of pressure rise on a sealed-off sample chamber at a constant room temperature with no pumping.

The experimental setup is basically the same as in Ref. 6. In order to identify the residual gas components of the mild steel chamber, a residual gas analyzer (Prisma, Balzers) was installed on the roughing pump system composed of a sputter ion pump with a nominal pumping speed of 10 *l/s* (Gamma vacuum), a turbomolecular pump with a nominal pumping speed of 60 *l/s* (Pfeiffer vacuum) and a small stainless steel four-way cross chamber with CF35 Conflat flanges. The stainless steel and mild steel test chambers were connected to the roughing pump system through an all-metal right-angle valve. The residual gas spectra on the roughing pump system revealed that the dominant residual gas of the mild steel chamber was H_2 after bakeout.

III. Results and Discussion

1. Pumping curve and base pressure

Fig. 2 shows pumping behaviors of the mild steel and stainless steel 304 chambers. Since the roughing down curves in a vacuum system significantly depend on the humidity of venting gas and the duration of exposure before pumping, the sample chambers ware submitted to *in-situ* bakeout at 150°C for 48 h and subsequent nitrogen gas exposure for 5 h prior to the measurements [7]. The systems were pumped for 24 h before 48 h baking at 150°C. At the final stage of baking, the NEG pump was activated at 450°C for 1 h and the roughing valve was closed to get the base pressure.

The roughing down curves during initial stage of pumping for both chambers closely followed the well-known power law, $P_0\ t_h^{-a}$ with a nearly unity. As for the machined mild chamber (see Fig. 2(red)), the pressure was only three times larger than that of the STS vacuum chamber, Fig. 2(blue). One can expect a big difference in vacuum pressure between the two materials. However the vacuum pressure difference between the two chambers were not as big as we think as measured in this study. The roughing down curves followed the power law as $1.5\times10^{-6}\ t^{-1.05}$ mbar (10-40 h, t in hour), while $9.8\times10^{-7}\ t^{-0.96}$ mbar for STS304. These measured roughing down curves show typical surface desorption characteristics of water vapor ($a\sim1$) limited by diffusion through the surface oxide layer [7,8]. The higher water outgassing seems to be related to the natural oxide

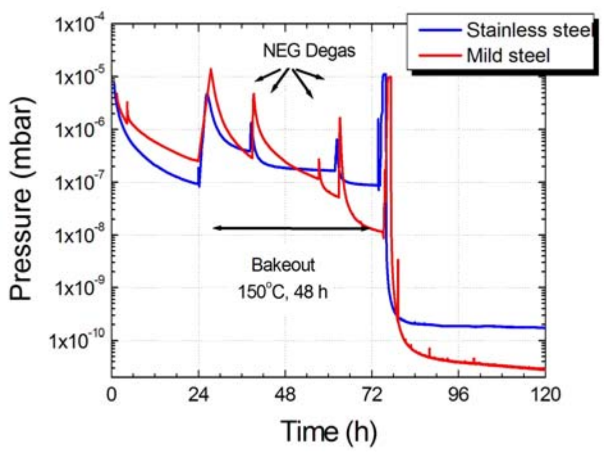


Figure 2. Pumping curves of the mild steel (red) and stainless steel 304 (blue) chambers.

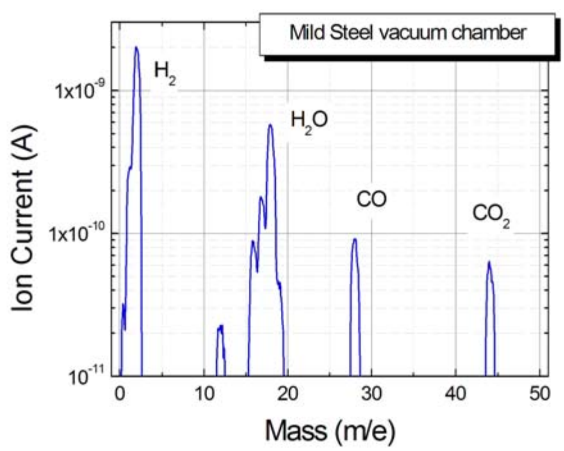


Figure 3. RGA spectrum of the mild steel taken at the end of bakeout.

thickness, as the mild steel is easily corroded and the oxide is continuously growing in contact with air.

Residual gas compositions at various stages of roughing down and bakeout showed that the desorbed molecules were mainly water vapor with traces of carbon and hydrogen containing molecules. The RGA spectra obtained from the steels showed no noticeable differences in residual gas compositions compared with ordinary vacuum chambers made of stainless steels (see Fig. 3).

After 48 h from the end of bakeout, the base pressure of the mild steel chamber installed on the pumping system reached to 2.8×10^{-11} mbar, while the stainless steel chamber showed the base pressure of 1.2×10^{-10} mbar. Considering the base pressure 1.4×10^{-11} mbar of the pumping system, we may conclude that the base pressure of the mild steel chamber would be about 8.8×10^{-12} mbar. In contract to common knowledge in vacuum science and technology, the results in this study is quite remarkable with a vacuum chamber made of a mild steel. The results clearly show that the mild steel is highly suitable for constructing UHV systems or even extremely high vacuum (XHV) systems. The cause of this desirable vacuum characteristic is under study and will be published elsewhere.

2. Outgassing rates

After the base pressure measurements, we utilized the RoR technique to measure hydrogen outgassing rates. A spinning rotor gauge (SRG, Oerlikon Leybold Vacuum) was used to prevent significant errors caused by either pumping or outgassing action of an ion gauge [6]. Because the temperature control is important for proper operation of the SRG, the temperature was maintained at $24\pm0.1^{\circ}$C during RoR measurements. The temperature stability enabled us to measure a RoR as low as 2×10^{-6} mbar/day without regard to the noise.

The stainless steel and mild steel chambers were baked again at 150°C for 48 h, and the SRG gauge was baked at 200°C to remove the water vapor which were adsorbed on the surface during venting. RoR data sets for the stainless steel 304 are given in Fig. 4(a) and 4(b). The pressure in the stainless steel 304 chamber increased by 1.64×10^{-4} mbar for 24 hours as shown in Fig. 4(a), and the linear fit of the data yielded an outgassing rate of 5.1×10^{-12} mbar l s^{-1} cm^{-2}. Then, the stainless steel chamber was degassed at 450°C for 36 hours in a vacuum furnace. Fig. 4(b) shows a RoR data set for the degassed stainless steel chamber. The data was also fitted linearly to yield an

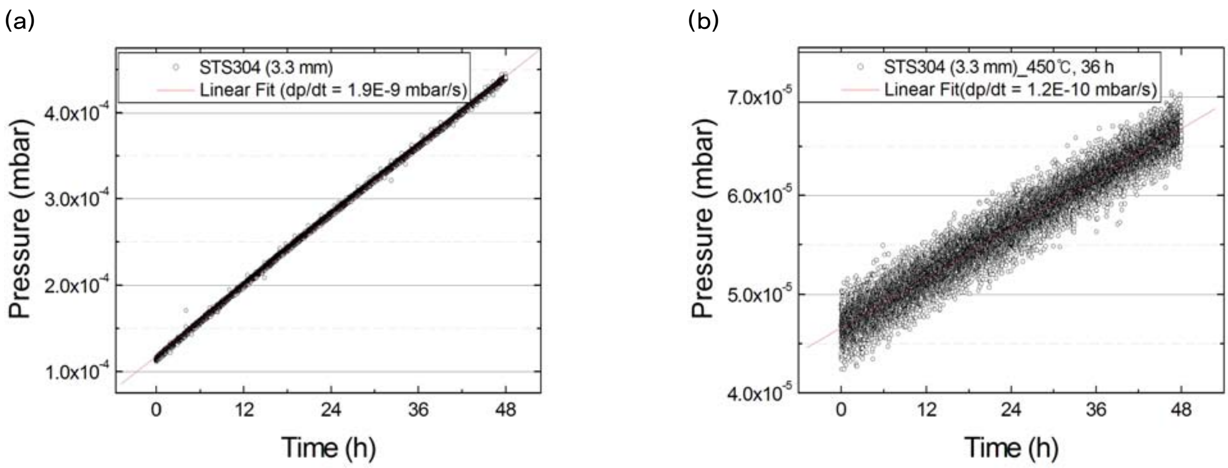


Figure 4. RoR data sets for the stainless steel 304 vacuum chamber; (a) untreated and (b) after heat treatment at 450°C for 36 h.

outgassing rate of 3.4×10^{-13} mbar l s^{-1} cm^{-2}; the value was consistent with our previous measurements and was within the typical range of reported values [6,9].

The mild steel chamber showed a slow increase of pressure, 3.46×10^{-6} mbar for 24 hours (Fig. 5(a)), compared to the undegassed stainless steel chamber. The linear fit of the RoR data yielded an outgassing rate of 2.6×10^{-13} mbar l s^{-1} cm^{-2}, which was about one order of magnitude smaller than that of the undegassed stainless steel chamber and comparable to that of pretreated stainless steel chambers at a high temperature (see Fig. 4(b)). The RoR measurements were carried out two times for the stainless steel 304 chamber and three times for the mild steel chamber. Again, this is another marked result of this study. The hydrogen outgassing rate is so low that the mild steel is a good material for constructing UHV systems, as discussed in the previous section.

Machining generates internal stress in magnetic materials, causing degradation of the magnetic characteristics. To eliminate such negative effect of internal stress and to recover the magnetic permeability, the mild steel chamber was annealed at 850°C in a vacuum furnace. The annealing time was increased from one hour, a normal period in electron microscopy society, to 24 hours because we expected that the vacuum annealing would degas the mild steel chamber.

The pressure in the vacuum-annealed mild steel chamber increased by 1.81×10^{-6} mbar for 24 hours and the linear fit of the data yielded an outgassing rate of 8.8×10^{-14} mbar l s^{-1} cm^{-2}, which is about one third of the outgassing rate of untreated mild steel. The heat treatment for the mild steel resulted in a relatively small decrease in outgassing rates compared to the vacuum firing method for the stainless steels which is usually resulted in the reduction of the outgassing rate by more than two orders of magnitude [10]. This may be understood as the H_2 concentration in the bulk is low enough, the limiting step for desorption becomes the surface recombination rather than the bulk diffusion.

Considering that the base pressure of a UHV system is greatly affected by the outgassing rate of the pump, we plan to replace the non-evaporable getter pump with a titanium sublimation pump with a liquid nitrogen cold trap which was known to generate XHV more reliably. A cold field emission electron gun is also planned to be fabricated because its operation requires UHV or XHV environments with magnetic shielding.

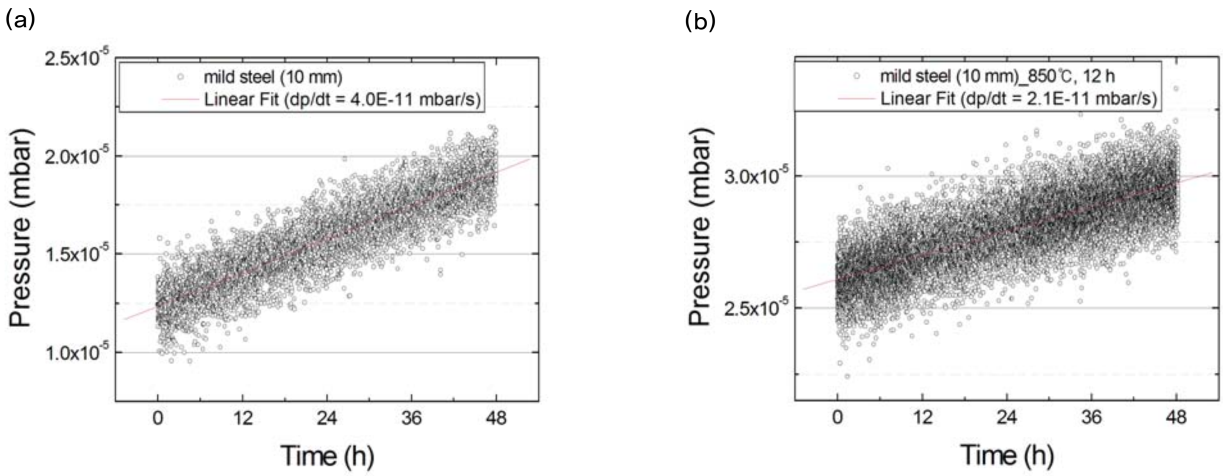


Figure 5. RoR data sets for the mils steel; (a) bare sample and (b) after annealing at 850°C for 12 h in a vacuum furnace.

IV. Summary

The outgassing rate of a mild steel chamber was measured and compared to those of a stainless steel chamber by using the rate of the rise method. The mild steel presented an extremely low outgassing rate of 2.6×10^{-13} mbar l s^{-1} cm^{-2}, while the outgassing rate of the stainless steel was 5.1×10^{-12} mbar l s^{-1} cm^{-2}. Vacuum annealing of the mild steel at 850°C reduced the outgassing rate further to 8.8×10^{-14} mbar l s^{-1} cm^{-2}, indicating that the mild steel would be adequate for XHV uses. The mild steel chamber was then pumped by a combined sputter-ion and non-evaporable getter pump with a nominal pumping speed of 490 *l/s* and the base pressure was 2.7×10^{-11} mbar.

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