Vacuum Furnace for Degassing Stainless-Steel Vacuum Components

James A. Fedchak[a), Julia Scherschligt, Daniel Barker, Stephen Eckel, Alex P. Farrell, Makfir Sefa

National Institute of Standards and Technology, Gaithersburg, MD 20899

a) Electronic mail: james.fedchak@nist.gov

Ultra-high vacuum systems must often be constructed of materials with ultra-low outgassing rates to achieve pressure of 10^{-6} Pa and below. Any component placed into the ultra-high vacuum system must also be constructed of materials with ultra-low outgassing rates. Baking stainless steel vacuum components to a temperature range of 400 °C to 450 °C while under vacuum is an effective method to reduce the outgassing rate of vacuum components for use in ultra-high vacuum systems. The design, construction, and operation of a vacuum furnace capable of baking vacuum components to a temperature of 450° C while maintaining a pressure of 10^{-3} Pa or lower is described. The furnace has been used for extended bakes at 450 °C while maintaining pressures below 10^{-5} Pa. As an example, we obtained an outgassing rate of 1.2×10^{-9} Pa L s^{-1} for a gate valve baked for 20 days at a temperature of 420 °C.

I. INTRODUCTION

Ultra-high vacuum (UHV) systems operate at pressures below 10^{-6} Pa and are widely used in advanced research and manufacturing. To achieve and maintain UHV pressures, vacuum chambers and systems must be constructed from low-outgassing materials and any components placed inside of the UHV system must also have a low outgassing rate. Extreme-high vacuum (XHV) systems operate at pressures in the 10^{-10} Pa decade and below; for these systems, the requirement for low outgassing chambers and components is even more demanding. Hydrogen is typically the dominant outgassing species in UHV and XHV systems. This is due to the presence of trapped

atomic hydrogen within the materials from which the chamber and components are constructed, and can't be removed by cleaning or be easily pumped away. Atomic hydrogen diffuses through the bulk and combines on the surface to release H_2 gas. Outgassing rates per unit area (flux) in the range of 10^{-10} Pa L s^{-1}cm^{-2} to 10^{-12} Pa L s^{-1}cm^{-2} are typically required for UHV and below. Chambers with larger surface area must have lower outgassing flux to achieve the same outgassing rate. Increasing the pumping speed would result in an even lower pressure; however, it must also be recalled that real vacuum systems have valves, flanges, constricting nipples, and are typically loaded with equipment and materials needed for the application of interest, all of which both increase the surface area and reduce the effective pumping speed. An effective H_2 pumping speed of 10 L/s is a realistic order-of-magnitude estimate for many modestly sized vacuum systems. Type 304 and 316 stainless steels (UNS S30403, UNS S30400, UNS S316403, etc.) are common materials used in UHV systems; unfortunately, untreated stainless-steel contains large residual concentrations of H_2 from the steel production process. Post-production treatments can reduce the concentration of dissolved H_2, thus reducing the outgassing rate.

It has been demonstrated that a 400 °C to 450 °C heat-treatment in vacuum is an effective method for reducing the outgassing rate[1–5]. Such a medium-temperature (herein defined as 400 °C to 450 °C) vacuum bake on a stainless-steel vacuum chamber can be performed utilizing external heaters, such as heater tape, and covering the heaters and chamber with insulation. This method is simple, but has the disadvantage that flanges and seals are prone to leak at such elevated temperatures, and, in some cases, temperatures of 400 °C or greater may exceed the specifications of other components in the system. Additionally, it is often desirable to bake components that will be placed into the UHV system without necessarily baking out the entire vacuum system of interest. A further complication is that it can take days or weeks to degas typically sized vacuum components and chambers using the medium-temperature vacuum bake method. On the other hand, a dedicated vacuum furnace is a convenient way to bake out chambers and components to achieve low outgassing rates. Such a furnace is separate from the vacuum system of interest, and may be designed using a minimum number of seals, thus reducing leak problems. High temperature vacuum furnaces are commercially available, but are

expensive and impractical for most end users of UHV and XHV. A medium-temperature vacuum furnace can be an asset to laboratories routinely attempting to achieve UHV pressures and lower, especially those which often need to change the vacuum system configuration or need to load components into the system. In this shop-note, we describe how to construct and operate a vacuum furnace that can be used to perform medium-temperature vacuum heat-treatments in the range of 400 °C to 450 °C. Our design has demonstrated robustness and should be within the resources of labs with limited budgets. We use hardware and components commonplace in many laboratories. Finally, we will present results for the outgassing rate of a gate valve degassed in the vacuum furnace; the outgassing measurements of the gate valve are described in an appendix.

II. Theory of degassing

During the production process of stainless steel, a large amount of atomic hydrogen is dissolved in the bulk, and a post-process treatment must be performed to reduce the hydrogen concentration. We provide an overview of such treatments below; for more detail, the reader is encouraged to consult various texts on the subject[6—8]. For untreated 300 series stainless steel, the atomic hydrogen concentration is on the order of 2 $\times 10^{19}$ cm^{-3}, corresponding to an H_2 equivalent pressure of roughly 4.1 $\times 10^4$ Pa at 23 °C (approximately 4/10 of an atmosphere)[9]. In discussing gas evolution from materials, one often distinguishes outgassing due to gas dissolved within the material (absorbed gas) from desorption of gas adsorbed on the material surface. In practice, it is difficult to distinguish which processes produces outgassing, and most outgassing rate measurements represent the total gas evolved from a material from all possible processes. During the evacuation of a stainless-steel chamber initially containing air at atmospheric pressure, the most abundant outgassing product is H_2O desorbing from the stainless-steel surface. Over time, the water outgassing rate in an evacuated system will decrease as the water desorbs from the stainless-steel surface and is pumped away. In a typical size vacuum system, outgassing fluxes of 10^{-8} Pa L/cm^2 s are possible after several days of pumping,[10] corresponding to a base pressure of 10^{-6} Pa in a vacuum system with 1000 cm^2 of surface area and evacuated with a 10 L/s pump. As the water outgassing rate reduces over time (typically as a power law), desorption becomes slower. To achieve lower water outgassing rates and lower base pressures, a vacuum bake for at least 1 day to 3 days in

the temperature range of 120 °C to 150 °C is typically performed. This effectively removes most of the water on the surface of the stainless steel, and a total outgassing flux in the range of 10^{-10} Pa L cm^{-2} s^{-1} to 10^{-9} Pa L cm^{-2} s^{-1} can be expected[1]. Once reduced to this level, the predominant outgassing species is H_2.

Hydrogen atoms dissolved in the bulk stainless steel diffuse to the surface, recombine to form H_2, and subsequently desorb from the surface. It may also diffuse back into the stainless-steel bulk. If the hydrogen concentration in the bulk is high, the surface concentration of hydrogen will be high as well. In this case, nearly every H atom diffusing to the surface will recombine with another H atom, and the outgassing rate is thus governed by hydrogen diffusion through the bulk with no dependence on the surface reaction rate. The flux j_{diff} of hydrogen through the stainless-steel due to a concentration gradient is given by Fick's first law of diffusion

$$j_{\text{diff}} = D(T)\frac{dc}{dx} \ .$$

(1)

The density of hydrogen in the steel is given by the concentration c, and the temperature-dependent diffusion coefficient $D(T) = D_0 e^{-E_D/k_B T}$, where E_D is an activation energy. The reduction of the hydrogen concentration over time is given by Fick's second law of diffusion

$$\frac{\partial c}{\partial t} = D(t)\frac{\partial^2 c}{\partial x^2} \ .$$

(2)

For stainless steel parts loaded into a vacuum furnace, we consider a slab of thickness d with vacuum on both sides ($c = 0$). The solution of this equation with these boundary conditions has been discussed in many fundamental texts and papers on vacuum technology[8,9,11]. It yields an exponential time constant called the characteristic time t_d given by

$$t_{\text{d}} = \frac{d^2}{\pi^2 D(T)} \ .$$

(3)

This formula is only valid for diffusion-limited outgassing, it ignores surface recombination and assumes that all hydrogen reaching the surface will desorb. The

4

formula can be used to predict the time required to reduce the bulk hydrogen concentration; a bake time of $5t_d$ reduces the concentration by 99% in the diffusion limited outgassing regime. Table 1 gives the characteristic time for 304 stainless steel of various thickness commonly found in vacuum components. These are calculated using eq. (3) and the diffusion coefficient $D(T)$ determined using $D_0 = 1.22 \times 10^{-6}$ m^2/s and E_D = 0.57 eV from Grant *et al.*[12] It is easily seen from Table 1 that thinner components degas much more quickly than thicker ones, and that increasing the temperature greatly reduces the time to degas components. Similar degas times are found for 316 stainless steel. Hydrogen can also permeate into the stainless steel from the H$_2$ partial pressure in the background. As outlined in the appendix in Berg[13], we use the diffusion and permeation coefficients from Grant *et al.* to estimate that, at a temperature of 400 °C and a H$_2$ partial pressure of 1×10^{-3} Pa, the equilibrium hydrogen concentration is reduced by more than a factor of 5000 from that of typical unbaked stainless-steel (3 ppm/wt[9]).

TABLE I. The calculated time it takes to reduce the hydrogen concentration in 304 stainless steel by 99%. Various thicknesses of steel are given in the table, corresponding to those commonly used in vacuum chamber construction. The flange designations are defined in the standard document ISO 3669 (International Organization for Standardization).

Description	$5t_d$ (Days)			
	150 °C	375 °C	400 °C	425 °C
1.6 mm (1/16 in) plate	72	0.30	0.20	0.20
3.2 mm (1/8 in) plate	290	1.3	0.90	0.60
DN 16 (CF 1.33") Flange, 0.76 cm (0.3") thick	1700	7.4	5.1	3.6
DN 40 (CF 2.75") Flange, 1.3 cm (0.5") thick	4600	21	14	9.9
DN 63 (CF 4.5") Flange, 1.7 cm (0.68") thick	8500	38	26	18
DN 100 (CF 6") Flange, 2 cm (0.78") thick	11000	50	34	24
DN150 (CF 8") Flange 2.24 cm (0.88") thick	14000	64	44	31
DN 200 (CF 10") Flange, 2.5 cm (1.0") thick	17000	77	53	37
DN 250 (CF13.25") Flange, 2.84 cm (1.12") thick	23000	100	71	50

As the outgassing rate is further reduced, the surface concentration is also lower and hydrogen atoms diffusing from the bulk to the surface will no longer combine to form H_2 with unit probability. In that case, outgassing will be recombination limited instead of diffusion limited, and the characteristic time will be longer than predicted by eq. (3). It is difficult to give an *a priori* estimate of the characteristic time in the recombination limited regime because it depends on a surface reaction-rate for $H + H \rightleftharpoons H_2$ and other surface details such as the area and roughness. For a sample baked at a temperature T for a time t, a dimensionless time, the Fourier number, may be defined as

$$F = \frac{D(T)t}{d^2}.$$ (4)

A Fourier number greater than 1 is often taken as an indication that the sample outgassing is in the recombination limited regime. The characteristic times given in Table 1 represent a lower limit to the time necessary to bake to the components. In this discussion, we've made several simplifying assumptions, a rigorous treatment of the theory of outgassing must include other effects to properly model ultra-low outgassing rates in the recombination limited regime, such as the presence of hydrogen traps; *i.e.*, deeply bound sites for hydrogen adsorption that have large activation energies.[12] Surface chemistry leading to other desorbed gases such as CO_2 also contribute to ultra-low outgassing rates. Additionally, stainless steel surfaces have an oxide layer which may have a lower diffusion coefficient than that of the stainless steel, thus forming a diffusion barrier[14] and suppressing outgassing. Such considerations are beyond the scope of this shop-note, but illustrate the complexities of achieving ultra-low outgassing rates.

III. Construction of the Vacuum Furnace

A.   *Design considerations of the furnace vacuum chamber*

Our goal was to design a vacuum furnace that could reliably be used for vacuum firing in the range of 400 °C to 450 °C. Because seals can easily leak when heated this temperature range, our design has only one seal that is exposed to these temperatures.

The furnace diameter is large enough to accommodate a component 50 cm long with a DN 350 flange (13.25 CF). The system also required a vacuum pump, a gauge to monitor the vacuum level during the bake, and temperature sensors for monitoring and controlling the bake temperature that are mounted external to the chamber. We also included two internal temperature sensors in our design as well, although we consider the latter an optional feature. The pumping system and gauges are located outside of the hot-zone. A turbomolecular pump with a dry backing pump was chosen as the pumping system to maintain the system at 10^{-3} Pa or below during the bake. Our vacuum furnace easily maintains pressure below 10^{-4} Pa at 450°C, allowing us to reduce the equilibrium hydrogen concentration by greater than 5×10^5. This maximal pressure also avoids accidental overload of the turbomolecular pump and, by keeping pressure in the molecular flow regime, assures that outgas products and other contaminants are efficiently removed.

Figure 1, (a) – (d), depicts the furnace vacuum chamber which consists of three main stages: a medium-temperature stage (i.e., the "hot zone"), a low-temperature (<150 °C) stage, and the pumping stage. The hot-zone can reach temperatures up to 450 °C during use, and the low-temperature stage is maintained to between 100 °C and 150 °C. This is to prevent water or other condensable gases from collecting on cold surfaces during the bake. The pumping stage is unheated.

The medium-temperature stage is a large vacuum chamber made of 316 stainless-steel, 3.2 mm thick, and consisting of a main chamber and an arrow tube section or "snout" which connects it to the pumping manifold. The main chamber lies entirely in the hot-zone. It consists of a cylinder with outer diameter (OD) of 35.6 cm on a 41.7 cm OD (16.5 CF) knife-edge flange sealed using a silver-plated copper gasket. The 16.5 CF flange is the only flange in the hot-zone and provides access for loading the oven. To make removing the 16.5 CF flange easier (even silver-plated copper gaskets may stick to knife-edge flanges when heated to 450 °C), four tapped through-holes were machined into the blank flange between the existing bolt-holes, and outside of the knife-edge, thus providing "jack-screws" such that a bolt driven into these tapped holes lifts the blank flange and opens the chamber. During our first several operations of the vacuum furnace, three Belleville disc-springs where used as washers on each bolt. This was to prevent the

7

bolts from loosening as the flange was heated. After several operations, many of the disc-springs showed a deterioration in their spring-constant. Instead of replacing these with new disc-springs, we added lock-washers. In either case, we never experienced a leak in the large flange seal. The snout has 6.4 cm OD and ends in a DN 63 (4.5 CF) flange, and lies partly in the hot-zone and partly in the low-temperature zone.

The low-temperature stage consists of a standard DN 63 cross connected to the large vacuum chamber on one end, and the turbomolecular pump on the other end. One port of the cross is used for a cold-cathode gauge capable of measuring 10^{-1} Pa to 10^{-9} Pa, the other port is a feedthrough for thermocouples. Two thermocouples, insulated with alumina beads, are used to monitor the interior temperature of the chamber and the components within the chamber. These are optional to the operation of the oven, but are an excellent check of the true temperature of the components under bake. A cold-cathode gauge was chosen to monitor the pressure. These gauges tend to be more durable than hot-filament gauges, and gauge accuracy isn't critical in this application.
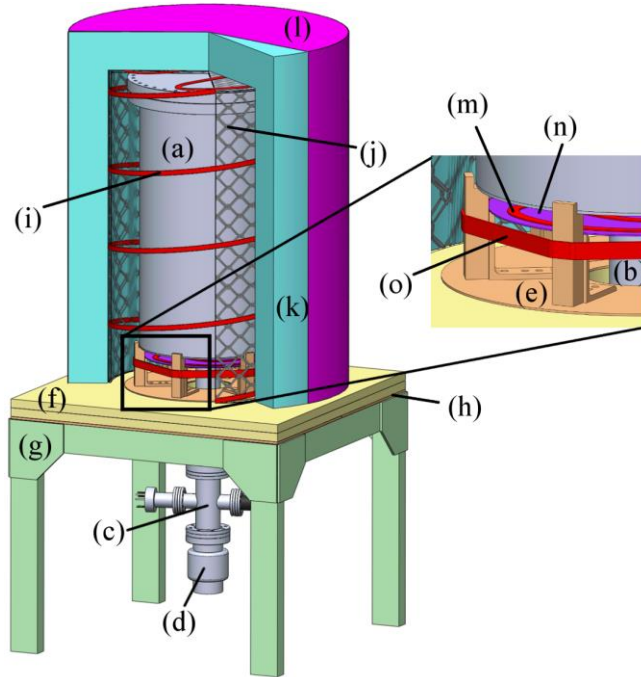

FIG. 1. (Color online) Vacuum furnace system showing cutaway view of oven shell. (a) Hot zone main chamber, (b) snout, (c) low-temperature pumping stage, (d) turbo pump, (e) supporting plate and pedestals, (f) insulating boards, (g) table support, (h) table top, (i) main heaters × 4, (j) heavy aluminum screen, (k) four-inch thick insulation, (l) foil outer shell, (m) bottom heater, (n) screen disk for bottom heater, (o) pedestal heater.

The vacuum chamber is supported on 6 pedestals made of carbon steel. These sit on a circular carbon steel plate, 3.2 mm thick, with a 10.2 cm center hole that the snout passes through. Two 5.1 cm thick square slabs of ceramic fiber (Fiberfrax®[15]) boards insulate the supporting plate from a table structure. The table legs and cross supports are constructed from rectangular extruded aluminum hardware and the table top is a 6.4 mm thick carbon-steel plate. See Figure 1.(e) – (h).

The pumping system is shown in Figure 2 and consists of a 60 L/s turbomolecular pump backed by a dry scroll-type rough pump. A thermocouple gauge between the turbo-pump and rough-pump is used to monitor the backing pressure. We also included a vent port to easily vent the system when the bake is complete. We included a valve on top of the rough-pump so that the rough-pump may be replaced during use. Once the chamber pressure is below 10^{-4} Pa, the rough pump may be disconnected (to swap out for service) for several minutes without affecting the base pressure. For laboratories where short-lived power outages are a possibility, we suggest operating the rough pump on a power back-up line, such as an uninterruptable power supply and/or using a safety valve between the rough pump and turbo that will automatically shut upon loss of power, and re-open when the rough pump starts again. Otherwise, the oven is slow to cool down both because of its insulation and because it's mass gives it significant heat capacity, and the turbomolecular pump will take time to spin-down and thus be able to maintain high-vacuum during short-lived outages.
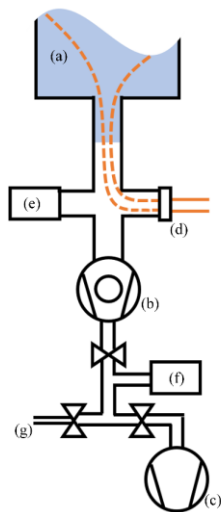
FIG. 2. (Color online) Pumping system. (a) Main chamber and snout, (b) turbo pump, (c) dry scroll-type backing pump, (d) feedthrough for internal thermocouples, (e) cold-cathode high vacuum gauge, (f) thermocouple-type low vacuum gauge, (g) vent.

B. Oven Design

The oven design is simple, inexpensive, yet effective. Heat is provided by fiberglass-insulated heat-cable (heater-tapes) rated to 750 °C. The hot-zone is heated by a total of 7 heater-tapes, each drawing about 300 W when powered. Four heater-tapes are wrapped around a cylindrical frame constructed of heavy aluminum screen which defines the oven shell. These are directly attached to the screen using steel wire. Each heater tape is paired with a thermocouple used to control the heater. These are also attached to the wire screen with steel wire. The sensing end is place near, but not directly under, the heater tape, and does not touch the wire screen. A four-inch thick layer of Fiberfrax[15] insulation blanket surrounds the screen cylinder on the sides and top, secured with wire, and a layer of aluminum foil is wrapped around the insulation. This final layer is mainly to contain the insulation fibers and hold everything neatly in place. Figure 1(i)–(l) depicts the oven shell.

The bottom of the large portion of the chamber is heated by a heater tape wound to a washer-shaped aluminum screen. The screen disk has a slot cut into it so that it can be fit around the small part of the chamber. The screen disk is slightly flexible and shaped such that it can be held in place by being wedged between the supporting legs of the chamber, and that is all that is necessary to support it. We found that the single heater tape on the screen disk was not quite enough for the bottom of the chamber, therefore we wrapped a heater-tape once around the six supporting pedestals. Both of these heater tapes were controlled using the same thermocouple sensor, which was attached to the screen disk. See Figure 1.(m)–(o) for the bottom heater assembly.

The portion of the apparatus between the turbo pump and the table-top (Figure 1.(b),(c)) was maintained between 100 °C and 125 °C during operation. We used custom designed heating boots for this, but conventional heater-tape wrapped around the components would have also sufficed.

C.   Temperature Control

Individual temperature controllers and type-K thermocouples were used to operate the four heater tapes on the main oven, and an additional controller and thermocouple operated both bottom heaters: the heater tape on the screen disk, and the heater tape on the pedestals. These individual controllers (Omega CN7223[15]) were integrated into a unit with transistors used as switches to drive a DC (direct current) relay. A schematic is shown in Figure 3. When the transistor base-emitter junction is biased through a resistor, it acts like a saturated switch which effectively grounds the negative terminal of the relay. The relay then becomes energized and this closes the contact to allow 120 Volts AC (alternating current) to energize the heater tape.
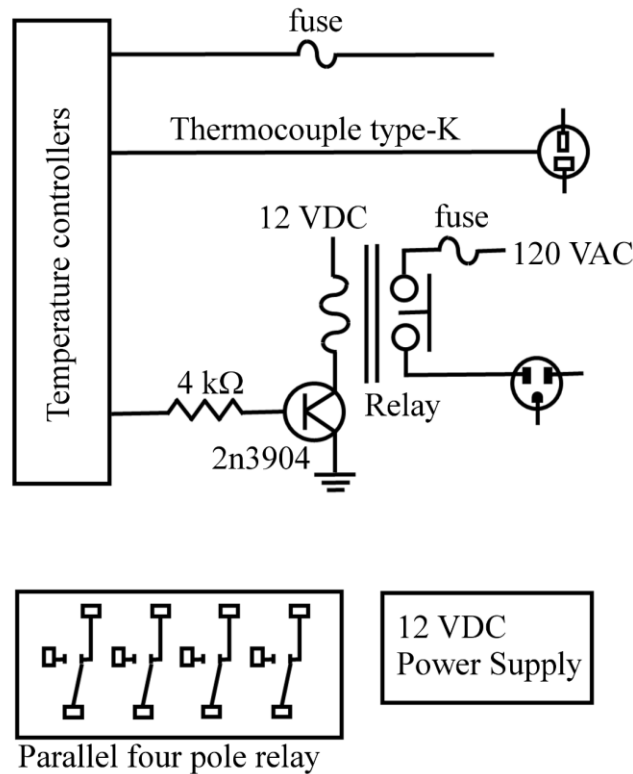

FIG. 3. Schematic diagram of a single temperature control unit used to drive a 120 V heater tape.

The transistor is required because the particular controller used was not capable of directly driving this relay; different controllers with a higher current output may be selected and this may eliminate the need for the transistor. The 12 V DC relay has four poles which are connect in parallel to improve the current capability and extend the life of the contacts under tight temperature regulation which results in rapid cycling of the current. Output fuses are added to protect the relay and heater tape due to an output overload (heat tapes shorted-out, etc.). We designed our control system to use 2.5 A fuses, and therefore all of the heater tapes used in the system were rated at ≤ 2.5 A.

IV. Operating the Furnace

The vacuum furnace can be loaded and under vacuum in about 1 hour. We use a torque wrench to secure the 16.5 CF flange to the rated torque of 35.3 Nm. We typically allowed the furnace to be evacuated overnight to reach a pressure below 10^{-4} Pa before starting the heaters.

As the chamber warms, the pressure increases. After the final temperature is reached, the chamber base pressure will stabilize and eventually begin to drop. To keep the pressure below about 10^{-3} Pa, we ramped the hot-zone temperature slowly, at a rate of 5 °C/h. The low temperature zone was heated much more quickly to about 100 °C. Figure 4 shows the pressure versus time for one of our 20 day bakes. The pressure steadily dropped over time, but after 20 days, the reduction rate was very slow. One method of determining bake times is to use the suggested times in Table 1, another way is to bake until the base pressure stops decreasing by a relevant amount.
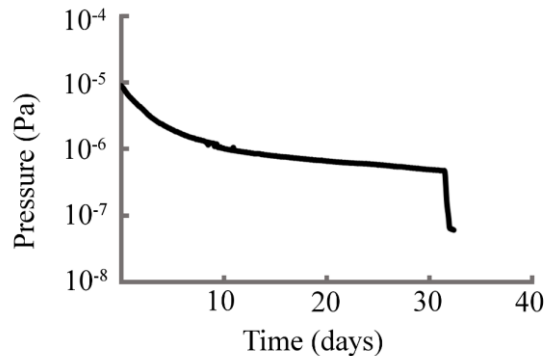
FIG. 4. Pressure versus time in the vacuum furnace during one of our early operations. The furnace was operated at 425 °C from day 0 until day 30, after which it was cooled to room temperature.

For our initial bake, in order to characterize the system and avoid any pressure or temperature anomalies, we controlled the ramp rate by manually adjusting the controller set points. Our first bake was with an empty chamber. This allowed us to debug our system with minimal risk. In addition, this allowed outgassing of the chamber itself before using it to bake other components. Subsequent bakes were computer controlled through an RS485 interface and LabVIEW software. In that case, the set points where changed by the LabView software t`o control the ramp rate.

Up to present, we have operated the furnace many times. For example, we baked a commercial gate valve with 28 mm ports for 20 days at a temperature of 420 °C. We obtained an outgassing rate for the gate valve of roughly 1.2×10^{-9} Pa L s^{-1}. This is an excellent result, it is equivalent to a partial pressure of 1.2×10^{-10} Pa when evacuated by a vacuum pump with a speed of only 10 L/s. Details of the gate-valve outgassing measurements are given in the appendix

V. SUMMARY

We have constructed a vacuum furnace capable of achieving temperatures of up to 450°C while under vacuum at pressures of 10^{-3} Pa or less. We have now operated this system many times without failure of the vacuum. This type of vacuum furnace is relatively inexpensive and within the budgets of many laboratories. For laboratories not requiring large components to be baked, our main vacuum chamber can easily be scaled down, making construction less expensive. The usefulness of the vacuum furnace is nicely demonstrated by achieving ultra-low outgassing rates with a gate valve.

Appendix: Ultralow Outgassing Gate Valve

Description of the Gate Valve and Vacuum Bake

We used the vacuum furnace described in the main article to perform a medium-temperature (400 °C to 450 °C) bake on a commercially available UHV gate-valve to reduce its outgassing rate to ultra-low levels. The gate valve was

pneumatically actuated and had 38 mm ports and a standard DN 40 flanges. It was constructed of stainless steel with copper bonnet seals and a Kalrez[15] O-ring for the gate seal. The O-ring was removed and the gate valve was disassembled before placing in the vacuum furnace. All the components external to the part of the gate valve that sees the vacuum were removed, if possible, and not vacuum baked. The pneumatic control mechanism was removed and not baked. All components (except the O-ring) that comprise or reside in the vacuum portion of the gate valve were vacuum baked. A picture of the disassembled gate valve is shown in Figure 5. The exterior of the gate valve was thoroughly cleaned with ethanol, acetone, and detergent before placing it in the vacuum furnace. There was a significant amount of lubricant on the exterior of the gate mechanism that had to be removed. The vacuum components of the gate valve were then baked at 420 °C for 20 days.

After the vacuum bake, the valve was reassembled and fitted with a new O-ring. It was stored for several weeks in atmosphere before the outgassing measurements were made. The O-ring was included in the outgassing measurements.

FIG. A1. (Color Online) Disassembled gate valve.

Description of the Outgassing Rate Measurements

A schematic of the apparatus used to measure the outgassing rate of the gate valve is shown in Figure 6. The spinning rotor gauge (SRG) and all-metal right-angle valve (RAV) had been baked between 400 °C to 430 °C for 12 days under vacuum to reduce their outgassing rate. A rate-of-rise measurement technique

was used to determine the outgassing rate of the vacuum baked gate valve (GV). This technique has been used previously by the authors and is described in detail in other publications.[1,16] Briefly, the gate valve was placed between an all-metal valve and a spinning rotor gauge as shown in Figure 6(b). The GV remained in an open position and never closed during the outgassing measurement. The entire system was evacuated and baked at 125 °C to 150 °C for 5 days to remove water. The RAV was then closed, and the pressure rise due to outgassing was recorded using an SRG over a period of several hours. The outgassing is due to three components of the system: the GV, the RAV, and the SRG. The RAV was then reopened and the process repeated to get several rate-of-rise curves. The pressure rate-of-rise of the GV+RAV+SRG system is obtained from the slope of a linear fit applied to the pressure rate-of-rise curve. The slopes from several such curves are averaged to obtain the final pressure rate-of-rise $\dfrac{dp_{\text{GV+RAV+SRG}}}{dt}$ of the GV+RAV+SRG SRG system. To obtain the outgassing rate of the gate valve
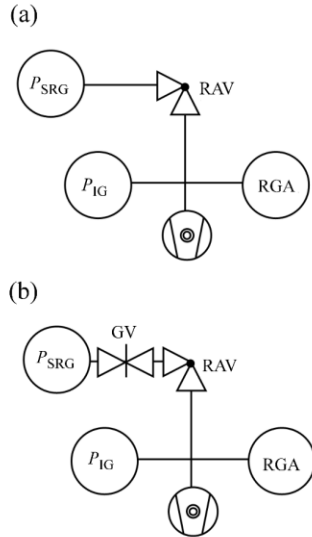
(a)

(b)

FIG. A2. Schematic of apparatus used to measure the gate valve outgassing rate. GV is the gate valve, RAV is a right-angle valve, SRG is the spinning rotor gauge, IG is an ionization gauge, RGA is a residual gas analyzer. (a) is the apparatus configured without the gate valve to measure background; (b) is the apparatus configured with the gate valve, used to determine the gate valve outgassing rate.

alone, it was necessary to obtain background outgassing rate due to the RAV+SRG. The combined outgassing rate of the SRG and right-angle valve were determined separately, as shown in Fig. 6(a). The same procedure was used to measure the background (SRG+RAV) as was used with the gate valve (GV+RAV+SRG) system. Finally, the outgassing rate of the gate valve is given by

$$q_{GV} = \frac{dp_{GV+RAV+SRG}}{dt} V_{GV+RAV+SRG} - \frac{dp_0}{dt} V_0$$

The second term in the measurement equation is associated with the background measurement. The background volume V_0 is the internal volume of the SRG+RAV and was determined approximately by dimensional measurements. The total volume $V_{GV+RAV+SRG}$ is given by V_0 plus the volume of the gate valve interior. This was determined by filling the gate valve with water. We determined the gate valve interior volume to be 148 cm^3 using this technique. Note that his may be a little less than that of a fully open gate valve because air actuation was not applied to the pneumatic gate valve during the outgassing measurement and so the gate valve was not fully open. Finally, using eq. (4), we get an H$_2$ outgassing rate of 1.2×10^{-9} Pa L s^{-1} with an estimated expanded uncertainty of 16% (i.e. a coverage factor of 2, or a 99% confidence interval). We verified the outgassing products was mostly H$_2$ gas by collecting outgassing data with the RAV closed for 24 hours and then opening the RAV and recording the subsequent gas burst using the RGA.

[1]M. Sefa, J. A. Fedchak, and J. Scherschligt, J. Vac. Sci. Technol. A **35**, 041601 (2017).

[2]Y. Tito Sasaki, J. Vac. Sci. Technol. A **25**, 1309 (2007).

[3]C. D. Park, S. M. Chung, X. Liu, and Y. Li, J. Vac. Sci. Technol. A **26**, 1166 (2008).

[4]A. A. Mamun, A. A. Elmustafa, M. L. Stutzman, P. A. Adderley, and M. Poelker, J. Vac. Sci. Technol. A **32**, 021604-1 (2014).

[5]K. Battes, C. Day, and V. Hauer, J. Vac. Sci. Technol. A **33**, 021603-1 (2015).

[6]G. Lewin, *Fundamentals of Vacuum Science and Technology* (McGraw-Hill, New York, 1965), pp. 24–33.

[7]J. Crank, *The Mathematics of Diffusion*, 2nd ed. (Clarendon, Oxford, 1975),

[8]*Handbook of Vacuum Technology*, edited by K. Jousten (Wiley-VCH Verlag, Weinheim, 2008), pp. 237–239.

[9]Y. Ishikawa, V. Nemanic, Vacuum **69,** 501–512 (2003).

[10]M. Li and H. F. Dylla, J. Vac. Sci. Technol. A **11**, 1702 (1993); doi: 10.1116/1.578482.

[11]R. Calder and G. Lewin, Brit. J. Appl. Phys. **18**, 1459 (1967).

[12]D. M. Grant, D. L. Cummings, and D. A. Blackburn, J. Nucl. Mater. **149**, 180 (1987).

[13]R. F. Berg, J. Vac. Sci. Technol. A **32**, 031604 (2014); doi: 10.1116/1.4869962,

[14]Y. Ishikawa, T. Yoshimura, J. Vac. Sci. Technol. A**13**, 1847 (1995).

[15]Commercial materials and equipment are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

[16]M. Sefa, Z. Ahmed, J. A. Fedchak, J. Scherschligt, N. Klimov, J. Vac. Sci. Technol. A **34**, 061603 (2016). doi: 10.1116/1.4965304