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Measurement of outgassing rates of steels

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Abstract:	Steels are commonly used materials in the fabrication of vacuum systems because of their good mechanical, corrosion, and vacuum properties. A variety of steels meet the criterion of low outgassing required for high or ultrahigh vacuum applications. However, a given material can present different outgassing rates depending on its manufacturing process or the various pretreatment processes involved during the fabrication. Thus, the measurement of outgassing rates is highly desirable for a specific vacuum application. For this reason, the rate-of-pressure rise (RoR) method is often used to measure the outgassing of hydrogen after bakeout. In this article, a detailed description of the design and execution of the experimental protocol involved in the RoR method is provided. The RoR method uses a spinning rotor gauge to minimize errors that stem from outgassing or the pumping action of a vacuum gauge. The outgassing rates of two ordinary steels (stainless steel and mild steel) were measured. The measurements were made before and after the heat pretreatment of the steels. The heat pretreatment of steels was performed to reduce the outgassing. Extremely low rates of outgassing (on the order of 10−11 Pa m3 s−1 m−2) can be routinely measured using relatively small samples.
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SHORT ABSTRACT:
A protocol for the measurement of outgassing rates of hydrogen from ordinary steel vacuum

chambers using the rate-of-pressure rise method is presented.

LONG ABSTRACT:
Steels are commonly used materials in the fabrication of vacuum systems because of their good mechanical, corrosion, and vacuum properties. A variety of steels meet the criterion of low outgassing required for high or ultrahigh vacuum applications. However, a given material can present different outgassing rates depending on its manufacturing process or the various pretreatment processes involved during the fabrication. Thus, the measurement of outgassing rates is highly desirable for a specific vacuum application. For this reason, the rate-of-pressure rise (RoR) method is often used to measure the outgassing of hydrogen after bakeout. In this article, a detailed description of the design and execution of the experimental protocol involved in the RoR method is provided. The RoR method uses a spinning rotor gauge to minimize errors that stem from outgassing or the pumping action of a vacuum gauge. The outgassing rates of two ordinary steels (stainless steel and mild steel) were measured. The measurements were made before and after the heat pretreatment of the steels. The heat pretreatment of steels was performed to reduce the outgassing. Extremely low rates of outgassing (on the order of 10^{-11} Pa m^3 s^{-1} m^{-2}) can be routinely measured using relatively small samples.

INTRODUCTION:
Steels are routinely used in construction because of their good mechanical properties. Certain steels (ferrous steels, in particular) are preferred materials for applications involving vacuum. Depending on the type and grade, these steels have sufficiently low outgassing rates essential for high vacuum (HV, $10^{-7} < p < 10^{-5}$ Pa) or ultrahigh vacuum (UHV, $10^{-9} < p < 10^{-7}$ Pa) systems. Further, extensive research has been conducted toward the development of special pretreatment procedures that reduce outgassing[1-3]. The pretreatment measures are designed to minimize the pumping investment or to improve the vacuum from HV to UHV or from UHV to extreme-high vacuum ($p < 10^{-10}$ Pa).

Although many practical methods have been proposed to reduce the outgassing rate of ferrous steels, recent methods are focused on reducing the time and temperature required to obtain a lower outgassing rate. Heat treatment at 350 °C–450 °C rather than vacuum firing at 800 °C–950 °C, is a good example of this approach.[1,4,5] Furthermore, choosing the ideal material for a specific vacuum application is critical; for example, selecting a ferritic material with a very low outgassing rate for use in magnetic field shielding.[6,7]

During such investigations, precise measurement of the outgassing rate is a prerequisite for the screening of candidate materials or verifying the effectiveness of various pretreatment procedures.[8,9] The most common experimental techniques used for the measurement of outgassing are the throughput and rate-of-pressure rise methods.[10] Recently, various experiments have been conducted to measure the hydrogen outgassing rate based on the ROR method using spinning rotor gauge (SRG).[1, 11-13] The RoR method using SRG is highly suitable for measuring very low hydrogen outgassing rates that often limit the lowest pressure achievable in a vacuum system made of steel. This is because the SRG has negligible pumping or outgassing action. Further, the SRG also has excellent accuracy and good linearity in high vacuum and

ultra-high vacuum range.[14]

Given that the published literature on RoR experiments is limited, it is worthwhile to describe the experimental details to develop a deeper understanding of the method. In this video article, we describe in detail the process of setting up the experiment and provide detailed instructions to perform outgassing measurements using the RoR method. To demonstrate the efficacy of the method, the outgassing rates of two commonly used steels (stainless steel 304 and mild steel S20C) were measured before and after a preheat treatment to reduce the hydrogen outgassing rate. The pre- and post-treatment values were compared. Typical experimental results using a rather simple setup are presented to demonstrate the efficacy of the method optimized for evaluating low hydrogen degassing rates.

PROTOCOL:

Caution: Please follow all appropriate safety practices while assembling the equipment and sample chambers. Please wear personal protective equipment (safety glasses, gloves, safety shoes, etc.).

1. Fabrication of a sample vacuum chamber

1.1) Design and fabrication of the vacuum chamber

1.1.1) Prepare and submit design drawings to a commercial vendor or an in-house machine shop for manufacturing the sample vacuum chamber. A representative example of the design drawing for a vacuum chamber made of S20C steel is shown in Figure 1. The chamber designed in this experiment is very basic and is commonly employed by vacuum companies.

Note: The manufacturer should have a basic knowledge of UHV systems.

1.1.2) Inspect all dimensions to ensure compliance with the drawing.

1.1.3) After shaping (machining) the vacuum chamber, cover the end CF flanges with plastic to prevent damage during transportation.

1.2) **Cleaning**

Note: Follow local environment, health, and safety regulations during chemical cleaning. Wear personal protective equipment. Handle the parts using vinyl gloves. Do not touch the parts with bare hands.

1.2.1) Clean the steel parts following the UHV cleaning procedure for steels. A typical cleaning procedure is described below.

1.2.2) Degrease the parts using a solvent, such as acetone, at room temperature for 5 min.

1.2.3) Clean the parts in an ultrasonic bath for 20 min using a BN- cleaner (pH 13).

1.2.4) Rinse the parts with tap water for 10 min, followed by a thorough rinse with deionized

water for 20 min.

1.2.5) Rinse thoroughly with alcohol and blow dry using dry nitrogen gas.

1.2.6) Wrap the parts in clean, lint-free paper and allow the parts to air-dry for one day.

1.3. **Welding**

Note: Do not touch the parts with bare hands. The welder should be trained in UHV welding.

1.3.1) Place the parts on a welding bench.

1.3.2) Preassemble the parts and align the parts as per the design drawing.

1.3.3) Backpurge with argon gas (5 L/min) to prevent corrosion during welding.

1.3.4) Tack weld the end flanges using the Tungsten inert gas (TIG) weld technique (argon flow rate: 8-9 L/min).[15]

1.3.5) Weld the end flanges completely using the TIG technique and a turning jig. Allow the heat-affected zone to cool down to room temperature. Stop the argon gas flow.

1.4. **Leak test**

1.4.1) Seal one end of the chamber with a CF blank flange.

1.4.2) Connect the other end to a helium leak detector (HLD).

1.4.3) Pumpdown the sample vacuum chamber using the HLD.

1.4.4) Place the heat-welded seam in a vinyl bag and fill the bag with helium gas.

1.4.5) Measure any change in the helium level. Ensure that the chamber is leak-proof. The helium leak rate should be $<1 \times 10^{-11}$ Pa m^3 s^{-1} (1×10^{-10} mbar l s^{-1}).

1.4.6) If no leak is observed, vent the chamber. Otherwise, reweld the vacuum chamber after venting (repeat steps 1.3.3 through 1.4.5).

2. Fabrication of the oven

2.1 Prepare and submit the design drawings to a vendor or an in-house machine shop for manufacturing the oven. Refer to the picture shown in Figure 2.

2.2 Procure the required parts and equipment described in Temperature Control of List of Specific Materials/Equipment.

2.3 Connect the cooling line.

2.4 Supply cooling water to a chiller. Run the chiller and check for water leaks. Stop the chiller.

3. Experimental setup for ROR measurements

3.1 Collect the required vacuum equipment/hardware specified in the List of Specific Materials/Equipment. The test setup primarily consists of a SRG, a residual gas analyzer (RGA), a turbomolecular pump (TMP) equipped with a roughing pump (RP), an all-metal angle valve (AV_{Ch}), a tee (CF35), and a reducer (CF35 to CF63). Additional items may include a helium leak detector and an UHV gauge, as listed in the List of Specific Materials/Equipment. The AV_{Ch} should have a rotatable flange on the seat (sealing) side to level off the SRG.

3.2 Collect the wrenches (M6 and M8), copper gaskets (CF35 and CF63), and bolts/nuts (M6 and M8) required for the assembly.

3.3 Use an industrial level meter to assemble the SRG.

3.4 Handle the parts using vinyl gloves. Do not touch the surface that is exposed to vacuum with bare hands. Wear safety shoes.

3.5 Assembly of the experimental apparatus

3.5.1 Assemble the vacuum components sequentially using copper gaskets from the pump side to the sample side, as shown in Figure 3. Always place the valve seat side facing the sample chamber. Tighten all flange joints face-to-face with a copper gasket (CF35) using a wrench (M6), except the joint between the sample chamber and the chamber isolation valve (AV_{Ch}).

3.5.2 Adjust the SRG flange assembly and the sample chamber so that the axis of the SRG head is vertical using the level meter; within ±2° (max) (Figure 4). Tighten the flange joint between the sample chamber and the AV_{Ch}, face-to-face, while maintaining the SRG flange's level. Refer to the SRG user's manual for detailed instructions.

3.5.3 Connect the RP and the HLD with isolating valves (AV_{Ro}, AV_{HLD}) to the clamp flange (KF) port of the exhaust end of the TMP.

Caution: Ensure that there is no mechanical shock to the SRG flange assembly or rotor.

3.6 Leak test

3.6.1 Turn on the HLD and wait until the detector is ready. Open the AV_{HLD} and close the AV_{Ro}.

3.6.2 Pump down the setup using the HLD. Refer to the HLD manual for the correct operating procedure. Wait for ~30 min to pump out the residual helium gas from the setup. Ensure that the helium level is within the minimum detectable limit of the HLD.

3.6.3 Spray helium gas through the leak-test groove on the flanges.

3.6.4 Measure any change in the helium level. Ensure that the chamber is leak tight. The helium leak rate should be $<1 \times 10^{-11}$ Pa m^3 s^{-1} (1×10^{-10} mbar l s^{-1}).

3.6.5 In case of leakage from the flanges, retorque the flanges.

3.6.6 If no leaks are found, stop the leak testing and vent the vacuum system. Open the AV_{Ro} and close the AV_{HLD}.

4. Measurement of outgassing rates

4.1. Pump down procedure

4.1.1) Pump down the vacuum system by switching on the TMP and the RP at the same time.

4.1.2) While the pumping process is on, collect the necessary items for bakeout; electrical heater tapes, heater controllers, handheld multimeter, Al foil, and temperature sensors/cables.

4.2. Bakeout procedure

4.2.1) Remove the SRG head from the flange assembly. Wrap vacuum components (between the SRG flange assembly and the inlet flange of the TMP) in band heaters.

4.2.2) Check and ensure that there is no electrical short circuit between the heaters and the vacuum parts using handheld multimeter.

4.2.3) Connect the heaters to the respective controllers and wrap the chamber in Al foil.

4.2.4) Raise the temperature to 150 °C at a ramp rate of 1 °C–2 °C/min.

4.2.5) Hold the chamber at 150 °C for 24–48 h using bakeout program controller. Maintain the temperature of the RGA electronics under 50°C using cooling fan.

4.2.6) Degas each of the RGA filaments by electron bombardment for at least 5 min.

4.2.7) Measure the RGA spectrum from 1 to 50 m/e to ensure that the H_2O peak (m/e = 18) is less than one-half of the H_2 peak (m/e = 2). If not, continue the bakeout.

4.2.8) Allow the system to cool down to room temperature at a ramp rate of 1 °C–2 °C/min. Check for leaks referring to the RGA spectrum measured during the cool down.

4.2.9) Analyze the residual gas in the sample chamber. Measure the RGA spectrum. Close the AV_{Ch} and measure the RGA spectrum again. The RGA spectrum of the sample chamber corresponds to the difference between the spectra acquired before and after closing the AV_{Ch}.

4.2.10) Verify that the sum of all impurity gases, such as H_2O, CO, and CO_2, is below 5%; otherwise, repeat the bakeout again.

4.3. Operating the SRG

Note: Proper operation of the SRG is very important. Refer to the SRG operating manual for instructions.

4.3.1) Assemble the SRG head on the SRG flange assembly.

4.3.2) Ensure that the axis of the SRG head is within ±2° (max) (Figure 4). Use a level meter for reference.

4.3.3) Start the SRG and wait for stabilization of the residual drag, a pressure-independent signal of the SRG, which usually takes a few hours.

4.3.4) Enter the proper input parameters, such as gas (H_2), temperature (24 °C), and measurement interval (10 s).

4.4. Procedure for initiating temperature control

4.4.1) While waiting for the signal to stabilize, stabilize the temperature of the sample. Switch on the chiller to run cooling water through the system. Set the fluid temperature to 15 °C.

4.4.2) Start the heater controller for the sample. Set the target temperature to 24 °C. Wait for the temperature to stabilize within ±0.1 °C after closing the door of the oven.

4.5. Procedure for acquiring the SRG signal

4.5.1) Verify that the variation of the offset value of the signal is within $\pm 1 \times 10^{-9}$ Pa/s; otherwise, disassemble the SRG from the system and change the rotor or flange assembly, then repeat 3.5-4.4. (If this is not possible, acquire the slope of the offset for 8–24 h. This will be subtracted from the measured outgassing rate data.)

4.5.2) Check the signal level provided by SRG controller; it should be at least −10 dB. Ideally, it should be between 0 and 6 dB. However, values of up to 12 dB are acceptable. If the signal is >14 dB, stop the SRG operation. Detach the head and heat the thimble to 200°C. Repeat the entire operation starting from step 4.5.1.

4.5.3) Check the damping level provided by SRG controller; the optimum value is between −35 and −60 dB, which is normally satisfied in the system using TMP and scroll pump which is laid on a rubber pad. Otherwise, stop all running equipment and remove sources of any mechanical vibration.

4.6. Acquiring the RoR data

4.6.1) Gently close the AV to start the pressure buildup. Be careful not to subject the SRG to a mechanical shock.

4.6.2) Close the door of the oven and acquire pressure data for 8–24 h using a computer.

4.6.3) Precheck the measured data to verify that the variation in temperature is within ±0.1°C after stabilization and that the pressure rise is linear within 10% error. If these criteria are met, stop the measurement. Otherwise, continue to measure until the pressure rise becomes linear within 10% error for at least 16 h.

4.6.4) Turn off all equipment.

4.7. Calculation of the outgassing rate

4.7.1) Select the pressure set after the temperature stabilization.

4.7.2) Fit the pressure rise data using linear least squares fitting and calculate the slope. The slope d*P*/d*t* is the measured rate of pressure rise after the valve is closed.

4.7.3) Calculate the outgassing rate, q (H_2 equivalent), using the equation

$$q = (V/A)\,(dP/dt) \quad [\text{Pa m}^3\ \text{s}^{-1}\ \text{m}^{-2}],$$

where *V* is the volume of the sample chamber (m^3) and *A* is the geometrical surface area of the chamber (m^2).

REPRESENTATIVE RESULTS:

As expected, the residual gas after the bakeout was mostly hydrogen.[7] The pressure rise measured using the SRG was linear over a long period of time (Figure 5). Thus, the readsorption effect might be negligible and the intrinsic outgassing rate (*q*) for the steels tested in this study can be evaluated using the RoR method.[10] The measured pressure rise data was analyzed using the linear least squares fitting method. The outgassing rates of the sample chambers were determined from the slope (Figure 5).

The measured outgassing rate for untreated STS304 steel (sample 1) was 5.1×10^{-9} Pa m^3 s^{-1} m^{-2}, which is consistent with the reported values.[1-7] A ~22-fold reduction in outgassing was achieved with a medium-temperature heat pretreatment in vacuum furnace for 36 h at 450 °C (Table I). This demonstrates the effectiveness of heat pretreatment in reducing the hydrogen outgassing rate of stainless steel, further indicating that the degassing of hydrogen during the heat treatment is governed by a bulk diffusion mechanism. While the outgassing rates for untreated mild steels were very low (<~4×10^{-10} Pa m^3 s^{-1} m^{-2} (samples 2 and 3), the outgassing rates were second to the rates of stainless steels after intensive heat treatment.[1,3,4] In addition, the outgassing rate for mild steel (sample 2) decreased by only 66% following heat treatment at 850 °C for 12 h in vacuum furnace (Table I), and no significant reduction in outgassing was observed.

The findings from these measurements strongly suggest that the difference in outgassing between stainless steels and mild steels can be attributed to the differences in the steel making processes, and in particular, the secondary metallurgy processes, during which impurity gases are extracted.[16] A vacuum degassing process, such as the Ruhrstahl–Hausen process, is generally employed during the production of mild steels. Thus, mobile hydrogen is completely

degassed during the steel-making process. In contrast, mixed-gas refining, such as argon–oxygen decarburization at atmospheric pressure, is primarily used during the production of stainless steels. This provides a reasonable explanation for the observed lower hydrogen outgassing rate of untreated mild steel compared to untreated stainless steel.[7]

Figure 1. **Sample chamber.**
An example of a vacuum chamber made of steel. A steel cylinder and two end plates with flanges (CF35) were directly welded. The area of the inner surface is ~2,400 cm^2 and the volume is ~7 l.

Figure 2. **Oven.**
A bird-eye view of the oven, together with the experimental setup and the sample vacuum chamber. A simple, box-shaped oven is adequate for this experiment.

Figure 3. **Experimental setup.**
A schematic of the experimental setup for the measurement of outgassing rates using the RoR method. A cylindrical sample chamber is placed inside a simple oven and pumped through an all-metal angle valve (AV). After bakeout, the SRG pickup head is attached and is switched on. The active temperature control is then initiated.
CF: flange, KF: clamp flange, RGA: residual gas analyzer, and TMP: turbomolecular pump.

Figure 4. Mounting of the SRG head on the vacuum chamber.
The axis of the SRG head should be vertical within ±2° (max) as shown. A level meter should be used to align the head.

Figure 5. Representative raw RoR data (dotted line) measured using the SRG after bakeout. The solid line (in blue) is the least-squares fit of the data. The slope of the curve corresponds to an outgassing rate of 4×10^{-10} Pa m^3 s^{-1} (H_2 equivalent). The solid line at the bottom (in red) shows the measured temperature variation, which is within ±0.1 °C.

Figure 6. Modification of a commercial SRG flange.
The flange is thinned as per the design drawing and heat treated at 400 °C for 72 h (*Fo* ~6.4) to reduce outgassing. The measured gas load on the SRG flange, together with the angle valve (from the surface exposed to the SRG side), was 8.3 (± 0.1) × 10^{-12} Pa m^3 s^{-1}, which amounts to 15%–28% of the outgassing from the samples after heat treatment (Table I). This background gas load must be subtracted from the total gas load on the sample vacuum chamber.

Table 1: Measured outgassing rates.
The rates (*q*) are total outgassing rates, in hydrogen equivalent units, and measured after an *in situ* bakeout at 150°C for 48 h. *Fo* represents the intensity of heat treatment (dimensionless); $Fo = 4Dt/d^2$, where *D* is the diffusion constant at the heat pretreatment temperature and *d* is the thickness of the chamber.[12,13]

DISCUSSION:

Numerous methods for the measurement of outgassing rates have been reported in the literature. Experimental methods include the throughput, conductance modulation, two-path, RoR, and variations of these methods. However, no one method is ideal for obtaining the necessary outgassing data.[10] The RoR method using SRG, however, became the method of choice for measurement of low outgassing materials.[11-13] SRG[17] is often used as a secondary standard in high vacuum systems without erroneous pumping or outgassing action. The RoR method using SRG is particularly suitable for measuring hydrogen outgassing at room temperature after bakeout. In contrast, other UHV gauges can cause significant errors generated by the gauges themselves. An extractor gauge, for instance, is a type of UHV ion gauge with low outgassing. However, the gauge itself and the surrounding walls generate a gas load as large as 1×10^{-11} Pa m^3 s^{-1}.[18] This amounts to 14%–30% of the gas load from the samples following the heat treatment (Table 1).

The outgassing from SRG flange (CF35) must be taken into account when measuring samples with a small area. Though small in size, the hydrogen outgas from the flange is as large as 7.5×10^{-12} Pa m^3 s^{-1} and the flange is too thick to degas hydrogen without firing. This amounts to approximately 12%–26% of the outgassing from the samples after heat treatment (Table I). Thus, this systematic error in the measured gas load must be corrected. Thinning the commercial SRG flange (Figure 6) and performing an appropriate heat treatment in vacuum will help minimize the outgassing. However, in a real situation, the combined background gas loads from the SRG flange assembly and the angle valve must be measured and corrected before the main measurements. Furthermore, using a thimble without a flange that is directly welded on the sample chamber is another good technique for measuring outgassing from very small samples (surface area <500 cm^2) using a pinch-off copper tube instead of an angle valve.[12,13]

In addition, proper operation of the SRG is crucial to ensure the precise measurement of extremely low outgassing rates. The pressure range that the measurement is taken over is from 10^{-8} Pa to 10^{-3} Pa. The temperature control is especially important. A slow, constant temperature change of 0.14°C/h causes a 10% error in the measured values.
Thus, the active temperature control unit, comprising a copper cooling coil at a constant temperature of 15°C and a proportional-integral-derivative controlled heater, was deployed in this study. The temperature was stabilized to within ±0.1°C during the measurements (Figure 5). At this temperature stability, RoR measurements as low as 1×10^{-3} Pa/day could be made in a single day.

Fabrication of individual parts of the sample chamber with the same thickness is another important factor affecting the outgassing rate following heat treatment (Figure 1). As stated earlier, bulk diffusion governs the degassing of mobile hydrogen, at least in the initial stage of heat treatment. In the RoR method, the outgassing rate depends not only on the duration of the heat treatment but also strongly on the sample thickness.[19] Thus, reporting the outgassing rate with respect to the intensity of heat treatment (For example, $Fo = 4Dt/d^2$, Table 1)[12,13] is recommended; simply reporting the duration of the heat treatment is misleading with respect to the intensity of heat treatment.

Using the protocol reported in this study that uses commercial parts to the extent possible, an outgassing rate lower than 1×10^{-10} Pa m^3 s^{-1} m^{-2} can be routinely measured from vacuum chambers made of steel. With careful design and under optimum experimental conditions, such a low rate can be measured from samples with a relatively small area. The surface area of the vacuum chamber used in this study is only 2,400 cm^2, which is one-third of the surface area of the chambers (7,600 cm^2) used in previous experiments for making similar measurements.[5] The equipment identified in this protocol is specific to the most suitable commercial ones. It should be noted that with a proper, carefully designed experimental setup and protocol, other equipment or methods can be used for the same purpose.

Furthermore, although ferrous steels were used in this video protocol, the same techniques are applicable to the measurement of outgassing rates from numerous other materials that can be used for the fabrication of vacuum chambers.

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The authors have no conflict of interest to declare.

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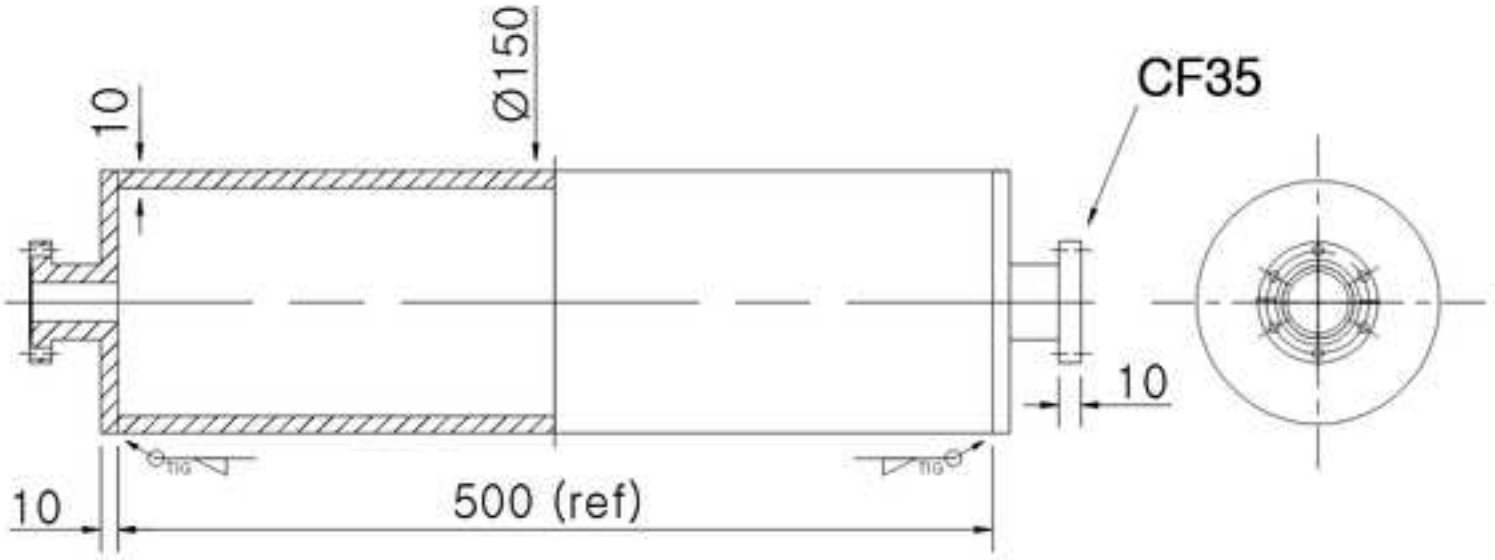

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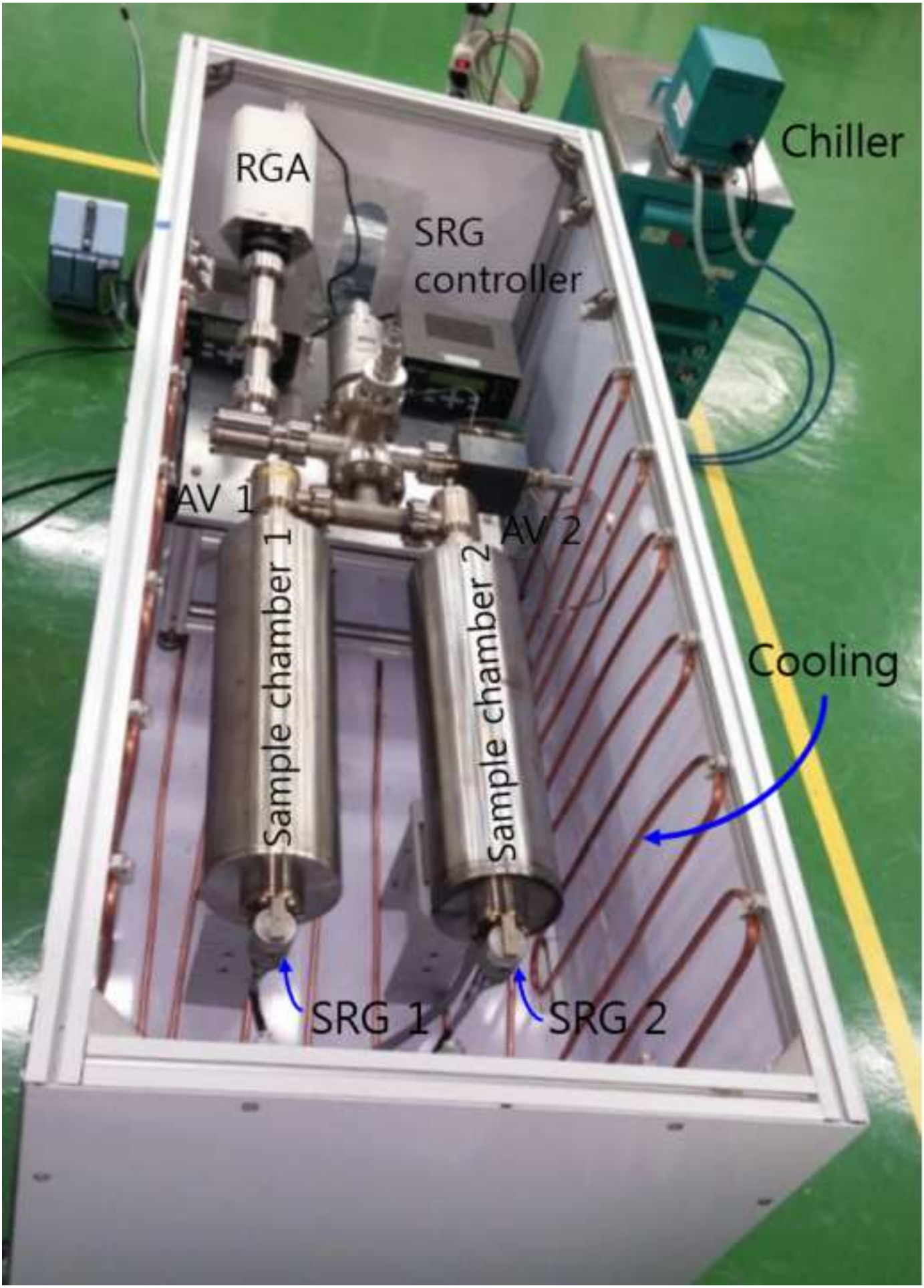

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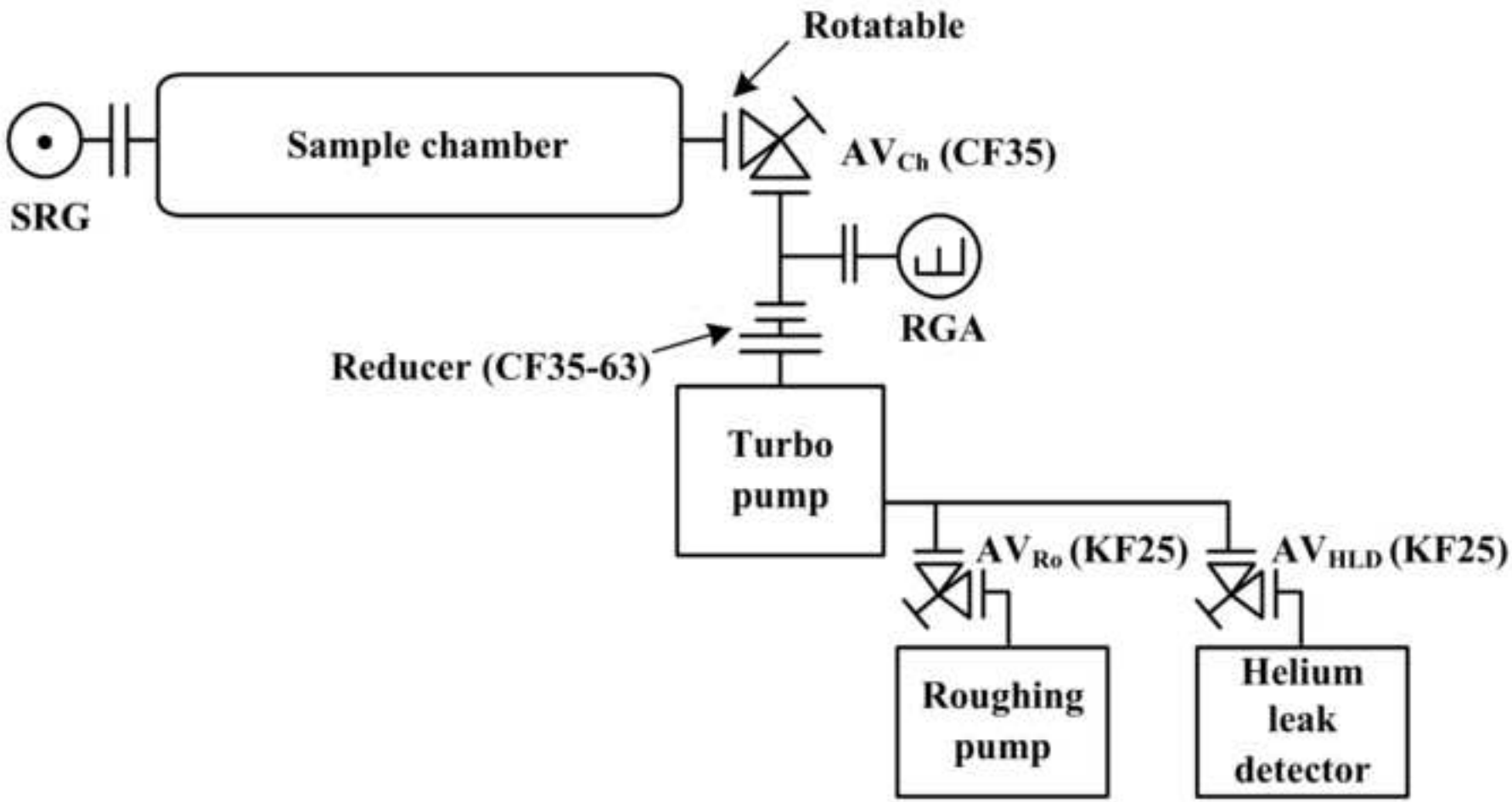

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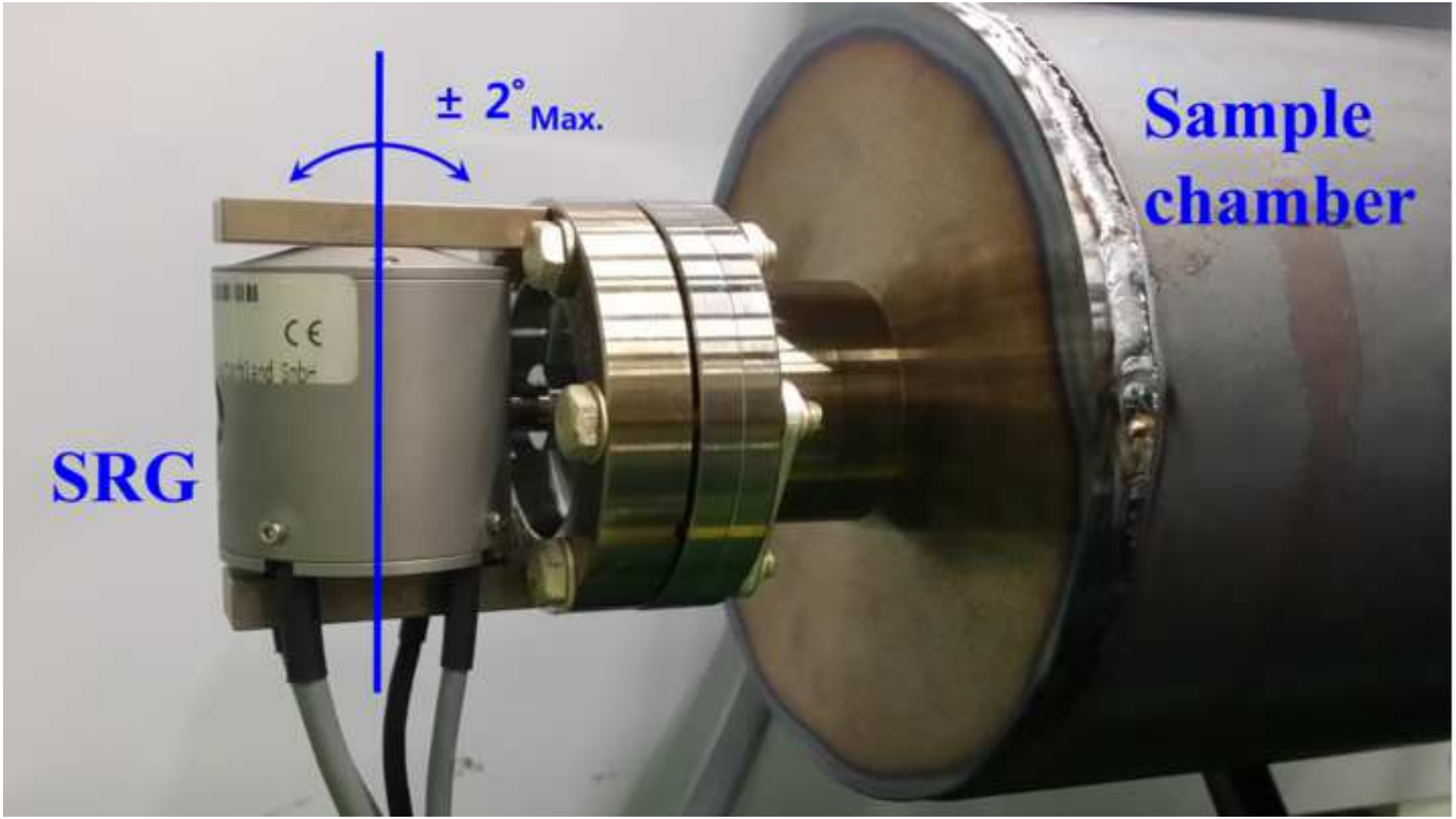

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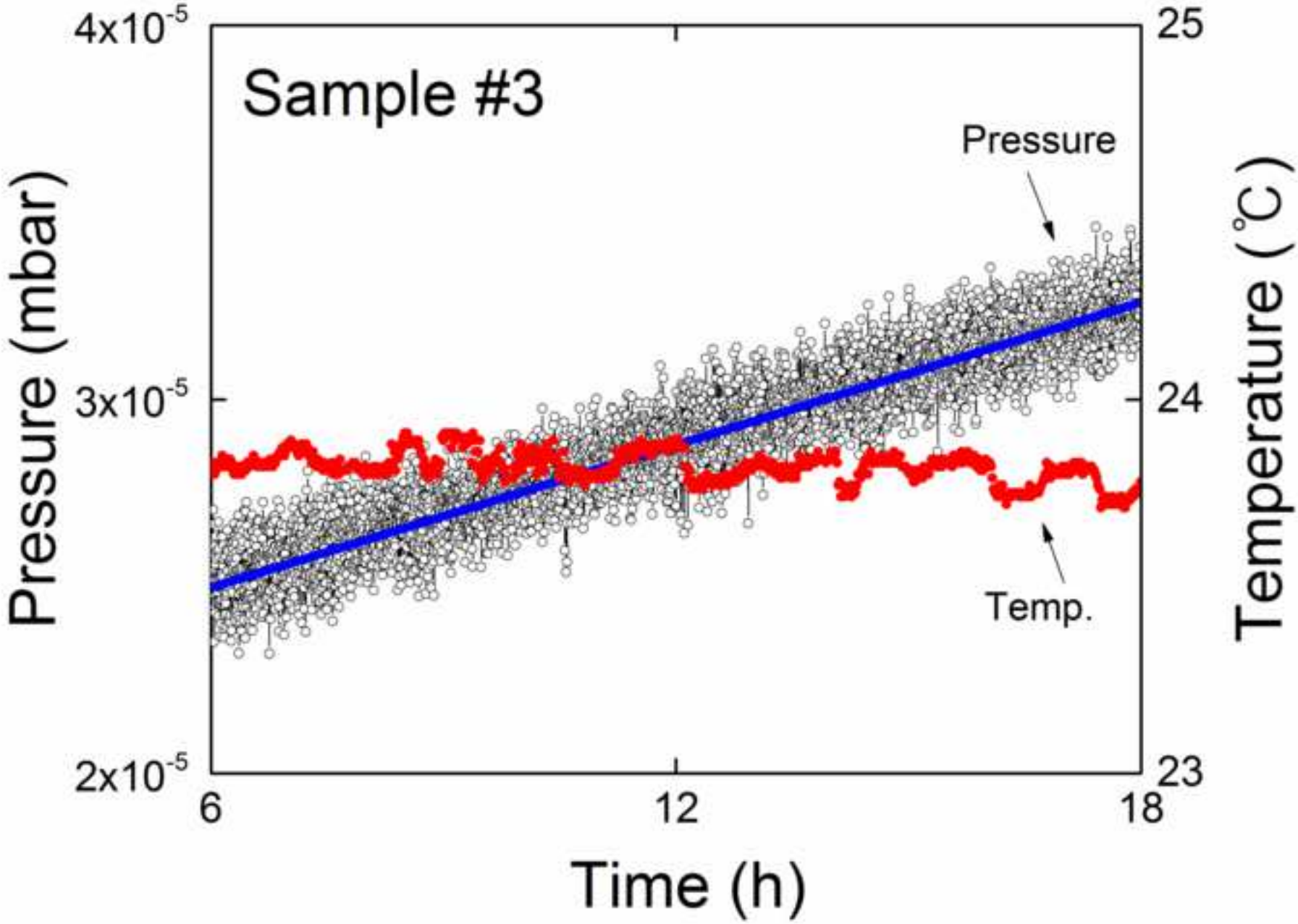

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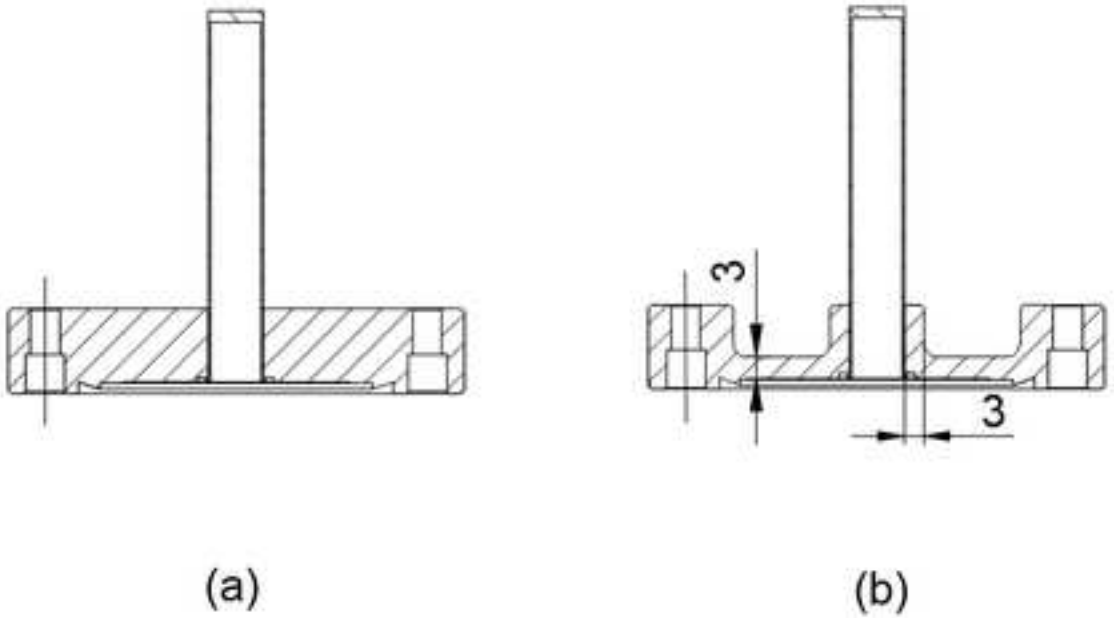

Table Ⅰ.

Material	Sample no.	*d* (mm)	D ((cm2/s)	Heat treatment
Stainless steel (304)	1	3.3	5×10^{-7}	— 450 ℃, 36 h
Mild steel (S20C)	2	10	1×10^{-4}	— 850 ℃, 12 h
	3	10		—

Fo	q (Pa m^3 s^{-1} m^{-2})
	5.1×10^{-9}
2.4	2.3×10^{-10}
	2.6×10^{-10}
17	8.8×10^{-11}
	4.0×10^{-10}

Excel Spreadsheet- Table of Materials/Equipment

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Materials

Name	Company	Catalog Number	Comments
Sample chamber			
Stainless steel, 304	POSCO (www.posco.co.kr)		
Mild steel, D3752	Xiangtan Iron&Steel co.,LTD (http://www.hnxg.com)		
Mild steel, D3752	SeAh Besteel (www.seahbesteel.co.kr)		
Cleaning			
Cleaning bath	Samill IDS		Ultrasonic cleaning, heating, timer, concentration control
Acetone	Samchun Chemical (www.samchun.com)	A1759	HPLC GRADE (99.7%)
Tekusolv	NCH Co. (www.nch.com)	0368-0058J	Solvents
BN cleaner	Henkel surface technologies (na.henkel-adhesives.com)	6610263775	Akkaline, pH 13
Ethanol	Fisher Scientific (www.fishersci.com)	A995-4	HPLC Reagent(99.9%)
Deionized water (Electro deionizer SYSTEM)	A.T.A (www.atagroup.co)	EDI SYSTEM	
Liquid N_2 gas	Hanyoung (www.gasmaster.co.kr)	B/T 176 L	LN2 dewar, purity 99.999%
Welding			

Tungsten Inert Gas wedling machine	Thermal Arc (www.victortechnologies.com/thermalarc)	400GTSW	Ar gas prefllow&postflow 8 liter/min, backflow 5 liter/min
turning jig	Vactron (www.vactron.co.kr)	Made to order	Made to order
Ar gas	Lindekorea (www.lindekorea.com)		Purity 99.999%
Leak test			
Leak detector	Adixen (www.adixen.fr/en/)	ASM380	Pumping Speed(air): 9.7 l/s
He gas	Lindekorea (www.lindekorea.com)		Purity 99.999%
Vacuum equipment			
Spinning rotor gauge	MKS Instruments (www.mks.com)	SRG-3	Controller, head, and thimble set
Industrial level meter	MKS Instruments (www.mks.com)	SRG-3	For SRG assemble ± 1˚
Oscilloscope	Tektronix (www.tek.com)	TDS2012B	
Residulal gas analyser	Balzers	QMA200	m/e 0-100
TMP(HiPace 80)	Pfeiffer Vacuum (www.pfeiffer-vacuum.com)	PMP03941	Pumping Speed(N_2): 67 l/s
Scroll pump	Anest Iwata (www.anest-iwata.co.jp)	ISP 90	Pumping Speed(Air): 1.8 l/s
All-metall easy close angle valve(CF35)	VAT Inc. (www.vatvalve.com)	54032-GE02-0002	Rotatable flange
Angle valve(KF25)	MDC Vacuum Inc. (www.mdcvacuum.com)	KAV-100	

Five-Way Crosses	MDC	Made to order	CF4-1/2 Spool-rotatable 1-way to CF2-3/4 Nipple 3ea, Vacuum degassed at 400℃ for 3 days
Reducing Tees	MDC	Made to order	CF4-1/2 Flange to CF2-3/4 Tees(Half flange), Vacuum degassed at 400℃ for 3 days
Temperature control			
Chiller	JEIO Tech (www.jeiotech.com)	RW-2025G	
Cooling line	LS Metal (www.lsmetal.biz)	C1100	Level Wound Coil, Diameter 10mm
Heater controllers	HMT	Made to order	Bakeout program controller
Electrical heater tapes	Brisk heat (www.briskheat.com)	BIH101080L	
Thermocouple(K type)	miraesensor (www.miraesensor.com)	MR-2290	
Handheld multimeter	Saehan (www.saehan.co.kr)	3234	
Data recorder(Temp.)	Yokogawa (www.yokogawa.com)	GP10-1E1F-UC10	

Materials

Name	Company	Catalog Number
Acetone	Fisher Scientific	A/0520/17
Isopropanol	Fisher Scientific	P/7500/15
Electron Beam resist	Marubeni Europe plc.	ZEP520A
Xylene	Fisher Scientific	X/0100/17
Microposit S1818 G2	Chestech Ltd.	10277866
Microposit Developer MF-319	Chestech Ltd.	10058721
Hydrofluoric Acid	Fisher Scientific	22333-5000
Microposit 1165 Remover	Chestech Ltd.	10058734
Sulphuric Acid	Fisher Scientific	S/9120/PB17
Hydrogen Peroxide	Fisher Scientific	BPE2633-500

Equipment

Silicon-on-Insulator wafer	Soitec	G8P-110-01
Diamond Scribe	J M Diamond Tool	HS-415
Microscope slides	Fisher Scientific	FB58622
Beakers	Fisher Scientific	FB33109
Tweezers	SPI Supplies	PT006-AB
Ultrasonic Bath	Camlab	1161436
Spin-Coater	Electronic Micro Systems Ltd.	EMS 4000
Pipette	Fisher Scientific	FB55343
E-beam Lithography	Raith Gmbh	Raith 150
Reactive Ion Etching System	Proprietary In-house Designed	--
UV Mask Aligner	Karl Suss	MJB-3
ASE source	Amonics	ALS-CL-15-B-
Single mode fibers	Thorlabs	P1-SMF28E-
3 dB fiber splitters	Thorlabs	C-WD-AL-50-H-
Aspheric lenses	New Focus	5720-C

XYZ stages	Melles Griot	17AMB003/MD
Polarizing beamsplitter cube	Thorlabs	PBS104
IR detector	New Focus	2033
100× Objective	Nikon	BD Plan 100x
Oscilloscope	Tektronix	TDS1001B
Optical Spectrum Analyzer	Advantest	Q8384
IR sensor card	Newport	F-IRC2
TLS source	Agilent	81940A
IR Camera	Electrophysics	7290A
IR Detector	New Focus	2153
Digital Multimeter	Agilent	34401A
Illumination	Stocker Yale	Lite Mite
Monochromator	Spectral Products	DK480
Array Detector	Andor	DU490A-1.7
GIF Fiber	Thorlabs	31L02

Comments

CAUTION: flammable, use good ventilation and avoid all ignition sources.

CAUTION: flammable, use good ventilation and avoid all ignition sources.

CAUTION: flammable, harmful by inhalation, avoid contact with skin and eyes.

CAUTION: flammable and highly toxic, use good ventilation, avoid all ignition sources, avoid contact with skin and eyes.
CAUTION: flammable and causes irritation to eyes, nose and respiratory tract.

CAUTION: alkaline liquid and can cause irritation to eyes, nose and respiratory tract.

CAUTION: extremely corrosive, readily destroys tissue; handle with full personal protective equipment rated for HF.
CAUTION: flammable and causes irritation to eyes, nose and respiratory tract.

CAUTION: corrosive and very toxic; handle with personal protective equipment and avoid inhalation of vapours or mists.
CAUTION: very hazardous in case of skin and eye contact; handle with personal protective equipment.

CAUTION: invisible IR radiation.

CAUTION: invisible IR radiation.

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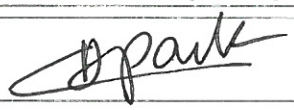
Signature:	 Date: 2016/5/20

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Response to reviewer's comments:

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→ We have changed as recommended.

3. All tables should be uploaded separately to your Editorial Manager account in the form of an .xls or .xlsx file.
→ We have changed as recommended.

4. Formatting:
-Please submit figures in an accepted format and not in a Word document. Each figure should be an individual file.
→ We have changed as recommended.

-Step 4.1.3 should be before 4.1.2.
→ We have revised the step 4.1.3 to be before 4.1.2.

5. Length exceeds 2.75 pg of highlighted material and must be reduced accordingly.
→ We have revised the length of highlighted material to be less than 2.75 pages. To reduce the length, we used abbreviations "RP" and "Al" for "roughing pump" and "aluminum", respectively.

6. Grammar:
-Line 49 – "A variety of steels meets" – should be plural verb.
→ We have revised "A variety of steels meets" to "A variety of steels meet".

7. Additional detail is required:
4.3.2 – "aluminum foils"
→ We have revised "aluminum foils" to "Al foil" in 4.1.2.

4.5.3 – Please use imperative tense.
-Line 463 – "recommended.; simply"
→ We have changed as suggested.

7. Additional detail is required:
-1.3.4 – Please include a citation.
→ We have included the citation as recommended. Please refer to the reference #15.

-4.1.1 – This diagram is not clear on the position. Please describe using text where it should

be placed. It seems as though it was already assembled and placed in prior steps.
→ We have changed in accord with the recommendation as described at Fig 3 and step 3.5.

-4.4.1 – Which components are wrapped? All of them?
→ The vacuum components between the SRG flange assembly and the inlet flange of the TMP are wrapped. We have revised as 4.2.1.

-4.4.2 – How is this checked?
→ We check by measuring the resistance between the chamber and the heater wire with the multimeter. If the resistance is infinite then there is no electrical short circuit. We have revised as 4.2.2.

-4.4.3 – Are the heaters wrapped? How many heaters are used?
→ The number of heaters depends on the size of the sample chamber. We used 3~4 heaters for this experiment. The heaters wrapping the chamber are also wrapped by Al foil.

-4.4.6 – How is degassing performed?
→ The degassing of the RGA ion source is performed by the electron bombardment (which is the default function of commercial RGAs) as described in 4.2.6.

-4.4.7, 4.4.9 – How is this scan performed?
→ We have revised as “Measure the RGA spectrum from 1 to 50 m/e” in 4.2.7.

-4.4.10 – When was the AV closed?
→ We obtain the residual gas spectrum of the sample by subtracting the spectrum of the vacuum components excluding the sample from the spectrum of the whole vacuum components (the sample + other vacuum components). The time of closing the AV is described in 4.2.9.

-4.5.2 – Wasn’t the SRG already placed on the flange assembly in Section 4.1? When was it removed?
→ The SRG head should be removed before baking. We have added this step in 4.2.1.

-4.6.2 – Which heater controller? There were multiple ones in 4.4.3.
→ The heater controller is used for both temperature controls during bake out and measurement. We have revised “Start the heater controller” to “Start the heater controller for the sample.” as noted in 4.4.2.

-4.7.3 – How is this checked? How does one remove mechanical vibration?

→ The roughing (mechanical) pump is the most vibration inducing component in this experimental setup. The vibration is reduced by placing a rubber pad between the floor and the mechanical pump. This point is addressed in 4.5.3 in the revised manuscript.

8. Branding should be removed from 1.1.3, Figure legends - ConFlat should be removed, CF without being defined is fine on its own.
→ We have revised as recommended.

9. Results: Please show any data demonstrating that hydrogen was released. How was this determined?
→ It is usually accepted that the major residual gas after bake out is hydrogen as described in ref. 7. This point can be also clearly recognized from the spectrum obtained in the step 4.2.9.

Response to comments of reviewer #1:

Manuscript Summary:
The authors technique is common in vacuum science but is not described or shown in detail in many places. This should be useful to those wanting to make outgassing measurements in the high vacuum, but are not familiar with spinning rotor gauges. However, there are several significant flaws that must be fixed.
→ We thank to the reviewer for many detailed suggestions which are helpful to improve our manuscript. We have revised the manuscript in accord with the suggestions.

Major Concerns:
General/overall:
1) The authors discuss historical aspects of outgassing measurements and rate-of-rise measurements in several places, but this discussion is inaccurate and not relevant to the paper.
→ We have removed the ambiguous parts of the manuscript as recommended.

2)I think the authors contextual discussions are a little weak and should be revised. The advantages/disadvantages are not clearly or precisely laid out. In the reviewer's opinion: The throughput method is more complicated, not practical in all situations, and requires a known pumping speed or conductance. The rate-of-rise method does not work well with gases that adsorb or condense. Other gauges can be used for rate of rise, but for high vacuum and ultra-high vacuum pressures, ion gauges and SRGs are pretty much all you have. Ion gauges generally have HIGHER outgassing rates than SRGS (this is incorrectly stated as having NO outgassing rate in several places) and can cause a temperature rise. SRGs are very linear (this is never mentioned) in the high vacuum, which makes them ideal for outgassing rate measurements. Etc. etc. etc.

→ We have revised the manuscript (INTRODUCTION and DISCUSSION sections) as recommended by the reviewer.

3)The authors do not discuss the accuracy of the SRG (which is necessary for the slope and the absolute outgassing rates they quote).
→ We have included the statement about the accuracy of the SRG in the line 88 of the manuscript.

4)They also do not discuss the frequency dependence of the residual drag (RD). This can cause a potential error. Perhaps the authors SRG had no appreciable slope on the RD, but this is not generally true. Someone following this procedure may get erroneous results if this is not correctly taken into account. This must be included in the protocol and the analysis.
Detailed comments:
→ We appreciate the reviewer that he/she reminded us that the frequency dependence of the RD. When we check the change of RD from our measured data during 24 h, we find that the change of RD is ranging from low 10^{-10} Pa/s to middle 10^{-9} Pa/s depending on the condition of the rotor. $1x10^{-9}$ Pa/s corresponds to 10% error of the minimum meaningful value (1×10^{-3} Pa/day; in line 431) with this experimental setup. We set this value as the criterion for acceptance to proceed to the next step. In this regards, we have revised the protocol as 4.5.

5)Line 86
The spinning rotor gauge dates back to well before the 1990's. This statement needs to be deleted or changed. See "The spinning rotor Gauge" J.K. Fremerey, JVSTA 1715 (1985) for the modern version of the SRG. Experimental versions exist much earlier. Fremerey Rev. Sci. Inst. 44, 1396 (1973) is but one example.
→ We agree with the reviewer's comment. We have revised the manuscript.

6)Line 88
This is a false statement and needs to be changed or deleted. The SRG certainly can and will outgas. Its absorption rate for H2 may be negligible at vacuum pressures, but surface absorption of water is still an issue.
→ We revised the false statement. Please refer to the revised manuscript.

7)Line 3.6 and other places:
1 degree seems a little tight and unnecessary. Two degrees is a more common rule-of-thumb. See "Recommended practices for the use of spinning rotor gauges in inter-laboratory comparisons" Measurement 66 (2015) 176-183. If this is your procedure, then I'm ok with leaving it, but you should be aware that this is probably not necessary.
→ We agree the reviewer's comments and revised the protocol.

8) 4.4.7 and 4.4.6 All of the RGS's I've ever seen cannot be operated at 150 C. If this is a special RGA, or has special bake-out cables, please state. Otherwise, change your protocol as the levels cannot be checked and the filaments cannot be degassed at this temperature.
→ The electronics of the RGA is cooled by the external air fan during the experiment. We revised the protocol as 4.2.5.

9)4.5.1 Give details on how the re-magnetization is done. I actually doubt this is generally necessary procedure as I've had experience baking SRGs many times and have never had a problem re-suspending the ball. In any case, because you cannot re-magnetize the rotor to its original moment, I'm not sure the statement is precisely correct. please re-write.
→ We have removed the part describing the re-magnetization as recommended.

10)4.7.2 Please give details on how to check the signal level. Is this from the controller? The processed signal usually reads decrement, not dB. dB is a relative unit, do you mean dBm?
→ The signal level is provided by the controller and the manual of SRG explains the unit as dB. If this is not appropriate, we have to change the unit.

11)Protocol section 4.9:
The residual drag of the SRG is frequency dependent. This can have a positive, negative, or zero slope. It varies and is unpredictable. For the low-outgassing rates you are measuring, this frequency dependence can easily have an effect on the outgassing rate (in some cases in can be mistaken for a pressure rise due to outgassing). The authors must explain how to take this into account in the analysis and data acquisition.
→ We have revised the protocol as shown in step 4.5. Please refer to the revised manuscript.

12) Discussion: The historical parts of the discussion about the throughput method and rate-of-rise method are neither true nor necessary for the paper. Please remove.
→ We agree. We have removed as recommended in the DISCUSSION section.

13) Discussion: I would not necessarily characterize an extractor gauge as a "ion gauge with low outgassing". It is an ion gauge with a lower x-ray limit than traditional BA gauges.
→ The filament current of BA gauge is 4 mA while that of extractor gauge is 1.6 mA, which results in the difference of the temperature of filament and the vacuum chamber surrounding the gauges. We believe that, from this difference, the outgassing from the BA gauge and its surrounding vacuum chamber is larger than those of the extractor gauge.

14) Line 435: "Although in principle the SRG has no outgassing" is a false statement and is unnecessary, please delete.
→ We have deleted as recommended.

15) please state the pressure range that the measurement is taken over.
→ The pressure range that the measurement is taken over is from 10^{-8} Pa to 10^{-3} Pa. This statement has been added in line 426.

Minor Concerns:
16) Abstract

Line two "steels meets" should be "steels meet"
→ We have changed as recommended.

17) Line 49
"steels meets" should be "steels meet"
→ We have changed as recommended.

Additional Comments to Authors:
N/A

Response to comments of reviewer #2:

Manuscript Summary:
General comments:
The authors should add, perhaps in the 'Introduction' section, the followings:
-A comparison of measurement accuracy using cold cathode gauge vs. SRG. The CCG gauge does have small pumping effect and the measured outgassing rate needs to be corrected for it. However, the simplicity of using CCG as compared with the complexity of using SRG may warrant the substitution even the measurement error bars increase.
→ We agree the reviewer's recommendation of CCG for the simple outgassing measurement gauge. However, we considered that it is more appropriate to use the SRG for the measurement of very low outgassing rate in spite of the complexity of using SRG because of its negligible outgassing rate, excellent accuracy, good linearity and stability in high vacuum range.

-The needs to control temperature ramping during bakeout and cooldown to 1-2o C/hr, which seems to be extremely slow (#264 and #273).
→ It was our mistake. We changed the unit from hour to minute.

Specific Comments:
#71: XHV is commonly defined as P < 10-10 Pa
→ We have changed as the reviewer's comment.

#145, #147: lower case for liter, i.e. l/min
→ We have changed as the reviewer's comment in #146, #149 and #224.

#173: ….described in Temperature Control of List of ….
→ We have changed as recommended.

#188: CF63 flanges and AV are not mentioned in any step, nor in the figures.

→ We have deleted the CF63 flanges and the AV which are not mentioned in any step, nor in the figures.

#191, #294, #395: suggest to remove 'plumb line', since it is very difficult to align to 1o using plumb line.
→ We have removed ‘plumb line’ as recommended by the reviewer.

#212: Why will you want to pump down with HLD while connected to TMP exhaust end? Both TMP and HLD can be operated at the same time while by-passing scroll pump.
→ We have revised the protocol as described in the revised figure 3. We added isolation valves (AV_{HLD} and AV_{Ro}) for the HLD and the scroll pump. Accordingly, the protocol has been changed as described in 3.5.3, 3.6.6 and 4.1.1.

#214: ….minimum detectable limit of the HLD.
→ We have revised as recommended.

#218: ….is leak tight.
→ We have revised as recommended.

#233 to #237: repeated statements, can you combine them?
→ We have revised as recommended. Please refer to the revised step 3.5.

#244: Disconnect and remove…..
→ We have revised the protocol as step 3.6.6.

#246: Scroll pump should be connected already, as a common practice or as part of a pump station shown in Figure 3.
→ We agree. We have revised the protocol as described in the revised figure 3. We have added isolation valves (AV_{HLD} and AV_{Ro}) for the HLD and the scroll pump and both are connected to the system together. Accordingly, the protocol has been changed as described in the revised step 3.6.6.

#262: ….wrap the chamber in aluminum foil.
→ We have revised as 4.2.3 as recommended. We used abbreviation “Al” for “aluminum” to reduce the length of the highlighted materials.

#266: Hold….for 24-48 h using bakeout program controller.
→ We have revised as 4.2.5 as recommended.

#333: What steps to take, if these criteria are not met?
→ Because maximum 10% error due to the frequency dependence of the residual drag is already included in this protocol as described in the revised step 4.5.1, we set the criterion for linearity of the measured outgassing rate to be within 10% error. From our experiences, the outgassing rate data within 10% error is obtained routinely with this experimental setup. However, if this criterion is not met, we suggest to continue to measure the outgassing rate for another 16 h for more stabilization. In this regards, we have revised as "…the pressure rise is linear within 10% error. If these criteria are met, stop the measurement. Otherwise, continue to measure until the pressure rise becomes linear within 10% error for at least 16 h." in the step 4.6.3.

#341: ….after the valve is closed.
→ We have revised as 4.7.2 as recommended.

#358 and #364: Are both heat treatments done in vacuum furnace?
→ Yes, both heat treatments are done in vacuum furnace. We have revised as #340 and #347.

#406: round up to 1 digit, i.e. 8.3±0.1, since the accuracy of these measurements can't be that great.
→ We have revised as recommended.

#406 and #432: both SRG and extractor have similar contribution to outgassing? How is it compared with outgassing/pumping of CCG?
→ The outgassing rate for the SRG described in the text is sum of the outgassing of the SRG and the isolation angle valve, whereas the outgassing rate of the extractor gauge is from its own as described in ref. 18. We believe that the outgassing rate of the SRG itself is much lower than the value in the text. We have not measured the outgassing or pumping effect of CCG which we consider as out of the scope of this work.

#463: define 'intensity'
→ 'intensity' is defined in line 395.

Table of Materials/Equipment:
Sputter Ion Pump, Cold Cathode Gauge, CF63 AV are not mentioned in the text.
Copper gaskets and bolt/nut set can be left out in the table
→ We have removed Sputter Ion Pump, Cold Cathode Gauge, CF63 AV which are not mentioned in the text as recommended by the reviewer.

Major Concerns:
N/A

Minor Concerns:
N/A

Additional Comments to Authors:
N/A

Reviewer #3:
Manuscript Summary:
Well written detailed description of the techniques used for outgassing rate measurements. This should enable others to reproduce results, and is a clear, concise explanation of the technique and results.
→ We appreciate the reviewer for the positive comments.

Major Concerns:
Well written overall.

Minor Concerns:
N/A

Additional Comments to Authors:
N/A

Flaw corrections:

- Mistakes in unit conversion:

#406: $8.34 (\pm 0.052) \times 10^{-8}$ → $8.3 (\pm 0.1) \times 10^{-12}$ (#389 in revised manuscript)

432: 9×10^{-8} → 1×10^{-11} (#410 in revised manuscript)

437: 7.5×10^{-8} → 7.5×10^{-12} (#415 in revised manuscript)

454: 1×10^{-5} → 1×10^{-3} (#432 in revised manuscript)