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# Outgassing characteristics analysis of mechanical cryocooler materials

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**Abstract.** Three types of outgassing rate measurements were conducted for the constituent materials of a single-stage Stirling (1ST) cooler: outgas measurement by total mass loss in a vacuum environment; outgassing rate measurement using the Quartz Crystal Microbalance (QCM) in a vacuum environment; and outgassing rate measurement using the Atmospheric Pressure Ionization Mass Spectrometer (API-MS) in an argon gas flowing environment. Relative comparisons of the adsorption energy and outgassing rate were made based on these measurement results. It was confirmed that the outgassing rate of the cold head polyimide part and compressor voice coil was dominant.

## 1. Introduction

The reliability and lifetime of mechanical cryocoolers are understood to depend on mechanical wear of the piston seal and valve seal, and working gas contaminated by impurity outgases such as H<sub>2</sub>O and CO<sub>2</sub>. Outgas analysis of both 20K-class two-stage Stirling coolers and a 4K-class Joule Thomson cooler was previously investigated[1]. In this investigation, outgassing rate measurements were conducted for the materials and components of the 1ST cooler[2], focusing on non-metallic materials as impurity gas sources. As the latest application of the 1ST cooler, the cooler is used to cool the sensor of the GCOM-C (Global Change Observation Mission- Climate) spacecraft to about 50K. The 1ST cooler has been used for several spacecrafts, such as SUZAKU, KAGUYA, AKATSUKI, and HITOMI [3]. Both lower temperature and reliable operation for a long lifetime were required for the GCOM-C spacecraft in comparison with these earlier spacecraft requirements. When the cooler generates low temperature, H<sub>2</sub>O outgas solidifies at the low temperature stage and hinders driving of the displacer of the cold head, which posed a problem in this investigation. The outgas measurements were made to quantitatively assess the amount of outgas in order to evaluate the validity of the cooler manufacturing process and improve the accuracy of the H<sub>2</sub>O contamination prediction model. We conducted several outgas measurements in early 2017, after the launch of the GCOM-C spacecraft on 23 December 2017, and found no apparent problems in the operation of the 1ST cooler after seven months on orbit.



**Table 1** Test method for each test sample

No.	Test pieces compressor	Outgas E595-07	QCM	API-MS	No.	Test pieces cold head	Outgas E595-07	QCM	API-MS
1	Voice Coil	-	-	C-1	8	Voice Coil	-	B-4, B-5	-
2	Magnet	-	-	C-2	9	Polyimide parts	-	-	C-4
3	GFRP parts	A-1	B-1, B-2	C-3	10	Polyimide covering	A-5	-	-
4	Piston seal	-	B-3	-					
5	PTFE parts	A-2	-	-					
6	Polyamide-imide covering	A-3	-	-					
7	Teflon parts	A-4	-	-					

## 2. Outgas measurement procedure

Outgassing rate measurement is used to obtain the outgassing properties of materials. Three types of outgassing rate measurements were made for the constituent materials of the 1ST cooler: outgas measurement by total mass loss and collected volatile condensable materials in a vacuum environment as per standard test method ASTM E595-07[4], outgas measurement using the QCM in a vacuum environment as per standard methods ASTM E1559-09 [5] and ECSS-Q-TM-70-52A [6], and outgas measurement using API-MS in an argon gas flowing environment.

Table 1 lists the materials and components used for measurement, such as the coil bobbin, piston seal, permanent magnet, and glass fiber reinforced plastic (GFRP). Based on previous research results obtained on outgas measurement [7], all metal materials had outgassing rates that are generally one to two orders of magnitude lower than those of resin materials, so metal materials are excluded from all measurements. In addition, the selected resin material has a relatively large surface area and mass. The combination of test pieces and test methods was set by considering the measurement of four test pieces judged to have high H<sub>2</sub>O outgas using the highly accurate API-MS test.

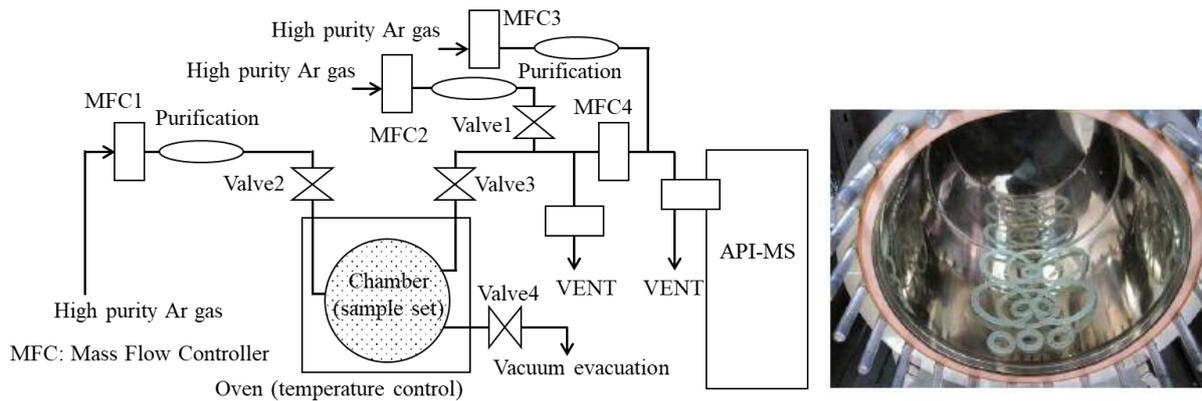
Figure 1 shows an overview of the device and the setup state of the measured material. In this test, highly accurate measurement on the order of ppb is possible while circulating argon gas as the material to be measured. In contrast, QCM test can be conducted at low cost, and has been used for many tests including the influence of air exposure time and evaluation of long-term trends of outgas in the major test pieces. Figure 2 shows an overview of the device and the setup state of the measured material. The effusion cell temperatures were 25 °C, 50 °C, 65 °C and 80 °C for each test. When outgas adheres to the QCM, the QCM-measured frequency increases. In addition, the average frequency value of QCM 3 and QCM 4 was subtracted from the average frequency value of QCM 1 and QCM 2 to estimate the value of the H<sub>2</sub>O outgassing by utilizing the difference in gas adsorption due to the difference in each QCM temperature. The resultant value is multiplied by both view factor between the orifice and the QCM and the coefficient considering the mass and surface area of the sample, and then the differentiated value is taken as the outgassing rate. Three samples were used, and one sample was measured with the same sample as in the API-MS measurement to clarify the quantitative differences between different measurement methods. Five samples presumed to have a relatively small amount of H<sub>2</sub>O release were confirmed by using the outgas measurement (Test-A). The test pieces were manufactured using equivalent processes to real cryocooler components, including surface treatment and material level bake-out processes. This investigation resulted in an exposure time in the ambient air being regulated of less than 120 hours, so as to effectively suppress the absorption of gases in ambient air during the cooler manufacturing. Time trends of the H<sub>2</sub>O outgassing rate were evaluated by fitting a mathematical model to predict the total amount of H<sub>2</sub>O. The test pieces were baked at each baking temperature (75 °C ~ 150 °C) for 100 h, exposed in ambient air for 120 hours except for Test B-2, Test B-5 and Test C-3 to simulate the effect of air adsorption during the actual cryocooler manufacturing process. Based on the measured outgas data, the outgassing rate was evaluated using the following equation. The outgassing rate is generally expressed by equation (1), and adsorption energy  $E$  is calculated from the outgas rate

at different temperatures using equation (2). The adsorption energy is calculated within the temperature range at the time of measurement and then is extended and used for estimating the outgas rate at a temperature outside the measurement temperature range. Calculate coefficients  $\alpha$  and  $\beta$  by fitting outgas rate measurement data with equation (3). Here,  $R$  is the gas constant.

$$\frac{dm}{dt} \propto \exp\left(-\frac{E}{RT}\right) \tag{1}$$

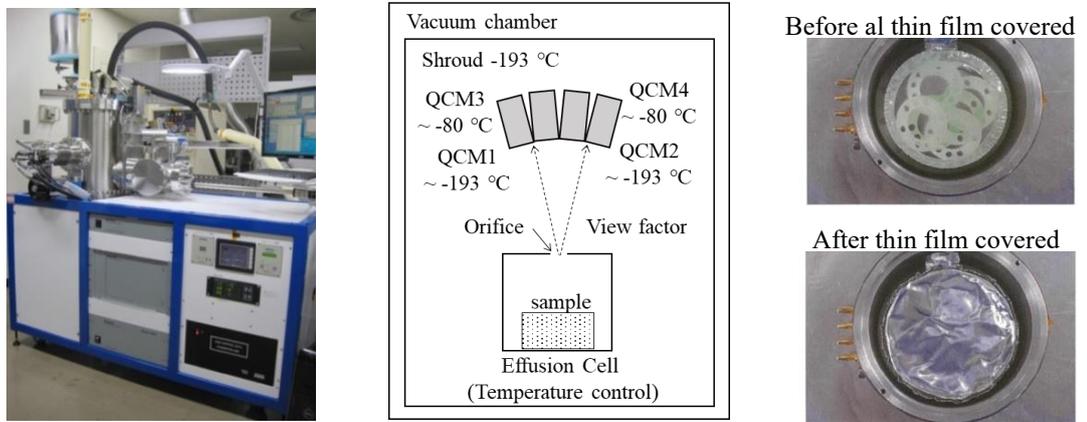
$$E_{T_1 T_2} = R \left( \frac{T_1 T_2}{T_1 - T_2} \right) \ln(k) \quad \text{where, } k = \frac{\left(\frac{dm}{dt}\right)_{T_1}}{\left(\frac{dm}{dt}\right)_{T_2}} \tag{2}$$

$$\frac{dm}{dt} = \alpha(t + t_0)^{-\beta} \tag{3}$$



(a) System outline (b) Sample setup view

**Figure 1.** Outgassing rate measurement system by using API-MS



(a) Apparatus outer view[8] (b) System outline (c) Sample setup view

**Figure 2.** Outgassing rate measurement system by using QCM

### 3. Outgas measurement results and discussion

This section describes the representative results obtained by the three types of outgas measurement tests.

Figure 3 (a) shows the outgassing rate measured by Test B-1. Figure 3 (b) shows the outgassing rate measured by Test C-3. As shown in the figure 3 (a), approximate expressions are obtained by fitting data of 10 hours before the temperature change of outgas rate at each temperature. And as shown in the figure 3 (b), several gases other than water can be detected in the API-MS test by this measurement, which shows that the outgassing rate is at least one order smaller than that of water. QCM test can only evaluate TML, ignoring other components thought that moisture release is dominant, but this can be said to be reasonable. Figure 4 shows the outgassing rate measurement results of GFRP parts after 120 hours of air exposure and without exposure. Although the initial outgassing rate is different, once the

temperature has been increased to 80°C, there is little difference between parts previously exposed to air and parts not exposed. Therefore, in the case of baking more than a certain amount, exposure it can be said to have hardly any influence, even if it is exposed to the atmospheric environment at the time of assembly, etc. It can be said that there is no problem if sufficient baking is carried out after assembly.

Figure 5 shows the estimated adsorption energy from 25 °C to 75 °C based on the outgassing measurement results for both the QCM test and the API-MS test. The adsorption energy of the measured cooler constituent material was in the range of 43.4 kJ/mol to 54.4 kJ/mol.

Figure 6 shows the prediction result of the outgassing rate when baking at 75 °C for 100hours was added. This time the acceleration factor of each material at room temperature is taken in to account, considering that baking was performed with 75 °C from the outgassing rate at room temperature obtained by measurement. The outgassing rate of the API-MS test (C-3) is about 2.3 times larger than the outgassing rate of the QCM test (B-2), which is likely to be affected by the difference in air ambient environment at the time of measurement (QCM: High vacuum condition, API-MS: Argon gas circulation condition). In addition, it was confirmed that the outgassing rate of the cold head polyimide part and compressor voice coil was dominant. The figure shows that the outgassing rate exceeded the acceptable outgassing rate level (1 ppb NL/min). Although the level can be reduced by gas purification after assembly, additional baking is needed for more efficient gas purification.

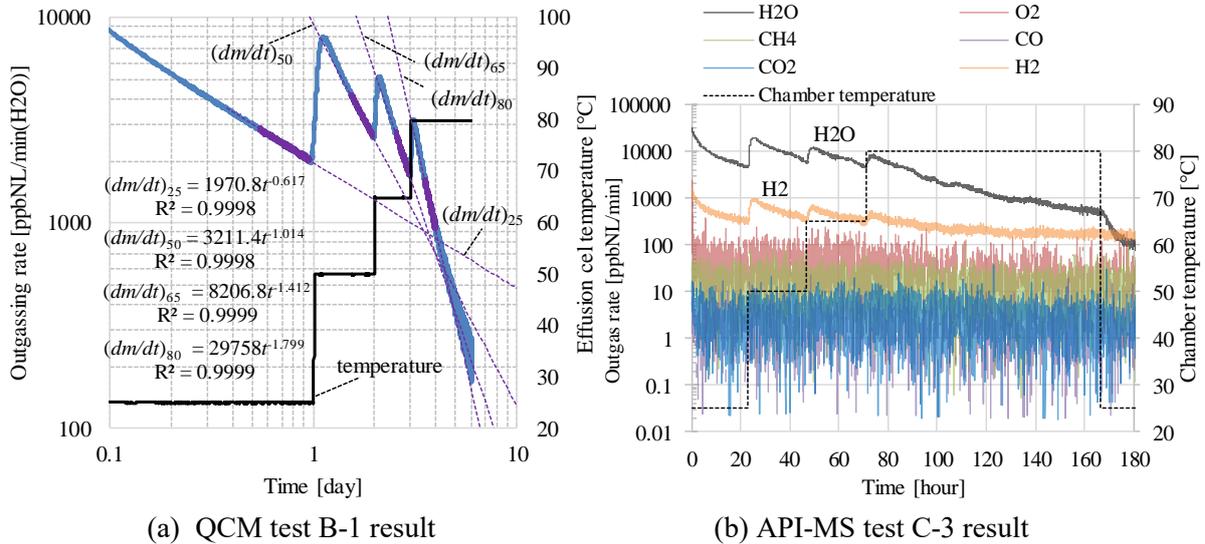


Figure 3. GFRP parts outgassing rate measurement results

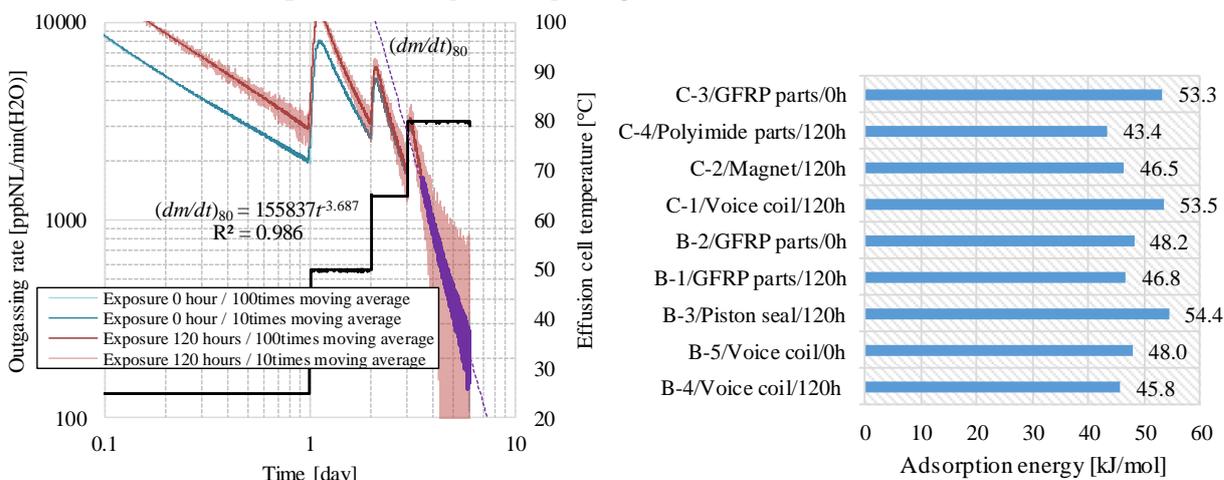
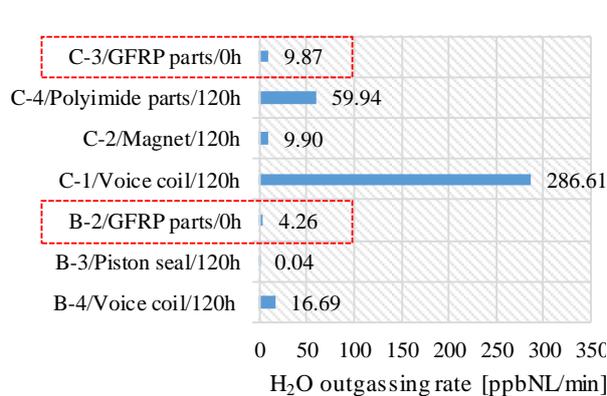
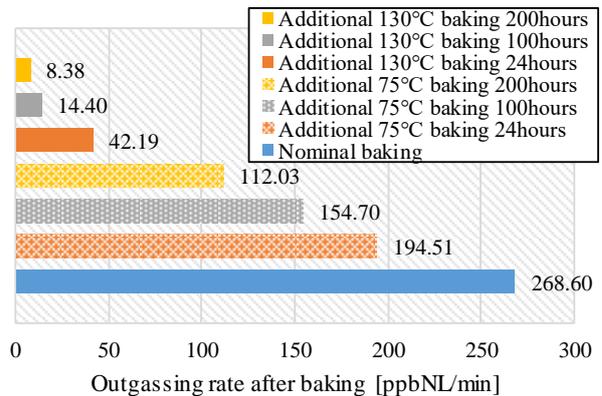


Figure 4. Outgassing rate comparison of difference in ambient air exposure time

Figure 5. Estimated adsorption energy from 25 °C to 75 °C by the results of outgassing measurement



**Figure 6.** Prediction result of outgassing rate when baking at 75 °C for 100hours was added



**Figure 7.** Compressor voice coil outgassing rate changes after additional baking

Figure 7 shows the outgassing rate changes of the compressor voice coil after additional baking. With the addition of baking at 130 °C, the use of materials with high heat resistant temperature is expected significantly reduce the outgassing rate. And regarding the materials for which raising the temperature above 75°C is difficult, the baking time is increased and the H<sub>2</sub>O content may be reduced gas substitution by discharge cleaning with inert gas such as argon. Materials with high outgassing rates were estimated based on the model in which additional baking proved effective in shortening the gas purification period after cooler assembly.

#### 4. Conclusions

Three types of outgas measurement tests were conducted for constituent materials of the 1ST cooler, and then relative comparisons were made of the adsorption energy and outgassing rate. We clarified the high outgassing rate materials and such components as the compressor voice coil and cold head Polyimide parts. In addition, we clarified the effectiveness of additional baking. As the temperature rises, it becomes clear that baking is more effective, and thus we will focus on further improving the manufacturing process. And since the inside of the cooler is an environment filled with helium gas, the API-MS test makes accurate measurement possible with high accuracy.

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