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## Experimental analysis of vacuum pressure and gas flow rate in structured-core transparent vacuum insulation panels

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### Abstract

The notion that modern buildings should strive to be net-zero energy buildings (NZEBs) is widely accepted. One of the causes leading to high energy usage for space heating, resulting in avoidable carbon emissions, is heat loss via building windows. In order to improve window's insulation in existing buildings, structured-core transparent vacuum insulation panels (TVIPs) are proposed. TVIPs mainly consist of the structured core material, the low-emissivity film, and the transparent gas barrier envelope. TVIPs have high insulation performance and are inexpensive to manufacture and can be easily installed. Therefore, TVIPs have the potential to improve window's insulation in existing buildings at a low cost. However, it is necessary to overcome the issue of preventing the pressure rise inside TVIP after vacuum sealing. The authors constructed an experimental setup to quantify the effect of reduction of gas flow rate in TVIP after evacuation by applying the pressure-rate-of-rise-method. In this experiment, a gas barrier film with a straw was used as the vacuum chamber. This could reproduce the pressure increase in the TVIP after sealing and the gas flow rate in the TVIP is evaluated. The experimental result shows that the coated core material and the enclosing getter agent lowered the pressure rise and gas flow rate in TVIP by combining concurrent evacuation and heating. Furthermore, after extending the simultaneous vacuuming and heating period to 8 h and applying the coated core material, and enclosing the getter agent, the internal pressure in TVIP may be lowered to around 1 Pa after 30 min after halting vacuuming. It was confirmed that this pressure satisfied the performance required for TVIPs, and the result was much closer to the realization of TVIPs.

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**Keywords:** Structured-core transparent vacuum insulation panel; Outgassing; Internal pressure; Gas flow rate; Pressure-rate-of-rise method

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## 1. Introduction

A significant improvement to insulation is a necessary step in converting existing buildings to net-zero energy buildings (ZEB), in particular, improvement of window's insulation plays a significant part [1,2]. The authors have been developed transparent vacuum insulation panels (TVIPs) to improve the window's insulation [1,3,4]. TVIPs have high insulation performance and are inexpensive to manufacture and can be easily installed. Therefore, they have the potential to improve the overall heat transfer coefficient of single-pane glass to less than  $1.5 \text{ W}/(\text{m}^2 \text{ K})$  [1]. On the other hand, to realize TVIPs, it is necessary to overcome the issue of preventing the pressure rise inside TVIP after vacuum sealing.

Outgassing from the core material is the most common source of pressure rise inside the TVIP after vacuum sealing. Methods to reduce outgassing from materials in a vacuum include baking [5] and coating of core materials [6]. Baking is the most popular method and the reduction of outgassing by baking is based on the temperature dependence of the diffusion coefficient according to Arrhenius' law [7]. Thus, the outgassing is reduced larger when the baking temperature becomes higher. On the other hand, the baking temperature is limited in the case of TVIPs because of the use of plastic as the core material. The authors showed that heating (baking) at a low temperature of  $60 \text{ }^\circ\text{C}$  when fabricating TVIPs reduces the effective thermal conductivity of TVIPs after vacuum sealing [1]. However, the effect of reducing outgassing has not been quantified. The effectiveness of the coating in reducing gas emissions has been quantified by Kwon et al. in experiments using polycarbonate core materials [6]. The coating is a promising method for reducing outgassing from the core material of TVIP along with low-temperature baking, according to the experimental results of Kwon et al. Moreover, the method of absorbing the gas released from the core material may also be effective in preventing pressure increase. The authors showed that enclosing getter agent when fabricating TVIPs can reduce the effective thermal conductivity of TVIPs after vacuum sealing [1].

Based on the aforementioned studies, there is a research gap in experimentally achieving the combination of heating, covering core material, and encapsulating getter agent so that it maintains the vacuum pressure within the TVIP from rising. In this paper, the authors constructed an experimental setup to quantify the effect of reduction of gas flow rate in TVIP by applying Pressure-rate-of-rise-method [5]. In this experimental setup, the authors used a gas barrier envelope with a straw as a vacuum chamber. This made it possible to reproduce the pressure change and the gas flow rate in the TVIP after vacuum sealing by measuring the pressure change after the vacuum pump was stopped. Using this experimental setup, the authors quantified the effect of each gas flow rate reduction method and also evaluated the effect of combining the reduction methods.

## 2. Manufacturing setup of TVIP

Conventional VIPs with aluminum envelope barriers [8,9] have no commercially available option that can be applied to the existing windows. The vacuum glazing windows can be applied to windows [10,11], however, they are relatively expensive and difficult to be used for retrofitting in buildings due to their single purpose in-flexibility. Therefore, the authors have proposed TVIPs be attached to the windows in existing buildings. Fig. 1 shows (a) manufacturing steps, (b) real images, and (c) the proposed real field application of the TVIP for the existing windows. The manufacturing steps start with preparing and drying the core material, the low-emissivity film, and the transparent gas barrier envelope. The core material shall be in a shape that allows a vacuum layer to be formed after vacuum sealing. In the previous paper, the peek spacer, the mesh spacer, and the frame spacer was proposed [1]. The low-emissivity film, whose emissivity on one side is approximately 0.3, is used to reduce radiative heat transfer in a vacuum layer. After drying, the core material and the low-emissivity films are inserted into the transparent gas barrier envelope. Then, the transparent gas barrier envelope including the core material and the low-emissivity films is installed in a vacuum chamber in the sealing machine and evacuated with the chamber. After the chamber is evacuated and the pressure reaches the set value, the gas barrier envelope is sealed. The proposed TVIPs could assist in enhancing the insulation ability of the windows in the existing buildings with sustaining the light transparency close to the conventional vacuum glazing windows. In addition, the TVIPs can be applied as thermal insulating transparent curtains. The TVIPs have the potential to improve the overall heat transfer coefficient of single-pane glass to less than  $1.5 \text{ W}/(\text{m}^2 \text{ K})$ . In addition, the manufacturing cost is expected to be less than one-third compared to the conventional vacuum glazed window. On the other hand, the realization of TVIP involves the issues of preventing pressure increase inside the TVIP after vacuum sealing and long-term durability. In particular, the pressure increase inside the TVIP after vacuum sealing is the largest challenge to overcome.

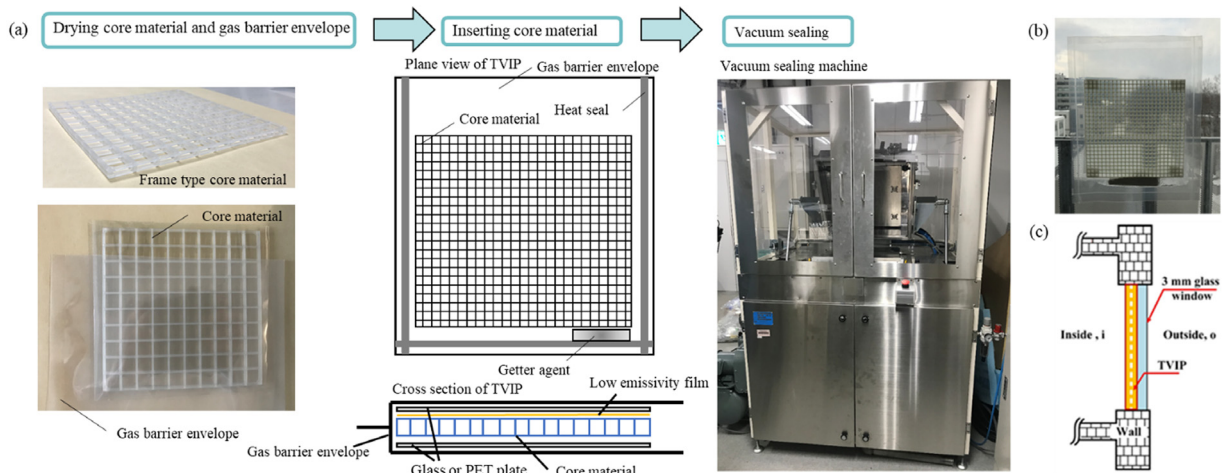


Fig. 1. (a) Manufacturing steps of TVIP (b) Real image (c) Proposed real field application.

### 3. Experimental methodology in applying the Pressure-Rate-of-Rise method

#### 3.1. Outlines of the experimental setup

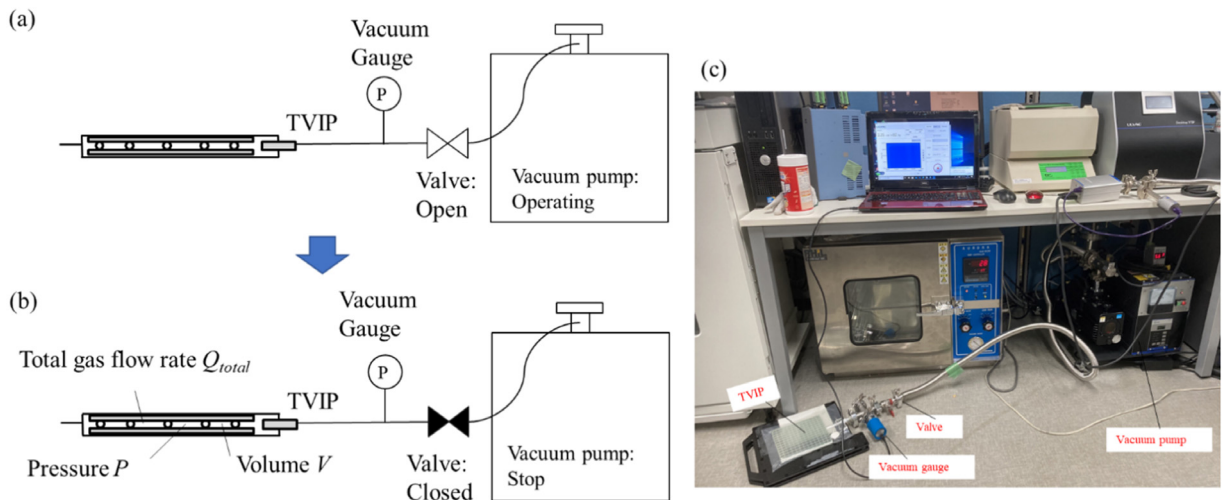
In order to quantify the change in outgassing rate after vacuum sealing, the Pressure-Rate-of-Rise Method is experimentally applied [5]. Fig. 2 shows outlines of the experimental apparatus and Fig. 3 shows an appearance of TVIP used in the experiment. A core material, a low-emissivity film, and glasses were inserted into the transparent gas barrier envelope with straw. Here, the core material, the low-emissivity film, and glasses were dried at a set temperature of 70 °C for 24 h. Fig. 4 illustrates the structure of the transparent gas barrier film used as the gas barrier envelope. The gas barrier film used in the preceding articles [1,3] is the same one employed in this research. The TVIP is directly connected to the vacuum pump as shown in Fig. 1. The valve between the TVIP and the vacuum pump is closed when the TVIP has been evacuated, and the vacuum pump is turned off. The total gas flow rate may be calculated using the following equation when the rise in pressure is measured [5];

$$Q_{total} = \frac{V dp}{dt} \tag{1}$$

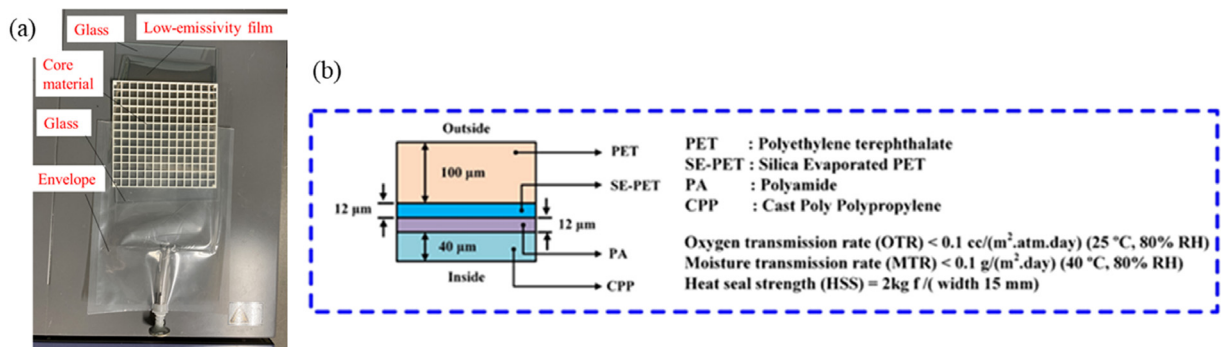
Here,  $V$  [m<sup>3</sup>] is the internal volume of the TVIP and the pipe between the TVIP and valve, and  $dp/dt$  [Pa/s] is the rate of pressure rise. In this study, the TVIP with a gas barrier envelope with a straw was used as the vacuum chamber for experiments on the Pressure-rate-of-rise method. This made it possible to measure the pressure inside a full-scale TVIP. It also enabled quantification of the reduction in gas emissions due to heating during the evacuation at high vacuum pressure, the use of coated core materials, and the attachment of adsorbents (Getters). In addition, the gas barrier envelope with straw is the same as the one used in previous papers except for the straw part, therefore, the pressure changes after closing the valve and stopping the vacuum pump can almost reproduce the pressure change in the TVIP after vacuum sealing. The vacuum gauge is a Pirani vacuum gauge that was connected to a data logger and has a measurable pressure range from  $5.0 \times 10^{-2}$  to  $1.0 \times 10^5$  Pa with a measurement accuracy of  $\pm 20\%$ . A rotary pump and a turbomolecular pump were used simultaneously. The standard configurations of the TVIPs used in the experiments are shown in Fig. 4. When a frame-type core material is used, the core material is covered with a low-emissivity film and sandwiched between two sheets of glass. In the case of peak type core material, the core material is covered with a low-emissivity film and sandwiched between glass and acrylic plate. The peak-type core material was newly produced by using a 3D printer. Although this peak core material is not transparent, it is possible to produce a transparent peak-type core material by using the transparent material.

#### 3.2. Experimental conditions

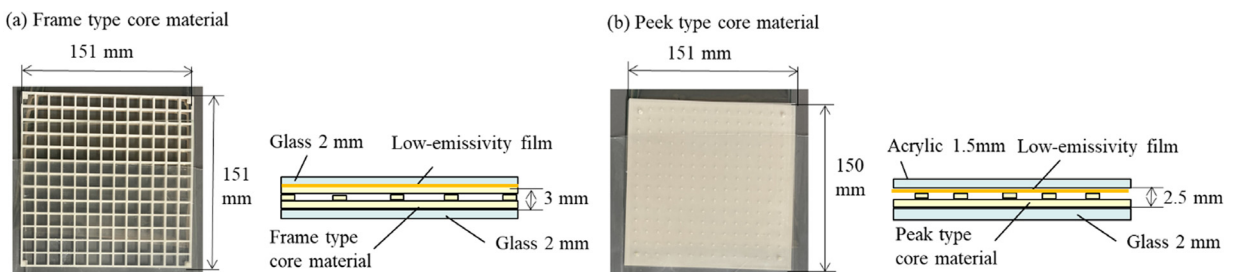
Experimental conditions are shown in Table 1. The empty samples in A, H, and J were composed of only the gas barrier envelope without the core and other materials from the basic TVIP configuration in order to quantify



**Fig. 2.** (a) Schematic diagram showing evacuation of TVIP, (b) pressure measurement in TVIP after vacuuming and, (c) pictorial illustration of the experimental measurement setup.



**Fig. 3.** (a) The constructional components of TVIP (b) Structure of the transparent gas barrier film for envelope.



**Fig. 4.** Standard configurations of (a) Frame type core and (b) Peek type core.

outgassing from the gas barrier envelope and gas penetration through gaps. In case B, a frame type core material was used, and the configuration was standard. The evacuation time after reaching 2 Pa was for 30 min. In case C, the double-stretched PET plates were used instead of glasses. In case D, heating started at a temperature setting of 60 °C simultaneously with the start of evacuation, and stopped when the valve was closed and the vacuum pump was stopped. In case E, organic polysilazane was used as coating agent and the core material were dipped in the organic polysilazane, and dried before being placed in the drying chamber. Then, the effect of organic polysilazane

**Table 1.** Implemented experimental boundary conditions.

	Vacuuming time (h) after reaching 2 Pa	Heating	Coating	Getter agent
A. Empty	0.5			
B. Standard	0.5			
C. Glass → Double-stretched PET	0.5			
D. Simultaneous heating	0.5	○		
E. Coating core material	0.5		○	
F. Enclosing getter agent	0.5			○
G. Heating + Coating + Getter	0.5	○	○	○
H. Empty, 4 h vacuuming	4			
I. Heating + getter + coating, 4 h vacuuming	4	○	○	○
J. Empty, 8 h vacuuming	8			
K. Heating + getter + coating, 8 h vacuuming	8	○	○	○
L. Coating + getter, 8 h vacuuming	8		○	○
M. Heating + coating, 8 h vacuuming	8	○	○	
N. Peak type core, heating + coating + getter, 8 h vacuuming	8	○	○	○

coating was verified. In case F, the getter agent, which consists of alloy and CaO (Calcium Carbonate) [12], was enclosed. In case G, I, K, and N, all of the simultaneous heating, the coating core material, and the enclosing getter agent were carried out. The vacuuming time after reaching 2 Pa was 30 min for G, 4 h for I, and 8 h for K, respectively. G, I, and K were compared to verify the effect of changing the vacuuming and heating time on the outgassing. Case L is K minus the simultaneous heating and M is K minus the getter agent. The effects of the simultaneous heating and the enclosing getter agent on the reduction of gas emission were verified by comparing K to L and M, respectively. In case N, the core material was changed from the frame type to the peak type and the other conditions were the same as case K.

In the experiment applying the Pressure-rate-of-rise method, the total gas flow rate was determined by using the obtained internal pressure data per second,

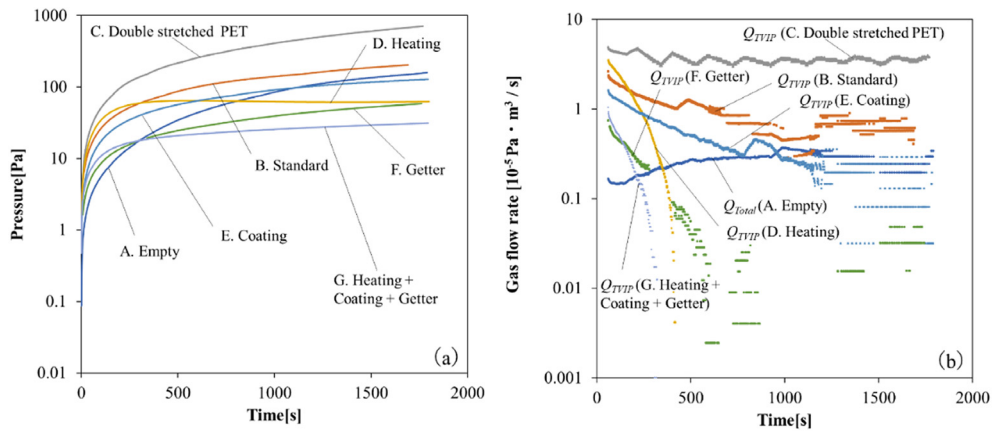
$$Q_{total} = \frac{V}{\Delta t} (p_1 - p_2) \quad (2)$$

$Q_{total}$  is the total gas flow rate [Pa·m<sup>3</sup>/s],  $V$  is the volume [m<sup>3</sup>],  $\Delta t$  is the time [s], and  $(p_1 - p_2)$  is the pressure change between  $\Delta t$  [Pa]. The volume  $V$  differs between empty and otherwise. In the empty case (A, H, and J),  $V$  is equal to the volume  $V_1$  of the cross-shaped vacuum pipe to which the vacuum gauge and valve are connected. When a TVIP with a core material is connected, the TVIP's volume  $V_2$  was determined from the thickness of 3 mm and side lengths of 151 mm × 151 mm for the frame type and from the thickness of 2.5 mm and side lengths of 150 mm × 150 mm for the peak type. Then the volume  $V$  was the sum of the volume of the cross-shaped vacuum pipe  $V_1$  and TVIP  $V_2$  ( $V = V_1 + V_2$ ). The gas flow rate inside the TVIP  $Q_{TVIP}$  was calculated by subtracting  $Q_{total}$  for empty case (A or H or J) from the total gas flow rate  $Q_{total}$  obtained in each experiment. However, if the calculated  $Q_{TVIP}$  is lower than 0 ( $Q_{TVIP} < 0$ ),  $Q_{TVIP}$  is regarded as 0 ( $Q_{TVIP} = 0$ ).

## 4. Results and discussion

### 4.1. Short-term pressure rise and gas flow rate

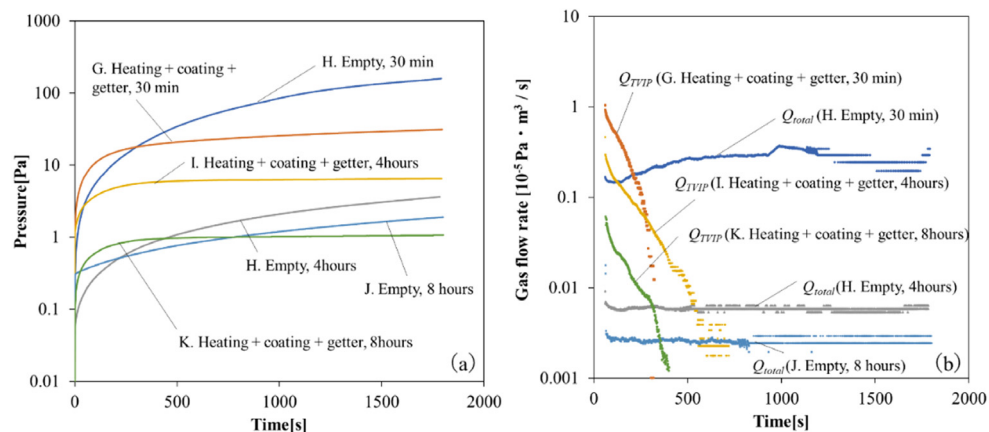
Fig. 5 shows (a) pressure change in case A, B, C, D, E, F, and G and (b) gas flow rate  $Q_{total}$  (case A) or  $Q_{TVIP}$  (case B, C, D, E, F, and G) after closing the valve. Compared to the pressure in case B, the pressures in all cases except in case C were lower. The result of case C suggests that the outgassing increases in the case of double-stretched PET plates. From cases D, E, and F, it was confirmed that the simultaneous heating, the coating core material, and the enclosing getter agent can reduce the pressure increase and  $Q_{TVIP}$ . The reduction by the simultaneous heating or the enclosing getter agent were more effective than the coating core material in the case of a 30-min vacuuming. From G, the combination of the simultaneous heating, the coating core material, and the enclosing getter agent reduced the pressure increase and  $Q_{TVIP}$  the most. The gas flow rate  $Q_{TVIP}$  dropped less



**Fig. 5.** (a) Evacuation pressure change in case of A, B, C, D, E, F, and G. (b) The comparison of gas flow rate  $Q_{total}$  (for case A) with  $Q_{TVIP}$  (for case B, C, D, E, F, and G).

than  $10^{-8} \text{ Pa} \cdot \text{m}^3 / \text{s}$  and reached almost zero by 500 s for case D and case G, where the simultaneous heating was conducted, indicating that more gas has been desorbed from the core material due to the simultaneous heating.

Next, the effects of changing the evacuation and heating time regimes on the outgassing were examined. Fig. 6 shows (a) pressure change in case A, G, H, I, J, and K and (b)  $Q_{total}$  (case A, H, and J) or  $Q_{TVIP}$  (case G, I, and K). The gas flow rate  $Q_{total}$  (case A, H, and J) varies with the vacuuming time even in the empty case and is almost constant during the 30-min measurement. A comparison of G, I, and K confirms that the pressure increase is suppressed by extending the vacuuming and heating time. Especially in K, the internal pressure was suppressed to approximately 1 Pa. This pressure met the performance requirements for TVIPs, and the end result was significantly closer to TVIP implementation. In cases, G, I, and K, the initial  $Q_{TVIP}$  after closing the valve became smaller as the vacuuming and heating time was increased.



**Fig. 6.** (a) Pressure change for the case of A, G, H, I, J, and K. (b) Gas flow rate  $Q_{total}$  (for case A, H, and J) in comparison to  $Q_{TVIP}$  (for case G, I, and K).

The effects of the simultaneous heating and the enclosing getter agent on the gas flow rate are discussed under the longer vacuuming and heating conditions. The result of changing the type of core material was discussed. Fig. 7 shows (a) pressure change in case K, L, M, and N and (b)  $Q_{TVIP}$ . Compared to case K, where all of the simultaneous heating, the coating core material, and the enclosing getter agent were carried out, case L, where the simultaneous heating was omitted, and case M, where the enclosing getter agent was omitted, showed the larger increase in pressure. In addition,  $Q_{TVIP}$  were more than  $10^{-8} \text{ Pa} \cdot \text{m}^3 / \text{s}$  and did not reach almost zero. The results show that simultaneous heating can significantly reduce the outgassing from core material and that the enclosing

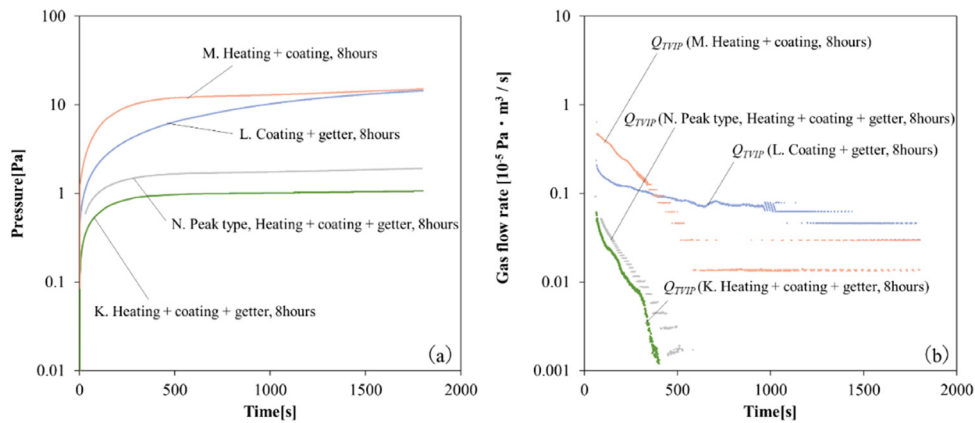


Fig. 7. (a) Pressure change in case K, L, M, and N, (b) Gas flow rate  $Q_{TVIP}$  (case K, L, M, and N).

getter agent reduces the gas flow rate over a long period of time due to the adsorption. Furthermore, the combination of the simultaneous heating and the enclosing getter agent has a synergy effect. Eventually, it was shown that both of the simultaneous heating and the enclosing getter agent were necessary to stabilize the pressure at a low value. For case N, in which the core material was changed, the pressure was a little higher than in case K. However, it was confirmed that the pressure stabilized after 600 s, as in case K.

#### 4.2. Short-term pressure rise and gas flow rate

The pressure was measured in cases K to N not only 30 min after the valve was closed, but also the next day after several days had passed. Table 2 shows the measured pressure, according to the number of elapsed days after closing the valve and stopping the vacuum pump, from case K to case N. It was confirmed that case K and case N, with all of the simultaneous heating, the coating core material, and the enclosing getter agent were able to maintain a vacuum pressure of less than 4 Pa even after about 5 days. On the other hand, the pressure increased in case L and case M. These results indicated that both the simultaneous heating and the enclosing getter agent were necessary to stabilize the pressure at a low value as explained in the previous section.

Table 2. Measured pressure according to the number of elapsed days after closing the valve and stopping the vacuum pump from case K to case N.

	Elapsed time after valve closed	Pressure [Pa]
K. 8 h heating + getter + coating	4 days 17.5 h	2.04
L. 8 h coating + getter	2 days 20 h	70.2
M. 8 h heating + coating	3 days 17 h	250
N. 8 h peaked heating + coating + getter	4 days 17.5 h	3.69

Finally, the effective thermal conductivity of the vacuum layer in case K was measured by applying the heat flux meter method, and 8.9 mW/(m K) was obtained. This value was lower than 11 mW/(m K), which was the lowest effective thermal conductivity of the vacuum layer in TVIP after vacuum sealing [1].

### 5. Conclusions

In this paper, the authors used a gas barrier envelope with a straw as a vacuum chamber and constructed an experimental setup to quantify the effect of reduction of gas flow rate in TVIP after evacuation by applying Pressure-rate-of-rise-method. As the result, the following are the conclusive remarks.

(1) The simultaneous evacuation and heating, the coating core material, and the enclosing getter agent reduce the pressure rise and gas flow rate in TVIP. After evacuation, the combination of simultaneous vacuuming and heating, the coated core material, and the enclosing getter agent lowered the pressure increase and gas flow rate in TVIP.

(2) The combination of the simultaneous evacuation/vacuuming and heating for 8 h, the coating core material, and the enclosing getter agent can suppress the internal pressure in TVIP to approximately 1 Pa. It was confirmed that this pressure satisfied the performance required for TVIPs. Even when the frame-type core material was changed to the peak-type core material, the internal pressure in TVIP after vacuuming and lapse of a certain time was kept constant. Also, both simultaneous heating and the enclosing getter agent were necessary to stabilize the pressure at a low value.

(3) The effective thermal conductivity of the vacuum layer in the case where the simultaneous vacuuming and heating for 8 h, the coating core material, and the enclosing getter agent were combined was measured by applying the heat flux meter method, and 8.9 mW/(m K) was obtained.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

### Data availability

Data will be made available on request.

### Acknowledgments

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