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A modified pump-out technique used for fabrication of low temperature metal sealed vacuum glazing

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Abstract

A modified pump-out technique, incorporating a novel pump-out hole sealing process, has been developed that enables a high level of vacuum to be achieved between the panes of a vacuum glazing. The modified pump-out method provides several potential opportunities for the fabrication of a vacuum glazing with improved thermal performance. In particular improved flexibility for production of a wide range of glazing sizes may allow a lower cost of manufacture to be achieved by avoiding the expense of a high vacuum oven which would otherwise be required for commercial production of high performance, large-scale vacuum glazings.

The thermal performance of the vacuum glazing fabricated using the pump-out technique was characterized using a guarded hotbox calorimeter and theoretically analyzed using a finite volume model. The excellent experimentally determined thermal performance of the fabricated vacuum glazing was in good agreement with that predicted theoretically.

Keywords: Vacuum glazing, pump-out, thermal performance

1. Introduction

Vacuum glazing consists of two planar sheets of glass sealed together around the edges and separated by an array of tiny support pillars on a regular square grid to form a narrow evacuated space. Vacuum glazing can provide good thermal insulation while maintaining high levels of visual transmittance and thus provides significant potential for energy saving. A high internal vacuum (< 0.1 Pa) between the glass sheets is required to achieve predicted theoretical performance. Several preparation methods have been reported and patented since the first vacuum glazing was detailed in a German patent (Zoller, 1913). Among these, Collins *et al* have fabricated vacuum glazing using solder glass to form a contiguous edge seal at a temperature of around 450 °C, after which evacuation was undertaken through a pump-out tube attached to the upper glass pane (Robinson and Collins, 1989; Collins and Simko, 1998; Collins *et al*., 1999). An attempt to form vacuum glazing within an evacuated chamber using a laser edge sealing technique at high temperature was not successful (Benson *et al*., 1990). Techniques used for the edge seal production at high temperature either using solder glass or applying a laser exclude the use of tempered glass which would loose its tempered qualities at these high sealing temperatures. To overcome this exclusion, as the use of tempered glass is now a mandatory requirement in many construction and related applications, the University of Ulster has developed a low temperature technique to create a novel edge seal, comprised of an inner vacuum seal formed from indium and an outer adhesive seal (Griffiths *et al*., 1998; Hyde *et al.*, 2000).

After the formation of a good vacuum edge seal, the evacuation of the vacuum glazing is the key to making a high quality vacuum glazing. Collins suggested several design options based on evacuation through a small pump-out tube or aperture (Collins, 2000). The technique used by Collins makes vacuum glazing with a stub on the surface of the upper glass pane; this stub may provide a possible mechanical weakness and is not visually attractive. A single stage process based on a low temperature sealing technique developed by the University of Ulster overcomes some drawbacks of the high temperature process, but due to the lower temperature used, the evacuation process needs to be improved to prevent outgassing. The single stage process, described in Fig 1, uses a high vacuum oven to evacuate and seal the glazing in a single operation. The sequence of operations involved in the fabrication process is glass cleaning, edge seal application, pillar placement, assembly of the glazing and location in the vacuum oven. The single stage process is characterised by the evacuation and sealing of the glazing in a high vacuum oven in a single operation, effectively 'sealing in' the vacuum. Combining the single stage low temperature seal technique with a modified pump-out method may solve the outgassing problem and is expected to allow manufacture of high quality vacuum glazing. In this work, vacuum glazing samples have been fabricated using this combined technique, experimental results indicate that the combination of a low temperature seal with a modified pump-out technique produce high performance glazing.

The thermal performance of the vacuum glazing samples fabricated was characterized using a guarded hotbox calorimeter and theoretically analyzed using a finite volume model (Fang *et al.*, 2006). The experimentally determined U-value for the vacuum glazing was also compared with that predicted theoretically.

2. Fabrication process

Fig. 2 shows a schematic diagram of a vacuum glazing. The size of the 4 mm thick glass panes chosen for this work was 400 mm by 400 mm, with low emittance coatings on one side of each glass pane which face each other inside the glazing. The glass sheets, separated by an array of tiny support pillars on a regular square grid, are sealed together around the edges to form a narrow evacuated space. The support pillars manufactured from stainless steel have a diameter of 0.3-0.4 mm and a height of 0.15 mm.

Fig. 3 illustrates the developed two stage manufacturing process of a vacuum glazing. Stage one includes glass preparation, indium seal deposition, location of support pillars array and edge seal formation within the vacuum chamber at low temperature. Stage two is the subsequent evacuation of the glazing using a modified pump-out system and sealing of the pumpout hole in the vacuum glazing in the bake-out oven.

Prior to fabrication, a 3 mm-diameter hole for glazing evacuation was drilled into one of the glass panes, this was sealed using an indium precoated glass cover disc when the required level of vacuum was established. Using such an access hole avoids incorporating a small glass tube into the glass pane for evacuation and the formation of a tube stub afterwards as in the technique used by Collins’s group (Collins and Simko, 1998). The glass panes were cleaned using acetone and isopropyl alcohol and baked inside the bake-out oven at 200 °C. The edge seal material used is indium, a malleable metal with excellent fatigue resistance which may be used to join materials with greatly mismatched coefficients of thermal expansion (Griffiths *et al.*, 1998). Thus with respect to vacuum glazing the indium edge seal can withstand the stresses induced in the glazing due to a thermal gradient between the glass panes when the glazing is subject to thermal cycling.

The first stage, formation of the edge seal is performed in a high vacuum stainless steel chamber with an halogen infra red heating system, this is based on the low temperature seal technique described in detail elsewhere (Hyde *et al.*, 2000). After ultrasonically applying indium around the edge of the glass sheets, the support pillars are positioned onto the lower glass sheet in a 25 mm x 25 mm array using a vacuum pick and place tool that has a nozzle size down to 0.2 mm in diameter, which is ideal for handling the tiny support pillars (0.4 mm in diameter) used inside the evacuated glazing. The upper glass sheet is then located to align with the lower glass sheet. The initial outgassing of the internal surface and the edge seal of the glazing are carried out within the vacuum chamber at a temperature below 200 ºC and at a pressure of around 10^{-5} Pa.

The second stage of the manufacture process of the vacuum glazing was a modified pump-out system to achieve a high vacuum inside the glazing. Fig. 4 shows a schematic diagram of the modified pump-out system that can be connected to the high vacuum chamber to achieve evacuation. The evacuation cup is manufactured from stainless steel and has three flanged ports, of which one is connected to the high vacuum system; the other two are for electrical feedthroughs providing power to a heating element and a thermocouple sensor. An O-ring sealed view port on the top of the cup allows visual inspection of the heating block inside the cup, which consists of a heating cartridge and a thermocouple. The O-ring sealed cup is positioned over the access hole on the upper glass sheet during evacuation. All O-rings including those used for the flanged port connections are silicone O-rings that can withstand a temperature of up to 250 °C. After formation of the edge seal in the vacuum chamber, the glazing sample is transferred into a bakeout oven and the heating block is then placed onto the indium precoated glass cover disc over the access hole. The assembly is baked out at 120 °C in the oven, and the annular region of the cup and the internal space of the sample are evacuated. The natural variability of the indium precoating of the glass cover disc and the corresponding area surrounding the access hole provides a sufficiently large gap to permit evacuation of the glazing. When the internal space between the two glass panes is evacuated to a pressure below 10^{-5} Pa, the heating block is then activated. The temperature of the heating block is measured by a K-type thermocouple fitted into the heating block and controlled by a PID temperature controller. Heat is transferred from the block to the indium precoated glass cover disc; the access hole is sealed when the temperature reaches the melting point of the indium.

3. Thermal performance

To assess the quality and compare the thermal performance of vacuum glazings produced by the two-stage and single stage processes, two typical samples were characterized using a guarded hotbox calorimeter (as shown in Fig.5). The guarded hotbox allows accurate measurement of heat flow through the vacuum glazing for given air temperatures within its cold and hot chambers. Heat transfer across the vacuum glazing including contributions from pillar conduction, radiation, and edge seal conduction were calculated using a finite volume model described in detail elsewhere (Fang *et al*., 2006), and compared with experimental measurements of heat transport through the vacuum glazing sample.

In this work, the thermal performance of sample *A* produced by the two-stage process is compared with that of sample *B* made by the single stage process. Samples A and B consisted of two 400 mm by 400 mm low-e coated glass panes with an emissivity of 0.16, separated by a 0.15 mm wide evacuated space supported by a 0.4 mm diameter array of support pillars spaced at 25 mm. Measured and predicted exposed glass surface temperature profiles along the center lines are shown in Fig. 6 (a) for sample *A* and 6 (b) for sample *B*. Air temperatures measured in the hot and cold chambers were 27.7 °C and 7.3 °C for Fig. 6 (a), and 27.8 °C and 8.2 °C for Fig. 6 (b). The convective heat transfer coefficients for the hot and cold sides of the mask wall surface are 12.01 and 22.02 W m^{-2} K^{-1} respectively. Detailed calculations of the heat transfer through the mask wall and flanking losses are given elsewhere (Fang et al 2006). Convective heat transfer coefficients on the glass surface for samples A and B are given in Table 1.It can be seen that measured temperatures on the glass surfaces agree with the predictions to within an experimental error of 4 %. Comparing the results obtained from sample *B*, the increased temperature difference between the hot and cold side of the vacuum glazing clearly indicates that the thermal insulating property of sample *A* is better than that of sample *B*.

Table 1 gives the experimentally determined and predicted overall heat transfer coefficients of the total window areas and the central glass regions. The overall heat transfer coefficient in the central glass region determined by experiment was 1.07 ± 0.09 W m^{-2} K^{-1} for sample *A* and 1.27 ± 0.11 W m^{-2} K^{-1} for sample *B*. For sample *A*, the experimentally determined overall heat transfer coefficient is in good agreement with the predictions made with the model to within experimental error.

Evidence of a good vacuum level inside the vacuum glazing can be obtained by using infrared thermography. With a high vacuum level heat transfer occurs due to radiation across the vacuum space, conduction through the edge seal and conduction through the support pillars resulting in temperature variations on the glass surface which can be observed using infrared thermography. Internal outgassing lowering the vacuum pressure increases gaseous heat transfer, reducing the temperature difference between the glass surfaces and the heat flow through the pillars. Thus the pillar induced temperature variations on the external glass surface are reduced and their appearance less obvious on an infrared image. Fig. 7 shows infrared images in the center region and near the edge area of sample *A* produced using the two-stage process. For this measurement, the cold and hot side air temperatures were 10.1 ºC and 36.4 ºC. The surface temperature gradient around each support pillar clearly shows the heat transfer through the pillar array and the edge area.

4. Vacuum stability

The stability of a vacuum glazing depends on the quality of the edge seal, efficiency of evacuation and the bake-out process, in particular the prevention of outgassing after manufacture from the internal glass surface to the vacuum space. Although the U-value of the vacuum glazing fabricated by the two-stage process was better than that of the single-stage process, it was found that when repeating the thermal measurements after several months, the vacuum level had partially degraded. This can be attributed to outgassing from the internal glass surface and the indium seal. To solve the outgassing problem, three methods have been investigated.

1) The application a new type of indium based multilayer seal to reduce outgassing from the edge seal and to stop migration of atmospheric gases through any weakly bonded or defective areas of the seal. Results obtained for vacuum glazing sealed by an indium based multilayer seal evacuated using a pump-out system showed that outgassing from the inner indium seal had been reduced efficiently. Vacuum glazing samples manufactured in this way demonstrated excellent thermal performance with an overall heat transfer coefficient in the central glass region of down to 1.00 W m^{-2} K^{-1}, which matches the lowest U-value predicted by theoretical calculation. This new multilayer seal may provide an increase in the overall strength and durability of the seal compared to the basic seal used initially (Zhao *at al*. 1, 2006).

2) Introduction of a getter inside the vacuum glazing to absorb any gases outgassed from the internal glass surfaces preventing degradation of system performance and providing long-term

vacuum stability of the glazing. Early results show a good level of vacuum has been maintained in the glazing after the sample was fabricated (Zhao *at al*. 2, 2006).

3) Bake-out of the assembly in an inert gas atmosphere in a system designed to reduce potential outgassing in formed vacuum glazing by effective removal of residual gases.

Further investigation of these areas is currently ongoing at the University of Ulster with detailed results to be reported in future papers.

5. Conclusions

Based on a low temperature edge sealing technique for evacuated glazing developed at the University of Ulster, a pump-out technique has been developed. The pump-out method provides several opportunities for the fabrication of evacuated glazing which include: rapid effective evacuation during the pump-out process in a bakeout oven; no requirement for an additional glass tube to be soldered onto the glass for evacuation; evacuation and bake-out can be undertaken at the same time; potential to form the basis of a flexible production system for a wide range of glazing sizes. When using a new type of indium based multilayer seal the internal gas pressure in the vacuum glazing can be reduced and maintained to provide a high performance vacuum glazing. The experimental characterization of a vacuum glazing fabricated using the modified pump-out technique demonstrates excellent thermal performance with an overall heat transfer coefficient in the central glass region of 1.07 W $m^{-2} K^{-1}$, 16 % less than that of a similar glazing produced by the single stage method. The thermal performance of the vacuum glazing determined experimentally is in good agreement with theoretical predictions.

Acknowledgement

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Fig. 1 Single stage manufacturing process for vacuum glazing

Fig. 2 Schematic diagram of a vacuum glazing using a pump-out method.

Fig. 3 Two stage manufacture process of a vacuum glazing

Fig. 4 Schematic diagram of the modified pump-out system including vacuum cup, heating block, and thermocouple.

Fig. 5 Schematic diagram of a guarded hotbox for thermal characterization and durability testing of the vacuum glazing.

Fig. 6 Measured and predicted glass surface temperature profiles along the center lines of vacuum glazing: (a) sample *A*, air temperatures in the hot and cold chambers were 27.7 ºC and 7.3 ºC; and (b) sample *B*, air temperatures in the hot and cold chambers were 27.8 ºC and 8.2 ºC.

Fig. 7 Infrared images of the central and the edge areas of vacuum glazing sample *A* clearly show the presence of increased heat transfer through the pillar array and the edge seal.

Table 1 Convective heat transfer coefficients and U-values of the central glass and total window areas of vacuum glazing sample A prepared by the two-stage process and sample B prepared by the single stage process.

Sample No.	Heat Transfer Coefficients on the glass surface (W m^{-2} K^{-1})		**Predicted *U*-values** (W m^{-2} K^{-1})		**Experimentally measured *U*-values** (W m^{-2} K^{-1})	
	Hot side	Cold side	U_{centre}	U_w	U_{centre}	U_w
Sample A	3.41	7.4	1.00	1.19	1.07 ± 0.09	1.19 ± 0.10
Sample B	3.21	6.76	1.00	1.19	1.27 ± 0.11	1.42 ± 0.12

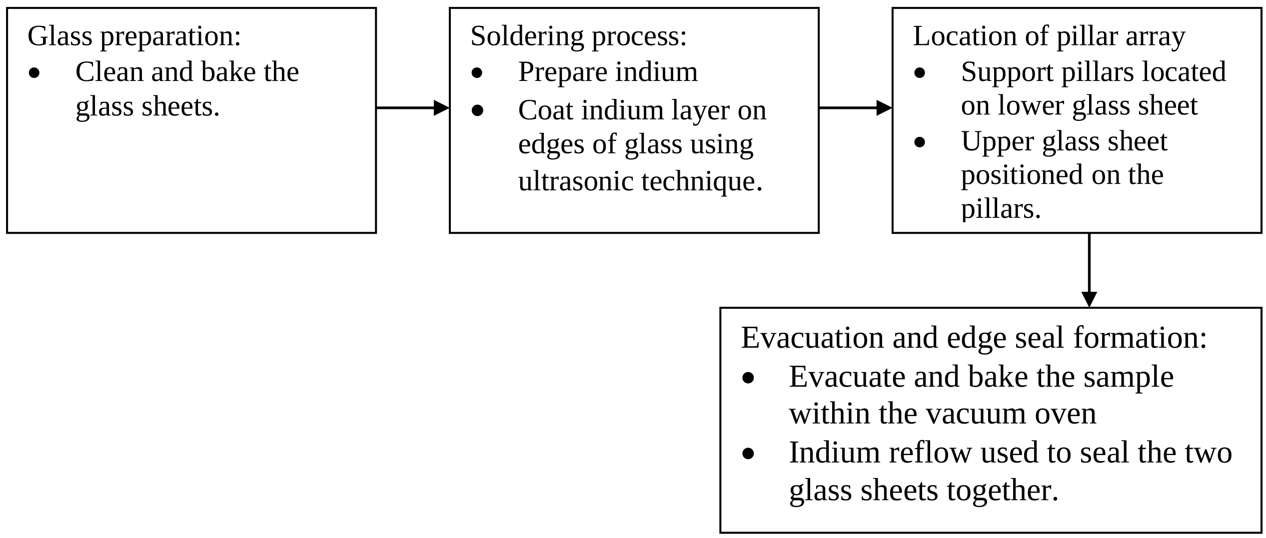

Glass preparation:
• Clean and bake the glass sheets.
Soldering process:
• Prepare indium
• Coat indium layer on edges of glass using ultrasonic technique.
Location of pillar array
• Support pillars located on lower glass sheet
• Upper glass sheet positioned on the pillars.
Evacuation and edge seal formation:
• Evacuate and bake the sample within the vacuum oven
• Indium reflow used to seal the two glass sheets together.

Fig. 1

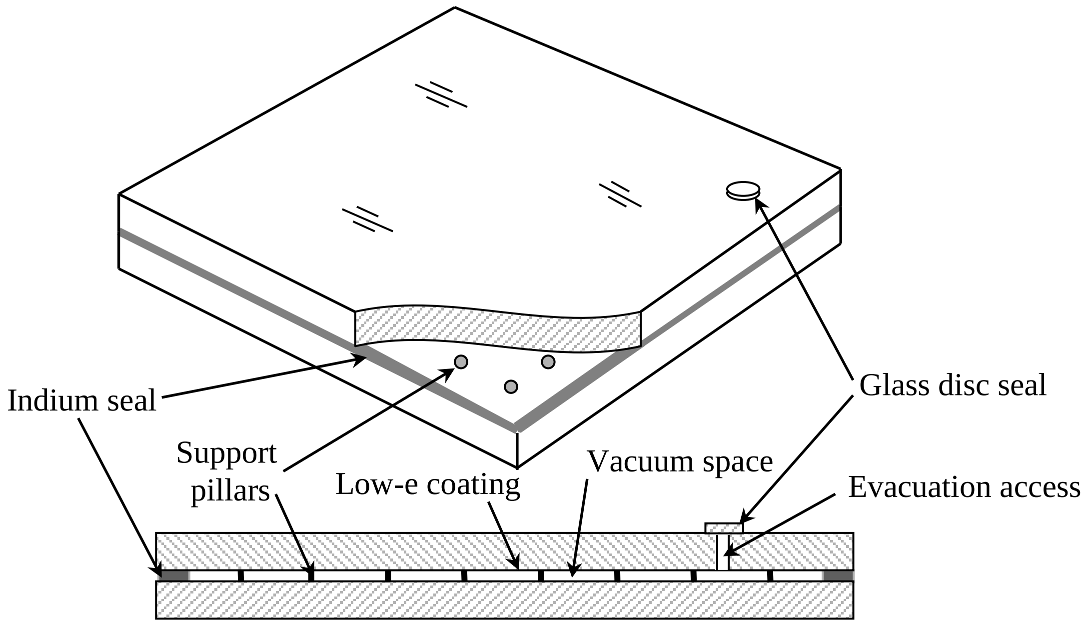

Indium seal
Support
pillars
Low-e coating
Vacuum space
Glass disc seal
Evacuation access

Fig.2

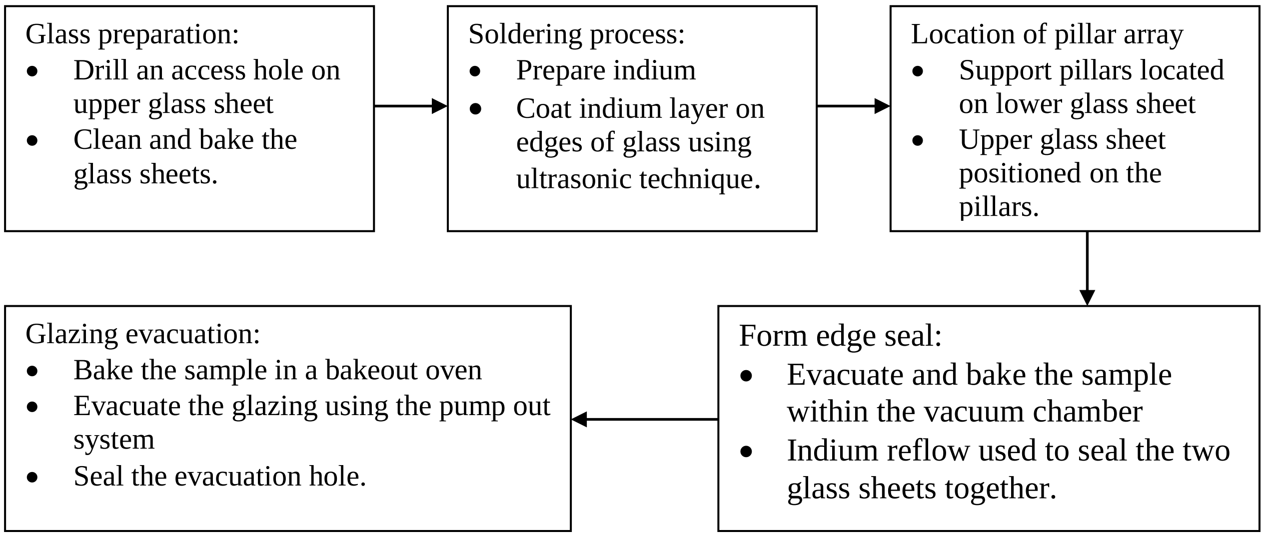

Glass preparation:
• Drill an access hole on upper glass sheet
• Clean and bake the glass sheets.
Soldering process:
• Prepare indium
• Coat indium layer on edges of glass using ultrasonic technique.
Location of pillar array
• Support pillars located on lower glass sheet
• Upper glass sheet positioned on the pillars.
Form edge seal:
• Evacuate and bake the sample within the vacuum chamber
• Indium reflow used to seal the two glass sheets together.
Glazing evacuation:
• Bake the sample in a bakeout oven
• Evacuate the glazing using the pump out system
• Seal the evacuation hole.

Fig. 3

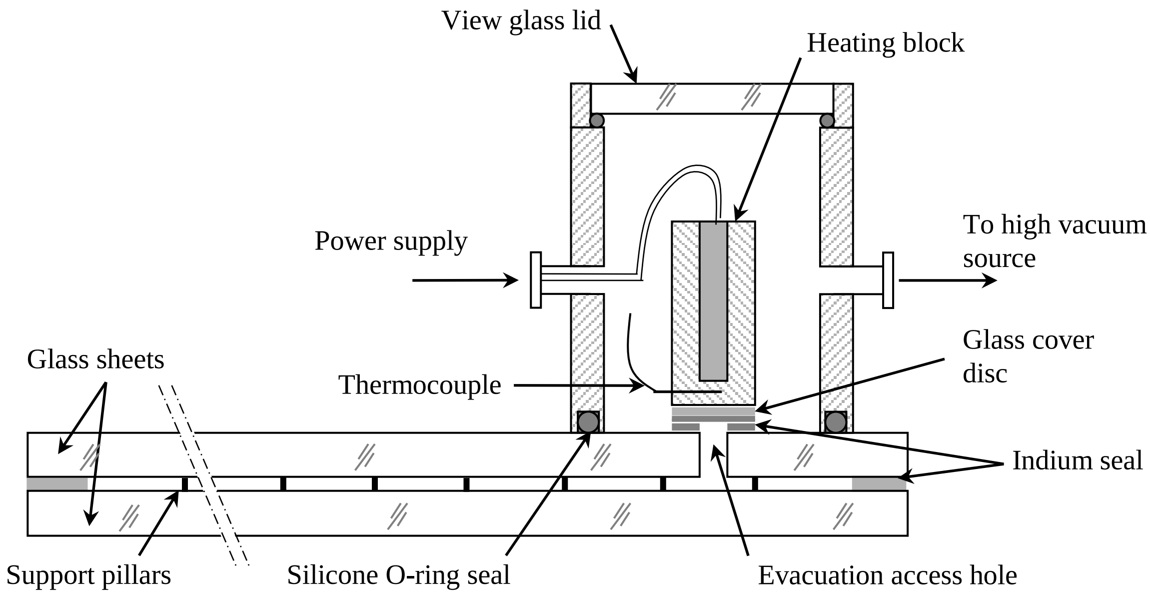
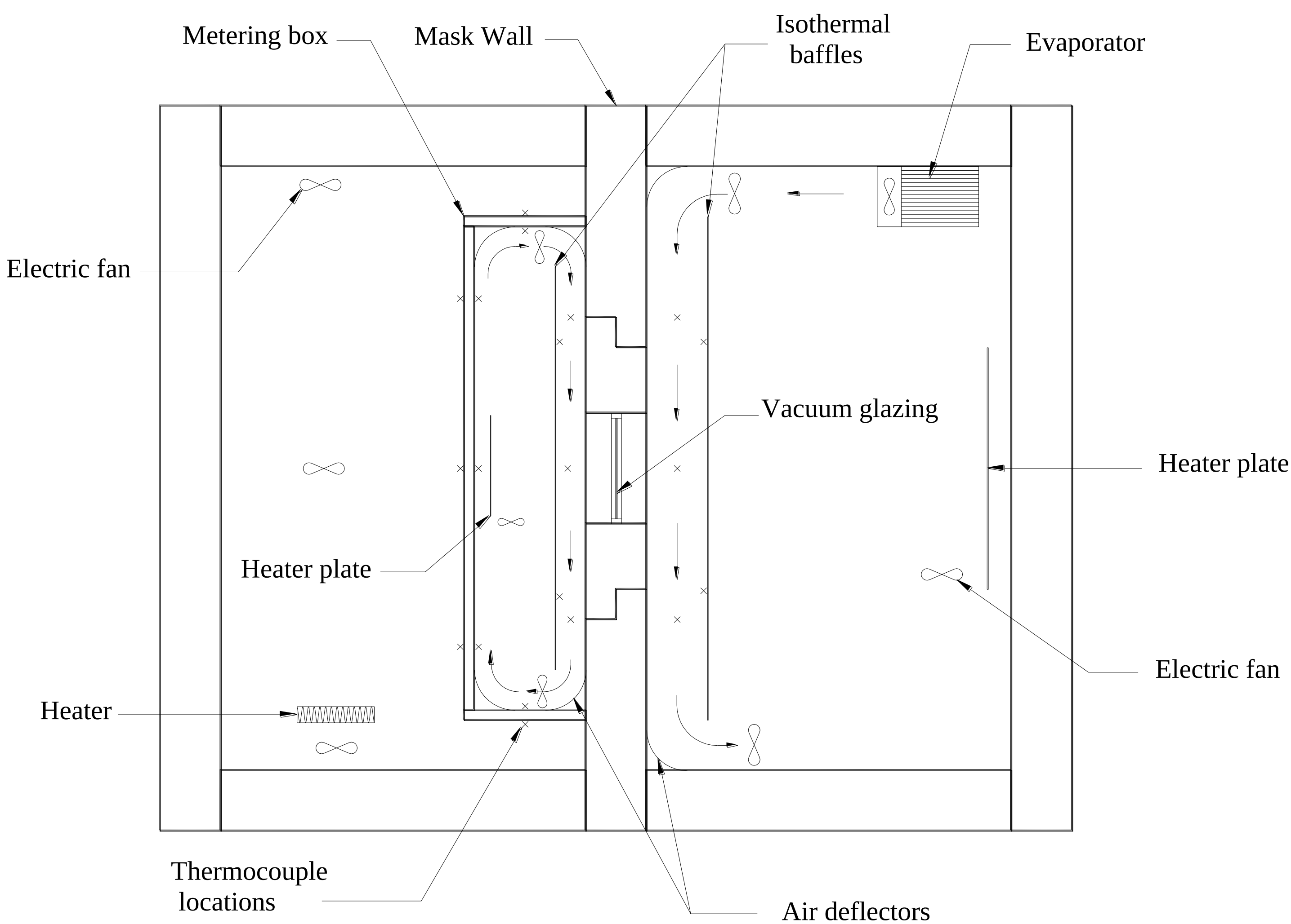
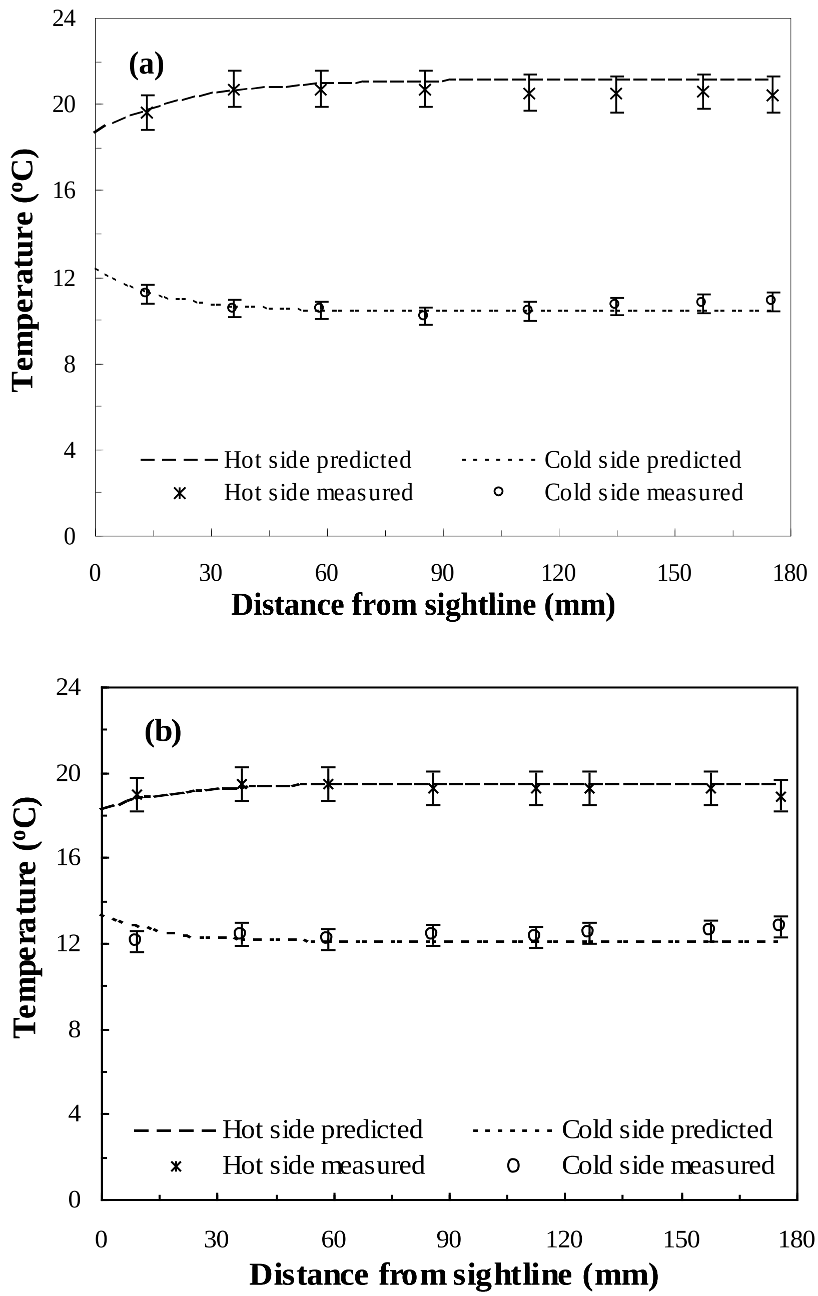

View glass lid
Heating block
Power supply
To high vacuum source
Glass cover disc
Glass sheets
Thermocouple
Indium seal
Support pillars
Silicone O-ring seal
Evacuation access hole

Fig. 4



Metering box
Mask Wall
Isothermal
baffles
Evaporator
Electric fan
Vacuum glazing
Heater plate
Heater plate
Electric fan
Heater
Thermocouple
locations
Air deflectors

Fig. 5



(a)
Temperature (ºC)
Distance from sightline (mm)
Hot side predicted
Cold side predicted
Hot side measured
Cold side measured
0
4
8
12
16
20
24
0
30
60
90
120
150
180
(b)
Temperature (ºC)
Distance from sightline (mm)
Hot side predicted
Cold side predicted
Hot side measured
Cold side measured
0
4
8
12
16
20
24
0
30
60
90
120
150
180

Fig. 6

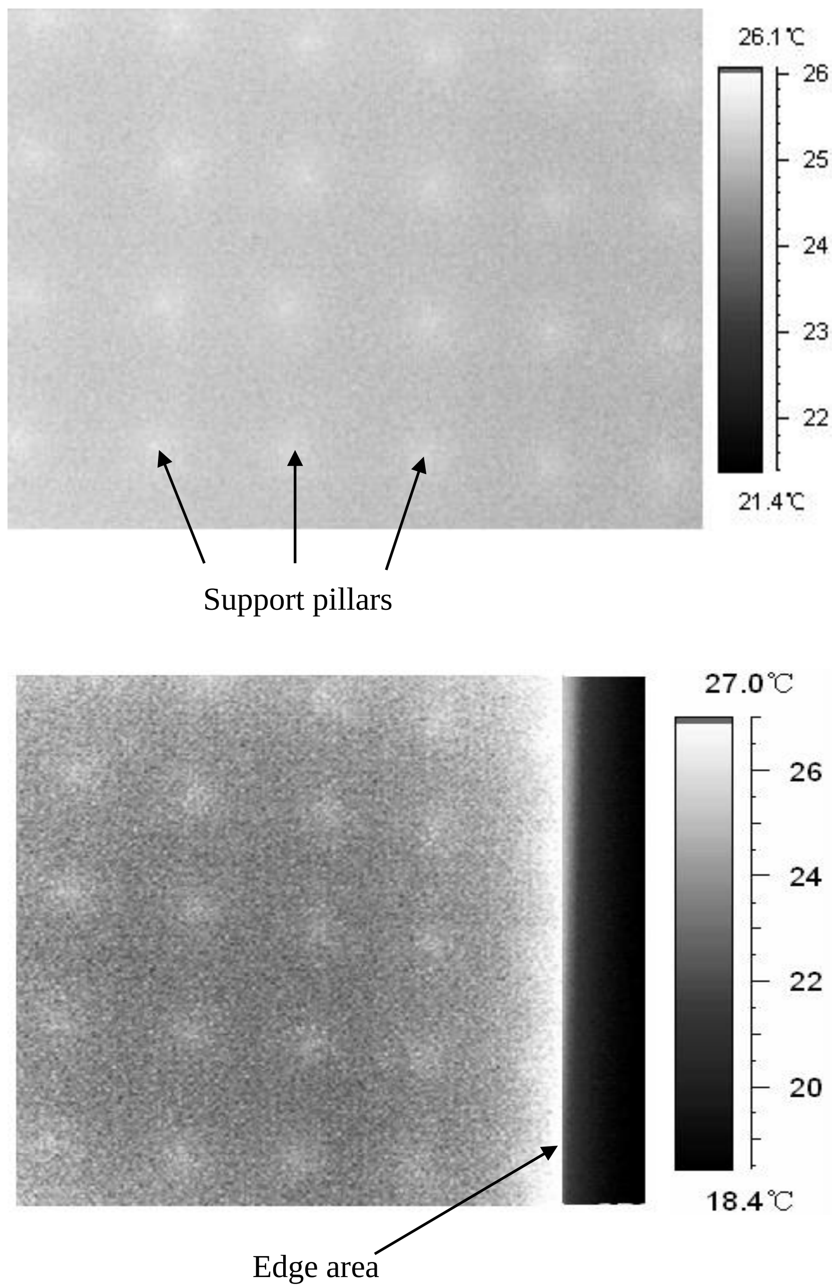

26.1℃
26
25
24
23
22
21.4℃
Support pillars
27.0℃
26
24
22
20
18.4℃
Edge area

Fig. 7