An Investigation on Edge Sealing Materials for the Fabrication of Vacuum Glazing

Saim Memon
Centre for Renewable Energy Systems Laboratory,
School of Electronic, electrical & Systems Engineering,
Loughborough University, Loughborough, UK
S.Memon@lboro.ac.uk

Prof. Philip C Eames (Supervisor)
Centre for Renewable Energy Systems Laboratory,
School of Electronic, electrical & Systems Engineering,
Loughborough University, Loughborough, UK
P.C.Eames@lboro.ac.uk

***Abstract*— This study aims at experimental durability investigation of low-temperature edge sealing material for the fabrication of vacuum glazing. A new technique offers a number of advantages in terms of cost for direct glass to metal sealing over conventional methods such as indium based vacuum glazing. The present study shows the results of three samples that were fabricated with different techniques and sizes. Sample A-170mmx170mm achieved a vacuum pressure of less than 0.001Pa. Sample B-400mmx400mm achieved a vacuum pressure of down to 0.1Pa. Sample C-300mmx300mm achieved a vacuum pressure of 0.088Pa. The edge seal bond was proven to be rigid; the small leakage around the edge can be avoided by improving the seal material uniformity around the periphery. A crack at one of the sides of the glass occurred due to the intense tensile stress resulting from the hot plate surface temperature of 120°C.**

Keywords- Vacuum glazing; Glass; sealing material; Thermal insulation

I. INTRODUCTION

Vacuum glazing allows low heat loss while maintaining transparency, regardless of the tiny pillar supports, and it is slim due to the narrow (0.15mm) vacuum gap when compared to conventional glazings. Vacuum glazing consists of two sheets of glass, an air-tight seal around the edge of the glass and an evacuated cavity with a pressure of 0.1 Pa or less to reduce heat transfer by gaseous conduction and convection to a negligible level. Radiative heat transfer can usually be reduced by using low emittance coatings on the surfaces of the glass sheet. An array of stainless steel support pillars, typically 0.15mm high and 0.3mm diameter, maintains the separation of the two glass sheets. A number of sealing materials and techniques have been reported in the literature. The edge sealing materials to date include solder glass and indium/ indium alloy materials. The solder glass edge seal, disclosed in US pat. 6,083,578, made with a high temperature (more than 400°C) process achieved a centre of pane thermal transmittance of 0.8 W/(m^2.K) (Collins and Simko 1998). It is now commercially available from NSG/Pilkington and is called SPECIA. The edge sealing process is crucial as it requires heating of the whole glazing to 450°C, this impose restrictions on the use of soft low emittance coatings. A low temperature (157°C) method of edge sealing using an indium alloy material was investigated theoretically and experimentally by the group at Ulster University and achieved a centre of pane thermal transmittance of 0.86 W/(m^2.K) (Eames, 2008) (Griffiths *et al*, 1998) & (Fang et al, 2007). However, the cost of indium is an issue. In a recent publication by Koebel *et al* (2011) an hermetic glass sealing method using anodic bonding of activated tin solder alloys (SnAl0.6) is reported, the idea was initially reported in Wallis and Pomerantz 1969, in which two small samples were fabricated and characterised under vacuum at a pressure of down to 0.05Pa. It was suggested that the method could be used for the fabrication of vacuum glazing. However, samples were fabricated without support pillars since the samples were very small in size. The making of large samples using this method could be an issue due to an increase of internal and external stress levels causing a sample to be at risk of glass fracture or breakage of the edge seal.

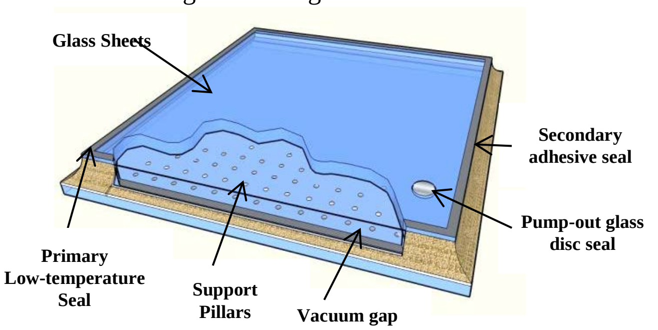

Figure 1: 2D graphical representation of the vacuum glazing using dual sealing technique.

For the successful fabrication of vacuum glazing, it has to go through five stages. First is to achieve an evacuation pressure of less than 0.01Pa between the two glass sheets. Second a successful fabrication of vacuum glazing; it substantiates edge sealing will sustain vacuum pressure for a long period of time. Third is to test the thermal performance of vacuum glazing, to comprehend the thermal transmittance U value. Fourth is to characterise the sample and understand the degradation of the sample when exposed to real weather conditions and any degradation of the vacuum over time. In this paper, the first two stages are discussed that focuses on the fabrication of vacuum glazing samples using alternative edge sealing material. A number of materials had been tried and designed; two materials, one low temperature and one high temperature melting have shown good performance achieving a vacuum pressure of down to 0.02Pa. In this paper, a dual

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sealing technique is also discussed and is shown illustratively in figure 1.a new low temperature sealing material samples were discussed among which the dual sealing technique used in the sample C, as shown in Fig. 1.

II. VACUUM SYSTEM DESIGN

A vacuum system was designed for the fabrication of vacuum glazing. A dry type turbo-molecular with backing pump with an achievable pressure of $5x10^{-6}$Pa was chosen. It has a pumping speed of 200-300litre/min which is much larger than the required speed e.g. with the sample size of $0.5m^2$ and a cavity height of 0.15mm with a volume of $37.5cm^3$ equals 0.0375 litre. A vacuum gauge measures the vacuum pressure near the vacuum cup in order to determine the approximate cavity pressure. This pressure gauge is connected to a PDR 900 transducer for the digital output of pressure in Pa and interface with a computer for monitoring purposes. In order to regulate or isolate the gas load either from the vacuum pump or Nitrogen gas introduced through a Swagelok adapter, angle valves were located at the two positions of U joint as shown in Fig. 2. CF (conflat) metal seal flanges were used to provide an ultra-high vacuum (more than $1x10^{-7}$Pa) seal between the two components. Only two CF flanges were used on the vacuum cup with an O ring seal on other joints.

The modified pump-out mechanism, as illustrated in Fig. 2, requires a circular glass disc (18mm) or square cover slip (around 10x10mm) pre-soldered with indium or alternative sealing material to be placed over the pump-out hole. An heating block, cartridge heater and thermocouple mounted on a metallic rod controlled through a supporting Y shaped block provides up and down motion of up to 10mm. A K type thermocouple fixed to the heating block measures the approximate glass disc/square temperature. Heat transfer at high vacuum occurs through both radiation and conduction due to the contact of the heating block over the glass disc. The required temperature should be approximately 40˚C more than the melting temperature of the pump-out sealing material to seal the pump-out hole.

III. FABRICATION PROCESS

A multiple stage fabrication method has been used with the modified pump-out system and techniques much similar to those detailed in Zhao *et al* (2007). Three samples were fabricated by using a new low temperature (Less than 200 □C) sealing material. A method described in Fig. 3, but not limited to the steps, uses a heating oven for the initial bake out and for making the edge seal between the two glass sheets. The use of a hot plate was considered advantageous for easier pump-out sealing and for achieving a better evacuation pressure. The addition of a metal wire gasket improves the sealing and may reduce problems of out gassing and can provide stability of the vacuum over a long term time period.

Preparing glass Sheets

- Drill a pump-out hole on the upper glass sheet.
- Clean the glass sheets with water, isopropanol and acetone.
- Bake the glass sheets to 200°C for up to 2 hours.

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Applying Edge Sealing Material

- Solder the sealing material around the periphery of glass sheets.
- Preparation of same material gasket square wire loop.

↓

Placing Support Pillars

- Support pillars positioned on lower glass sheet
- Upper glass sheet situated on the pillars

↓

Heating the sample in the Heating Oven

- A sealing temperature was less than 200°C

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Evacuation & pump-out hole sealing

- When the sample reached 120°C in the heating oven, the sample was taken out and placed on the hot plate surface in order to maintain the temperature and evacuate the sample.
- Seal the pump-out hole using glass square/disc soldered on one side with same sealing material.

Figure 3. A block diagram of the fabrication process for the development of vacuum glazing

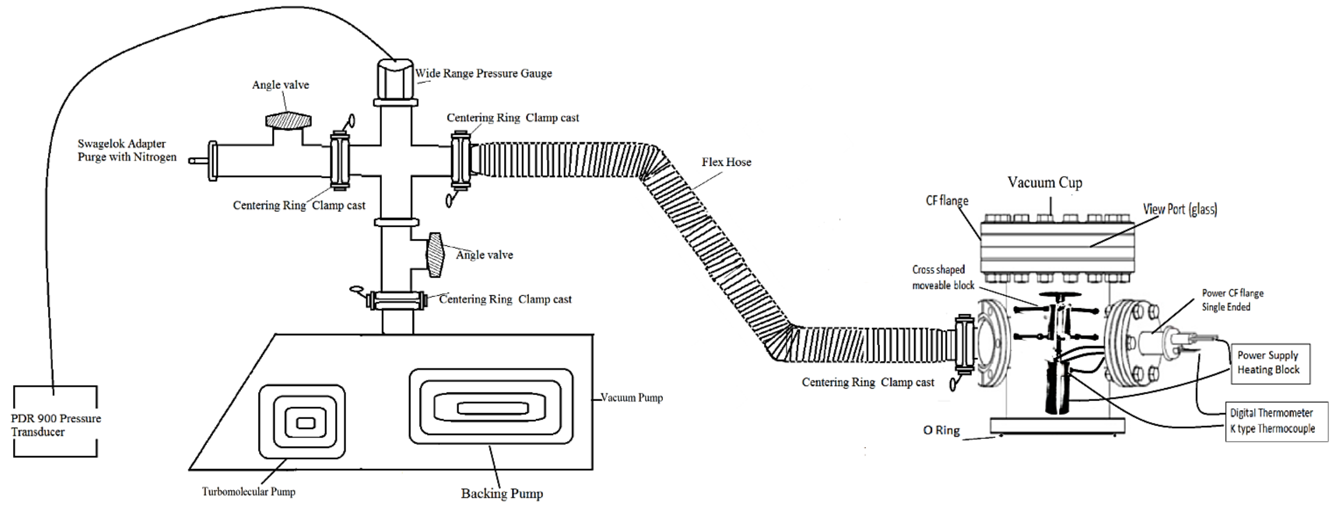


Figure 2. A detailed sketch of the design of the vacuum system for the fabrication of vacuum glazing

IV. EXPERIMENTAL RESULTS & DISCUSSION

In this on-going investigative research work, three samples were fabricated using a low temperature sealing material with different sample sizes. Sample A-170mmx170mm, shown in Fig. 4, achieved a vacuum pressure less than 0.001Pa. Due to its small size, the level of stresses on the periphery of the sample was low. It was confirmed the selected low-temperature sealing material has significant potential to sustain the required vacuum pressure. This experiment was repeated and it was found that similar vacuum pressure was achieved. Sample B-400mmx400mm, shown in Fig. 5, achieved a vacuum pressure of down to 0.1Pa. Sample B was fabricated followed the fabrication process, detailed in Fig. 3. Although the seal was successfully fabricated during evacuation a small crack occurred around the pump out sealing area due to thermal stresses. This may be due to the temperature gradient around the pump-out hole and the rest of the sample. Sample C-300mmx300mm achieved a vacuum pump pressure of 0.088Pa, as shown in Fig 6(a). Sample C was fabricated with a different technique but with the addition of an adhesive around the periphery of the sample. The pressure drop over time data for sample C is presented in the table 1.

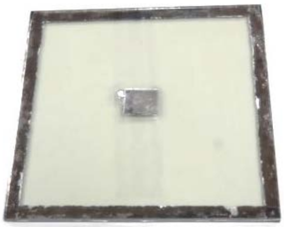
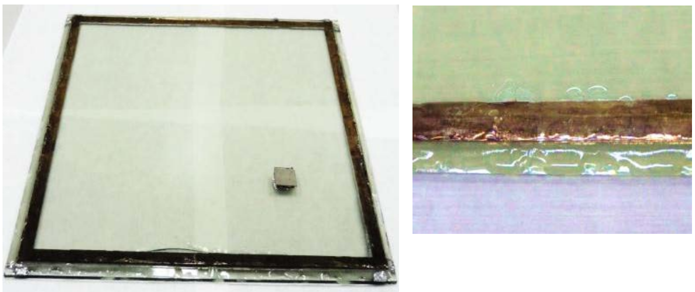


Figure 4. Sample A-170mmx170mm sample with pump-out pressure of less than 0.001 Pa.

Figure 5. Sample B-400mmx400mm sample with pump-out pressure down to 0.1 Pa.



(a) (b)

Figure 6. (a) Sample C-300mmx300mm sample, pump-out pressure of 0.086 Pa. (b) A small crack at one side of the edge that was later sealed with the adhesive used as a secondary sealing material.

When the evacuation of the sample C was started the vacuum pump pressure rapidly went down to $1.12x10^3$ Pa. Sample C was evacuated at around 120°C on the surface of the hot plate. It was observed that the glass bends and stretches due to the hot plate surface temperature. At an evacuation pressure of 30.9 Pa, a glass cracking noise was heard which indicated the possibility of leakage from around the edges. In order to determine the leakage location, an adhesive was applied around the periphery. A small leak of vacuum around one of the edge seal segment was observed as shown in Fig 6(b). After applying the adhesive as a secondary seal the pump-out pressure went down to 0.088 Pa. At this point, a high level of internal compressive stresses and external tensile stresses were observed. When the heating element was switched on, for pump-out hole sealing, a crack occurred on one side of the glass. The crack was not in the edge sealing area. The edge seal bond was strong and no cracks were observed around the seal except the tiny leakage that occurred due to the non-uniformity of the edge sealing material. Crack on the sides of the glass may have occurred due to the temperature gradient in the sample.

TABLE 1: EVACUATION PRESSURE DROP OCCURRED FOR THE SAMPLE C.

Time (sec)	0	50	100	150	200	250	300	350
Pressure (Pa)	$1.07x10^3$	$1.12x10^3$	30.9	11.3	3.1	0.45	0.089	0.088
Sample C			*A small leak*		*Adhesive applied as secondary seal*			

V. CONCLUSIONS & RECOMMENDATIONS

This paper proposes the development process for a new low temperature edge sealing material that has the potential to withstand the high vacuum pressure of down to 0.001Pa. The current research presents the results for three samples of different size that were fabricated with different sealing techniques. It was observed that the small sample had limited tensile stresses and didn't suffer breakage due to vacuum thermal or mechanical pressure stresses. The edge sealing needs to be uniform to prevent the small leak that was observed. The heating block temperature used for sealing must be controlled to accurately math the melting point of the sealing material.

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