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### A new low-temperature hermetic composite edge seal for the fabrication of triple vacuum glazing



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**Der** 

Saim Memon<sup>a,\*</sup>, Farukh Farukh<sup>b</sup>, Philip C. Eames<sup>a</sup>, Vadim V. Silberschmidt<sup>b</sup>

<sup>a</sup> Centre for Renewable Energy Systems Technology (CREST), School of Electronic, Electrical and Systems Engineering, Loughborough University, Loughborough, LE11 3TU, UK

<sup>b</sup> Mechanics of Advanced Materials, Wolfson School of Mechanical and Manufacturing Engineering, Loughborough University, Loughborough, LE11 3TU, UK

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#### ABSTRACT

High performance low-cost vacuum glazing is a key development in the move to more energy-efficient buildings. This study reports the results of experimental and theoretical investigations into the development of a new low-temperature (less than 200 °C) composite edge seal. A prototype triple vacuum glazing of dimensions 300mmx300 mm was fabricated with a measured vacuum pressure of  $4.8 \times 10^{-2}$  Pa achieved. A three-dimensional finite-element model for this prototype triple vacuum glazing with the composite edge seal was also developed. Centre-of-pane and total thermal transmittance values for this small prototype of the triple vacuum glazing were predicted to be 0.33 Wm<sup>-2</sup>K<sup>-1</sup> and 1.05 Wm<sup>-2</sup>K<sup>-1</sup>, respectively. It was predicted using the developed model that the thermal performance could be improved by reducing the width of the composite edge seal and by the use of soft low-emissivity coatings on the glass surfaces. Detailed three-dimensional isothermal contour plots of the modelled triple vacuum glazing are presented.

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#### 1. Introduction

In buildings, where day-lighting and solar gains are advantageous, large window sizes are a desired feature to increase daylighting and solar gains [1]. In order to increase the window size, without increasing the space-heating load, low heat loss glazing is required of which vacuum glazing is one option [2]. For large area vacuum glazing the use of tempered glass is advantageous which necessitates a method of low-temperature edge sealing, this is due to the loss of glass temper at temperatures greater than 300 °C [3]. A low temperature method of fabricating double vacuum glazing was first developed at the University of Ulster [4,5]. This method used indium or one of its alloys to seal the edges of the glass sheets hermetically at a temperature of less than 200 °C. The predicted, and experimentally determined, thermal transmittance of an indium sealed double vacuum glazing was reported to be less than  $1 \text{ Wm}^{-2}\text{K}^{-1}$  for the central glazing area [6–8]. To reduce the heat loss to a level where the thermal transmittance of the central glazing area is less than 0.5  $Wm^{-2}K^{-1}$  [9], the concept of triple

E-mail address: S.Memon@nescol.ac.uk (S. Memon).

vacuum glazing was introduced [10]. This consists of three sheets of glass, a vacuum tight seal around the periphery of the three glass sheets, and two evacuated cavities at a pressure below 0.1 Pa to reduce heat transfer by gaseous conduction and convection to a negligible level. Radiative heat transfer can be reduced to a low level by using soft low-emittance coatings such as silver (Ag) on the surfaces of the glass sheets [4]. An array of stainless steel support pillars, typically 0.15 mm high and 0.3 mm diameter, maintain the separation of the three glass sheets.

The scarcity and cost of indium are challenges in advancing indium-sealed vacuum glazing technology to the mass production level. The abundance of indium in the Earth's crust was estimated [11] to be about 0.05 ppm for the continental and 0.072 ppm for the oceanic crust, respectively, which is much lower than the abundance of Sn, i.e. 2 ppm in the earth's crust [12]. There thus exists a requirement to identify or develop alternative low-temperature edge sealing materials that do not suffer from these challenges prior to this technology being suitable for mass production.

A selection of different metals and alloys were assessed and tested prior to the successful development of a new low-temperature method for hermetically sealing the glass edges in a vacuum glazing. These included Sn wires (purity of 99.95% and 99.999%), Sn63PB37, Sn90In10, Cerasolzer (type 297, 246, 224 and



<sup>\*</sup> Corresponding author. School of Engineering, Science & Technology, North East Scotland College, Altens Industrial Estate, Aberdeen, AB12 3LE, UK.

186) and combinations of these alloys with an embedded annealed copper wire gasket. From laboratory experiments it was apparent that a good hermetic bond can be formed using several different materials but because of stresses developed during the fabrication process, the glass bends and stretches during heating and evacuation, leading to cracks in the seal area occurring when subject to the pressure difference between the vacuum cavity (0.1Pa or less) and atmosphere (100.000Pa) [13]. To increase the mechanical rigidity of the primary edge seal, a secondary edge seal (i.e. an adhesive such as araldite or J-B weld epoxy steel resin) was employed to provide increased bond strength and rigidity. After many experiments a clear understanding of the behaviour of Cerasolzer CS186 at a range of different temperatures was gained. This enabled a process to produce a composite glass edge seal suitable for fabrication of triple vacuum glazing to be developed, as illustrated schematically in Fig. 1.

The primary edge seal material selected, Cerasolzer CS186, is a composite of Sn(56%), Pb(39%), Zn(3%), Sb(1%) and Al–Ti–Si–Cu (1%) alloys [14]. This metal alloy composition was disclosed in the Japanese patent 20098/1968 [15] and is a commercial product of the Asahi Glass Co., Ltd [16,17]. The secondary edge seal material used is a steel reinforced epoxy known under the commercial trade name of J-B Weld epoxy steel resin. It is capable of sustaining constant temperatures of up to 260 °C with a maximum temperature threshold of up to 316 °C for 10 min [18].

A new stepped arrangement of the three glass sheets was developed, as shown in Fig. 1, which allows primary and secondary seals of 10 mm and 4 mm width to be used. With such a wide edge seal, edge seal conduction and its influence on the thermal performance of the triple vacuum glazing are important in determining the performance of this new design for triple vacuum glazing. In the work presented in this paper, a three-dimensional finite-element model for the fabricated prototype of triple vacuum glazing with this edge seal design was developed using the measured composite edge seal thermal properties. This enabled the influence of wider edge seals on the predicted thermal performance to be investigated. The simulation results were compared with those reported for the thermal performance of triple vacuum glazing in the literature.

#### 2. Cerasolzer and Indium surface analysis

The use of ultrasonic soldering an approach used previously for Indium sealed vacuum glazing [4] was initially found to be more challenging when forming a Cerasolzer with significant difficulty in maintaining the surface consistency and smoothness. After many trials, it was found that the best results were obtained with ultrasonic vibrations of approximately 25 kHz and a temperature setting of 186 °C, this led to a contiguous mechanical bond being formed between the Cerasolzer and the glass surface. Due to the mechanical bond formed Cerasolzer was considered to be a suitable material for use in an edge seal for triple vacuum glazing. Samples with Indium and Cerasolzer ultrasonically soldered to the glass surfaces of two 1 mm thick slide cover slips, each of area 20 mm  $\times$  20 mm, as illustrated in Fig. 2 were produced. A DualBeam (FIB-SEM) microscope was used to comparatively analyse the surface microstructure, smoothness and consistency of both the indium and Cerasolzer surfaces. This comparison with indium was made because it is a material successfully used for hermetically-sealing the edges of glass sheets in a vacuum glazing [4]. It can be seen that the Cerasolzer coated sample shown in Fig. 2a has a similar smooth appearance to an identically prepared sample using indium shown in Fig. 2b. The uniform surface obtained with these materials is a key feature for obtaining a viable vacuum tight edge seal.

An X-ray high-resolution CT (Computed Tomography) system was used to analyse the homogeneity of the indium and the Cerasolzer layers when used to bond two 4 mm thick k-glass samples of area 10 mm  $\times$  10 mm together. Views through the crosssection at the interface between the glass and indium and glass and Cerasolzer are presented in Fig. 3. It can be seen from Fig. 3a that the glass-indium bond has several micro pinholes or voids with air trapped inside, which may affect the hermeticity of the edge seal when used for vacuum glazing. This phenomenon was also detected and discussed by Zhao et al. [19]. It can be seen from Fig. 3b that there are fewer, smaller micro pin holes (with air trapped inside) for the Cerasolzer-glass bond sample than in the indium-glass bond shown in Fig. 3a. It was reported by Griffiths et al. [4], Eames [9] and Zhao et al. [6] that an indium-glass sealed sample should not be overheated since this leads to the indium flowing excessively and that a secondary adhesive edge seal is required to avoid moisture ingress and seal degradation. Due to the apparent similarities between Cerasolzer and Indium glass bonds it is probable that vacuum glazing with a Cerasolzer seal will require a secondary seal also.

To assess the continuity of the glass-Cerasolzer bond, a 10 mm

wide joint between two 4 mm thick k-glass sheets (each of area

 $20 \text{ mm} \times 20 \text{ mm}$ ) was made using an ultrasonic soldering iron at a

#### 3. Glass-Cerasolzer bond



Fig. 1. A schematic diagram of a triple vacuum glazing showing the primary edge seal made of Cerasolzer CS186 alloy and a secondary edge seal of J-B weld epoxy steel resin.

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